Town of Simsbury

933 HOPMEADOW STREET SIMSBURY, CONNECTICUT 06070

Watch Board of Selectmen meetings LIVE and rebroadcast on Comcast Channels 96, 1090, Frontier Channel 6071 and LIVE streamed or on-demand at www.simsburytv.org

SIMSBURY BOARD OF SELECTMEN
Main Meeting Room – Simsbury Town Hall – 933 Hopmeadow Street, Simsbury
Special Meeting – September 12, 2018 – 5:00 p.m.

CALL TO ORDER

EXECUTIVE SESSION

a) Pursuant to CGS §1-200(6)(B) concerning Pending Claims and Litigation – Deepwater Wind Appeal, Petition 1313

RETURN FROM EXECUTIVE SESSION

SELECTMEN ACTION

a) Proposed Settlement Agreement and Option to Purchase Agreement with Deepwater Wind and Possible Vote

ADJOURN

Town of Simsbury
933 HOPMEADOW STREET SIMSBURY, CONNECTICUT 06070

BOARD OF SELECTMEN MEETING
AGENDA SUBMISSION FORM

1. **Title of Submission:** Proposed Settlement Agreement and Option to Purchase Agreement – Deepwater Wind

2. **Date of Board Meeting:** September 12, 2018 (special meeting)

3. **Individual or Entity Making the Submission:**
Maria E. Capriola, Town Manager Maria E. Capriola

4. **Action Requested of the Board of Selectmen:**
If the Board of Selectmen supports approving the settlement agreement and option to purchase agreement with Deepwater Wind as presented, the following motion is in order:

Move, effective September 12, 2018 to approve the settlement agreement as presented, and to authorize Town Manager, Maria E. Capriola, to execute the agreement following final review and approval by Town Attorney.

Move, effective September 12, 2018 to approve the option to purchase agreement as presented, and to authorize Town Manager, Maria E. Capriola, to execute the agreement following final review and approval by Town Attorney.

5. **Summary of Submission:**
At the policy direction of the Board of Selectmen, legal counsel and staff have completed negotiations with Deepwater Wind. Attached is a proposed settlement agreement and option to purchase agreement.

A presentation detailing the highlights of the settlement agreement has been attached and will be given on Wednesday evening.

6. **Financial Impact:**
To date, the Town has spent approximately $90,284 on legal and consulting fees related to this initiative, not including work conducted for which invoices have yet to be received. The settlement agreement could commit the Town to paying for up to $25,000 towards the well receptor survey and the water testing. Other future consulting and legal costs may be incurred due to construction, water and soil monitoring.

7. **Description of Documents Included with Submission:**
   a) Proposed Settlement Agreement with Deepwater Wind, including proposed Option to Purchase Agreement with Deepwater Wind
   b) Presentation, Summary of Background and Proposed Agreements, dated September 12, 2018

RELEASE AND SETTLEMENT AGREEMENT

This Release and Settlement Agreement ("Agreement") is made between DWW Solar II, LLC, its parent companies, successors-in-interest, assigns, affiliates, and any corporation or entity to which or with which it may merge or consolidate ("DWW"), the Town of Simsbury, Connecticut ("the Town") and the following seven individuals: Christine Kilbourn-Jones, Laura Nigro, Lisabeth Shlansky, Zhenkui Zhang, John Marktell, Rob Perissi and Ed Wrobel and their heirs, successors-in-interest and assigns. These seven individuals shall be referred herein as "the Abutters," and the Abutters, DWW and the Town shall collectively be referred to herein as the "Parties," and each individually as a "Party."

RECITALS

A. DWW is the developer of a 26.4 MW AC solar photovoltaic array proposed to be located in the Hoskins Road area in Simsbury, Connecticut ("the Project").

B. In connection with its development of the Project, on June 29, 2017, DWW filed a Petition for Declaratory Ruling with the Connecticut Siting Council ("Siting Council") seeking the Siting Council's approval of the Project. This Petition was labeled as Petition 1313 by the Siting Council.

C. During the course of the proceedings before the Siting Council in Petition 1313, the Town and the Abutters were granted party status in Petition 1313 by the Siting Council.

D. The Siting Council issued its approval of Petition 1313 on December 21, 2017, with the mailing of said approval on December 22, 2017 ("Final Decision").

E. The Town and the Abutters believe that they have been wrongfully aggrieved by the Final Decision, and further believe that they have the right to appeal the Final Decision.

F. DWW believes that the Town and the Abutters have not been wrongfully aggrieved by the Final Decision, and that the Siting Council correctly decided all matters before it in connection with Petition 1313. DWW further believes that the Town and the Abutters have no valid basis for an appeal.

G. On February 1, 2018, the Town filed an administrative appeal concerning the Final Decision and effected service in accordance with General Statutes § 4-166 *et seq.*, currently pending in the Connecticut Superior Court, judicial district of New Britain, as Docket No. HHB-CV18-6042321-S ("Town Appeal").

H. On Februrary 5, 2018, the Abutters filed an administrative appeal concerning the Final Decision and effected service in accordance with General Statutes § 4-166 *et seq.*, currently pending in the Connecticut Superior Court, judicial district of New Britain, as Docket No. HHB-CV18-5021660-S ("Abutters Appeal").

I. Since the issuance of the Final Decision, the Parties have discussed, in good faith, resolution of these issues.

J. The Parties desire to settle fully and finally all differences or disputes between them related in any way to the Final Decision.

Now, Therefore, in consideration of the promises exchanged pursuant to the terms of this Agreement, and for other good and valuable consideration, the sufficiency of which the Parties acknowledge, the Parties agree as follows:

1. **DWW's Representations and Warranties.** DWW hereby represents and warrants that it shall undertake the following in connection with the Project, in exchange for the withdrawal of the Town Appeal and the Abutters Appeal:

a) Soil testing – DWW agrees to submit the Soils & Materials Management Plan, which is attached as Exhibit A hereto, to the Siting Council as part of DWW's Development and Management Plan ("D&M Plan"). DWW shall comply with the Soils and Materials Management Plan approved by the Siting Council.

b) Water testing – DWW agrees to submit the Drinking Water Well Testing Protocol, which is attached as Exhibit B hereto, to the Siting Council as part of DWW's D&M Plan. DWW shall comply with the Drinking Water Testing Protocol approved by the Siting Council. DWW shall use a consultant, that is approved by the Town, to enact the Drinking Water Well Testing Protocol ~~that is approved by the Town~~. Such approval from the Town shall not be unreasonably withheld, conditioned or delayed. As part of the Drinking Water Well Testing Protocol, DWW shall perform a well receptor survey. The Town will reimburse DWW for the cost of this well receptor survey for the actual costs of the survey, such costs not to exceed five thousand dollars ($5,000.00). ~~; and t~~The Town will also reimburse DWW for fifty percent (50%) of the costs of all well testing performed in accordance with the Drinking Water Well Testing Protocol, such costs not to exceed twenty thousand dollars ($20,000).

c) Historical research – DWW has provided the Town with the results of DWW's research, including the resources and data underlying the research, relating to Dr. Martin Luther King Jr.'s association with the Town and the farm associated with the Project Site. A copy of these results is attached hereto as Exhibit C. The Town does not adopt the results of the research performed by DWW by executing this Agreement.

d) 400 watt panels – DWW agrees that it will evaluate such panels, but DWW does not affirmatively agree to use such panels. As part of its D&M Plan submittal, DWW shall provide a written memorandum explaining the results of its evaluation, including the feasibility of the 400 watt panels for the Project.

e) Limitation of Project footprint – DWW will redesign the Project so that once the construction of the Project has been completed, the Project will not contain any solar panels or other permanent equipment related to the Project that will be located in the parcel that is south of Hoskins Road, otherwise known as Parcel # H05-103-024. Parcel # H05-103-024 may be used by the Project for temporary staging and laydown during the construction of the Project or during periods of major maintenance (such as the replacement of panels and/or inverters) and decommissioning. The temporary laydown area will be situated as far away from any residential properties as practically feasible, but in no circumstances shall the temporary laydown area be closer than one hundred (100) feet from any residential property. This redesign shall be incorporated into the D&M Plan to be submitted to the Siting Council. DWW

shall restore the temporary laydown area, which shall include re-seeding and tilling. After completion of temporary staging, Parcel # H05-103-024 shalll be maintained as a grassland, subject to occasional mowing as needed, or shall be used for pollinator habitat and/or agricultural purposes, consistent with its prior use as an agricultural site.

f) Future development – DWW shall have the right to operate a solar project on the Project Site. DWW covenants that at the earlier of 1) such time as DWW ceases to use the Project Site for the purpose of converting sunlight into electricity, or 2) ninety-nine (99) years from the day of the execution of this Agreement, DWW: (i) will not develop the Project Site for another purpose ~~(i.e., the Project Site will revert to an agricultural use or will remain vacant)~~ and (ii) will provide to the Town an option to purchase the Project Site for $1.00. The Town shall have six (6) months in which to exercise its option to purchase. The Town acknowledges that DWW does not currently own the parcels making up the Project Site. If DWW exercises its option to acquire the parcels making up Project Site, DWW represents that it will execute the form of the use restriction and the option to purchase ~~is~~ included hereto as Exhibit D.

~~The form of the use restriction and the option to purchase is included hereto as Exhibit D.~~

g) The Siting Council's approval of the Project permitted two barns to be removed from the Project Site. DWW will remove only one barn, as indicated on the map included hereto as Exhibit E. The remaining four barns on the Project Site will not be removed from the Project Site, unless they present a danger to life or property. Notwithstanding the foregoing, DWW will install and/or maintain cabling, weather proofing and otherwise maintain the two barns adjacent to Hoskins Road to the extent that the structures are a component of the visual screening measures pursuant to Paragraph 1.h. below. DWW will periodically inspect these two barns to evaluate their condition relative to said dangers noted above.

h) Visual screening – DWW has submitted a redesign of the visual screening for the Project, which has been reviewed by the Town. A copy of a rendering of this visual screening is included hereto as Exhibit F, and DWW shall submit a visual screening program, in the form of Exhibit F as part of its D&M Plan submittal.

i) Decommissioning – DWW will submit a decommissioning plan to the Siting Council as part of its D&M Plan. The decommissioning plan will provide: On or before June 30, 2029, DWW shall provide security sufficient to pay for decommissioning costs in the form of a performance bond, letter of credit or another form of financial security acceptable to the Siting Council and the Town, to ensure the availability of funds for such decommissioning costs (the "Financial Assurance"). The Financial Assurance shall be maintained in effect by Lessee (including renewals, replacements and extensions) for the remainder of the terms for the DWW PPAs. The amount of the Financial Assurance for the decommissioning work will be based on the all inclusive costs of decommissioning associated with the solar arrays and all related equipment and improvements thereto, and the legal and proper disposal of all equipment and waste. The calculations shall include all professional costs, remediation, labor costs, trucking, hauling, disposal costs, landscaping costs and other cost which is reasonably

expected to be incurred LESS the estimated salvage value of the solar arrays. The amount of the Financial Assurance shall be determined by (i) mutual agreement of DWW and the Town, each using reasonable judgment or (ii) by an independent Professional Engineer licensed in the State of Connecticut. To initially establish the required amount of Financial Assurance, DWW shall provide the Town with an estimate of the decommissioning costs and estimated salvage value. For a period of thirty (30) days following the Town's receipt of DWW's estimate, DWW and the Town shall work in good faith to reach a mutually acceptable amount for the Financial Assurance. If DWW and the Town are unable to reach agreement on the amount of Financial Assurance as provided above, then the amount for the Financial Assurance shall be established by an independent Professional Engineer selected by DWW and reasonably acceptable to the Town. The determination of such Professional Engineer shall be binding upon DWW and the Town. The costs for such Professional Engineer's establishment of the amount for the Financial Assurance shall be paid by DWW. The Town may request that the amount of the Financial Assurance be revaluated once every two years thereafter in the manner set forth above to ensure sufficient funds for decommissioning. If upon revaluation, the amount of the estimated decommissioning costs changes, the amount of the Financial Assurance will be adjusted within thirty (30) days of such determination.

j) Perimeter Road – Given that DWW will not be constructing any Project facilities on Parcel # H05-103-024, the Parties agree that DWW can construct arrays in the area of the Project's perimeter road. DWW will submit amendments reflecting this construction in its D&M Plan.

k) Copies: – DWW will provide the Town and its consultants with copies of any submissions or reports that DWW provides to the Siting Council.

2. **Withdrawal of the Town Appeal and Abutters Appeal.** The Town and the Abutters will withdraw their respective Appeals upon an approval by the Siting Council of a D&M Plan incorporating each and every element of sub-paragraphs a, b, d, e, g, h, i and j of Section 1, above, and after the expiration of any appeal rights which might be attendant thereto. The Town and the Abutters will file a withdrawal of their respective Appeals within seven (7) days of the expiration of the abovementioned appeal rights.

3. **Release.** The Town and the Abutters, for themselves and all those who claim through them or could claim through them, will not, now or in the future, file any appeal, actions, causes of action, or lawsuits in the Superior Court of Connecticut or any other court of

competent jurisdiction contesting the Final Decision (any, a "Claim" and collectively, the "Claims"). For the avoidance of doubt, nothing in this Release shall be construed to release DWW from any breach of this Agreement or any debt, demands, actions, causes of action, damages, claims or liabilities of any nature relating to DWW's construction, operation, maintance or decommissioning of the Project.

4. **Effect of Release.** Once executed by the Parties, this Agreement will be binding upon and inure to the benefit of the Parties, their heirs, predecessors, parents, successors, assigns, affiliates, employees, agents, and any corporation or entity to which or with which they may merge or consolidate with respect to all Claims.

5. **Applicable Law.** This Agreement will be governed by and construed in accordance with the laws of the State of Connecticut.

6. **Preparation of Release.** The Parties acknowledge and agree that this Agreement has been prepared, reviewed, studied, and executed without compulsion, fraud, duress, or undue influence and without circumstances which would overcome the free will of the signatory, and that it is expressly made by the Parties with the requisite experience and that each party to this Agreement acknowledges that it has had the benefit of advice of competent legal counsel with respect to its decision to enter into this Agreement. Accordingly, the normal rule of construction to the effect that any ambiguities are to be resolved against the drafting Party shall not be employed in the interpretation of this Agreement or any amendment of it. It is the intent of the Parties that no part of this Agreement be construed against any of the Parties because of the identity of the drafter.

7. **Costs and Fees.** The Parties agree that each will bear its respective costs, fees,

and expenses, including, but not limited to, attorneys' fees, incurred in connection with the Claims, the negotiation, preparation, and execution of this Agreement, and the performance of obligations contemplated by this Agreement.

8. **Non-Assignment of Claims.** Each of the Town and the Abutters represents and warrants to DWW that the rights and claims released pursuant to this Agreement have not in any way been assigned, transferred, hypothecated, or otherwise encumbered, and that they are the sole and absolute owners of such rights and claims.

9. **Mechanical Tracking Devices.** Each party acknowledges that DWW may request that the Siting Council allow DWW to use mechanical tracking devices on the solar panels to allow the panels to move to increase the amount of time the panels are fully exposed to the sun. No party shall challenge this request by DWW, provided that DWW demonstrates to the Council that the addition of such tracking will not result in any of the following occurring: a) increase the amount of sound from previously-approved levels in the Final Decision by more than ten percent (10%), b) increase the amount of glare, as measured by reflectance, in accordance with ASTM standard methods or other applicable standards or laws for solar reflectance, from previously-approved levels in the Final Decision by more than ten percent (10%), or c) an increase in the height of any panels in excess of fourteen (14) feet. In the event that DWW's request exceeds one or more of these parameters, the parties may challenge DWW's request before the Siting Council. The parties recognize that the Siting Council is not bound by the terms of this Agreement, and will have the full ability to issue a decision on this request, regardless of whether there is opposition to the request. The parties will honor the final decision of the Siting Council with respect to this request and will not appeal it once it has been rendered.

10. **Execution Authorized.** The Parties represent and agree that all actions required by them to authorize and approve the execution, delivery, and performance of the terms of this Agreement has been duly taken and the same shall constitute their valid and binding obligations.

The Parties have the full power and authority to execute and deliver this Agreement. The undersigned represent that they have been duly authorized to enter into this Agreement on behalf of the Party on whose behalf he or she has signed.

11. **Severability.** The Parties agree that if any of these provisions should be deemed invalid or unenforceable by any court of competent jurisdiction, such invalidity or unenforceability will not affect the whole Agreement, and the Agreement will be construed as if not containing the particular provision held to be invalid or unenforceable, and the obligations of the Parties will be construed and enforced accordingly.

12. **Waiver.** The waiver by a Party of another Party's breach of any provision of this Agreement shall not be construed as a waiver of any subsequent breach of thisAgreement. The failure of a Party to enforce any provision of this Agreement, or to exercise any right or privilege hereunder, shall not be construed as a waiver of any such provision, right, or privilege.

13. **Entire Agreement.** This Agreement constitutes the entire agreement between the Parties relating to the Claims and the Final Decision. Except as expressly stated above, this Agreement supersedes and replaces any and all prior or contemporaneous agreements or understandings, whether written or oral, with regard to the matters set forth herein. This Agreement may not be amended or modified in whole or in part, nor any of its provisions waived, except by an agreement in writing signed by authorized representatives of all Parties.

14. **Counterparts.** The Parties agree that this Agreement may be executed in counterparts and that execution of counterparts shall have the same force and effect as if the Parties had signed the same instrument. Any signature made and transmitted electronically or via facsimile for the purposes of executing this Agreement shall be deemed an original signature for

purposes of this Agreement and shall be binding on the signing Party.

[*Remainder of page intentionally left blank*]

IN WITNESS WHEREOF, the Parties have executed this Release and Settlement Agreement as of the dates set forth below.

DWW Solar II, LLC

By: _____

Name: _____

Title: _____

The Town of Simsbury, Connecticut

By: _____

Name: _____

Title: _____

The Connecticut Department of Agriculture

By: _____

Name: _____

Title: _____

Christine Kilbourn-Jones

By: _____

Name: _____

Title: _____

Laura Nigro

By: _____

Name: _____

Title: _____

Lisabeth Shlansky

By: _____

Name: _____

Title: _____

Zhenkui Zhang

By: _____

Name: _____

Title: _____

John Marktell

By: _____

Name: _____

Title: _____

Rob Perissi

By: _____

Name: _____

Title: _____

Ed Wrobel

By: _____

Name: _____

Title: _____

Proactive by Design

DRAFT

SOIL & MATERIALS MANAGEMENT PLAN
TOBACCO VALLEY SOLAR
SIMSBURY, CONNECTICUT

~~July~~ August 2018
File No. 05.0045765.01

PREPARED FOR:
DWW Solar II, LLC
c/o Deepwater Wind
56 Exchange Terrace, Suite 300
Providence, RI 02903

GZA GeoEnvironmental, Inc.
655 Winding Brook Drive, Suite 402 | Glastonbury, CT 06033
860-286-8900

29 Offices Nationwide
www.gza.com

Proactive by Design

GEOTECHNICAL
ENVIRONMENTAL
ECOLOGICAL
WATER
CONSTRUCTION MANAGEMENT

655 Winding Brook Drive
Suite 402
Glastonbury, CT 06033
T: 860.286.8900
F: 860.652.8590
www.gza.com

~~July~~ August ~~16~~17, 2018 Draft
File No. 05.0045765.01

DWW Solar II, LLC
c/o Deepwater Wind
56 Exchange Terrace, Suite 300
Providence, RI 02903

Attn: Aileen Kenney

Re: Soil & Materials Management Plan
Tobacco Valley Solar
Simsbury, Connecticut

Dear Ms. Kenney:

GZA GeoEnvironmental, Inc. (GZA) is pleased to provide the attached Soil & Materials Management Plan for the proposed Tobacco Valley Solar project in Simsbury, Connecticut (the Site). This plan provides guidance on managing soil that may be encountered during the construction activities. This plan is subject to the limitations included in Appendix A.

We trust this plan satisfies your present requirements; should you require additional information, please contact the undersigned.

Very truly yours,

GZA GEOENVIRONMENTAL, INC.

Adam T. Henry, LEP
Associate Principal

Gordon T. Brookman, LEP
Consultant / Reviewer

FIGURES

APPENDICES

1.00 SOIL AND MATERIALS MANAGEMENT PLAN OUTLINE

1.10 PURPOSE, BACKGROUND AND SCOPE

The purpose of this Soil and Materials Management Plan (SMMP) is to define the program for handling, segregating, stockpiling, sampling and reusing or disposing of soil/material encountered during upcoming regrading and construction activities at the proposed Tobacco Valley Solar project in Simsbury, Connecticut (the Site). The location of the Site is shown on Figure 1.

The Site, as currently configured, consists of five (5) adjacent parcels (totaling approximately 289 acres) located on County Road, Hopmeadow Street and Hoskins Road in a residential and agricultural section of Simsbury, Connecticut. The Site layout is shown on Figure 2. According to GZA's 2016 Phase I Environmental Site Assessment report, the Site appears to have consisted of undeveloped wooded land and agricultural fields since at least 1934. Three of the parcels (Parcels 1, 3 and 5) have historically been used for tobacco farming while two of the parcels (Parcels 2 and 4) have historically consisted of undeveloped wooded land. Five barns (unused or used for miscellaneous storage) were present on Parcels 1 and 5 at the time of GZA's 2016 Phase I report, and small unnamed ponds are located on Parcels 1, 3 and 5. Agricultural fields at the Site appear to have been most recently used to grow vegetables (squash, pumpkins, and tomatoes) and tobacco.

Properties adjoining the Site consist of residential dwellings or apartments, vacant residential land and vacant commercial land.

DWW Solar II, LLC has proposed to construct a 26.4-megawatt solar photovoltaic electric generating facility on the Site. Based on plans, dated June 2017 and designed by VHB, GZA understands the project consists of the installation of ground-mounted solar panels, equipment pads, and underground utilities. Other improvements will include access roads covered with crushed stone and fences. Some of these improvements will require penetrations to the subsurface.

The information presented in this Plan provides procedures/requirements for materials management during the project construction based on the current understanding of the Site and project parameters. The specific details and logistics for implementation of the SMMP shall be the responsibility of the Contractor. The Contractor, with the support of GZA, will be responsible for the proper management and reuse/disposal of excavated material in accordance with applicable laws.

The scope of the SMMP relates to the handling and management of at-grade and below-grade soils, water, and other materials. This Plan is not intended to be used for guidance relating to demolition, handling, removal, management, and disposal of buildings or other above-grade structures or materials (including foundation elements). This Plan is subject to the limitations in Appendix A.

1.20 PROJECT REPRESENTATIVES

Owner:	DWW Solar II, LLC c/o Deepwater Wind 56 Exchange Terrace, Suite 300

	Providence, RI 02903 Attention: Aileen Kenney
Civil Engineer:	Vanasse Hangen Brustlin, Inc 100 Great Meadow Road, Suite 200 Wethersfield, CT 06109 Tel: (860) 807-4300 Attention: Paul Vitaliano
Owner's Representative:	TBD
Environmental Consultant:	GZA GeoEnvironmental, Inc. 655 Winding Brook Drive, Suite 402 Glastonbury, CT 06033 Tel: (860) 858-3166 Attention: Adam Henry
Site Contractor:	TBD

1.30 RESPONSIBILITIES

Soils and other materials may be encountered during regrading associated with the planned Site development. Based on the anticipated shallow depths of excavations (generally less than 4 feet below ground surface (fbgs) but up to 10 fbgs in limited locations), and the depth to groundwater (> 40 fbgs) reported by previous investigators, dewatering is not considered to be a potential issue during this project. However, if shallow groundwater is encountered, it should be managed as described in Section 5.0.

The following is a description of certain key tasks relating to the soils and material management.

A. The Contractor(s) shall be responsible for:

1. All aspects of implementing the SMMP including all costs associated with excavation, management, segregation, stockpiling and onsite reuse of soil, and if necessary, testing, transportation, and offsite disposal of soil.

2. Compliance with the conditions of the CTDEEP General Permit for the Discharge of Stormwater and Dewatering Wastewaters from Construction Activities. A copy of the General Permit is provided in Appendix B. We note that based on the size of the proposed site disturbance area (>5 acres), registration under the permit will be required, and a Stormwater Pollution Control Plan (SWPCP) will need to be developed and implemented for the project. The Contractor must adhere to the SWPCP and follow the Connecticut Guidelines for Soil Erosion and Sediment Control and the Connecticut Stormwater Quality Manual.

3. Compliance with other CTDEEP General Permits, if applicable and when necessary, including the CTDEEP General Permit for Contaminated Soil and/or Sediment Management and the CTDEEP General Permit for Discharge of Groundwater Remediation Wastewater. Copies of these General Permits are provided in Appendix B.

4. Determining the project schedule, construction sequencing, and other operational parameters of the project

and communicating such information to the project team.

5. Overseeing all earth-related construction work for the project including installation and maintenance of soil erosion and sediment controls in accordance with the Sediment and Erosion Control Plan as prepared by the Civil Engineer. A copy of the Soil Erosion and Sediment Control Plan is provided in Appendix C.

6. Establishing on-Site material stockpiling location(s) in accordance with the Soil Erosion and Sediment Control Plan and soil staging and transfer general permit, if required.

7. Compliance with the necessary environmental and non-environmental permits, approvals, authorizations, Site health and safety plan (HASP) and all other applicable state, and federal health and safety standards for the performance of the construction work.

8. Providing all labor, materials, equipment, and other services required for handling, segregating, and stockpiling of materials encountered during construction.

9. If necessary, documentation and waste shipping paperwork; identification of and obtaining approval from appropriate off-Site disposal facility for disposal/recycling of impacted soils; and loading, transport, and disposal of impacted soils (with support from GZA). The Owner must approve all Contractor proposed disposal and or recycling facilities prior to off-site shipment of soils or other materials.

10. Protecting the health of workers, other on-Site personnel, the general public and minimizing impacts to the environment.

B. GZA will be responsible for the following:

1. Coordinating with the Contractor to facilitate proper segregation of materials as requested.

2. Reviewing Contractor's documents related to compliance with applicable environmental permits, approvals, and authorizations.

2.00 EXECUTION

2.10 REGULATORY COMPLIANCE

The Contractor shall conduct all work in accordance with all applicable ~~federal and State of Connecticut codes, ordinances, statutes,~~ regulations~~, and permits~~ that may apply to the project, including but not limited to:

- Applicable CTDEEP General Permits; and
- All other applicable ~~State and federal statutes and~~ regulations pertaining to environmental impact assessment, air pollution control, safe drinking water, water pollution control, solid and hazardous waste management, and toxic substances control.

2.20 PLANNED EXCAVATION ACTIVITIES

The Site work associated with this project will require earthwork activities, including but not limited to the following:

- Installation of erosion and sedimentation controls;
- Excavation and stockpiling of certain soils;
- Alterations of surficial coverings including grading and tree removal;
- Installation of utilities; and
- Site grading and drainage.

The sequence of these activities will be in accordance with project phasing plans developed by the Civil Engineer.

Estimated quantities of soils requiring stockpiling during earthwork activities are currently unknown. The Contractor shall review associated bid documents and specifications to make its own determination on the quantities of material for excavation and stockpiling. Quantities of material may be more or less than the quantities referenced in this document and final quantity estimates are the responsibility of the Contractor. GZA notes that no analytical data is available for Site soils; however, material potentially requiring special management (such as a farm dump or buried debris) may be encountered during the excavations. The Contractor shall be responsible for testing soil that is sent off-site for disposal/reuse. If suspect materials are encountered during excavation activities, the Contractor ~~should~~ will immediately notify GZA and the Owner.

Soil and erosion control requirements for soils must comply with the CTDEEP General Permit for the Discharge of Stormwater and Dewatering Wastewaters from Construction Activities. Based on the size of the proposed site disturbance area (>5 acres), registration under the permit will be required and a SWPCP will need to be developed and implemented for the project. The Contractor must adhere to the SWPCP and follow the Connecticut Guidelines for Soil Erosion and Sediment Control and the Connecticut Stormwater Quality Manual.

2.30 EXCAVATION ENVIRONMENTAL CONTROLS

2.30.1 Dust Control

The Contractor shall employ dust control measures necessary to minimize the creation of airborne fugitive dust from soils during performance of this work. Such measures shall include the containment of soils through implementation of soil transfer and stockpile best management practices and other suitable methods (i.e., wetting and covering stockpile/trucks) to limit dust, as necessary. Certain contaminants if present in Site soils at high concentrations could present a particulate inhalation hazard when contaminated soil becomes airborne with dust if site conditions are dry. An aggressive approach towards dust suppression shall be employed. Work areas shall be wetted with a water mist to control dust generation resulting from vehicle and personnel traffic and from soil handling activities. Should visible dust be generated from site operation, additional wetting shall be implemented.

2.30.2 Vapor and Odor Control

Contractor shall monitor the work area in accordance with the requirements of the Site HASP as prepared in accordance with Section 6.2 of this SMMP. Contaminant vapors at significant concentrations that might require

respiratory protection for Site workers are not anticipated during the project. However, in the event, that excavation or other Site activities encounter unanticipated contaminants, vapors or odors, as determined through air monitoring and/or direct observations, GZA should be notified and the Contractor shall be prepared to employ control measures necessary to minimize the generation of such contaminant vapors and odors. Such measures shall include: restricting work in a particular area, use of temporary mats or coverings, use of odor-suppressant foam, containment of a particular work area, and other feasible means of controlling contaminants, vapors and odors, as necessary, including remediation.

2.40 SOIL STOCKPILING

At least one business day prior to the commencement of excavation activities, the stockpile area shall be prepared to receive the materials. The location(s) of the stockpiles shall be in accordance with the Soil Erosion and Sediment Control Plan as prepared by the Civil Engineer and General Permits, if applicable. The stockpile areas shall be cleared and then fenced off, if Site access is not already restricted. The following minimum stockpile criteria shall apply to stockpiles.

Stockpile areas shall be graded such that stormwater run-on and runoff is diverted around the stockpiled materials. At a minimum, a snow-fence and haybales with silt fence shall be placed continuously around the perimeter of each stockpile area. The stockpile area shall be underlain with a minimum ten (10)-mil-thick black polyethylene sheeting. In the event excavated materials are excessively wet (saturated), earth berms shall be placed around the perimeter of the stockpile area, if necessary, to contain drainage from the stockpiles. Stockpile side slopes shall be no steeper than 3 horizontal (H) to 1 vertical (V).

Drainage effluent from the stockpiles~~,~~ shall be contained within perimeter berms, and infiltrated.

Stockpiled materials shall be placed within the designated stockpile areas, graded to shed water, and covered prior to inclement weather and at the end of each work day with a minimum ten (10)-mil-thick black polyethylene cover overlapped and weighted to form a continuous waterproof barrier over the material. The cover shall be maintained throughout the stockpile period to prevent water from entering the stockpiled materials and to prevent blowing dust. Stockpile locations shall be placed as approved by the Owner, Engineer, or GZA in advance of construction.

The transfer of materials from the excavation to the stockpile area shall be conducted in such a manner as to prevent loss of or spread of materials or dust across the Site.

If suspect contaminated materials are encountered by the Contractor (such as a farm dump or buried debris, stained, unnaturally colored or odorous soil), those materials shall be stockpiled separately.

The Contractor is responsible for all construction, protection, movement, and maintenance of stockpiles for the duration of the project work or until directed otherwise by the Owner or the GZA.

The clearing and preparing of stockpile areas and the grading, polyethylene barriers, berms, and all other materials, equipment, and labor required for protection of the excavated material will be considered part of the work.

3.00 SAMPLING AND DECONTAMINATION PROCEDURES

Sampling of soil stockpiled during earthwork activity that is intended for onsite reuse is not required. If off-site disposal is planned for some or all of this stockpiled material, representative soil samples will be collected from the stockpiled material by GZA. Because previous environmental reports have indicated that the Site is not in a CTDEEP remediation program, post-excavation sampling of the underlying residual material is not planned. If suspect contaminated materials (such as those associated with a farm dump or buried debris) are encountered, they will be stockpiled separately and sampled for purposes of off-site disposal/reuse and/or prior to on-Site reuse, and post-excavation sampling may be conducted as necessary after consultation with GZA and the Owner.

For stockpiled material that is planned for off-site disposal, including suspected contaminated material as described in Section 2.40, representative soil samples will be collected for characterization purposes by GZA (with assistance from the Contractor) at a frequency of one sample per 500 cy of soil stockpiled. Each stockpile characterization sample will consist of a composite made of a minimum of five discrete grab samples collected at various depths and locations within the stockpile. The sampling and decontamination procedures are further detailed below.

3.10 SOIL SAMPLING PROCEDURES

Proper soil sampling technique requires, on the part of the field representatives, understanding of the objective of the sampling program and adhering to the following guidelines.

The following equipment will be required:

- Photo-ionization detector (PID)
- Stainless steel auger(s), trowel(s), and/or shovel(s)
- Stainless steel bowl(s) and spoon(s)
- Reagent grade methanol and wash bottle
- Sample containers of appropriate size and preservative (if required) for each constituent to be analyzed according to EPA protocols
- Buckets, water, Alconox or equivalent
- Paper towels and garbage bags
- Coolers and ice packs
- Sample labels and waterproof markers
- Chain of custody forms and custody seals

The samples will be collected using an auger, trowel or shovel, field screened using the PID, placed in the appropriate container, and labeled according to the procedures outlined below. The soil sampling equipment will be decontaminated between each sample location.

For stockpile sampling, a minimum of one composite soil sample will be collected for every 500 cy of soil stockpiled for offsite disposal. The soil samples will be obtained in the following manner:

- Samples for volatile organic compounds (VOCs) will be discrete grab samples (one per 500-cubic yards). The grab sample chosen for analysis will be the sample with the highest PID field screening results.

- For other constituents, composite samples will be prepared by combining equivalent volumes of soil from individual grab samples. Between 5 and 10 grab samples will be retrieved from each 500-cy stockpile and used to make a composite sample.

- Grab samples will be collected at different depths and locations in the stockpile utilizing a stainless-steel hand auger or stainless-steel trowel to ensure variations in the soil types are proportionally represented in the composite. Successive grab samples will be placed in the same stainless-steel bowl after field screening and subsampling (as appropriate) for VOCs.

- After the 5-10 grab samples have been placed into the bowl, the contents of the bowl will be fully mixed with a decontaminated spoon and a representative sample of the contents will be transferred into the appropriate sample containers.

- Samples will be labeled according to the procedures outlined below.

Spoils derived from the stockpile sampling program will be placed back into the stockpile.

3.20 SAMPLING EQUIPMENT DECONTAMINATION

Decontamination of soil sampling equipment is the responsibility of the field personnel. Decontamination of the sampling equipment will be performed as follows:

1. Scrub the surface of the sampling equipment with a brush that is consistently submerged in a bucket containing Alconox mixed with tap water.

2. Rinse the scrubbed sampling equipment with tap water contained in adjacent bucket.

3. Rinse the sampling equipment with methanol over a third bucket to capture the methanol.

4. Rinse again with distilled water over a collection bucket.

5. Contain all decontamination liquids for management in a drum or similar container located at the stockpile area.

6. Prevent the cleaned sampling equipment from coming into contact with any potentially contaminated media prior to use for sampling.

3.30 SAMPLE STORAGE

Proper storage following sample collection is important in maintaining sample quality. Soil samples will be placed promptly into a chilled/iced cooler and maintained at approximately 4 degrees Celsius (C) until delivery to the laboratory. The sample collector will transport the samples directly to the laboratory at the end of the sampling day or will arrange to have the laboratory courier pick up the samples within 24 hours.

3.40 DOCUMENTATION OF FIELD WORK

3.40.1 Field Log Book/Sampling Log

Complete and thorough logging of field work is essential to a timely and accurate completion of the project. Field personnel are responsible for recording actions and times of major events and of sampling in a field log book and/or field sampling log. Also, sample identification (numbers and descriptions) of field samples will be accounted for on the field sampling log. For each sampling event, the field book and/or field sampling log will contain the following:

- GZA field person's name(s), GZA equipment used, weather, date, time, and location at start of day.
- Descriptions and sketches of the sample/stockpile location, stockpile dimensions/estimated volume, origins of materials that comprise the stockpile.
- Descriptions of the number of grab samples retrieved from stockpile, locations/depths of these grab samples, observed soil types, conditions (staining, odors, fill, debris, etc.) and PID readings at each grab sample location, and identification of the grab sample location selected for the discrete VOC sample.
- Other comments would include: Description of any unusual conditions; Record of Health and Safety monitoring - time, equipment, and results; Record of site accidents or incidents; Record of any visitors; Causes and duration of any delays; and any other data that may be construed as relevant information at a later date.

3.40.2 Chain-of-Custody Forms

GZA will be responsible for filling out chain-of-custody forms when samples are collected. The chain-of-custody form is a document that tracks the samples collected from the field to the laboratory and indicates the custodian of the samples at any time and also which laboratory analyses are to be performed on which samples. Each sample will be clearly labeled and listed on the chain-of-custody. The chain of custody will be filled out as samples are collected and will accompany the samples to the laboratory. The sampler will be the first custodian of the samples and will sign the chain-of-custody when he/she relinquishes custody of the samples to another individual or to the laboratory which will also sign and date the document.

3.50 LABORATORY ANALYSIS

Stockpile samples will be analyzed by a CT-certified laboratory for extractable total petroleum hydrocarbons (ETPH), VOCs, semi-VOCs, total and leachable RCRA 8 metals, copper, nickel, zinc, polychlorinated biphenyls (PCBs), herbicides, pesticides, and any other analytical parameter that may be required by the designated offsite disposal/reuse facility.

Laboratory turnaround will typically be on a 5-day basis, adjustable to the schedule of construction.

3.60 QUALITY ASSURANCE/QUALITY CONTROL PROGRAM

The following sections provide descriptions of the QA/QC program which will be followed during the sampling and analysis. This program will be followed to generate analytical data of known and defensible quality.

Functioning of the sample collection and analysis process is facilitated by communication between the field sampler and the laboratory and clear identification of the tasks and responsibilities of each. Adherence to the protocol presented herein will minimize problems in maintaining data quality and integrity.

Field representatives will conduct the following activities prior to initiating the collection of samples:

1. Select the analytical tests to be performed and schedule the analyses with the laboratory.

2. Determine the type, size, and quantity of sample containers required, the amount of required preservative, and the maximum field holding times for each sample.

3. Determine the equipment required for sampling, and make sure that it is available.

4. Obtain sample containers, preservatives, and trip blanks (if necessary) from the laboratory.

5. Obtain chain-of-custody forms, sampling log, shipping forms, and sealing tapes.

6. Determine that all sampling equipment and accessories have been decontaminated as described in Section 3.20.

4.00 STOCKPILED MATERIAL CLASSIFICATION, HANDLING, AND DISPOSAL

The results of the stockpile sampling (Section 3.00) will be provided to the Contractor to evaluate potential off-site disposal/reuse options and/or potential on-Site reuse. The Contractor will coordinate with GZA to complete any additional required waste characterization, identify appropriate off-Site disposal facilities for impacted soils, and arrange for loading, transport, and disposal of impacted soils. Owner will sign all transportation and disposal documents (Material Shipping Records [MSRs] or Bills of Lading [BOLs]) as the Generator. Contractor shall provide copies of soil disposal weight tickets, manifests or Bills of Lading, and disposal facility acceptance letters to the Owner.

5.00 MANAGEMENT OF GROUNDWATER

5.10 DESCRIPTION

No dewatering is planned. If the Contractor encounters surface water or groundwater which must be dewatered for construction, the Contractor shall properly contain groundwater discharge and manage all dewatering in accordance with State and federal regulations. Dewatering activities must comply with the CTDEEP General Permit for the Discharge of Stormwater and Dewatering Wastewaters from Construction Activities, which applies to all discharges of stormwater and dewatering wastewater from construction activities which result in the disturbance of *one or more* total acres of land area on a site regardless of project phasing. Note that registration is required under this general permit for projects disturbing greater than 5 acres of land (such as the Site) and the permittee must develop and implement a SWPCP for the project, and adhere to the Connecticut Guidelines for Soil Erosion and Sediment

Control, the Connecticut Stormwater Quality Manual, and soil and erosion control land use regulations. The Contractor shall comply with the requirements of other General Permits as applicable.

Water quality testing shall be conducted by GZA prior to the discharge of any wastewater generated by dewatering activities. If the results of water quality testing indicate that the wastewater is not suitable for discharge to the ground, it shall be containerized and removed for off-site disposal.

5.20 SUBMITTALS

The Contractor shall consult with GZA prior to the start of any dewatering activities and shall provide GZA with an appropriate Dewatering Plan at least 5 business days prior to the start of dewatering activities. Contractor shall not start dewatering activities without prior authorization from GZA. The Dewatering Plan shall indicate the purpose, location, and estimated duration of the proposed dewatering activities. In addition, the Dewatering Plan shall include the Contractor's proposed dewatering methodology. If necessary, permit registrations and/or approvals shall be completed, and water quality testing shall be conducted by GZA prior to the discharge of any water generated by dewatering activities.

5.30 RESPONSIBILITY OF THE CONTRACTOR

Minimum precautions noted in this Section shall in no way relieve the Contractor of his responsibility for implementing stricter health and safety precautions as warranted by the Work.

The Contractor shall be responsible for adhering to permits, regulations, specifications, and recognized standard practices related to both contaminated and uncontaminated soil and groundwater/stormwater handling during excavation and dewatering for construction.

The Contractor shall be responsible to remove and transmit groundwater and stormwater under an approved Dewatering Plan. Contractor shall complete dewatering activities in accordance with all applicable State and federal regulations and permits.

The Contractor shall make reasonable effort to minimize the volumes of groundwater dewatered from the excavation.

6.00 DECONTAMINATION OF EQUIPMENT AND HEALTH AND SAFETY

6.10 DECONTAMINATION OF EQUIPMENT

In the event that contaminated soil or groundwater is encountered at the Site, the Contractor is responsible to clean all tools and equipment before they are taken from the Site. Contractor's tools and equipment which are to be taken from the Site shall be decontaminated on-Site. This shall include all tools, heavy machinery and excavating and hauling equipment used during excavation, stockpiling and any re-handling of impacted soil or groundwater.

6.20 HEALTH AND SAFETY

The Contractor and any subcontractors performing work within the contract limits shall have a Site specific written HASP developed by a qualified person designated by the Contractor. The Contractor shall establish protocols and provide procedures to protect worker's health and safety as it relates to the proposed construction activities when performed in the presence of contaminated materials or otherwise environmentally sensitive conditions. The HASP shall be developed and implemented to addresses the relative risk of exposure to documented hazards present within the contract limits. The HASP shall establish health and safety protocols which address the relative risk of exposure to regulated substances in accordance with 29 CFR 1910.120 and 29 CFR 1926.65. Such protocols shall only address those concerns directly related to Site conditions.

The Contractor shall utilize available information and existing records and data pertaining to chemical and physical hazards associated with any of the regulated substances to develop the HASP. Further information can be made available to the Contractor upon request.

The requirements set forth herein pertain to the provision of workers' health and safety as it relates to proposed project activities when performed in the presence of hazardous or regulated materials or otherwise environmentally sensitive conditions. The provision of worker health and safety protocols which address potential and/or actual risk of exposure to Site specific hazards posed to Contractor employees is solely the responsibility of the Contractor.

GZA GeoEnvironmental, Inc.

GZA

Proactive by Design

GEOTECHNICAL
ENVIRONMENTAL
ECOLOGICAL
WATER
CONSTRUCTION MANAGEMENT

655 Winding Brook Drive
Suite 402
Glastonbury, CT 06033
T: 860.286.8900
F: 860.652.8590
www.gza.com

August 31, 2018
File No. 05.0045765.01

DWW Solar II, LLC
c/o Deepwater Wind
56 Exchange Terrace, Suite 300
Providence, RI 02903

Attn: Aileen Kenney

Re: Drinking Water Well Testing Protocol
Tobacco Valley Solar, Simsbury, CT

Dear Ms. Kenney:

GZA GeoEnvironmental, Inc. (GZA) has prepared this Drinking Water Well Testing Protocol to describe the methods and procedures for sampling potable wells at certain residences near the proposed Tobacco Valley Solar (TVS) project in Simsbury, CT (Site). This letter is subject to the Terms & Conditions of our contract.

The Site consists of five (5) adjacent parcels (totaling approximately 289 acres) located on County Road, Hopmeadow Street and Hoskins Road in a primarily residential section of Simsbury, Connecticut. DWW Solar II, LLC has proposed to construct a 26.4-megawatt solar photovoltaic electric generating facility on the Site. Based on plans, dated June 2017 and designed by VHB, GZA understands the project consists of the installation of ground-mounted solar panels, equipment pads, and underground utilities. Other improvements will include access roads covered with crushed stone and fences. Some of these improvements will require penetrations to the subsurface.

Historically, portions of the Site and adjacent areas were used for tobacco farming. Around 1990, remediation of a pesticide disposal area located off-site to the east was conducted by the Connecticut Department of Energy and Environmental Protection (CTDEEP). Subsequent to the remedial action, CTDEEP conducted annual testing of certain potable wells in the Site area for 1,2-dibromoethane (EDB), 1,2-dibromo-3-chloropropane (DBCP), and 1,2,3-trichloropropane (123TCP). The testing program was discontinued around 2013 due to lack of funding and monitoring data that showed the concentration of pollutants in the wells did not exceed what the Department of Public Health considered protective over the long-term.

As part of the TVS project, DWW Solar II, LLC will conduct a potable well receptor survey within 500 feet of the Site to identify the locations and owners of potable wells. Based on the results of the survey, DWW Solar II, LLC will ~~plans to~~ contact well owners~~,~~ and conduct testing of the private potable wells within the survey area shown on the

attached figure if consent is provided by the well owners. A list of the locations of the potable wells that are adjacent to or near ~~to~~ the Site is attached and would be revised based on the results of receptor survey. The purpose of this Drinking Water Well Testing Protocol is to ensure that the data generated by the testing are sufficient for comparison to the drinking water standards and for the protection of human health. For those wells for which consent has been provided, one testing event will be conducted prior to construction activities and one testing event will be conducted once construction activities have concluded. Well samples will be tested for the same constituents (EDB, DBCP and 123TCP) that CTDEEP selected for their long-term monitoring program since they are the most soluble pesticide compounds and a baseline of monitoring data exists. In addition, well samples will be tested for pesticides by EPA Method 8081, volatile organic compounds (VOCs) by EPA Method 524.2, and the metals arsenic and copper by EPA Method 6010C.

Sample Collection

DWW Solar II, LLC personnel will coordinate each sampling event by contacting the owners and/or residents prior to the event to arrange access to the property to sample the well(s). The sample collector/handler will wear latex gloves during the sampling procedures. Certified-clean sample containers will be supplied by the analytical laboratory. Sample containers are labeled at the time of collection in order to identify and track the sample according to its origin, date and time of collection. To facilitate sample collection and to ensure proper labeling, labels may be partially completed prior to sample collection. Sample labels should be completed with waterproof ink.

Sampling personnel shall confirm with each resident generally how much household water use has occurred prior to sampling and include a sampling note stating whether this confirmation occurred and how many minutes water was subsequently purged. If sampling staff cannot confirm that significant water use has occurred (e.g., via showering or laundry), or if the house is unoccupied, a 15-minute purge will be required provided that the well can sustain such a withdrawal. The samples will be collected in laboratory-provided pre-preserved containers including three ~~preserved~~ 40-milliliter vials, one 250-milliliter plastic jar, and two 500-milliliter amber glass jars for laboratory analysis. Vials shall be filled completely with no headspace remaining in the vial. Sampling personnel shall document the sample location and identify whether any water filtration equipment is present. Samples shall be stored on ice in coolers and/or in a refrigerator to a target temperature of <4 degrees Celsius upon arrival at the laboratory. Samples shall be delivered to the laboratory within 4 days of their collection.

A trip blank sample shall accompany all potable well samples to the laboratory to assess any cross contamination of VOCs between bottles. A trip blank consists of vials filled at the laboratory with laboratory grade reagent water. These bottles are delivered along with the sample containers prior to sampling. They are stored in the sample cooler during sampling and transported to the laboratory in the coolers with the samples. Per the laboratory standard operating procedures, attached, the laboratory will analyze one duplicate sample for every 20 samples analyzed.

A chain of custody is completed to create an accurate written record that can trace the sample from the moment of collection through analysis. This document includes project reference names or codes, the

sampler's name, the type of sample, the number and type of bottles collected, the sample analyses required, the date and time of the sample, and the signatures of individuals relinquishing and receiving the samples including the sampler, courier, and laboratory receiver.

Laboratory Analysis

All samples shall be analyzed by a CT DPH-certified and NELAP-accredited laboratory. Laboratory analyses to be performed include EDB, DBCP and 123TCP by EPA Method 504.1 or Method 524.3, VOCs by EPA Method 524.2, pesticides by EPA Method 8081, and the metals arsenic and copper by EPA Method 6010C. Laboratory Quantitation Limits (QL) and Minimum Detection Limits (MDL) shall be sufficient to achieve the required DPH action levels for the target compounds. Relevant laboratory and action levels for these compounds are summarized in Table 1 and ~~a~~ typical laboratory standard operating protocols for the test methods ~~is~~ are attached. The laboratory analyses will be conducted in accordance with CTDEEP Reasonable Confidence Protocols (RCP).

Trip blanks will be analyzed for VOCs with each sample delivery group. If any target compounds are detected in a trip blank, the associated samples will be reviewed to determine if cross-contamination has occurred. In the event that cross-contamination has occurred, sample handling procedures will be audited and improved.

Data Review and Analysis

A licensed environmental professional (LEP) will oversee the sampling program, review the laboratory results and RCP reports and evaluate whether any additional actions should be undertaken. Analytical results will be provided to the homeowner with 72 hours after receipt of the laboratory reports by GZA. GZA will provide a summary of results, findings and recommendations in a written report after each sampling event. Such actions may include re-sampling. If target compounds are detected in the water samples at concentrations exceeding the MDLs, notification to the CTDEEP within 30 days may be required by the homeowner in accordance with the Significant Environmental Hazard (SEH) regulations and additional water testing may be warranted or required.

We hope this information is useful to you. If you have any questions, please contact us.

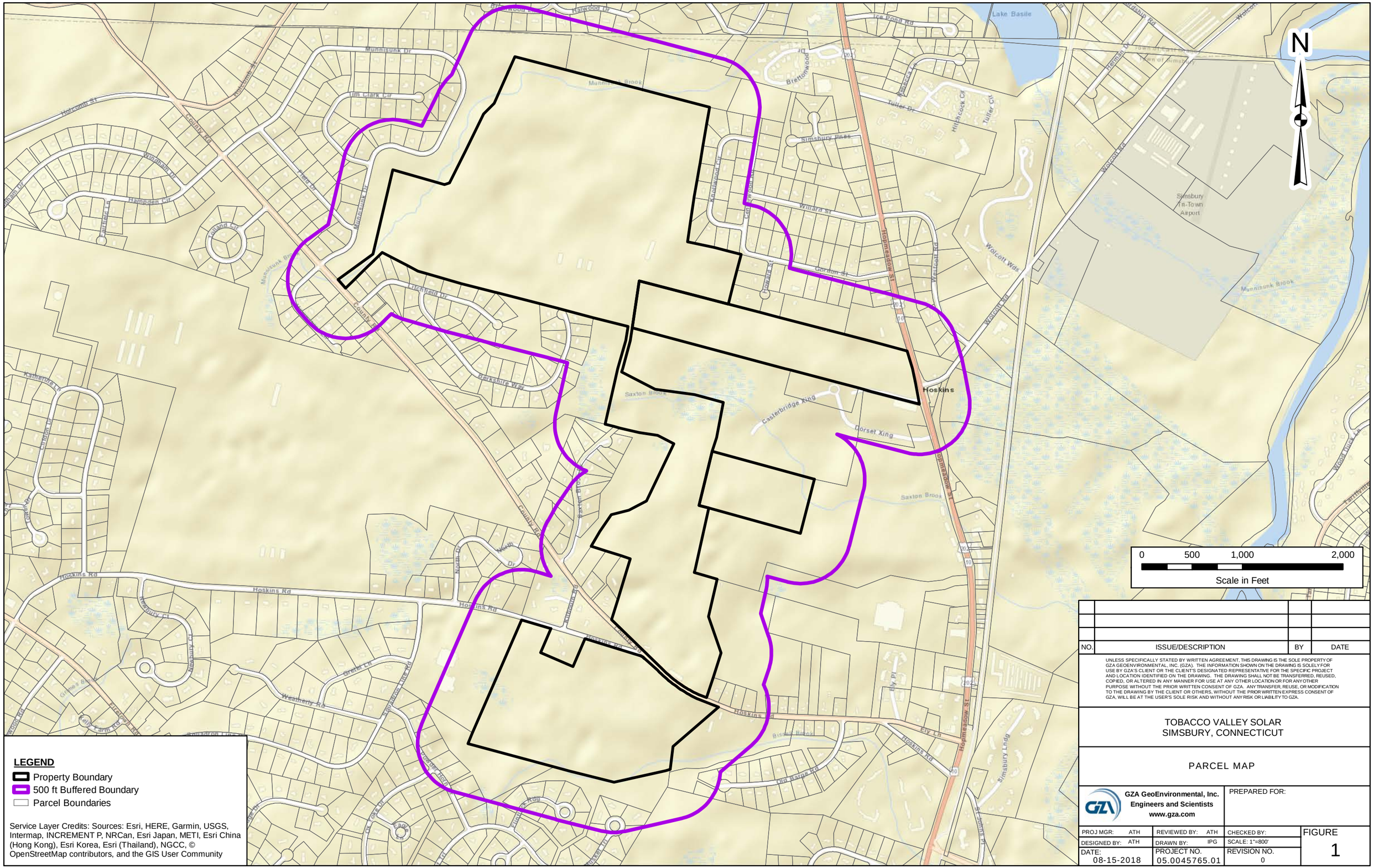
Very truly yours,
GZA GEOENVIRONMENTAL, INC.

Adam T. Henry, LEP
Associate Principal

Gordon T. Brookman, LEP
Principal

Attachments: Figure 1 Parcel Map

List of Adjacent and Nearby Potable Wells
Table 1 Project Action Limits and Laboratory Reporting Limits, EDB, DBCP, TCP
Table 2 Project Action Limits and Laboratory Reporting Limits, VOCs, Pesticides, Metals
Fact Sheet on EPA Method 524.3
ESS Laboratory Standard Operating Procedures: ~~– EPA Method 504.1~~
EPA Method 504.1
EPA Method 524.2
EPA Method 8081
EPA Method 6010C



LEGEND

- Property Boundary
- 500 ft Buffered Boundary
- Parcel Boundaries

Service Layer Credits: Sources: Esri, HERE, Garmin, USGS, Intermap, INCREMENT P, NRCan, Esri Japan, METI, Esri China (Hong Kong), Esri Korea, Esri (Thailand), NGCC, © OpenStreetMap contributors, and the GIS User Community

NO.	ISSUE/DESCRIPTION	BY	DATE

TOBACCO VALLEY SOLAR
SIMSBURY, CONNECTICUT

PARCEL MAP

GZA GeoEnvironmental, Inc.
Engineers and Scientists
www.gza.com

PREPARED FOR:

PROJ MGR: ATH	REVIEWED BY: ATH	CHECKED BY:	FIGURE
DESIGNED BY: ATH	DRAWN BY: IPG	SCALE: 1"=800'	1
DATE: 08-15-2018	PROJECT NO. 05.0045765.01	REVISION NO. 0	

 \\gzaglastonbury\Jobs_45,500-45,999\45765.h65 Deepwater Wind\45765-01.ath\GIS\mxd\F1 PARCELS.mxd, 8/31/2018, 9:24:08 AM, marvin.revere

List of Adjacent or Nearby Potable Wells

1 Centerwood Road

10 County Road
14 County Road
16 County Road
20 County Road

25 Gordon Street

85 Hoskins Road
100 Hoskins Road

3 Howard Street
5 Howard street

3 Knollwood Road
5 Knollwood Road
7 Knollwood Road
9 Knollwood Road
11 Knollwood Road
13 Knollwood Road
15 Knollwood Road
17 Knollwood Road
19 Knollwood Road

TABLE 1
PROJECT ACTION LIMITS AND
LABORATORY REPORTING LIMITS

Cas Number	Analyte	Method	Units	MDL	POTABLE WATER		
					GWPC	CTDPH Action Level	Federal MCL
106-93-4	1,2-dibromoethane (EDB)	504.1	ug/L	0.003	0.05	0.05	0.05
96-12-8	1,2-dibromo-3-chloropropane (DBCP)	504.1	ug/L	0.003	*0.2*	NE	0.2
96-18-4	1,2,3-trichloropropane (123TCP)	504.1	ug/L	0.009	NE	0.05	NE

Notes:
µg/L = microgram per liter
MDL= Minimum Detection Limit
GWPC = CTDEEP Groundwater Protection Criteria
CTDPH = Connecticut Department of Health Action Level
MCL = Federal Maximimum Contaminant Level

TABLE 2
PROJECT ACTION LIMITS AND
LABORATORY REPORTING LIMITS
VOCs, Metals, Pesticides

Compound	Units	MRL	2013-GA GWPC	2008-GWPC
VOCs EPA Method 524.2				
1,1,1,2-Tetrachloroethane	ug/L	0.5	1	NE
1,1,1-Trichloroethane	ug/L	0.5	200	NE
1,1,2,2-Tetrachloroethane	ug/L	0.5	0.5	NE
1,1,2-Trichloro-1,2,2-trifluoroethane	ug/L	1	NE	1000
1,1,2-Trichloroethane	ug/L	0.5	5	NE
1,1-Dichloroethane	ug/L	0.5	70	NE
1,1-Dichloroethene	ug/L	0.5	7	NE
1,1-Dichloropropene	ug/L	0.5	NE	NE
1,2,3-Trichlorobenzene	ug/L	0.5	NE	NE
1,2,3-Trichloropropane	ug/L	0.5	NE	NE
1,2,4-Trichlorobenzene	ug/L	0.5	NE	70
1,2,4-Trimethylbenzene	ug/L	0.5	NE	350
1,2-Dibromo-3-Chloropropane	ug/L	5	NE	0.2
1,2-Dibromoethane	ug/L	0.5	0.05	NE
1,2-Dichlorobenzene	ug/L	0.5	600	NE
1,2-Dichloroethane	ug/L	0.5	1	NE
1,2-Dichloropropane	ug/L	0.5	5	NE
1,3,5-Trimethylbenzene	ug/L	0.5	NE	350
1,3-Dichlorobenzene	ug/L	0.5	600	NE
1,3-Dichloropropane	ug/L	0.5	NE	NE
1,4-Dichlorobenzene	ug/L	0.5	75	NE
2,2-Dichloropropane	ug/L	0.5	NE	NE
2-Butanone	ug/L	10	400	NE
2-Chlorotoluene	ug/L	0.5	NE	14
2-Hexanone	ug/L	10	NE	NE
4-Chlorotoluene	ug/L	0.5	NE	14
4-Isopropyltoluene	ug/L	0.5	NE	210
4-Methyl-2-Pentanone	ug/L	10	350	NE
Acetone	ug/L	10	700	NE
Acrylonitrile	ug/L	0.4	0.5	NE
Benzene	ug/L	0.5	1	NE
Bromobenzene	ug/L	0.5	NE	NE
Bromodichloromethane	ug/L	0.4	NE	0.56
Bromoform	ug/L	1	4	NE
Bromomethane	ug/L	1	NE	3.5
Carbon Disulfide	ug/L	1	NE	700
Carbon Tetrachloride	ug/L	0.5	5	NE
Chlorobenzene	ug/L	0.5	100	NE
Chloroethane	ug/L	1	NE	1000
Chloroform	ug/L	0.5	6	NE
Chloromethane	ug/L	2.4	NE	18.2
cis-1,2-Dichloroethene	ug/L	0.5	70	NE
cis-1,3-Dichloropropene	ug/L	0.4	0.5	NE
Dibromochloromethane	ug/L	0.4	0.5	NE
Dibromomethane	ug/L	0.5	NE	NE
Dichlorodifluoromethane	ug/L	1	NE	1000
Ethylbenzene	ug/L	0.5	700	NE
Hexachlorobutadiene	ug/L	0.4	NE	0.45
Isopropylbenzene	ug/L	0.5	NE	700
Methyl tert-Butyl Ether	ug/L	0.5	100	NE
Methylene Chloride	ug/L	4	5	NE

TABLE 2
PROJECT ACTION LIMITS AND
LABORATORY REPORTING LIMITS
VOCs, Metals, Pesticides

Naphthalene	ug/L	1	280	NE
n-Butylbenzene	ug/L	0.5	NE	61
n-Propylbenzene	ug/L	0.5	NE	61
sec-Butylbenzene	ug/L	0.5	NE	61
Styrene	ug/L	0.5	100	NE
tert-Butylbenzene	ug/L	0.5	NE	61
Tertiary-amyl methyl ether	ug/L	1	NE	NE
Tertiary-butyl Alcohol	ug/L	25	NE	100
Tetrachloroethene	ug/L	0.5	5	NE
Tetrahydrofuran	ug/L	5	NE	4.6
Toluene	ug/L	0.5	1000	NE
trans-1,2-Dichloroethene	ug/L	0.5	100	NE
trans-1,3-Dichloropropene	ug/L	0.4	0.5	NE
Trans-1,4-Dichloro-2-Butene	ug/L	5	NE	NE
Trichloroethene	ug/L	0.5	5	NE
Trichlorofluoromethane	ug/L	1	NE	1000
Vinyl Chloride	ug/L	0.5	2	NE
Xylene O	ug/L	0.5	530	NE
Xylene P,M	ug/L	1	530	NE
Xylenes (Total)	ug/L	1	530	NE
Total Metals EPA Method 6010C				
Arsenic	ug/L	5	50	NE
Copper	ug/L	10	1300	NE
Pesticides EPA Method 8081				
4,4´-DDD	ug/L	0.05	NE	0.1
4,4´-DDE	ug/L	0.05	NE	0.1
4,4´-DDT	ug/L	0.05	NE	0.1
Alachlor	ug/L	0.05	2	NE
Aldrin	ug/L	0.05	NE	0.05
alpha-BHC	ug/L	0.05	NE	NE
beta-BHC	ug/L	0.05	NE	NE
Chlordane (Total)	ug/L	0.002	0.3	NE
delta-BHC	ug/L	0.05	NE	NE
Dieldrin	ug/L	0.001	0.002	NE
Endosulfan I	ug/L	0.05	NE	4.2
Endosulfan II	ug/L	0.05	NE	4.2
Endosulfan Sulfate	ug/L	0.05	NE	4.2
Endrin	ug/L	0.05	NE	2
Endrin Aldehyde	ug/L	0.05	NE	2
Endrin Ketone	ug/L	0.05	NE	2
gamma-BHC (Lindane)	ug/L	0.05	0.2	NE
gamma-Chlordane	ug/L	0.05	NE	0.3
Heptachlor	ug/L	0.05	0.4	NE
Heptachlor Epoxide	ug/L	0.05	0.2	NE
Hexachlorobenzene	ug/L	0.05	NE	NE
Methoxychlor	ug/L	0.05	40	NE
Toxaphene	ug/L	1.29	3	NE

VOLATILE ORGANIC PESTICIDES (DBCP, EDB, 1,2,3-TCP)	
Test Description	Determination of purgeable organic compounds in drinking water
Test Use	Useful for evaluating finished drinking water.
Test Department	Organic Chemistry: Phone 860-920-6581/6666 Fax 860-920-6703
Methodology	EPA Method 524.3-SIM: Capillary Column GC/MS
Availability	Year-round
Sample Requirements	Three (3) 40-mL samples. Two (2) Field Blanks (containing lab-provided reagent water) per sampling trip.
Container type /Preservative	40-mL amber glass vials with caps equipped with PTFE-lined septa and containing the following preservatives: 25 mg Ascorbic Acid preservative 200 mg Maleic Acid preservative
Collection Instructions (Note 1)	For taps, remove aerators and let water run 4-5 minutes. For outdoor locations, sampling location should be in accordance with a preapproved quality assurance project plan. Make sure no air bubbles are present.
Sample Holding Time & Transport	Samples are iced or refrigerated and kept at 4°±2°C from time of collection until analysis. Samples must be analyzed within 14 days of collection.
Unacceptable Conditions	Incomplete requisition form. Insufficient sample volume. Samples received beyond the 14-day holding time. Improper collection/container/preservative.
Requisition Form	Use the Organics/Radiation Water Examination request form.
Required Information	Fill out entire requisition form.
Limitations	Samples with air bubbles larger than a pea cannot be analyzed. These samples will be rejected for analysis and the collector will be notified.
Additional Comments	The CT PHL can use this method to determine the following compounds: 1,2,3-Trichloropropane (TCP) 1,2-Dibromoethane (EDB) 1,2-Dibromo,3-chloropropane (DBCP)

Note 1: See *New England States Environmental Sampling Guide*, latest edition.
https://www.epa.gov/sites/production/files/2015-06/documents/NE-States-Sample-Collection-Manual.pdf

ESS LABORATORY
DIVISION OF THIELSCH ENGINEERING, INC.
185 FRANCES AVENUE
CRANSTON, RHODE ISLAND 02910

SOP NO. 60_5041
EDB, DBCP and 1,2,3-Trichloropropane
(EPA Methods 504 and 8011)

APPROVED BY:

Operations Manager	Date 9/13/11
QA Officer	Date 8/12/11
Laboratory Director	Date 9/13/11

SOP NO. 60_5041
EDB, DBCP and 1,2,3-Trichloropropane
(EPA Methods 504 and 8011)

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of the following compounds in finished drinking water and groundwater:

Analyte	Chemical Abstract Services Registry Number
1,2-Dibromoethane (EDB)	106-93-4
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
1,2,3-Trichloropropane (TCP)	96-18-4

1.2 The method detection limits (MDL) for EDB was determined to be 0.003 µg/L. The MDL for DBCP was determined to be 0.003 µg/L. The MDL for TCP was determined to be 0.009 µg/L. The method is useful for these analytes over a concentration range from approximately 0.02 to 200 µg/L.

2.0 METHOD SUMMARY

2.1 A volume of 35 mL of sample is extracted with 2 mL of hexane. Two µL of extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis. Aqueous calibration standards are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.2 Confirmatory evidence is obtained using a dissimilar column.

2.3 Drinking water samples are tested per EPA Method 504.1. Groundwater samples are tested per EPA Methods 504.1 or 8011. Soil samples are tested per EPA Method 8011. When component concentrations are sufficiently high, Method 524 may be employed for improved specificity.

3.0 HEALTH AND SAFETY

3.1 The toxicity and carcinogenicity of chemicals used in this method should be treated as a potential health hazard and exposure to these chemicals should be minimized.

3.2 EDB, DBCP and TCP have all been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard

solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

3.3 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used for sample analysis. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.4 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.5 Stock solutions and reagents used for sample preservation are used in a hood.

3.6 The MSDS's for the concentrated chemical used in the laboratory are kept on file in a central location where all employees can review them.

4.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

4.1 Samples are collected in 40 mL VOA vials, 2 per sample, and stored at 4°C for up to 14 days when being analyzed for EDB, DBCP and/or TCP.

4.2 Trip Blanks: Replicate field reagent blanks (FRB) must be taken along with each sample set, which is composed of the samples collected from the same general sampling site at approximately the same time. At the laboratory, fill a minimum of two sample bottles with reagent water, seal and ship to the sampling site along with sample bottles. Wherever a set of samples is shipped and stored, it must be accompanied by the FRB.

4.3 Samples that may contain residual chlorine (especially drinking water by Method 504) should be preserved with sodium thiosulfate solution. Add 75 ul of freshly prepared sodium thiosulfate solution (40 mg/ml) to empty 40 ml VOA vials just prior to sample collection. There must be zero headspace (no air bubbles).

4.4 Ground water samples (Method 8011) can be preserved with 1:1 HCl to pH < 2. If residual chlorine is present also preserve with sodium thiosulfate per Para 4.3 above. Soil samples are collected in unpreserved containers and stored at 4°C.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Impurities contained in the extracting solvent usually account for the majority of the analytical problems.

5.2 Solvent blanks should be analyzed on each new bottle of solvent before use.

5.3 Whenever an interference is noted in the reagent water blank, the analyst should reanalyze the extracting solvent. If necessary, it is generally more economical to obtain a new source of solvent.

5.4 Interference-free solvent is defined as a solvent containing less than 0.015 µg/L individual analyte interference. Protect interference-free solvents by storing in an area free of organochlorine solvents.

5.5 This liquid/liquid extraction technique efficiently extracts a wide boiling range of non-polar organic compounds and, in addition, extracts polar organic components of the sample with varying efficiencies. When possible, note sample matrix that have interferences.

5.6 Current column technology suffers from the fact that EDB at low concentrations may be masked by very high levels of dibromochloromethane (DBCM), a common disinfecting byproduct of chlorinating drinking water.

6.0 EQUIPMENT/APPARATUS

6.1 **Sample Containers**: 40 mL screw cap vials each equipped with a Teflon-lined cap. Individual vials shown to contain at least 40.0 mL are calibrated at the 35.0 mL mark so that volumetric measurements of sample volumes can be performed. Precleaned 40ml VOA vials are used.

6.2 **Vials**: Auto sampler, screw cap with Teflon faced septa, 1.8 mL

6.3 **Microsyringes**: 10, 25, 50 and 500 µL

6.4 **Pipettes**: 5.0 mL transfer

6.5 **Standard Solution Storage Containers**: 15 mL bottles with Teflon lined screw caps.

6.6 **Gas Chromatography System**

6.6.1 The gas chromatograph must be capable of temperature programming between 40°C and 275°C. GC is equipped with a linearized electron capture detector and a capillary column split/splitless injector.

6.6.2 Two gas chromatography columns are used. Column A, RTX CL Pesticides, provides separation of the method analytes without interferences from trihalomethanes. Column A is used as the primary analytical column unless routinely occurring analytes are not adequately resolved. Column B RTX Pesticide II, is used as the confirmatory column.

6.6.3 Columns: Restek® RTX CL Pesticide I, II 30mm x 0.25mm x 0.50µm film thickness fused silica capillary column or equivalent. The linear velocity of the helium carrier gas is set at 25cm/second at 100°C.

6.7 **100 mL Volumetric Flasks**

6.8 **1.0 L Bottle**, Wide Mouth, borosilicate glass w/ Teflon lined cap

6.9 **Nitrogen Evaporator**, Organomation® N-Evap

6.10 **Drying Oven** capable of maintaining 105°C, VWR® Series 1330F

6.11 **Graduated Cylinder**, 50 mL Glass

6.12 **Analytical Balance**, OHAUS® Precision Standard SP-400

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 **Hexane extraction solvent**: UV Grade, distilled in glass.

7.1.2 **Methanol**: ACS Reagent Grade, demonstrated to be free of method analytes above the MDLs.

7.1.3 **Sodium Chloride, NaCl**: ACS Reagent Grade, for pretreatment, before use, place in muffle furnace at room temperature. Increase temperature to 400°C for 30 minutes. Place in a bottle and cap.

7.1.4 **Sodium Thiosulfate, $Na_2S_2O_3$**: ACS Reagent Grade, for preparation of solution (40 mg/mL), dissolve 1 g of $Na_2S_2O_3$ in reagent water and bring to 25 mL volume in a volumetric flask.

7.2 **Reagent Water**: water free of interferences above the analyte MDLs.

7.2.1 Passing tap water through a filter bed containing active carbon generates reagent water. Change the activated carbon when there is evidence that volatile organic compounds are breaking through the carbon.

7.2.2 Reagent water may also be prepared by boiling water for 15 minutes. While still hot, transfer water to a narrow mouth crew cap bottle with a Teflon seal.

7.3 **Standards**:

7.3.1 **Primary Standards** are purchased from Restek at 200 mg/L.

7.3.2 **Second Source Standards** are purchased from Ultra at 200 mg/L for EDB and DBCP; and from Ultra at 1000 mg/L for TCP.

7.3.3 **Surrogate solution:** Pentachloroethane purchased from Ultra at 100mg/L.

7.3.4 **Working Surrogate**(0.2 ug/ml) by diluting 10 ul to 5 mls with Methanol.

7.3.5 **Working Standards**: Use the primary solutions to prepare working standards that contain EDB, DBCP and TCP in methanol at 0.2 ug/ml. Dilute 5ul of each of the primary standards to 5ml with Methanol. Store the working standards in 5ml vials with Teflon lined screw caps. Store the solutions with minimal headspace and check frequently for signs of degradation. For the second source standard, dilute 5ul of the primary EDB/DBCP standard and 40 ul an intermediate TCP standard (prepared by diluting 62.5ul of the primary standard to 2.5ml) to 5ml with Methanol.

7.3.6 **Laboratory Reagent Blank (LRB)** : Add 6-7 grams of NaCl to 35 ml of DI water. Add 35 ul of the working surrogate (0.2 ug/L final conc). Immediately extract like a sample and analyze.

7.3.7 **MDL Check Sample:** Add 6-7 grams of NaCl to 35 ml of DI water. Add 2 ul of an MDL check standard (0.2 ug/L EDB/DBCP and 0.5 ug/L TCP final conc), made by diluting 5 ul of EDB/DBCP standard (see 7.3.2) and 100ul intermediate TCP standard (see 7.3.5) to 5ml with methanol. Add 35 ul of the working surrogate (0.2 ug/L final conc). Immediately extract like a sample and analyze.

7.3.8 **BS/BSD: (Laboratory Fortified Blank; LFB):** Add 6-7 grams of NaCl to 35 ml of DI water. Add 35 ul of the primary working standard (0.20 ug/L final conc). Add 35 ul of the working surrogate (0.2 ug/L final conc). Immediately extract like a sample and analyze. NOTE: LFB is prepared in the same manner as a calibration verification standard and can be used to satisfy CCV requirement.

7.3.9 **Matrix Spike:** Add 6-7 grams of NaCl to 35 ml sample. Spike the 35 ml sample with 35 ul of second source working standard (Para 7.3.5). Add 35 ul of the working surrogate (0.2 ug/L final conc). Immediately extract like a sample and analyze.

7.3.10 **Calibration Standards** are made by adding an appropriate volume of a working standard to 35 ml of reagent water in a voa vial. First add 6-7 grams of NaCl to each vial. Use the appropriate syringe (10 ul, 25 ul, 100 ul, 250 ul, 500 ul) to rapidly inject the alcoholic standard into the water in the vial. Remove the needle as quickly as possible after injection. Mix by inverting the flask several times.

7.3.10.1 There are seven calibration standards used at the following concentrations:

Standard/ Surrogate amount (µL)	Final Volume Hexane (mL)	Sample Volume (ml)	On Column Conc (ug/L)	Final Conc. (µg/L)
2	2	35	0.2	0.011
5	2	35	0.5	0.0285
7	2	35	0.7	0.04
14	2	35	1.4	0.08
35	2	35	3.5	0.20
70	2	35	7.0	0.40
140	2	35	14.0	0.8
350	2	35	35.0	2.0

7.3.11 **Second Source standard:** (QCS) Add 6-7 grams of NaCl to 35 ml of DI water in a voa vial. Add 17.5 ul of the second source working standard (0.10 ug/L final conc). Add 35 ul of the working surrogate.(0.2 ug/L final conc) Immediately extract like a sample and analyze.

7.3.12 NOTE: Stock standards are stable for at least four weeks, when stored at 4°C and away from light.

8.0 CALIBRATION

8.1 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for each compound. The %RSD should be less than 20% (less than 10% for Method 8011), for each compound.

8.1.1 Linearity - If the %RSD of any compound is 20% or less (10% for Method 8011), then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.1.2 If the RSD of any compound exceeds 20% (10% for 8011), then one of the following options must be applied to the initial calibration in this situation or a new initial calibration must be performed.

8.1.2.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the required QC limit. This option would apply in those instances where a linear instrument response is expected.

8.1.2.2 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards; **do not** force the curve through zero. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 and the Coefficient of Determination (COD), r^2, must be greater than or equal to 0.99 (6 data points). This can easily be accomplished within the EnviroQuant software.

8.1.3 Immediately after the initial calibration has been generated then the initial calibration has to be verified using a second source standard (§7.3.11). The percent recovery for the independent calibration verification standard is 80-120% for all compounds.

8.1.4 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a calibration standard (§7.3.10) at 0.2 µg/L final concentration. (3.5 ug/L on column) Determine if the analytes have % Deviation less than 20%. NOTE: Method 504.1 requires that the CCV concentration be varied, so that several points in the calibration range are verified.

8.1.5 Retention time (RT) window position is to be established for each analyte and surrogate at method set-up and after any major maintenance, such as a column change and at the beginning of the analytical shift. RT width is ± 3 times standard deviation from each analyte RT from a 72 hour study. Verify for each calibration standard.

9.0 PROCEDURE

9.1 Aqueous Sample Preparation

9.1.1 Remove samples and standards from storage and allow them to reach room temperature.

9.1.2 Prepare a VOA vial with 35 ml of DI water (weigh out 34.9g DI water at room temperature, 22°C). Use this as a "go-by" to adjust the volume of samples to 35 ml. For samples and field reagent blanks in 40mL vials, remove the container cap. Line up the sample next to the go-by and,

using a 5 mL transfer pipette, discard enough water so that 35 ml remains.

9.1.3 Spike the samples with the appropriate surrogate and spike.

9.1.4 For each Batch of 20 samples prepare the following:

9.1.4.1 CCV/QCS
9.1.4.2 LRB (Method Blank)
9.1.4.2 Solvent (Calibration) Blank (not included in batch extraction)
9.1.4.3 LFB/LFBD (BS/BSD)
9.1.4.4 MS
9.1.4.5 DUP
9.1.4.6 Replicate FRBs (Trip Blanks – per client request)
9.1.4.7 MDL Check

9.2 Micro-extraction and Analysis

9.2.1 Remove the container cap and add 6 -7 g NaCl (15 ml vial with containing 6.5g NaCl may be used as a go-by) to the sample.

9.2.2 Recap the sample container and dissolve the NaCl by shaking for about 20 seconds.

9.2.3 Remove the cap and, using a transfer pipette, add 2.0 mL of hexane. Recap and shake vigorously for one minute. Allow the water and hexane phases to separate. (If stored at this stage, keep the container upside down.)

9.2.4 Remove the cap and carefully transfer 0.5 mL of the hexane layer into an autoinjector using a disposable glass pipette.

9.2.5 Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second autoinjector vial. Reserve this second vial at 4°C for a re-analysis if necessary.

9.2.6 Transfer the first sample vial to an autoinjector set up to inject 2.0 µL portions into the gas chromatograph for analysis.

9.2.7 Once extracted, the extracts must be analyzed within 24 hours.

9.3 Soil Sample Preparation

9.3.1 Weigh out about 5.0 g sample into a VOA vial.

9.3.2 Add 35.0 ml of Reagent Water

9.3.3 Continue as described in Sections 9.1.3 and 9.1.4.

10.0 CALCULATIONS

10.1 Identify the method analytes in the sample chromatogram by comparing the retention time of the suspect peaks to retention times of the calibration standards and the laboratory control standards analyzed using identical conditions.

10.2 The calibration curve is setup to calculate the final concentration of the sample in ug/L. Calculations are as follows:

10.2.1 Calculate the corrected sample contraction as:

$$\text{Concentration, } \mu g/L = C \times \frac{Ve}{Vs}$$

Use the calibration curve or calibration factor (Section 7.3.10) to directly calculate the uncorrected (on column) concentration (C) of each analyte in the sample . Ve is the extract volume (2mls) and Vs is the sample volume (usually 35mls)

10.3 Results should be reported to an MRL of 0.015 ug/L (0.025 ug/L for 1,2,3-TCP).

11.0 QUALITY ASSURANCE/QUALITY CONTROL

11.1 Use of this method requires a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of instrument performance check solutions (ICP), laboratory reagent blanks (LRB), laboratory fortified blanks (LFB), laboratory fortified sample matrix (LFM) and quality control samples (QCS) to evaluate and document data quality. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

11.1.1 The analyst must make an initial determination of the method detection limits and demonstrate the ability to generate acceptable precision with this method. This is established as described in Sect. 11.2.

11.1.2 Each day, the analyst must analyze a laboratory reagent blank (LRB) and, when provided, a field blank (Sect.7.3.6), to demonstrate that interferences from the analytical system are under control, before any samples are analyzed. In general, background interferences co-eluting with method analytes should be below the method detection limits.

11.1.3 The laboratory must, on an ongoing basis, demonstrate through the analyses of laboratory fortified blanks (LFB) that the measurement

system operation is in control. This procedure is described in Sect. 7.3.8. The frequency of the LFB analyses is equivalent to 10% of all samples analyzed. Recoveries must be 60-140% (70-130% for Method 504).

11.1.4 The laboratory should demonstrate the ability to analyze low-level samples weekly. The procedure for low-level LFB samples is described in Sect. 11.4. Recovery shall be 60-140% (70-130% for Method 504).

11.2 To establish the ability to achieve low detection limits and generate acceptable accuracy and precision, the analyst should perform the following operations:

11.2.1 Prepare seven samples at 0.02 µg/L by diluting 2 µL of the MDL check sample concentrate (Sect. 7.3.7) into 35 mL aliquots of reagent water in 40 mL bottles. Cap and mix well.

11.2.2 Analyze the well-mixed MDL check samples per Section 9.0.

11.2.3 Calculate the average concentration found (x) in µg/L and the standard deviation of the concentrations in µg/L for each analyte. Then calculate the MDL for each analyte.

11.2.4 For each analyte, x should be between 70% and 130% of the true value. Additionally, the calculated MDL should meet data quality objectives. If performance is acceptable and analysis is actual, then samples can begin. If any analyte fails to meet the data quality objectives on the basis of high variability, correct the source of the problem and repeat the test.

CAUTION: No attempts to establish low detection limits should be made before instrument optimization and adequate conditioning of both the column and the GC system. Conditioning includes the processing of LFB and LFM samples containing moderate analyte concentrations.

11.3 The laboratory must demonstrate on a frequency equivalent to 10% of the samples analyzed that the measurement system is in control by analyzing an LFB of the analytes at 0.20 µg/L concentration level.

11.3.1 Prepare an LFB sample (0.20 µg/L) by adding 35 µL of LFB concentrate (Sect. 7.5) to 35 mL of reagent water in a 40 mL bottle.

11.3.2 Immediately analyze the LFB sample according to Section 9.0 and calculate the recovery for each analyte. The recovery must be between 60% and 140% of the expected value. (70-130% for Method 504)

11.3.3 If the recovery for either analyte falls outside of the designated range, the analyte fails the acceptance criteria. A second LFB containing each

analyte that failed must be analyzed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test.

11.4 The laboratory will demonstrate the ability to analyze low-level samples at least once per week when analysis is being performed.

11.4.1 Prepare an MDL check sample (0.02 µg/L) as outlined in Sect. 7.3.7 and immediately analyze per Section 9.0.

11.4.2 The instrument response must be indicating that the laboratory's MDL is distinguishable from instrument background signal. If it is not, correct the problem and repeat the MDL test in Sect. 11.4.1.

11.4.3 For each analyte, the recovery must be between 60% and 140% of the expected value.

11.4.4 When either analyte fails the test, the analyst should repeat the test for that analyte. Repeated failure, however, will confirm a general problem with the measurement system or faulty samples and/or standards. If this occurs, locate and correct the source of the problem and repeat the test.

11.5 A quality control sample (QCS) shall be analyzed at least weekly for Method 8011 and at least quarterly for Method 504.1. This is the second source standard (Sect 7.3.11). If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem source. Recovery must be 60-140% (70-130% for Method 504).

11.6 At least once in every 20 samples, fortify an aliquot of a randomly selected routine sample with known amounts of analytes. The added concentration should not be less than the background concentration of the sample selected for fortification. To simplify these checks, it would be convenient to use LFC concentrations = 10x MDL. Over time, recovery should be evaluated on fortified samples for all routine sources. Recovery should be 65-135% (Method 504.1) or 30-150% (Method 8011).

12.0 DATA VALIDATION

12.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges in accordance with SOP 80.0040 and Section 12.4 of this SOP. The only exceptions made to ranges would be the following:

12.1.1 For Duplicates, the RPD should be ±30%. However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

12.1.2 For matrix spikes, the % Recovery should be 30-150% (65-135% for Method 504). If the matrix spike is outside limits, check the BS/BSD. If the BS/BSD is within limits, matrix interferences are present and should be noted in the narrative.

12.1.3 Analytical batches with method blanks > ½ the MRL will be re-prepped and re-analyzed with the following exceptions:

12.1.3.1 Samples that are at least twenty times higher than the method blank may be reported.

12.1.3.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

12.1.3.3 If the analyte is found in the method blank > ½ the MRL but is not in any of the associated samples, no corrective action is needed.

12.1.3.4 Any results that are reported with method blank contamination must be B-flagged.

12.2 For the BS/BSD, the %Recovery should be 60-140% (70-130% for Method 504). If the BS/BSD is outside this criteria, the analytical batch will be re-extracted and re-analyzed with the following exceptions: (Method 8011 only - No exceptions for Method 504)

12.2.1 For BS/BSD >130%, samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.

12.2.1.1 For BS/BSDs < 60%, RPD < 40% , samples with results above the regulatory limit may be reported.

12.2.1.2 In some instances there may be insufficient sample to re-extract. The client is to be contacted and the results, if reported, are to be reported as estimated values when this occurs.

12.2.1.3 Any samples that are reported with invalid BS/BSD data must have a notation in the case narrative.

12.3 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

12.4 All data is reviewed for accuracy by a second analyst. Results of this review are noted on the instrument run log in the second level review comment section per SOP 80.0040.

13.0 REFERENCES

13.1 SW-846 Method 8011, On-line Test Methods for the Analysis of Solid Waste

13.2 USEPA Analytical Methods, Water Science, Test Method 504.1

13.3 TNI Standard: Volume 1, Module 2 and Volume 1, Module 4.

13.5 Richard, J.J., G.A. Junk, "Liquid Extraction for Rapid Determination of Halomethanes in Water", Journal AWWA, 69, 62, January 1977.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15.0 METHOD PERFORMANCE

15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 80-120% Recovery and %RSD of $\leq$ 20%.

16.0 DEFINITIONS

16.1 Refer to TNI 2009 Standard, Module 2, Section 3 - Terms & Definitions, located in Network Directory Folders Q:\Lab\NELAC Standards.

16.2 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.3 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.5 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine

laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.6 **Blank Spike or Lab control sample (BS or LCS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.7 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.8 **Duplicate**: An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

16.9 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.10 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.11 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.12 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.13 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

16.14 **Trip Blank (Field Reagent Blank)**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 BA/BS Chemistry, Natural & Applied Science or equivalent experience.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 **Instrument Maintenance**: The following procedure is performed when the instrument is initially set up or when a continuing calibration has failed the QC criteria.

18.1.1 Set the GC system to room temperature.

18.1.2 Turn off oven.

18.1.3 Remove column by unscrewing the column in the injection port.

18.1.4 Remove septum nut and septa. Discard septa.

18.1.5 Remove the insert retainer nut. This will expose the O-ring and glass liner. Using a set of tweezers, remove O-ring and liner. If O-ring is not distorted then set aside for later use. Otherwise, replace O-ring. Remove the glass liner. Rinse liner with methanol and scrub with a cotton swab. If the liner is visibly stained, then replace with a new one.

18.1.6 With cotton swab dipped in methanol, clean the injection port and retainer nut.

18.1.7 Remove the gold seal nut located on the bottom of the injection port. With a cotton swab and methanol, clean the gold seal.

18.1.8 Replace all parts in the following order:

18.1.8.1 Gold seal nut. Hand tighten and 1/4 turn with wrench.

18.1.8.2 Insert clean or new glass liner.

18.1.8.3 Place O-ring over liner. Slide O-ring over and down the liner until it fits snug against the injection port.

18.1.8.4 Replace insert retainer nut.

18.1.8.5 Place a new green septa into insert retainer nut.

18.1.8.6 Replace septum nut. **Only hand tighten!**

18.1.8.7 Slide column nut and a new graphite ferrule over column.

18.1.8.8 Using a ceramic tile, cut 3-6 inches off the column. The cut must be square with no jagged edges.

18.1.8.9 Connect column to injection port by inserting 3 mm of column into the injection port and hand tighten column nut then adding 1/4 turn with a wrench.

18.1.9 Make sure all gases are flowing. (Measure flows with bubble meter.) The flow should be between 5 and 6 ml/min.

18.1.10 Turn on injection port temperature.

18.1.11 Set oven temperature to 120°C and allow the system to stabilize. Bake out the oven at 300°C for two hours. Reset back to 120°C.

18.2 Record all maintenance in the instrument's maintenance logbook.

19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS (Including TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA)

20.1 Table 1 - Method Quality Objectives for SW-846 8011.

TABLE 1 Method Quality Objectives for SW-846 8011

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Instrument set up. Each time the ICV or CCV cannot meet criteria.	• Minimum of 5 standards and contains all analytes • Low standard ≤ MRL • RSD≤10%, R≥0.995 R^2≥0.99 (Do not force through zero for LR).	• No allowance. Perform maintenance and recalibrate.
ICV – second source verification standard	Immediately following initial calibration.	• %Rec = 80-120% (***70-130% for 504)*** • Must contain all target analytes. • No allowances	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is within acceptable criteria then calibration is accepted, otherwise recalibrate. • Report exceedance in narrative
CCV	Prior to sample analysis, every 12 hours and every 20 samples and at the end of each analytical sequence.	• Concentration level near midpoint of curve • Must contain all target analytes. • DoD QSM requires all analytes be ≤20% • Recoveries between 60-140% (Method 8011 and Low Level Check): Recovery 70-130 % (Method 504).	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after invalid CCV. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***

Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes< ½ MRL	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20 x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate (BS/BSD) (aka LCS)	One per analytical batch of 20 or fewer samples.	• Prepared using standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Must contain all single component analytes • Recoveries between 60-140% (Method 8011 and Low Level Check): **Recovery 70-130 % (Method 504).** • Laboratories must develop in-house limits that are within above criteria.	If the recovery for either analyte falls outside the designated range, the analyte fails the acceptance criteria. A second LCS containing each analyte that failed must be analyzed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test. Results may not be reported without a valid LCS.
Matrix Spike Duplicate	One per analytical batch of 20 or fewer samples	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard • Must contain all single component analytes • Matrix specific • Recoveries between 30-150%. ***Method 504: Matrix spike % Recoveries are to be 65-135.*** • RPD should be < 30%	• Check BS/BSD, if recoveries are acceptable then note exceedance in project narrative.

Surrogates	Added to all samples and standards.	• Use a minimum of 1 surrogates. • Percent recovery of 30-150% • MCP requires that both columns and both surrogates be within criterion and reported.	• If a surrogate is diluted to a concentration below the lowest standard, then no corrective action is needed. • If surrogates are outside criteria for re-extract, note in project narrative. • If re-extract is within hold and within criteria, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client. • Note exceedance in project narrative. If sample is not re-analyzed due to obvious interference (e.g., UCM), the chromatogram is to be included in the final report.
Identification and Quantitation		• Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. • The lab must report the higher of the two results. • All QA/QC requirements must be met on secondary column as well. • Analytes must fall within retention time windows.	• Flag data that is >40% RPD between two columns. • IF high RPD can be attributed to interference on one of the two columns, the lab should report the lower value and provide a discussion in the case narrative that this approach was employed.
Dibromochloro-methane	Run a DBCM Standard quarterly	• Demonstrate that DBCM is not interfering.	• Flag data

ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 20.0002A
MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA METHOD 524.2)

REVIEWED BY:

Operations Manager	Date 11/17/6
QA Manager	Date 11/20/06
Laboratory Director	Date 11/20/06

Master copy in red. Controlled copies in green.

MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY CAPILLARY COLUMN GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA METHOD 524.2)

1.0 SCOPE AND APPLICATION

1.1 This method describes the technique utilized by ESS Labs in the qualitative and quantitative analysis of volatile samples based on EPA method 524.2. The following are compounds that ESS Laboratory currently determines by this method:

Analyte	Chemical Abstract Service Registry Number
Acetone	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon Disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Dibromochloromethane	124-48-1
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5

Analyte	Chemical Abstract Service Registry Number
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	590-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone	591-78-6
Isopropylbenzene	98-82-8
4-Isopropyltoluene	99-87-6
Methylene chloride	75-09-2
4-Methyl-2-Pentanone	108-10-1
Methyl-t-butyl ether	1634-04-4
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Tetrahydrofuran	109-99-9
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,3,5-Trichlorobenzene	108-70-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl chloride	75-01-4
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-24-3

1.2 This method is used to determine volatile organic compounds in drinking water samples.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The quantitation limit of this method for an individual compound is approximately 0.5 µg/L for drinking water. Method Reporting Limits (MRL) will be proportionately higher for samples that require dilution or reduced sample size to avoid off-scale peaks.

2.0 METHOD SUMMARY

2.1 This SOP provides gas chromatograph/mass spectrometer (GC/MS) conditions for the detection of volatile organic compounds (VOCs).

2.2 This SOP only addresses sample analysis by purge and trap.

2.2.1 Purged sample components are trapped in a tube containing suitable sorbent materials.

2.2.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb components. The analytes are desorbed directly to a bore capillary column for analysis.

2.2.3 Wide bore capillary columns require a jet separator while narrow bore columns can be directly interfaced to the ion source.

2.3 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS).

2.4 Tentative identifications are obtained by analyzing standards under the same conditions used for samples and comparing resultant GC Retention times. Absolute identifications are obtained by comparing the mass spectra of individual compounds to the reference spectra for that compound.

2.5 Concentrations of the identified compounds are measured by relating the response produced for the compound to the response produced by a compound that is used as an internal standard.

3.0 HEALTH AND SAFETY

3.1 Many chemicals used in this procedure are tentatively classified as known or suspected carcinogens. Extreme caution should be used in handling all chemicals in this procedure.

3.2 To minimize exposure, process samples in an exhaust hood or well-ventilated workspace. When working with samples and chemicals, wear gloves to minimize contact and possible absorption. Always wear appropriate eye protection.

3.3 The laboratory employee, prior to attempting this procedure, should review proper emergency response to spills or injury. This includes location of spill kits, emergency eyewash and showers, fire fighting equipment, as well as evacuation routes.

3.4 Material Safety Data Sheets are available for all chemicals used in this procedure. All laboratory employees are required to read these before handling these chemicals.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Non-chlorinated drinking water samples are preserved with 1:1 HCl at a ratio of 100 µL to 40 ml. All samples are collected in triplicate.

4.2 When sampling from an open body of water, such as surface water, wastewater and possible leachate samples, partially fill a 1 L beaker with sample from a representative area. Fill a sample vial with sample from the larger container and adjust the pH of the sample to about 2 by adding 1:1 HCl dropwise while stirring. Check the pH with narrow range pH paper and record the number of drops necessary to adjust the pH to 2.when collecting the actual samples, refill the beaker with fresh sample and pour the sample into sample vials following the filling instructions described in Attachments B or C. Add the appropriate number of drops of 1:1 HCl to each sample to adjust the pH to about 2. If samples are suspected to contain residual chlorine, add ascorbic acid or sodium thiosulfate as described in Attachments B or C.

4.3 When sampling from a faucet, flush for about 10 minutes before collecting the sample.

4.4 When samples are suspected to contain residual chlorine, as is the case for samples received from treatment facilities, 25 mg of ascorbic acid per 40 ml sample is added to the vials before they are sent to the client (samples are unlikely to have residual chlorine present in excess of 5 mg/L). After the vials are filled with sample, two drops of 1:1 HCl are added to each vial. Sampling instructions are provided in Attachments B and C. ***NOTE: Samples to be analyzed only for Trihalomethanes are only preserved with Sodium thiosulfate.***

4.5 Samples for analysis using this SOP must be stored in tightly sealed vials with Teflon-lined silicon septum seals in which the original sample was collected. All samples are chilled when collected and stored in a refrigerator at 4 ± 2 °C within the laboratory.

4.6 Samples for volatile analysis must be analyzed within 14 days of sample collection. If a sample foams vigorously when HCl is added, discard that sample. Client must then collect an un-acidified sample and inform the lab that

the sample must be analyzed within 24 hr of collection time if to be analyzed for any compounds other than THMs.

4.7 The trip blanks are prepared with organic free water and sample preservatives. This water comes from a carbon filtration system located in the VOA lab. A trip blank must accompany each batch of VOC samples at all times.

4.8 Refrigerator blanks are prepared like trip blanks and stored with samples. These blanks are analyzed weekly to check for cross contamination from samples. To prevent cross contamination, all sample containers must be airtight and all high level samples must be stored separately. If the blank is greater than the MRL, then notify the operations manager immediately

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 During analysis, major contaminant sources are volatiles in the laboratory and impurities in the inert purging gas and in the sorbent trap. Avoid Teflon tubing, Teflon thread sealant or flow controllers with rubber components in the purging device since they out-gas organic compounds which will be trapped during the purge operation. Analysis of reagent blanks can be indicative of contaminants. When potential interfering peaks are noted in reagent blanks, purge gas source should changed and the molecular sieve purge gas filter regenerated. Subtracting blank values from sample results in not permitted.

5.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between sample rinsing of the purging apparatus and sample syringes with two portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagents blanks should be analyzed to check for cross contamination.

5.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate Teflon tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

5.4 Traces of ketones, methylene chloride and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol.

5.5 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200 °C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular weight halogenated hydrogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

5.6 Meta- and para- xylenes are co-eluters. They are evaluated and reported as a combined result.

5.7 On the VRX column, 2-chlorotoluene and n-propylbenzene are not completely resolved and they share the same primary ion. The shoulder is integrated based on the secondary ion. These analytes are routinely evaluated and manually integrated when necessary.

6.0 EQUIPMENT/APPARATUS

6.1 Microsyringes: 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, and 1000 µL. These syringes should be equipped with a 20 gauge (0.006 ″ ID) needle.

6.2 Disposable micro-pipettes: 10, 50, 100, and 200 µL volumes. Purchased from Drummond.

6.3 Micro-reaction vessels: 1.0 ml and 5.0 ml purchased from Supelco, catalog. No 3-3293 with Mininert caps. Clean reaction vessels by placing in oven at 105 °C overnight.

6.4 Volumetric flasks: of various sizes with ground glass stoppers.

6.5 Vials: 40 ml, with pierceable Teflon screwcap top.

6.6 Disposable Pasteur pipettes.

6.7 Purge and Trap Devices: (A) Archon Auto-sampler. (B) Tekmar Dohrmann Purge and Trap Concentrator.

6.7.1 Archon autosampler: The Archon Autosampler is designed to automate the tedious sample handling procedures associated with purge and trap analyses for volatile organic compounds (VOCs) under current EPA methods. The Archon can be used for drinking water and waste water.

6.7.1.1 The Archon can be programmed to run up to 54 samples. All method parameters are preset by the analyst. The Archon automatically takes a 25 ml aliquot directly from the VOA vial, injects 1µL of Internal Standard/surrogate standard, and transfers

this to a 25 ml sparge tube to be purged by the Purge and Trap Concentrator.

6.7.2 The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. Purge flow of 40ml/min for 11 min.

6.7.3 The trap is at least 25 cm long and has an inside diameter of at least 0.105 ". This trap is commercially available from Sigma Aldrich/Supelco (Purge Trap K), Catalog No. 24920-U

6.7.4 The desorber is capable of rapidly heating to 265°C. Desorb preheat at 260°C. Desorb Temp 265°C. Desorb time of 4 min.

6.8 Gas Chromatograph: HP-5890. A complete analytical system with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories, including syringes, analytical columns, and gases.

6.9 Column: 60m x 0.25mm ID available from Agilent (DB-VRX).

6.10 Mass spectrometer HP 5971MSD. Capable of scanning from 35-200 amu every second or less utilizing 70 volts electron energy in the electron impact ionization mode producing mass spectra that meets all the method criteria when 25 ng 4-Bromofluorobenzene (BFB) is injected through the gas chromatography inlet.

6.11 Data system: HP MS Chemstation. This system is interfaced to the mass spectrometer detector and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows searching of any GC/MS data files for ions of a specified mass and plotting such ion abundance versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance of any EICP between specified time or scan number limits. The most current version of the EPA/NIST Mass Spectral Library is also available.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 **Methanol**: Purge and Trap Grade, purchased from Fisher (catalog no. A453-500).

7.1.2 **Reagent Water**: Water where interferent is not observed at the Method Detection Limit (MDL) of the parameters of interests. Water is purified using the Nanopure ultra-pure water system located in the VOA lab.

7.1.3 **Hydrochloric acid** (1+1): Carefully add measured volume of concentrated HCl to equal volume of reagent water.

7.1.4 **Sodium Thiosulfate –** Purchased vials containing 1.5 ml 10N solution are submitted to clients for sampling.

7.1.5 **Ascorbic Acid** - Crystalline Powder, U.S.P.-F.C.C. Grade, J.T. Baker

7.2 **Standards**

7.2.1 **Primary Standards**: obtained from a commercial source and stored in the freezer at –10 °C to –20 °C. After opening store in a vial with a mininert valve. All certificates of analysis are marked with the Primary standard ID and placed in a logbook. Primary standards are not to be used after the manufacturer's expiration date and once opened; they may not be used after 2 months. Methanolic solutions prepared for gaseous analytes are closely monitored for deterioration and are stored at less than -10°C. Typically, they are replaced about every two weeks. The gas standards are evaluated for stability by comparing the CCV (primary standard) to the Blank Spike control sample (Second source standard) daily. Inconsistencies (> 30%) require a new Gas ampule. The following primary standards are used:

Primary Standard	Manufacturer	Cat. #	Concentration
Ketones	Restek	30006	5000 µg/ml
Cal Mix #1	Restek	30633	2000 µg/ml
Custom VOA Additions	Restek	558360	2000 µg/ml
Oxygenates	Restek	30465	2000 µg/ml /10,000 µg (TBA)
Gases	Restek	30042-510	2000 µg/ml
Vinyl Acetate	Restek	30216	2000 µg/ml
1,4-Dioxane*	Accustandard	ALR-062N	Neat
1,3,5-Trichlorobenzene	Accustandard	AS-E0176	5000 µg/ml

*Prepare 1,4-Dioxane working standard by adding 10 µl into 1 ml of methanol; final concentration is 10,000 µg/ml

7.2.2 Working Calibration- In a 5 ml volumetric flask, add 62.5 µL Gases, 125 µL – Ketones, 62.5 µL – Cal mix #1, 62.5 µL – Custom VOA additions, 25 µL 1,3,5-Trichlorobenzene 62.5 µL Oxygenates, 237.5 µl of 1,4-Dioxane, 62.5 µL Vinyl Acetate. Volumize to 5ml with methanol. Final concentration will be 125 µg/ml Ketones and tert-Butyl Alcohol; 500 µg/ml 1,4-Dioxane and 25 µg/ml all other compounds.

7.2.3 Secondary Source Standards – obtained from a commercial source.

Secondary Standard	Manufacturer	Cat. #	Concentration
Custom standard	Ultra	CUS-10604	2000 µg/ml
2-CEVE standard	Accustandard	M-601-L0x	2000 µg/ml
VOC Mix	Ultra	DWM-592	2000 µg/ml

Vinyl Acetate	Supelco	40327	5000 µg/ml
502.2 Mix	Accustandard	M-502-10x	2000 µg/ml
Ketone Mix	Accustandard	Clp-022k-25X	2000 µg/ml
1,4-Dioxane*	Ultra	RCC-180	Neat
tert-Butyl Alcohol	Accustandard	S-410	2000 µg/ml

*Prepare 1,4-Dioxane working standard by adding 10 µL into1 ml of methanol. Final concentration is 10,000 µg/ml.

7.2.3.1 Initial Calibration verification /Laboratory Control /Matrix Spike standard(s) (ICV/LCS/MS) used to check the primary standard. Into a 5 ml volumetric flask add 62.5 µL each of Custom standard, 2-CEVE standard, VOC Mix #2, Vinyl acetate standard, and 502.2 Mix. Add 100 µL of Ketone mix, 312.5 µL tert-Butyl Alcohol and 250 µL of 1,4-Dioxane working standard. Volumize to 5 ml with methanol. Final concentration will be 125 µg/L for Ketones and tert-Butyl Alcohol, 500 µg/L for 1,4-Dioxane, and 25 µg/L for all other compounds.

7.2.4 Surrogate standards – The surrogate solution is made up of 4-Bromofluorobenzene and 1,2-dichlorobenzene-d4. The mixed stock surrogate is purchased from Supelco at 2000µg/ml catalog no. 4-8466. The stock solution is logged into the primary standard logbook upon receipt.

7.2.5 The Internal Standard is Fluorobenzene and is purchased from Restek at a concentration of 2000 µg/ml catalog no. 30030.

7.2.5.1 Surrogate/Internal Standard Mix prepared by adding 312.5 µL of the stock surrogate and internal standards to a 5 ml volumetric flask. Volumize to 5 ml with methanol, final concentration 62.5 µg/ml. Transfer to an Archon standard vial and put vial into the Archon standard well. The Archon injects 1µL into each 25 ml sample, final concentration 5 µg/ml.

7.2.6 4-Bromofluorobenzene (BFB) standard –purchased from Supelco at a concentration of 2000 µg/ml catalog no. 4-8083.

7.2.6.1 Add 62.5 µL of stock solution to a 5.0 ml volumetric flask. Volumize with methanol, final concentration 25 µg/ml. The Tune standard is prepared by adding 25uL to 50ml DI Water, inverting 3 times and transferring to a VOA vial. This affords a BFB standard at a concentration of 12.5 ug/L, which is suitable to check an instrument's tune.

7.2.7 Matrix Spiking Standard – See section 7.2.3.1.

7.2.7.1 Working matrix spike solution – Addition of 40 µL to 100 ml of sample yields a concentration of 50 µg/L for Ketones and 10 µg/L for all other target compound.

8.0 PROCEDURE

8.1 Initial calibration for purge-and-trap procedures:

8.1.1 GC/MS operating conditions: The following parameters are setup through Hewlett Packard MS EnviroQuant. A copy of a EnviroQuant method is in Attachment A.

8.1.2

Electron energy:	70 volts
Mass range:	35 amu - 260 amu
Scan time:	3.25 sec/scan
Initial column temperature:	40°C
Initial column holding time:	1.2 min.
Column temperature program:	7°C/min (Rate1), 7°C/min (Rate 2).
Final column temperature:	150°C/min (Rate1), 220°C/min (Rate 2).
Final column holding time:	0.0 min.
Injector temperature:	200°C
Transfer line temperature:	175°C
Carrier gas (Helium):	37.1 ml/min.

8.1.3 Assemble a purge-and-trap device that meets the specification in Section 6.7. New traps are conditioned one hour at 265°C in the bake mode.

8.1.4 Prior to any analysis of samples or standards, the run logbook must be filled out. Each instrument has its own run logbook. Any unusual observations are included in the comment section of the logbook.

8.1.5 A tune check must be performed before the initial calibration curve. The 4-Bromofluorobenzene standard is run on the GC/MS column as described in Section 7.2.6. Analysis is not to begin until the 4-BFB spectra meets the criteria in Section 11.1. The analysis of all calibration standards must be accomplished within 12 hours of this tune check.

8.1.6 Prepare the standards for generating the calibration curve using the Working Standards (see section 7.2.2) and Internal Standard/Surrogate solution (see Section 7.2.5.1) as follows:

8.1.6.1 To 50 ml of acidified (pH≤2) reagent water in a volumetric flask, inject 20 µl of the Working standard for a final concentration of 10 µg/L. Cap, invert three times, and transfer to VOA vial leaving no headspace. Seal tightly and place in Archon auto-sampler for analysis. Repeat this procedure seven times using 0.2, 1.0, 4.0, 8.0, 40, 80 and 160 µL for concentrations of 0.1,

0.5, 2, 4, 20, 40, and 80 µg/L, respectively, to complete the calibration curve.

8.1.7 Carry out the purge-and-trap analysis procedure as described in Section 8.3.

8.1.8 EnviroQuant software is used to tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and internal standard. It is also used to calculate response factors (RF) for each compound relative to its internal standards according to the calculations in Section 9.1.

8.1.8.1 NOTE: It is each analyst's responsibility to become familiar with the software by reading the available manuals. **After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout corresponds to the area account listed in the calibration method in Enviroquant.** Each analyte along with their retention times and the ions used for qualitative analysis are listed in Appendix B.

8.1.9 The average RF is calculated in the EnviroQuant Method for each compound.

8.1.10 Using the RFs from the initial calibration, EnviroQuant software calculates the percent relative standard deviation (%RSD) for all compounds using the equation in Section 9.2. The %RSD should be less than 20% for each compound.

8.1.10.1 Linearity - If the %RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

8.1.10.2 When the RSD exceeds 20%, the plotting and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

8.1.10.3 If a %RSD greater than 30 percent is measured for any method analyte, then corrective action to eliminate a system leak and/or column reactive sites is required before reattempting calibration. Poor purging compounds are listed in Table 2.

8.1.10.4 If the RSD of ANY compound exceeds 20% (never to exceed 30%), then one of the following options must be applied to the GC/MS initial calibration in this situation or a new initial calibration must be performed.

8.1.10.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 20% QC limit. This option would apply in those instances where a linear instrument response is expected.

8.1.10.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points.

8.1.10.4.3 Use a linear calibration. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards. In order to be used for quantitative purposes, the coefficient of determination (R^2) must be greater than or equal to 0.99. This can easily be accomplished within the EnviroQuant software.

8.1.10.4.4 Use a quadratic regression or non-linear calibration model, minimum of six points for this option. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range. (Not an option for South Carolina samples)

8.2 Daily GC/MS analysis: Just prior to all samples and standard analysis, the run logs are filled out according to 8.1.3.

8.2.1 Prior to the analysis of samples, inject 1 µL of the 4-Bromofluorobenzene standard (Section 7.2.6). The resultant mass spectra for BFB must meet all of the criteria in Section 11.1 before sample analysis begins. This criteria must be met each 12-hour shift.

8.2.2 The initial calibration curve must be verified each 12-hour shift. This is accomplished by analyzing a calibration standard at 10 µg/L. The acceptance criterion is ± 30% deviation or drift, for poor purging

compounds (Ketones, 2-CEVE), 50%. Compounds not analyzed for in the batch of samples do not need to meet the criteria.

8.2.3 The internal standard responses and retention times in the continuing calibration standard should be evaluated immediately after data acquisition. If the area for any of the internal standards changes by ± 30% (or – 50% to +100% from the initial calibration) or the retention time changes by ±0.5 minutes from the last daily calibration standard check, the mass spectrometer should be checked for defects. Analysis is not to continue until problem has been resolved.

8.2.4 Method blank analysis should be performed at the following frequency:

8.2.4.1 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour shift and must be analyzed before any samples.

8.2.4.2 A method blank for volatile analysis should contain less than or equal to the MRL of methylene chloride or 5 ppb in the case of 25 ml purge.

8.2.4.3 For all other TCL compounds not listed above, the method blank should be:

8.2.4.3.1 Less than ½ the MRL or less than the level of acceptable blank contamination specified in the approved quality assurance project plan.

8.2.4.3.2 Less than 5% of the regulatory limit associated with an analyte.

8.2.4.3.3 Or less than 5% of the sample result for the same analyte, whichever is greater.

8.2.4.4 If a laboratory method blank exceeds these criteria, the operator should consider the analytical system to be out of control. The source of the contamination should be investigated and appropriate corrective measures should be taken and documented before further sample analysis proceeds. All samples processed with a method blank that is out of control (i.e., contaminated) should be re-extracted/re-purged and reanalyzed. Any deviation will be noted on the customer's report. See section 11.0 for expanded criteria.

8.3 GC/MS analysis: All samples and standards are analyzed under the same conditions in the associated initial calibration. Run logs are filled according to 8.1.3.

8.3.1 All samples and standard solutions should be allowed to warm to ambient temperature before analysis.

8.3.2 Set up the GC/MS system as outlined in Section 8.1.1.

8.3.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 11.1) before analyzing samples.

8.3.4 Place sample in sealed VOA vial in Archon auto-sampler for analysis.

8.3.5 The Archon will automatically inject 1µL of surrogate/internal standard QC mix (Section 7.2.5.1) to the sample. The surrogate/internal standard QC mix is to be added to each continuing calibration standard, blank, sample and spiked sample.

8.3.6 Purge the sample for 11.0 $\pm$0.1 min at ambient temperature.

8.3.7 At the conclusion of the purge time, the purge-and-trap will sound a ready signal from the GC and begin to desorb the trap while initiating the chromatographic temperature program and the CHEMSTATION data acquisition.

8.3.8 The Archon auto-sampler will automatically rinse the purge chamber with reagent water.

8.3.9 After the sample has been desorbed for 3 minutes, the purge-and-trap will recondition the trap by heating to 265°C while back flushing with Helium. This step will take 4 minutes

8.3.10 When necessary to composite samples for analysis, the following procedure is used:

8.3.10.1 Add equal amounts of sample to a 50 ml volumetric flask. Pour off composited sample to a VOA vial leaving no headspace and seal tightly.

8.3.10.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

8.3.10.3 Place composited VOA vial in Archon autosampler for analysis.

8.3.11 If a sample or a dilution of a sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must be performed manually in a 50 ml volumetric flask using reagent water and an appropriate amount of sample.

8.3.11.1 When a sample is analyzed that has any target compound at a level exceeding twice the initial calibration range, this analysis should be followed by a blank reagent water analysis in a different purge chamber to demonstrate that system is clean. If the blank analysis is not free of interferences, the system should be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences. Analysis of sample in the contaminated chamber may not resume until the analysis of reagent water in the chamber is free of any target compounds.

NOTE: Since the analytical system is automated the analyst may not be aware that a high concentration sample has been analyzed. If analysis of subsequent samples occurs, then the presence of the high concentration analyte makes reanalysis of that sample necessary to determine if carryover has occurred.

8.3.12 For matrix spike and matrix spike duplicate analysis, add 40 µL of the matrix spike solution (Section 7.2.3.1) to 100 ml of sample. Disregarding any dilution, this is equivalent to a concentration of 10 µg/L of each matrix spike compound.

8.3.13 All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

8.4 Data interpretation

8.4.1 Qualitative identification

8.4.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the compound obtained on the user's GC/MS. These standard reference spectra are obtained through analysis of the calibration standards. To compare the sample and reference spectra, go into the EnviroQuant software (QEDIT). The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity - any ions over 30% if less than three such ions are present.

8.4.1.2 The requirements for qualitative verification by comparison of mass spectra are:

8.4.1.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a

compound-specific retention time will be accepted as meeting this criterion.

8.4.1.2.2 The RRT of the sample component is within ±0.06 RRT units of the RRT of the standard component.

8.4.1.2.3 The relative intensities of the characteristic ions agree within 20% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 30% and 70%).

8.4.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. ESS Laboratory currently reports total xylenes due to inability of resolving the isomers.

8.4.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

8.4.1.2.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are:

8.4.1.2.6.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

8.4.1.2.6.2 The relative intensities of the major ions should agree within ±30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).

8.4.1.2.6.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

8.4.1.2.6.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

8.4.1.2.6.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

8.4.2 Quantitative Analysis

8.4.2.1 Compounds which have been identified are quantified by the internal standard method, utilizing the integrated abundance of the primary ion. The internal standard used shall be those listed in Section 8.1.7. The EnviroQuant method is setup to calculate the concentration of the analytes of interest in ng/ml using the initial calibration results stored in the method. This raw data number does not account for deviations in sample volume, sample weight, dilution factors, and % moisture. To include these other factors, see section 9.0.

9.0 CALCULATIONS

9.1 Response Factor (RF)
RF=(As x Cis)/(Ais x Cs)

Where:

A_S = Peak area (or height) of the analyte or surrogate.
A_{IS} = Peak area (or height) of the internal standard.
C_S = Concentration of the analyte or surrogate, in µg/L.
C_{IS} = Concentration of the internal standard, in µg/L.

9.2 Percent Relative Standard Deviation (%RSD)
% RSD = (SD/ $RF_{Average}$) x 100%

Where:
RSD = Relative standard deviation.
RF = Mean of 5 initial RFs for a compound.
SD = Standard deviation of average RFs for a compound.

9.3 Percent Difference
%Difference = (($RF_{Average}$ – RFc)/ RF Average) x 100%

Where:
$RF_{Average}$ = Average response factor from initial calibration
RF_c = Response factor from current verification check standard.

9.4 Percent Drift
%Drift = ((C1-C0)/ C1) x 100%

Where:
C_1 = compound standard concentration
C_0 = measured concentration using selected quantitation method

9.5 Concentration of Target Analytes in Water and Water-Miscible Waste
Concentration:
ug/L = (Ax)(Is)(D) / (Ais)($RF_{Average}$)(Vs)

Where:
A_X, A_{IS} = same as in section 9.1.
I_S = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always unitless.
$RF_{Average}$ = Mean response factor from the initial calibration.
V_S = Volume of the aqueous sample extracted or purged (ml). If units of liters are used for this term, then multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/ml, which is equivalent to µg/L.

NOTE: The result on the raw data generated through Enviroquant is the final result in ug/L without any dilution factor applied.

9.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 **Accuracy and Precision**

All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file

QC Sample Preparation:

Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

Sample Analysis:

The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:

Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:

Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where :

s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

x = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision

Accuracy and Precision data should be presented with the following minimuminfo:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable Sample Analyzed by:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results:

Method 524.2 has established initial demonstration of accuracy precision ranges of 80-120% for all analytes and ≤ 20% RSD. If any of the accuracy and precision results do not fall within the criteria then re-prep and reanalyze all QC samples only for those analytes that were no good.

10.2 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, and a matrix spike duplicate. An analytical or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. Within the analytical batch are included individual QC requirements as defined by the analytical (determinative) method. For instance, each injection sequence would begin with a tune and CCV (or initial 8-point calibration and ICV), followed by a method blank, and then the other QC samples (normally a LCS, MS, MSD, etc.) and remaining field samples. Preparation batches of samples may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP. If all field and QC samples could not be completed within the 12-hour clock, then another tune, CCV, and method blank would be required. Each analytical sequence must be documented using the run log.

10.3 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 11.1. The computer software will evaluate the tune information. The analyst should be aware of the process used. The criteria specified in Section 11.1 would typically be derived using three scans, one at the apex, and one on both sides of the apex.

10.4 Run a 8-point initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 11.2.

10.5 Run an initial calibration verification (ICV) standard using secondary standards (7.2.3) following the initial calibration curve. Acceptance criteria are listed in Section 11.3.

10.6 Run a mid-point Continuing Calibration Verification (CCV) at 10 µg/L using the primary source standards on a daily basis before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 11.4. Also run a daily 0.5µg/L LCV (Low Level CCV) before sample analysis using the primary source standards. Trihalomethanes must meet 50 – 150% recovery criteria.

10.7 A method blank must be prepared with each batch of samples not to exceed 12 hours. The method blank should be prepared from organic-free water. The method blanks are to be related to each 12-hour sequence of samples injected. Acceptance criteria for these blanks are listed in Section 11.5. Instrument blanks may be injected at any time in the sequence to verify absence of contamination.

10.8 A laboratory control sample must be prepared and analyzed with each batch of samples. The LCS would be prepared using the second source standard and would contain all method target analytes. Control charts will be maintained bi-annually for the LCS for all target analytes and surrogate spikes. Surrogate spike recoveries from the method blanks will also be control charted along with the LCS surrogate values. Acceptance criterion is 70-130% (poor purgers are 50-150%).

NOTE: The CCV and LCS are considered to be equivalent. Only one will need to be analyzed. In the case of solids, the LCS is extracted similar to the samples.

10.9 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples.

10.9.1 The matrix spike is prepared as described in Section 7.2.7.1.

10.9.2 Matrix spike recovery limits are calculated for each compound and each matrix. The recoveries limits are 70-130% with %RPD of $\leq$ 20%.

10.10 Surrogates recoveries are calculated for each LCS, method blank, QC sample, and field sample analyzed. Acceptance criteria are listed in Section 11.7.

10.11 The relative retention times (RRTs) of identified compounds need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.8.

10.12 Ion abundance for target compounds and any tentatively identified compounds need to meet specific requirements. Acceptance criteria for ion abundance are presented in Section 11.9.

10.13 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

10.14 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high standard, are to be reported as estimated values. Corrective actions are described in Section 11.0.

10.15 MDLs are determined in either reagent water or organic-free sand / soil and verified annually. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.). MDLs (LODs) are determined in accordance with SOP 110.0013

11.0 DATA VALIDATION

All items shall be verified and documented using the data review checklist in Figure 4.

11.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

Table 1. BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-80.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

11.1.1 If the BFB acceptance criteria are not met, perform any or all of the following corrective actions:
Re-inject BFB.
Retune with PFTBA, then re-inject BFB.
Clean MS source, retune with PFTBA, re-inject BFB.

11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After a eight-point initial calibration curve is analyzed, ensure that the following criteria were met.

11.2.1 For the RFs, the %RSD must be less than 30% for all method target analytes, except for the poor purgers where the %RSD must be less than 50%. If acceptance criteria are not met, the following corrective actions should be performed:

11.2.1.1 If the %RSD of any method target analyte is 20%, or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.1.2 If the %RSD of any method target analyte is greater than 20%, calibration curves must be constructed using first or higher order regression fits of the seven calibration points (five points may be used if linear regression is used). The corresponding coefficient of determination must be 0.99 or greater.

11.2.1.3 If these acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using six standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.3 If a 8-point initial calibration was performed, verify that an initial calibration verification (ICV) was performed. The percent difference for all analytes must be within 30%. If not, reanalyze the ICV or prepare a new calibration curve as necessary.

11.4 After the continuing calibration verification (CCV) standard is analyzed, ensure it was run at the required frequency (every 12 hours or initially before daily analysis). All analytes must be within $\pm$ 30% difference or drift(poor purgers $\pm$50%). If criterion is not met, reanalyze the CCV or prepare a new calibration curve as necessary.

11.5 Assess the method blanks. The analyst shall confirm that this blank was analyzed at the required frequency and the criteria in Section 8.2.4 are met. Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed with the following exceptions:

11.5.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above the ½ MRL but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.6 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits on the appropriate blank spike control charts. If these conditions are not met, perform the following corrective actions as appropriate.

11.6.1 If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

11.6.2 If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. Report matrix interference in the project narrative.

11.7 Check the surrogate calculations for correctness for all samples, blanks, LCS, MS, and MSD. Maximum default range is 80 to 120%, for Method 524. If blank or LCS are outside criteria, then stop analysis until problem has been corrected. If samples are outside criteria, re-analyze sample once. If still outside criteria, then note exception in the project narrative. If re-analysis produces results within criteria, then report out valid results only.

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.8.1 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

11.8.2 If the retention time for any internal standard changes by more than 30 seconds from the last daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.9 The analyst must verify that ion abundance meet specific criteria for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

11.9.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.9.2 The relative intensities of ions specified in Section 11.9.1 must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 30 and 70 percent.)

11.9.2.1.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.2.1.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.9.2.1.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

11.9.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.9.3.1.2 The relative intensities of the major ions should agree within ±30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

11.9.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.9.3.1.4 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the internal standards changes by 70 to 130% from the last daily calibration standard check or 50-150% of the initial calibration. The mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.

11.10.1 Check to be sure there are no errors in internal standards preparation or addition. Also check instrument performance.

11.10.2 Re-analyze the sample if any of the above checks do not reveal a problem.

11.11 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.12 All manual integration must be printed when made for verification.

11.13 Besides the items listed in Sections 11.1 through 11.12, the analyst should also verify the additional items as noted in Figure 4.

12.0 REFERENCES

12.1 Methods for the Determination of Organic Compounds in Drinking Water—Supplement III, EPA-600/R-95-131. August 1995

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 80-120% Recovery and %RSD of ≤ 20%. Five (5) sporadic marginal exceedances are allowed.

14.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 1/12/2006

Compound Name	Spk Amt	Avg	RSD	%Rec	Compound Name	Spk Amt	Avg	RSD	%Rec
Dichlorodifluoromethane	10	11.718	7	117	1,1,2-Trichloroethane	10	10.188	3	102
Chloromethane	10	11.390	3	114	1,3-Dichloropropane	10	10.305	2	103
Vinyl Chloride	10	12.420	4	124	Tetrachloroethene	10	9.060	3	91
Bromomethane	10	11.943	3	119	2-Hexanone	50	49.243	3	98
Chloroethane	10	11.548	4	115	Dibromochloromethane	10	10.115	3	101
Trichlorofluoromethane	10	11.965	4	120	1,2-Dibromoethane	10	10.063	2	101
Acetone	50	54.395	5	109	Chlorobenzene	10	10.203	2	102
Methyl Iodide	10	11.918	5	119	1,1,1,2-Tetrachloroethane	10	9.975	2	100
1,1-Dichloroethene	10	11.815	4	118	Ethylbenzene	10	10.418	3	104
Carbon Disulfide	10	11.363	3	114	Xylene P,M	20	20.538	3	103
Methylene Chloride	10	11.733	2	117	Xylene O	10	10.325	3	103
Acrylonitrile	10	11.170	1	112	Styrene	10	10.453	2	105
Methyl tert-Butyl Ether	10	9.815	1	98	Bromoform	10	9.803	3	98

trans-1,2-Dichloroethene	10	10.600	4	106	Isopropylbenzene	10	10.290	2	103
1,1-Dichloroethane	10	10.593	2	106	1,2,3-Trichloropropane	10	9.885	3	99
cis-1,2 Dichloroethene	10	10.523	3	105	Bromobenzene	10	10.045	3	100
2,2-Dichloropropane	10	8.895	5	89	1,1,2,2-Tetrachloroethane	10	10.050	2	
2-Butanone	50	51.740	5	103	n-Propylbenzene	10	10.320	6	103
Bromochloromethane	10	10.935	2	109	2-Chlorotoluene	10	9.715	10	97
Tetrahydrofuran	10	9.123	4	91	4-Chlorotoluene	10	9.978	3	100
Chloroform	10	10.528	2	105	1,3,5-Trimethylbenzene	10	10.315	3	
1,1,1-Trichloroethane	10	9.810	3	98	tert-Butylbenzene	10	10.253	3	103
1,1-Dichloropropene	10	10.340	3	103	1,2,4-Trimethylbenzene	10	10.090	3	101
Carbon Tetrachloride	10	10.308	4	103	sec-Butylbenzene	10	10.345	4	103
Benzene	10	10.118	1	101	1,3 Dichlorobenzene	10	10.195	2	102
1,2-Dichloroethane	10	10.800	2	108	4-Isopropyltoluene	10	10.060	3	101
Trichloroethene	10	10.555	5	106	1,4 Dichlorobenzene	10	9.945	3	
1,2-Dichloropropane	10	9.898	2	99	n-Butylbenzene	10	10.418	3	104
Dibromomethane	10	10.430	2	104	1,2 Dichlorobenzene	10	4.755	2	95
Bromodichloromethane	10	10.473	3	105	DBCP	10	10.030	3	10
cis-1,3-Dichloropropene	10	9.520	1	95	1,2,4-Trichlorobenzene	10	8.905	1	89
Toluene	10	10.235	4	102	Hexachlorobutadiene	10	9.590	3	96
t-1,3-Dichloropropene	10	9.383	1	94	Naphthalene	10	9.985	4	100
4-Methyl-2-pentanone	50	49.858	4	100	1,2,3-Trichlorobenzene	10	9.678	2	9

16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte

concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

16.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

16.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 If BFB criteria are not met, the analysis must be repeated. Analysis can not begin until BFB meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

18.2 If manual or auto-tune does not produce BFB spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

18.3 Method blanks must not contain any target compound greater than ½ the MRL with the exception of methylene chloride. If the method blank does not meet

criteria, then system must be cleaned before processing samples. This includes washing the purge vessel and baking the trap.

18.4 See laboratory supervisor or operations manager for all other maintenance problems.

18.5 Record all maintenance in the instrument's maintenance logbook.

19.0 Data Management And Records

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

Table 2 – QC Summary for EPA Method 524.2

Attachment A – MS6 Run Log – VOA

Attachment B – Drinking Water Sampling Instructions using Sodium Thiosulfate

Attachment C – Drinking Water Sampling Instructions using Ascorbic Acid

Attachment D - Run Parameters

TABLE 2
Quality Control Summary for EPA Method 524.2

QC Element	Performance standard	Corrective action
GC/MS Tune with BFB	Criteria listed in section 11.1 Performed Every 12 hours	Perform instrument maintenance, re-tune. Suspend analyses until within tune criteria.
Initial Calibration	Minimum of 5 standards Low standard at MRL. %RSD≤20% or R^2>0.99 for all compound.	Recalibrate when ICV or CCV no longer meet criteria.
Continuing Calibration	Every 12 hours prior to sample analysis. Concentration at 10 ug/L. Must contain all target analytes. %D must be ≤ 30% for all analytes, ≤50 % for poor purgers. Can use as LCS if performed using source separate from Ical	Re-analyze once with freshly prepared CCV. If still outside criteria, re-calibrate.
ICV	Performed immediately after Ical. %D must be ≤ 30% for all analytes, ≤50 % for poor purgers.	Re-analyze once with freshly prepared ICV. If still outside criteria, re-calibrate.
Method Blanks	Every 20 samples prior to sample analysis and after CCV. Matrix and preservative specific. Target analytes < ½ MRL except for common lab contaminants (such as acetone, MEK, and methylene chloride) which must be < 5x MRL.	Locate source of problem, re-analyze method blank. Re-analyze associated samples unless sample is non-detect for the contaminant. See section 11.0.
Laboratory Control Sample (LCS)	Every 12 hours prior to sample analysis. Concentration same as MS and prepared from source different than Ical. Must contain all target analytes. 70-130% Recovery	Re-analyze the LCS Locate source of problem and re-analyze associated samples.
MS/MSD	Every 20 samples Prepared from source different than Ical. Percent recoveries 70-130% RPD ≤ 20%	Check LCS If LCS recoveries acceptable, note non-conformance in narrative.
Surrogates	80-120%	If outside criteria, re-analyze sample unless obvious interference is present.
Internal standards	Area counts in sample must be between 70-130% of the areas in the associated CCV or 50-150% of the Ical. Retention times must be within ± 30s of associated CCV.	If outside criteria, re-analyze sample unless obvious interference is present.

Attachment A

VOA ESS LABORATORY MS6 RUN LOG

DATE :	2/24/2015		Work orders reported: [redacted]						
ANALYST:	MD						Control# 20.0032		
METHOD:	DW021915		MATRIX		Element CAL ID:				
Surrogate Std:	5B18115		AQ		1502026				
Internal Std:	5B18117								

VIAL #	SAMPLE ID	METHOD	FILE ID	Dilution	STD ID/COMMENTS	pH	BATCH ID	Sequence ID	RW
1	TUN1	DW021915	MJ02474	1	5B24121		CB52453	CYB0298	MD
2	CCV1	DW021915	MJ02475	1	5B24122		CB52453	CYB0298	MD
3	BS1	DW021915	MJ02476	1	5B19081 20ul/50ml		CB52453	CYB0298	MD
4	BSD1	DW021915	MJ02477	1	5B19081 20ul/50ml		CB52453	CYB0298	MD
5	LCV1	DW021915	MJ02478	1	5B19001 2ul/50ml		CB52453	CYB0298	MD
6	TB	DW021915	MJ02479	1			CB52453	CYB0298	MD
7	BLK1	DW021915	MJ02480	1			CB52453	CYB0298	MD
8	RB022015R1	DW021915	MJ02481	1			CB52453	CYB0298	MD
9	1502320-01	DW021915	MJ02482	1		<2	CB52453	CYB0298	MD
10	SRM1	DW021915	MJ02483	1	5B24123-0.1PPB		CB52453	CYB0298	MD
11	SRM2	DW021915	MJ02484	1	5B24124-0.2PPB		CB52453	CYB0298	MD
12	SRM3	DW021915	MJ02485	1	5B24125-0.5PPB		CB52453	CYB0298	MD
13	SRM4	DW021915	MJ02486	1	5B24126-1.0PPB		CB52453	CYB0298	MD
14	SRM5	DW021915	MJ02487	1	5B24127-2.0PPB		CB52453	CYB0298	MD

QUADS:	None
SCV:	All Pass
BS/BSD:	All Pass
CCAL:	All Pass
CCAL%D:	Acetone>20%.All other compounds >20% are on Linear Regression
Comments:	All deletions made due to failed spectra/retention time-MD

Review (includes a check and review of manual integrations)

CLD 2/26/15

Attachment B: Drinking Water sampling instructions using Sodium Thiosulfate

ESS Laboratory VOC 524.2 Sample Collection Procedure for samples Containing Residual Chlorine

Application: 524.2 Volatile analysis when gases such as vinyl chloride or Table 7 analytes (see attached) **are not to be determined**. This procedure **may be used** for Trihalomethane (THM) samples and in the case of THMs, samples do not need to be acidified at the time of collection.

Number of Vials: **3 per sample**

Vial Description: VOA vial containing 1.5 ml 10N of Sodium Thiosulfate ($Na_2S_2O_3$).

Materials: VOA vial containing 1:1 HCl
Plastic transfer pipette

Sampling Procedure:

If samples, such as finished drinking water or waste water, are suspected to contain residual chlorine, fill sample vial to overflowing, but take care not to flush out the Sodium thiosulfate. No air bubbles should pass through the sample as the vial is filled, or be trapped in the sample when the vial is sealed. Adjust the pH of the each sample vial to <2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Seal the sample vials, Teflon face down, and invert three times to mix.

Sample Shipping:

Samples must be kept cool with ice or ice packs (4^{o} +/- 2^{o} C)

A **Trip Blank** must accompany each batch of samples. A Trip Blank is a VOA vial that has been filled in the laboratory with Type I water and contains 10% Sodium thiosulfate.

For additional information call ESS Laboratory: 401–461-7181

Attachment C: Drinking Water sampling instructions using **Ascorbic Acid**

ESS Laboratory VOC 524.2 Sample Collection Procedure for samples Containing Residual Chlorine

Application: Full 524.2 Volatile analysis. *Must acidify at the time of collection.*

Number of Vials: **3 per sample**

Vial Description: VOA vial containing 25 mg of Ascorbic Acid

Materials: VOA vial containing 1:1 HCl
Plastic transfer pipette

Sampling Procedure:

If samples, such as finished drinking water or waste water, are suspected to contain residual chlorine, fill sample vial to overflowing, but take care not to flush out the ascorbic acid. No air bubbles should pass through the sample as the vial is filled, or be trapped in the sample when the vial is sealed. Adjust the pH of the each sample vial to <2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Seal the sample vials, Teflon face down, and invert three times to mix.

.

Sample Shipping:

Samples must be kept cool with ice or ice packs (4^{o} +/- 2^{o} C)

A **Trip Blank** must accompany each batch of samples. A Trip Blank is a VOA vial that has been filled in the laboratory with Type I water and contains 25 mg of ascorbic acid.

For additional information call ESS Laboratory: 401–461-7181

TABLE 7. ACCURACY AND PRECISION DATA FROM SEVEN DETERMINATIONS OF METHOD ANALYTES IN REAGENT WATER USING WIDE-BORE CAPILLARY COLUMN 4[a]

Compound	True Conc. (µg/L)	Mean Conc. Detected (µg/L)	Rel. Std. Dev. (%)	Method Detect. Limit (µg/L)
Acetone	1.0	1.6	5.7	0.28
Acrylonitrile	1.0	0.81	8.7	0.22
Allyl Chloride	1.0	0.90	4.7	0.13
2-Butanone	2.0	2.7	5.6	0.48
Carbon Disulfide	0.20	0.19	15	0.093
Chloroacetonitrile	1.0	0.83	4.7	0.12
1-Chlorobutane	1.0	0.87	6.6	0.18
trans-Dichloro-2-Butene	1.0	1.3	8.7	0.36
1,1-Dichloropropanone	5.0	4.2	7.7	1.0
cis-1,3-Dichloropropene	0.20	0.20	3.1	0.020
trans-1,3-Dichloropropene	0.10	0.11	14	0.048
Diethyl Ether	1.0	0.92	9.5	0.28
Ethyl Methacrylate	0.20	0.23	3.9	0.028
Hexachloroethane	0.20	0.18	10	0.057
2-Hexanone	1.0	1.1	12	0.39
Methacrylonitrile	1.0	0.92	4.2	0.12
Methylacrylate	1.0	1.2	12	0.45
Methyl Iodide	0.20	0.19	3.1	0.019
Methylmethacrylate	1.0	1.0	13	0.43
4-Methyl-2-Pentanone	0.40	0.56	9.7	0.17
Methyl-tert-Butylether	0.40	0.52	5.6	0.090
Nitrobenzene	2.0	2.1	18	1.2
2-Nitrobenzene	1.0	0.83	6.2	0.16
Pentachloroethane	0.20	0.23	20	0.14
Propionitrile	1.0	0.87	5.3	0.14
Tetrahydrofuran	5.0	3.9	13	1.6

[a]Data obtained using Column 4 with the open split interface and an ion trap mass spectrometer.

Saver time: 1.00 min
Gas type: Helium

COLUMN 1
Capillary Column
Model Number: J&W DB-VRX
Max temperature: 260 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 1.00 um
Mode: constant pressure
Pressure: 12.12 psi
Nominal initial flow: 0.6 mL/min
Average velocity: 34 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

BACK DETECTOR (NO DET)

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 250 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0(Off)		

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

Back Injector:

Sample Washes	0
Sample Pumps	0
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
Nanoliter Adapter	Off
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

MS ACQUISITION PARAMETERS

General Information

Tune File	: BFB1.U
Acquistion Mode	: Scan

MS Information

Solvent Delay	: 1.00 min
EM Absolute	: True
Resulting EM Voltage	: 1505.9

[Scan Parameters]

Low Mass	: 35
High Mass	: 260
Threshold	: 500
Sample #	: 3 A/D Samples 8
Plot 2 low mass	: 50
Plot 2 high mass	: 550

[MSZones]

MS Quad	: 150 C maximum 200 C
MS Source	: 230 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\DW081511.M

Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
c:\database\nbs75k.l	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: No
    File: No

Generate Report During Run Method: Yes

ELEMENT ID: 1108009
Calibration Last Updated: Sat Sep 03 14:22:51 2011

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) Fluorobenzene (ISTD)

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 70.00	25.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	1690570
.5		5.000	1682534
2		5.000	1718448
4		5.000	1747504
10		5.000	1825568
20		5.000	1912901
40		5.000	1914540
80		5.000	2066169

Qualifier Peak Analysis ON ISTD conc: 5.000 ug/l
Curve Fit: Avg. RF

---

2) Dichlorodifluoromethane ( )

Ret. Time 1.33 min., Extract & Integrate from 0.83 to 1.83 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 87.00	32.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36960
2		2.000	149394
4		4.000	293456
10		10.000	668055
20		20.000	1409318
40		40.000	2846921
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Chloromethane ( )

Ret. Time 1.42 min., Extract & Integrate from 0.92 to 1.92 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 50.00			*** METH DEFAULT ***

Q1 52.00 31.90 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	45391
2		2.000	166918
4		4.000	323945
10		10.000	747394
20		20.000	1603576
40		40.000	3076897
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

4) Vinyl Chloride ( )

Ret. Time 1.52 min., Extract & Integrate from 1.02 to 2.02 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 64.00	32.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	11891
.5		0.500	42344
2		2.000	157435
4		4.000	282213
10		10.000	650908
20		20.000	1398475
40		40.000	-1
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

5) Bromomethane ( )

Ret. Time 1.71 min., Extract & Integrate from 1.21 to 2.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 94.00			*** METH DEFAULT ***
Q1 96.00	95.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	34956
2		2.000	122284
4		4.000	222754
10		10.000	474121
20		20.000	961781
40		40.000	1891085
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

6) Chloroethane ( )

Ret. Time 1.79 min., Extract & Integrate from 1.29 to 2.29 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	64.00			*** METH DEFAULT ***
Q1	66.00	32.80	30.0	*** METH DEFAULT ***
Q2	49.00	25.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	26265
2		2.000	100426
4		4.000	196041
10		10.000	451403
20		20.000	973394
40		40.000	1888351
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

7) Trichlorofluoromethane ( )

Ret. Time 2.12 min., Extract & Integrate from 1.62 to 2.62 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	103.00	64.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	54664
2		2.000	215043
4		4.000	427510
10		10.000	983929
20		20.000	2090455
40		40.000	4135057
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

8) Diethyl Ether ( )

Ret. Time 2.28 min., Extract & Integrate from 1.78 to 2.78 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	74.00			*** METH DEFAULT ***
Q1	59.00	130.40	30.0	*** METH DEFAULT ***
Q2	45.00	96.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	11727
2		2.000	46602
4		4.000	90300
10		10.000	210772

20	20.000	438586
40	40.000	908276
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

9) Acetone ( )

Ret. Time 2.21 min., Extract & Integrate from 1.71 to 2.71 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	350.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	5722
2		10.000	18760
4		20.000	35941
10		50.000	76997
20		100.000	160218
40		200.000	316000
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

10) Methyl Iodide ( )

Ret. Time 2.48 min., Extract & Integrate from 1.98 to 2.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 142.00			*** METH DEFAULT ***
Q1 127.00	41.20	30.0	*** METH DEFAULT ***
Q2 141.00	14.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	26301
2		2.000	140389
4		4.000	329007
10		10.000	892249
20		20.000	2054017
40		40.000	4200866
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

11) 1,1-Dichloroethene ( )

Ret. Time 2.46 min., Extract & Integrate from 1.96 to 2.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	164.40	30.0	*** METH DEFAULT ***

Q2 63.00 52.80 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	29614
2		2.000	114139
4		4.000	220331
10		10.000	516731
20		20.000	1114541
40		40.000	2223528
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

12) Carbon Disulfide ( )

Ret. Time 2.71 min., Extract & Integrate from 2.21 to 3.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	9.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	83841
2		2.000	321837
4		4.000	656523
10		10.000	1582942
20		20.000	3478192
40		40.000	7071409
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

13) 1,1,2-Trichloro-1,2,2-trifluoroethane ( )

Ret. Time 2.62 min., Extract & Integrate from 2.12 to 3.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 101.00			*** METH DEFAULT ***
Q1 151.00	88.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28596
2		2.000	112978
4		4.000	228356
10		10.000	524434
20		20.000	1099626
40		40.000	2231704
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

14) Allyl Chloride ( )

Ret. Time 2.65 min., Extract & Integrate from 2.15 to 3.15 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	41.00			*** METH DEFAULT ***
Q1	39.00	75.00	30.0	*** METH DEFAULT ***
Q2	76.00	33.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	52801
2		2.000	207549
4		4.000	408008
10		10.000	968400
20		20.000	2103511
40		40.000	4222910
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

15) Methylene Chloride ( )

Ret. Time 2.58 min., Extract & Integrate from 2.08 to 3.08 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	84.00			*** METH DEFAULT ***
Q1	86.00	64.40	30.0	*** METH DEFAULT ***
Q2	49.00	134.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36304
2		2.000	120018
4		4.000	227085
10		10.000	511490
20		20.000	1060423
40		40.000	2141972
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

16) Acrylonitrile ( )

Ret. Time 2.52 min., Extract & Integrate from 2.02 to 3.02 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	53.00			*** METH DEFAULT ***
Q1	52.00	87.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	3782
2		2.000	15516
4		4.000	29250
10		10.000	65748

20	20.000	139866
40	40.000	290625
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

17) Methyl tert-Butyl Ether ( )

Ret. Time 3.24 min., Extract & Integrate from 2.74 to 3.74 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 57.00	24.00	30.0	*** METH DEFAULT ***
Q2 41.00	29.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	40869
2		2.000	179899
4		4.000	360740
10		10.000	833379
20		20.000	1749135
40		40.000	3639731
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

18) trans-1,2-Dichloroethene ( )

Ret. Time 3.12 min., Extract & Integrate from 2.62 to 3.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	145.90	30.0	*** METH DEFAULT ***
Q2 98.00	63.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	13826
2		2.000	125636
4		4.000	248179
10		10.000	576571
20		20.000	1246875
40		40.000	2503492
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

19) 1,1-Dichloroethane ( )

Ret. Time 3.32 min., Extract & Integrate from 2.82 to 3.82 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***

Q1	65.00	32.20	30.0	*** METH DEFAULT ***
Q2	83.00	12.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	58632
2		2.000	231190
4		4.000	456871
10		10.000	1067804
20		20.000	2258809
40		40.000	4525649
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

20) Vinyl Acetate ( )

Ret. Time 3.47 min., Extract & Integrate from 2.97 to 3.97 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	86.00	10.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28743
2		2.000	103993
4		4.000	217175
10		10.000	515102
20		20.000	1054155
40		40.000	2335063
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

21) Di-isopropyl ether ( )

Ret. Time 3.71 min., Extract & Integrate from 3.21 to 4.21 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	45.00			*** METH DEFAULT ***
Q1	43.00	55.20	30.0	*** METH DEFAULT ***
Q2	87.00	26.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	116487
2		2.000	369008
4		4.000	725811
10		10.000	1665410
20		20.000	3479414
40		40.000	7052270
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

22) cis-1,2 Dichloroethene ( )

Ret. Time 3.75 min., Extract & Integrate from 3.25 to 4.25 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	96.00			*** METH DEFAULT ***
Q1	61.00	128.80	30.0	*** METH DEFAULT ***
Q2	98.00	63.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	39534
2		2.000	132799
4		4.000	264232
10		10.000	616511
20		20.000	1302211
40		40.000	2644750
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

23) 2,2-Dichloropropane ( )

Ret. Time 3.95 min., Extract & Integrate from 3.45 to 4.45 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	77.00			*** METH DEFAULT ***
Q1	97.00	19.40	30.0	*** METH DEFAULT ***
Q2	41.00	74.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	45851
2		2.000	169003
4		4.000	332646
10		10.000	796515
20		20.000	1744258
40		40.000	3518798
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

24) 2-Butanone ( )

Ret. Time 3.69 min., Extract & Integrate from 3.19 to 4.19 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	72.00			*** METH DEFAULT ***
Q1	43.00	464.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	7666
2		10.000	25258

4	20.000	48472
10	50.000	109303
20	100.000	242685
40	200.000	498826
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

25) Methyl Acrylate ( )

Ret. Time 3.98 min., Extract & Integrate from 3.48 to 4.48 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 55.00			*** METH DEFAULT ***
Q1 85.00	15.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	13314
2	2.000	50262
4	4.000	99671
10	10.000	228442
20	20.000	482427
40	40.000	998052
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

26) Methylacrylonitrile ( )

Ret. Time 3.74 min., Extract & Integrate from 3.24 to 4.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 67.00	59.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	10951
2	2.000	31644
4	4.000	58470
10	10.000	130631
20	20.000	293087
40	40.000	621971
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

27) Bromochloromethane ( )

Ret. Time 3.86 min., Extract & Integrate from 3.36 to 4.36 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

Q1	49.00	159.80	30.0	*** METH DEFAULT ***
Q2	130.00	130.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	14550
2		2.000	47300
4		4.000	97133
10		10.000	220117
20		20.000	460966
40		40.000	954488
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

28) Ethyl tertiary-butyl ether ( )

Ret. Time 3.99 min., Extract & Integrate from 3.49 to 4.49 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	59.00			*** METH DEFAULT ***
Q1	41.00	27.00	30.0	*** METH DEFAULT ***
Q2	87.00	43.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	79145
2		2.000	266041
4		4.000	537069
10		10.000	1231033
20		20.000	2566449
40		40.000	5330292
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

29) Tetrahydrofuran ( )

Ret. Time 4.14 min., Extract & Integrate from 3.64 to 4.64 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	42.00			*** METH DEFAULT ***
Q1	72.00	37.90	30.0	*** METH DEFAULT ***
Q2	41.00	58.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	4732
2		2.000	15733
4		4.000	31939
10		10.000	67195
20		20.000	132552
40		40.000	321731
80		not used for this compound	

Qualifier Peak Analysis ON

Curve Fit: Avg. RF

---

30) Chloroform ( )

Ret. Time 3.90 min., Extract & Integrate from 3.40 to 4.40 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	65.00	30.0	*** METH DEFAULT ***
Q2 47.00	26.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	67293
2	2.000	219876
4	4.000	434950
10	10.000	1009787
20	20.000	2143181
40	40.000	4311061
80	80.000	8994486

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

31) 1,1,1-Trichloroethane ( )

Ret. Time 4.40 min., Extract & Integrate from 3.90 to 4.90 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 97.00			*** METH DEFAULT ***
Q1 99.00	64.00	30.0	*** METH DEFAULT ***
Q2 61.00	50.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	50331
2	2.000	190189
4	4.000	431996
10	10.000	1053058
20	20.000	2349823
40	40.000	4797589
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

32) 1,1-Dichloropropene ( )

Ret. Time 4.52 min., Extract & Integrate from 4.02 to 5.02 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 110.00	34.70	30.0	*** METH DEFAULT ***
Q2 77.00	30.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

.5	0.500	52176
2	2.000	215991
4	4.000	429289
10	10.000	1027420
20	20.000	2246127
40	40.000	4561147
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

33) 1-Chlorobutane ( )

Ret. Time 4.56 min., Extract & Integrate from 4.06 to 5.06 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 56.00			*** METH DEFAULT ***
Q1 41.00	62.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	70255
2		2.000	292591
4		4.000	586952
10		10.000	1387207
20		20.000	3029018
40		40.000	6105790
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

34) Carbon Tetrachloride ( )

Ret. Time 4.62 min., Extract & Integrate from 4.12 to 5.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 117.00			*** METH DEFAULT ***
Q1 119.00	95.20	30.0	*** METH DEFAULT ***
Q2 121.00	30.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	42586
2		2.000	176418
4		4.000	372946
10		10.000	931815
20		20.000	2144871
40		40.000	4499321
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

35) Benzene ( )

Ret. Time 4.66 min., Extract & Integrate from 4.16 to 5.16 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 78.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	160427
2		2.000	645737
4		4.000	1294586
10		10.000	3021310
20		20.000	6440595
40		40.000	12339306
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

36) 1,2-Dichloroethane ( )

Ret. Time 4.34 min., Extract & Integrate from 3.84 to 4.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 98.00	9.60	30.0	*** METH DEFAULT ***
Q2 49.00	29.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	36374
2		2.000	118014
4		4.000	278606
10		10.000	598304
20		20.000	1333259
40		40.000	2706295
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

37) Tertiary-amyl methyl ether ( )

Ret. Time 4.79 min., Extract & Integrate from 4.29 to 5.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 43.00	37.00	30.0	*** METH DEFAULT ***
Q2 87.00	23.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	77542
2		2.000	245638
4		4.000	474123
10		10.000	1076359
20		20.000	2299783
40		40.000	4864454
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

38) Trichloroethene ( )

Ret. Time 5.05 min., Extract & Integrate from 4.55 to 5.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 97.00	63.40	30.0	*** METH DEFAULT ***
Q2 130.00	110.40	30.0	*** METH DEFAULT ***
Q3 132.00	108.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	42036
2	2.000	165582
4	4.000	323560
10	10.000	764549
20	20.000	1678117
40	40.000	3404760
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

39) 1,2-Dichloropropane ( )

Ret. Time 5.02 min., Extract & Integrate from 4.52 to 5.52 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 112.00	5.00	30.0	*** METH DEFAULT ***
Q2 61.00	14.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	34953
2	2.000	139627
4	4.000	283987
10	10.000	668898
20	20.000	1426581
40	40.000	2985914
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

40) Dibromomethane ( )

Ret. Time 5.00 min., Extract & Integrate from 4.50 to 5.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 93.00			*** METH DEFAULT ***
Q1 95.00	84.00	30.0	*** METH DEFAULT ***
Q2 174.00	114.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12269
2		2.000	48235
4		4.000	102153
10		10.000	238889
20		20.000	515181
40		40.000	1077910
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

41) Methyl Methacrylate ( )

Ret. Time 5.21 min., Extract & Integrate from 4.71 to 5.71 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	69.00			*** METH DEFAULT ***
Q1	41.00	152.10	30.0	*** METH DEFAULT ***
Q2	100.00	38.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	10014
2		2.000	39731
4		4.000	85246
10		10.000	200209
20		20.000	439437
40		40.000	954233
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

42) 1,4-Dioxane ( )

Ret. Time 5.18 min., Extract & Integrate from 4.68 to 5.68 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	88.00			*** METH DEFAULT ***
Q1	58.00	66.10	30.0	*** METH DEFAULT ***
Q2	43.00	32.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		10.000	3291
2		40.000	13800
4		80.000	26106
10		200.000	64098
20		400.000	135144
40		800.000	284023
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

43) Bromodichloromethane ( )

Ret. Time 5.08 min., Extract & Integrate from 4.58 to 5.58 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	85.00	66.20	30.0	*** METH DEFAULT ***
Q2	127.00	9.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	33933
2		2.000	137452
4		4.000	302081
10		10.000	741238
20		20.000	1655844
40		40.000	3542073
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

44) 2-Nitropropane ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	43.00			*** METH DEFAULT ***
Q1	41.00	91.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	2900
2		2.000	10592
4		4.000	23547
10		10.000	54007
20		20.000	116495
40		40.000	277934
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

45) cis-1,3-Dichloropropene ( )

Ret. Time 5.46 min., Extract & Integrate from 4.96 to 5.96 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	75.00			*** METH DEFAULT ***
Q1	77.00	32.30	30.0	*** METH DEFAULT ***
Q2	39.00	59.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	8289
.5		0.500	31105
2		2.000	139660
4		4.000	305908
10		10.000	775254

20	20.000	1779959
40	40.000	3871476
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

46) Toluene ( )

Ret. Time 5.88 min., Extract & Integrate from 5.38 to 6.38 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 92.00			*** METH DEFAULT ***
Q1 91.00	170.70	30.0	*** METH DEFAULT ***
Q2 65.00	20.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	96196
2	2.000	391179
4	4.000	800115
10	10.000	1921826
20	20.000	4182210
40	40.000	8381625
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

47) trans-1,3-Dichloropropene ( )

Ret. Time 5.70 min., Extract & Integrate from 5.20 to 6.20 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.60	30.0	*** METH DEFAULT ***
Q2 39.00	55.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	5943
.5	0.500	21572
2	2.000	94464
4	4.000	213249
10	10.000	559050
20	20.000	1305048
40	40.000	2892875
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

48) 4-Methyl-2-pentanone ( )

Ret. Time 5.54 min., Extract & Integrate from 5.04 to 6.04 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***

Q1	43.00	275.10	30.0	*** METH DEFAULT ***
Q2	85.00	43.30	30.0	*** METH DEFAULT ***
Q3	100.00	39.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	24697
2		10.000	103203
4		20.000	210621
10		50.000	482279
20		100.000	1043113
40		200.000	2251644
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

49) Ethyl Methacrylate ( )

Ret. Time 5.95 min., Extract & Integrate from 5.45 to 6.45 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	69.00			*** METH DEFAULT ***
Q1	41.00	81.70	30.0	*** METH DEFAULT ***
Q2	99.00	17.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	19903
2		2.000	83504
4		4.000	172830
10		10.000	417422
20		20.000	928791
40		40.000	2041841
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

50) 1,1,2-Trichloroethane ( )

Ret. Time 5.78 min., Extract & Integrate from 5.28 to 6.28 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	83.00			*** METH DEFAULT ***
Q1	97.00	118.00	30.0	*** METH DEFAULT ***
Q2	85.00	63.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	16002
2		2.000	60484
4		4.000	123861
10		10.000	284482
20		20.000	609828
40		40.000	1279606
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

51) 1,3-Dichloropropane ( )

Ret. Time 5.91 min., Extract & Integrate from 5.41 to 6.41 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	32.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	37545
2		2.000	144945
4		4.000	295111
10		10.000	684312
20		20.000	1459201
40		40.000	3043375
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

52) Tetrachloroethene ( )

Ret. Time 6.26 min., Extract & Integrate from 5.76 to 6.76 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 164.00			*** METH DEFAULT ***
Q1 129.00	83.70	30.0	*** METH DEFAULT ***
Q2 131.00	81.70	30.0	*** METH DEFAULT ***
Q3 166.00	128.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	43581
2		2.000	179022
4		4.000	345147
10		10.000	790720
20		20.000	1936176
40		40.000	3506601
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

53) 2-Hexanone ( )

Ret. Time 6.01 min., Extract & Integrate from 5.51 to 6.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 58.00	48.70	30.0	*** METH DEFAULT ***
Q2 57.00	18.20	30.0	*** METH DEFAULT ***
Q3 100.00	11.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	49467
2		10.000	200526
4		20.000	392586
10		50.000	899015
20		100.000	1979397
40		200.000	4157200
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

54) Dibromochloromethane ( )

Ret. Time 6.04 min., Extract & Integrate from 5.54 to 6.54 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	129.00			*** METH DEFAULT ***
Q1	127.00	77.30	30.0	*** METH DEFAULT ***
Q2	131.00	24.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	4709
.5		0.500	17627
2		2.000	71941
4		4.000	158920
10		10.000	402267
20		20.000	924702
40		40.000	2036515
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

55) 1,2-Dibromoethane ( )

Ret. Time 6.17 min., Extract & Integrate from 5.67 to 6.67 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	107.00			*** METH DEFAULT ***
Q1	109.00	94.60	30.0	*** METH DEFAULT ***
Q2	188.00	4.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	15080
2		2.000	61744
4		4.000	130132
10		10.000	305515
20		20.000	673239
40		40.000	1438591
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

56) Chlorobenzene ( )

Ret. Time 6.61 min., Extract & Integrate from 6.11 to 7.11 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 112.00			*** METH DEFAULT ***
Q1 77.00	59.60	30.0	*** METH DEFAULT ***
Q2 114.00	32.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	109324
2		2.000	432293
4		4.000	868996
10		10.000	2026834
20		20.000	4298947
40		40.000	8681321
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

57) 1,1,1,2-Tetrachloroethane ( )

Ret. Time 6.57 min., Extract & Integrate from 6.07 to 7.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 131.00			*** METH DEFAULT ***
Q1 133.00	95.80	30.0	*** METH DEFAULT ***
Q2 119.00	62.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	27157
2		2.000	114376
4		4.000	242600
10		10.000	600647
20		20.000	1357287
40		40.000	2922262
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

58) Ethylbenzene ( )

Ret. Time 6.71 min., Extract & Integrate from 6.21 to 7.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 106.00	32.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	188023
2		2.000	788584
4		4.000	1612705
10		10.000	3765172

20	20.000	7977305
40	40.000	13344160
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

59) Xylene P,M ( )

Ret. Time 6.81 min., Extract & Integrate from 6.31 to 7.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	192.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.200	39207
.5		1.000	149852
2		4.000	629108
4		8.000	1266040
10		20.000	2966623
20		40.000	6318075
40		80.000	12261430
80	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

60) Xylene O ( )

Ret. Time 7.01 min., Extract & Integrate from 6.51 to 7.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	211.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	20530
.5		0.500	74477
2		2.000	312937
4		4.000	627585
10		10.000	1472227
20		20.000	3145819
40		40.000	6466312
80	not used for this compound		

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

61) Styrene ( )

Ret. Time 6.97 min., Extract & Integrate from 6.47 to 7.47 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 104.00			*** METH DEFAULT ***
Q1 78.00	43.90	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	112400
2		2.000	442769
4		4.000	897779
10		10.000	2123551
20		20.000	4603885
40		40.000	9365958
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

62) Bromoform ( )

Ret. Time 6.86 min., Extract & Integrate from 6.36 to 7.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	173.00			*** METH DEFAULT ***
Q1	175.00	50.30	30.0	*** METH DEFAULT ***
Q2	254.00	14.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	2449
.5		0.500	9798
2		2.000	37330
4		4.000	80546
10		10.000	197613
20		20.000	472889
40		40.000	1103963
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

63) Isopropylbenzene ( )

Ret. Time 7.18 min., Extract & Integrate from 6.68 to 7.68 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	105.00			*** METH DEFAULT ***
Q1	120.00	28.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	202767
2		2.000	836722
4		4.000	1682632
10		10.000	3935012
20		20.000	8273088
40		40.000	13561272
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

64) Bromofluorobenzene (SURR) ( )

Ret. Time 7.20 min., Extract & Integrate from 6.70 to 7.70 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 174.00	89.00	30.0	*** METH DEFAULT ***
Q2 176.00	86.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	527028
.5		5.000	553882
2		5.000	589917
4		5.000	605718
10		5.000	628334
20		5.000	653945
40		5.000	683757
80		5.000	727953

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

65) 1,2,3-Trichloropropane ( )

Ret. Time 7.07 min., Extract & Integrate from 6.57 to 7.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	32.10	30.0	*** METH DEFAULT ***
Q2 110.00	36.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	17780
2		2.000	70126
4		4.000	142224
10		10.000	324133
20		20.000	732606
40		40.000	1606352
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

66) Bromobenzene ( )

Ret. Time 7.31 min., Extract & Integrate from 6.81 to 7.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 156.00			*** METH DEFAULT ***
Q1 77.00	145.40	30.0	*** METH DEFAULT ***
Q2 158.00	97.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	45940
2		2.000	171950
4		4.000	346822
10		10.000	794531

20	20.000	1696454
40	40.000	3609305
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

67) 1,1,2,2-Tetrachloroethane ( )

Ret. Time 7.00 min., Extract & Integrate from 6.50 to 7.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 131.00	10.80	30.0	*** METH DEFAULT ***
Q2 85.00	65.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	6625
.5	0.500	19972
2	2.000	75157
4	4.000	142720
10	10.000	322705
20	20.000	687452
40	40.000	1501234
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Linear

---

68) trans-1,4-dichloro-2-Butene ( )

Ret. Time 7.08 min., Extract & Integrate from 6.58 to 7.58 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 89.00			*** METH DEFAULT ***
Q1 77.00	83.60	30.0	*** METH DEFAULT ***
Q2 75.00	247.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1403
2	2.000	7354
4	4.000	15162
10	10.000	40075
20	20.000	106495
40	40.000	-1
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

69) n-Propylbenzene ( )

Ret. Time 7.40 min., Extract & Integrate from 6.90 to 7.90 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***

Q1 120.00 24.40 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	232625
2		2.000	959571
4		4.000	1957894
10		10.000	4503677
20		20.000	9315222
40		40.000	14828613
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

70) 2-Chlorotoluene ( )

Ret. Time 7.45 min., Extract & Integrate from 6.95 to 7.95 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	35.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	152808
2		2.000	580413
4		4.000	1104284
10		10.000	2647671
20		20.000	5646676
40		40.000	11189058
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

71) 4-Chlorotoluene ( )

Ret. Time 7.49 min., Extract & Integrate from 6.99 to 7.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	34.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	145869
2		2.000	555187
4		4.000	1140849
10		10.000	2490011
20		20.000	5157242
40		40.000	10504085
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

72) 1,3,5-Trimethylbenzene ( )

Ret. Time 7.54 min., Extract & Integrate from 7.04 to 8.04 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	105.00			*** METH DEFAULT ***
Q1	120.00	51.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	165875
2		2.000	692952
4		4.000	1399212
10		10.000	3272803
20		20.000	6889088
40		40.000	12771475
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

73) tert-Butylbenzene ( )

Ret. Time 7.69 min., Extract & Integrate from 7.19 to 8.19 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	119.00			*** METH DEFAULT ***
Q1	91.00	63.90	30.0	*** METH DEFAULT ***
Q2	134.00	27.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	159871
2		2.000	650834
4		4.000	1321966
10		10.000	3080440
20		20.000	6581018
40		40.000	12539394
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

74) Pentachloroethane ( )

Ret. Time 7.59 min., Extract & Integrate from 7.09 to 8.09 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	165.00			*** METH DEFAULT ***
Q1	167.00	129.30	30.0	*** METH DEFAULT ***
Q2	117.00	109.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12343
2		2.000	46411
4		4.000	102656
10		10.000	267484

20	20.000	470943
40	40.000	-1
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

75) 1,2,4-Trimethylbenzene ( )

Ret. Time 7.74 min., Extract & Integrate from 7.24 to 8.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	48.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	171825
2		2.000	691642
4		4.000	1395733
10		10.000	3242121
20		20.000	6862349
40		40.000	13151847
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

76) sec-Butylbenzene ( )

Ret. Time 7.80 min., Extract & Integrate from 7.30 to 8.30 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 134.00	22.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	225678
2		2.000	937207
4		4.000	1889398
10		10.000	4329423
20		20.000	9047247
40		40.000	14688099
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

77) 1,3 Dichlorobenzene ( )

Ret. Time 7.84 min., Extract & Integrate from 7.34 to 8.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	37.80	30.0	*** METH DEFAULT ***
Q2 148.00	63.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	98691
2		2.000	401233
4		4.000	755591
10		10.000	1675027
20		20.000	3612788
40		40.000	7418009
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

78) 4-Isopropyltoluene ( )

Ret. Time 7.89 min., Extract & Integrate from 7.39 to 8.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 134.00	27.10	30.0	*** METH DEFAULT ***
Q2 91.00	23.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	185017
2		2.000	773128
4		4.000	1582978
10		10.000	3680162
20		20.000	7753964
40		40.000	13898481
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

79) 1,4 Dichlorobenzene ( )

Ret. Time 7.88 min., Extract & Integrate from 7.38 to 8.38 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	35.70	30.0	*** METH DEFAULT ***
Q2 148.00	65.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	102707
2		2.000	340485
4		4.000	690244
10		10.000	1593261
20		20.000	3342611
40		40.000	7094610
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

80) Hexachloroethane ( )

Ret. Time 8.34 min., Extract & Integrate from 7.84 to 8.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 201.00			*** METH DEFAULT ***
Q1 166.00	87.20	30.0	*** METH DEFAULT ***
Q2 117.00	108.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	19465
2		2.000	87418
4		4.000	187323
10		10.000	499068
20		20.000	1222429
40		40.000	2795522
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

81) n-Butylbenzene ( )

Ret. Time 8.10 min., Extract & Integrate from 7.60 to 8.60 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 92.00	55.30	30.0	*** METH DEFAULT ***
Q2 134.00	30.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	154801
2		2.000	643897
4		4.000	1315328
10		10.000	3050251
20		20.000	6409277
40		40.000	12761514
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

82) 1,2-Dichlorobenzene-d4 ( )

Ret. Time 8.05 min., Extract & Integrate from 7.55 to 8.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 152.15			*** METH DEFAULT ***
Q1 150.15	166.50	30.0	*** METH DEFAULT ***
Q2 115.15	58.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	508403
.5		5.000	544533
2		5.000	565874

4	5.000	590994
10	5.000	602868
20	5.000	627811
40	5.000	663563
80	5.000	720117

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

83) 1,2 Dichlorobenzene ( )

Ret. Time 8.06 min., Extract & Integrate from 7.56 to 8.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	39.60	30.0	*** METH DEFAULT ***
Q2 148.00	65.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	91420
2	2.000	326167
4	4.000	629183
10	10.000	1398812
20	20.000	2972663
40	40.000	6221630
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

84) 1,2-Dibromo-3-Chloropropane ( )

Ret. Time 8.31 min., Extract & Integrate from 7.81 to 8.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 155.00	87.10	30.0	*** METH DEFAULT ***
Q2 157.00	111.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	2547
2	2.000	9762
4	4.000	20032
10	10.000	46884
20	20.000	111912
40	40.000	259327
80	not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

85) 1,3,5-Trichlorobenzene ( )

Ret. Time 8.76 min., Extract & Integrate from 8.26 to 9.26 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	97.20	30.0	*** METH DEFAULT ***
Q2	145.00	27.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	70443
2		2.000	248682
4		4.000	490427
10		10.000	1136746
20		20.000	2465208
40		40.000	5296527
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

86) 1,2,4-Trichlorobenzene ( )

Ret. Time 9.07 min., Extract & Integrate from 8.57 to 9.57 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	180.00			*** METH DEFAULT ***
Q1	182.00	95.10	30.0	*** METH DEFAULT ***
Q2	145.00	27.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	62590
2		2.000	223696
4		4.000	436329
10		10.000	998621
20		20.000	2161944
40		40.000	4690096
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

87) Hexachlorobutadiene ( )

Ret. Time 9.25 min., Extract & Integrate from 8.75 to 9.75 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	225.00			*** METH DEFAULT ***
Q1	223.00	62.70	30.0	*** METH DEFAULT ***
Q2	227.00	65.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	12825
.5		0.500	35249
2		2.000	147567
4		4.000	297982
10		10.000	683397
20		20.000	1476173
40		40.000	3273159
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

88) Naphthalene ( )

Ret. Time 9.22 min., Extract & Integrate from 8.72 to 9.72 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	81853
2		2.000	292288
4		4.000	585992
10		10.000	1349661
20		20.000	2942248
40		40.000	6471198
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

89) 1,2,3-Trichlorobenzene ( )

Ret. Time 9.35 min., Extract & Integrate from 8.85 to 9.85 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***
Q1 182.00	95.70	30.0	*** METH DEFAULT ***
Q2 145.00	29.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	53349
2		2.000	189403
4		4.000	372802
10		10.000	848609
20		20.000	1831399
40		40.000	4003204
80		not used for this compound	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Thu Sep 08 15:01:03 2011

COLUMN 1
Capillary Column
Model Number: J&W DB-VRX
Max temperature: 260 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 1.00 um
Mode: constant pressure
Pressure: 28.64 psi
Nominal initial flow: 1.5 mL/min
Average velocity: 55 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (NO DET)

BACK DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 280 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0	(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

Back Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File	: BFB.U	
Acquistion Mode	: Scan	

MS Information
-- -----------

Solvent Delay	: 0.50 min	
EM Absolute	: True	
Resulting EM Voltage	: 1352.9	

[Scan Parameters]

Low Mass	: 35	
High Mass	: 260	
Threshold	: 500	
Sample #	: 4	A/D Samples 16
Plot 2 low mass	: 50	
Plot 2 high mass	: 550	

[MSZones]

MS Quad	: 150 C	maximum 200 C
MS Source	: 230 C	maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\DW121107.M

Percent Report Settings

Sort By: Signal

Output Destination
    Screen: No
    Printer: Yes
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
c:\database\nbs75k.l	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: Yes
    File: No

Generate Report During Run Method: Yes

ELEMENT ID: 0710008
Calibration Last Updated: Fri Dec 14 13:11:04 2007

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent

Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) Fluorobenzene (ISTD)

Ret. Time 3.72 min., Extract & Integrate from 3.22 to 4.22 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 70.00	22.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		5.000	3033916
.5		5.000	2817685
2		5.000	2868259
4		5.000	2906919
10		5.000	2955227
20		5.000	3315924
40		5.000	3457593
80		5.000	3371630

Qualifier Peak Analysis ON ISTD conc: 5.000 ug/l
Curve Fit: Avg. RF

---

2) Dichlorodifluoromethane ( )

Ret. Time 0.79 min., Extract & Integrate from 0.29 to 1.29 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 85.00			*** METH DEFAULT ***
Q1 87.00	32.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	31332
2		2.000	122286
4		4.000	270669
10		10.000	650037
20		20.000	1316798
40		40.000	2705084
80		80.000	4849089

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Chloromethane ( )

Ret. Time 0.84 min., Extract & Integrate from 0.34 to 1.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 50.00			*** METH DEFAULT ***
Q1 52.00	32.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	31157
2		2.000	109487
4		4.000	295145
10		10.000	710391
20		20.000	1353030
40		40.000	3420633
80		80.000	5504260

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

4) Vinyl Chloride ( )

Ret. Time 0.89 min., Extract & Integrate from 0.39 to 1.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 64.00	30.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	7067
.5		0.500	25561
2		2.000	105700
4		4.000	214052
10		10.000	533840
20		20.000	1132807
40		40.000	1900927
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

5) Bromomethane ( )

Ret. Time 1.01 min., Extract & Integrate from 0.51 to 1.51 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 94.00			*** METH DEFAULT ***
Q1 96.00	90.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	14883
2		2.000	61310
4		4.000	119238
10		10.000	284703
20		20.000	598393
40		40.000	1198724
80		80.000	1977293

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

6) Chloroethane ( )

Ret. Time 1.06 min., Extract & Integrate from 0.56 to 1.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 64.00			*** METH DEFAULT ***
Q1 66.00	32.80	30.0	*** METH DEFAULT ***
Q2 49.00	31.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14250
2	2.000	57081
4	4.000	123175
10	10.000	287706
20	20.000	598018
40	40.000	1197318
80	80.000	2043237

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

7) Trichlorofluoromethane ( )

Ret. Time 1.25 min., Extract & Integrate from 0.75 to 1.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 101.00			*** METH DEFAULT ***
Q1 103.00	64.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	34584
2	2.000	143084
4	4.000	313090
10	10.000	789160
20	20.000	1737098
40	40.000	3526725
80	80.000	6142972

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

8) Diethyl Ether ( )

Ret. Time 1.35 min., Extract & Integrate from 0.85 to 1.85 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 74.00			*** METH DEFAULT ***
Q1 59.00	163.10	30.0	*** METH DEFAULT ***
Q2 45.00	139.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	5089
2	2.000	19226
4	4.000	41043
10	10.000	99068
20	20.000	244788
40	40.000	536924

80 80.000 1003652

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

9) Acetone ( )

Ret. Time 1.30 min., Extract & Integrate from 0.80 to 1.80 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	455.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	2.500	2449
2	10.000	9682
4	20.000	17614
10	50.000	45045
20	100.000	104247
40	200.000	237982
80	400.000	496854

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

10) Methyl Iodide ( )

Ret. Time 1.48 min., Extract & Integrate from 0.98 to 1.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 142.00			*** METH DEFAULT ***
Q1 127.00	58.20	30.0	*** METH DEFAULT ***
Q2 141.00	14.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	16873
2	2.000	69111
4	4.000	144238
10	10.000	359852
20	20.000	733260
40	40.000	1523178
80	80.000	2495939

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

11) 1,1-Dichloroethene ( )

Ret. Time 1.46 min., Extract & Integrate from 0.96 to 1.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	266.60	30.0	*** METH DEFAULT ***
Q2 63.00	83.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12013
2		2.000	50142
4		4.000	114346
10		10.000	279654
20		20.000	685285
40		40.000	1516319
80		80.000	2805853

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

12) Carbon Disulfide ( )

Ret. Time 1.61 min., Extract & Integrate from 1.11 to 2.11 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	76.00			*** METH DEFAULT ***
Q1	78.00	9.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	54773
2		2.000	227728
4		4.000	476891
10		10.000	1186209
20		20.000	2693364
40		40.000	5537404
80		80.000	9558042

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

13) 1,1,2-Trichloro-1,2,2-trifluoroethane ( )

Ret. Time 1.56 min., Extract & Integrate from 1.06 to 2.06 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	101.00			*** METH DEFAULT ***
Q1	151.00	69.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	12021
2		2.000	46688
4		4.000	99120
10		10.000	248258
20		20.000	648997
40		40.000	1560204
80		80.000	2929839

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

14) Allyl Chloride ( )

Ret. Time 1.57 min., Extract & Integrate from 1.07 to 2.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 39.00	81.30	30.0	*** METH DEFAULT ***
Q2 76.00	25.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	34976
2	2.000	139375
4	4.000	298824
10	10.000	734930
20	20.000	1782451
40	40.000	3884984
80	80.000	6891420

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

15) Methylene Chloride ( )

Ret. Time 1.53 min., Extract & Integrate from 1.03 to 2.03 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 84.00			*** METH DEFAULT ***
Q1 86.00	63.40	30.0	*** METH DEFAULT ***
Q2 49.00	174.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	21281
2	2.000	69958
4	4.000	132791
10	10.000	313715
20	20.000	664694
40	40.000	1386441
80	80.000	2605325

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

16) Acrylonitrile ( )

Ret. Time 1.49 min., Extract & Integrate from 0.99 to 1.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 53.00			*** METH DEFAULT ***
Q1 52.00	80.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1587
2	2.000	8482
4	4.000	17260
10	10.000	43371
20	20.000	99687
40	40.000	225584

80 80.000 466418

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

17) Methyl tert-Butyl Ether ( )

Ret. Time 1.93 min., Extract & Integrate from 1.43 to 2.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 [illegible].00	23.00	30.0	*** METH DEFAULT ***
Q2 [illegible].00	31.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	27474
2	2.000	102811
4	4.000	209175
10	10.000	491788
20	20.000	1125091
40	40.000	2622416
80	80.000	5452244

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

18) trans-1,2-Dichloroethene ( )

Ret. Time 1.85 min., Extract & Integrate from 1.35 to 2.35 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible].00			*** METH DEFAULT ***
Q1 [illegible].00	223.30	30.0	*** METH DEFAULT ***
Q2 [illegible].00	62.90	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	12826
2	2.000	50194
4	4.000	110111
10	10.000	261910
20	20.000	628136
40	40.000	1397512
80	80.000	2833769

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

19) 1,1-Dichloroethane ( )

Ret. Time 1.99 min., Extract & Integrate from 1.49 to 2.49 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 65.00	31.50	30.0	*** METH DEFAULT ***
Q2 [illegible].00	11.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	34660
2		2.000	132259
4		4.000	279701
10		10.000	678205
20		20.000	1579869
40		40.000	3456163
80		80.000	6817187

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

20) Vinyl Acetate ( )

Ret. Time 2.12 min., Extract & Integrate from 1.62 to 2.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 86.00	5.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	18181
2		2.000	72167
4		4.000	140440
10		10.000	349855
20		20.000	816515
40		40.000	1965356
80		80.000	4043601

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

21) Di-isopropyl ether ( )

Ret. Time 2.34 min., Extract & Integrate from 1.84 to 2.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 45.00			*** METH DEFAULT ***
Q1 43.00	54.20	30.0	*** METH DEFAULT ***
Q2 87.00	18.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	63818
2		2.000	253754
4		4.000	528629
10		10.000	1288781
20		20.000	3031529
40		40.000	6406725
80		80.000	11482441

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

22) cis-1,2 Dichloroethene ( )

Ret. Time 2.38 min., Extract & Integrate from 1.88 to 2.88 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 96.00			*** METH DEFAULT ***
Q1 61.00	193.30	30.0	*** METH DEFAULT ***
Q2 98.00	62.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14078
2	2.000	53634
4	4.000	111049
10	10.000	263945
20	20.000	613927
40	40.000	1353429
80	80.000	2800492

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

23) 2,2-Dichloropropane ( )

Ret. Time 2.60 min., Extract & Integrate from 2.10 to 3.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 77.00			*** METH DEFAULT ***
Q1 97.00	17.90	30.0	*** METH DEFAULT ***
Q2 41.00	63.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	25869
2	2.000	100110
4	4.000	235066
10	10.000	591975
20	20.000	1623971
40	40.000	4247692
80	80.000	8409848

Qualifier Peak Analysis ON
Curve Fit: Linear

---

24) 2-Butanone ( )

Ret. Time 2.31 min., Extract & Integrate from 1.81 to 2.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 72.00			*** METH DEFAULT ***
Q1 43.00	689.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	2.500	1547
2	10.000	10460
4	20.000	19835
10	50.000	54110

20	100.000	131310
40	200.000	331502
80	400.000	750914

Qualifier Peak Analysis ON
Curve Fit: Linear

---

25) Methyl Acrylate ( )

Ret. Time 3.41 min., Extract & Integrate from 2.91 to 3.91 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 55.00			*** METH DEFAULT ***
Q1 85.00	14.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	15232
2		2.000	65074
4		4.000	145982
10		10.000	350644
20		20.000	864079
40		40.000	2117269
80		80.000	4346878

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

26) Methylacrylonitrile ( )

Ret. Time 2.36 min., Extract & Integrate from 1.86 to 2.86 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 41.00			*** METH DEFAULT ***
Q1 67.00	18.50	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	3554
2		2.000	13452
4		4.000	35595
10		10.000	75007
20		20.000	157223
40		40.000	-1
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

27) Bromochloromethane ( )

Ret. Time 2.50 min., Extract & Integrate from 2.00 to 3.00 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***
Q1 49.00	191.60	30.0	*** METH DEFAULT ***
Q2 130.00	129.60	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	6886
2		2.000	19141
4		4.000	52553
10		10.000	147040
20		20.000	359263
40		40.000	687283
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

28) Ethyl tertiary-butyl ether ( )

Ret. Time 2.65 min., Extract & Integrate from 2.15 to 3.15 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 59.00			*** METH DEFAULT ***
Q1 41.00	21.60	30.0	*** METH DEFAULT ***
Q2 87.00	38.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	56677
2		2.000	196812
4		4.000	422791
10		10.000	1037258
20		20.000	2466713
40		40.000	5194375
80		80.000	9578866

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

29) Tetrahydrofuran ( )

Ret. Time 2.83 min., Extract & Integrate from 2.33 to 3.33 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 42.00			*** METH DEFAULT ***
Q1 72.00	39.10	30.0	*** METH DEFAULT ***
Q2 41.00	55.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	2668
2		2.000	8373
4		4.000	15331
10		10.000	39005
20		20.000	89966
40		40.000	210650
80		80.000	472056

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

30) Chloroform ( )

Ret. Time 2.54 min., Extract & Integrate from 2.04 to 3.04 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	63.10	30.0	*** METH DEFAULT ***
Q2 47.00	26.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	38728
2	2.000	119790
4	4.000	312154
10	10.000	774069
20	20.000	1840830
40	40.000	3923193
80	80.000	6446963

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

31) 1,1,1-Trichloroethane ( )

Ret. Time 4.81 min., Extract & Integrate from 4.31 to 5.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 97.00			*** METH DEFAULT ***
Q1 99.00	63.40	30.0	*** METH DEFAULT ***
Q2 61.00	64.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	14140
2	2.000	57782
4	4.000	120388
10	10.000	300464
20	20.000	688973
40	40.000	1516201
80	80.000	3170508

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

32) 1,1-Dichloropropene ( )

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 110.00	19.30	30.0	*** METH DEFAULT ***
Q2 77.00	31.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29134
2	2.000	122316

4	4.000	268159
10	10.000	691239
20	20.000	1659594
40	40.000	3681174
80	80.000	7390794

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

33) 1-Chlorobutane ( )

Ret. Time 3.41 min., Extract & Integrate from 2.91 to 3.91 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 77.00			*** METH DEFAULT ***
Q1 41.00	59.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	40829
2	2.000	175515
4	4.000	391117
10	10.000	955283
20	20.000	2336398
40	40.000	5695758
80	80.000	11403424

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

34) Carbon Tetrachloride ( )

Ret. Time 3.48 min., Extract & Integrate from 2.98 to 3.98 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 117.00			*** METH DEFAULT ***
Q1 119.00	94.70	30.0	*** METH DEFAULT ***
Q2 121.00	30.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	24687
2	2.000	106566
4	4.000	233912
10	10.000	586654
20	20.000	1553519
40	40.000	-1
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

35) Benzene ( )

Ret. Time 3.53 min., Extract & Integrate from 3.03 to 4.03 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration

Tgt 78.00 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	108632
2		2.000	430521
4		4.000	923313
10		10.000	2269155
20		20.000	5239596
40		40.000	10625262
80		80.000	16209715

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

36) 1,1-Dichloroethane ( )

Ret. Time 3.12 min., Extract & Integrate from 2.62 to 3.62 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 62.00			*** METH DEFAULT ***
Q1 98.00	7.10	30.0	*** METH DEFAULT ***
Q2 49.00	28.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	22310
2		2.000	88144
4		4.000	180900
10		10.000	445816
20		20.000	1065986
40		40.000	2313058
80		80.000	4590567

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

37) Tertiary-amyl methyl ether ( )

Ret. Time 3.71 min., Extract & Integrate from 3.21 to 4.21 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 73.00			*** METH DEFAULT ***
Q1 43.00	33.10	30.0	*** METH DEFAULT ***
Q2 87.00	23.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	78915
2		2.000	200961
4		4.000	376443
10		10.000	875306
20		20.000	2038681
40		40.000	4419668
80		80.000	8592300

Qualifier Peak Analysis ON
Curve Fit: Linear

38) Trichloroethene ( )

Ret. Time 4.00 min., Extract & Integrate from 3.50 to 4.50 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 97.00	64.30	30.0	*** METH DEFAULT ***
Q2 130.00	83.40	30.0	*** METH DEFAULT ***
Q3 132.00	79.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29105
2	2.000	109494
4	4.000	237956
10	10.000	604797
20	20.000	1413279
40	40.000	3073250
80	80.000	5821030

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

39) 1,2-Dichloropropane ( )

Ret. Time 3.97 min., Extract & Integrate from 3.47 to 4.47 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 63.00			*** METH DEFAULT ***
Q1 112.00	3.80	30.0	*** METH DEFAULT ***
Q2 61.00	13.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	28217
2	2.000	104601
4	4.000	215517
10	10.000	512026
20	20.000	1166358
40	40.000	2553892
80	80.000	4985269

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

40) Dibromomethane ( )

Ret. Time 3.93 min., Extract & Integrate from 3.43 to 4.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 93.00			*** METH DEFAULT ***
Q1 95.00	84.70	30.0	*** METH DEFAULT ***
Q2 174.00	78.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

.5	0.500	8937
2	2.000	36450
4	4.000	74293
10	10.000	182877
20	20.000	437511
40	40.000	952752
80	80.000	1951527

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

41) Methyl Methacrylate ( )

Ret. Time 4.22 min., Extract & Integrate from 3.72 to 4.72 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	129.40	30.0	*** METH DEFAULT ***
Q2 [illegible]	36.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1	not used for this compound		
.5		0.500	6424
2		2.000	28467
4		4.000	62844
10		10.000	167303
20		20.000	410847
40		40.000	994684
80		80.000	2053373

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

42) 1,4-Dioxane ( )

Ret. Time 4.14 min., Extract & Integrate from 3.64 to 4.64 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt [illegible]			*** METH DEFAULT ***
Q1 [illegible]	84.50	30.0	*** METH DEFAULT ***
Q2 [illegible]	36.10	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1	not used for this compound		
.5		10.000	324
2		40.000	4698
4		80.000	10710
10		200.000	28726
20		400.000	68133
40		800.000	151064
80		1600.000	297019

Qualifier Peak Analysis ON
Curve Fit: Linear

---

43) Bromodichloromethane ( )

Ret. Time 4.03 min., Extract & Integrate from 3.53 to 4.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 85.00	64.50	30.0	*** METH DEFAULT ***
Q2 127.00	8.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	24898
2	2.000	101803
4	4.000	223259
10	10.000	536192
20	20.000	1313914
40	40.000	3177622
80	80.000	6598428

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

44) 2-Nitropropane ( )

Ret. Time 4.04 min., Extract & Integrate from 3.54 to 4.54 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 41.00	90.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	857
2	2.000	3688
4	4.000	7711
10	10.000	22460
20	20.000	65526
40	40.000	195469
80	80.000	526451

Qualifier Peak Analysis ON
Curve Fit: Quadratic

---

45) cis-1,3-Dichloropropene ( )

Ret. Time 4.46 min., Extract & Integrate from 3.96 to 4.96 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.50	30.0	*** METH DEFAULT ***
Q2 39.00	55.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	7425
.5	0.500	29134
2	2.000	122316
4	4.000	268159
10	10.000	691239
20	20.000	1659594
40	40.000	3681174

80 80.000 7390794

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

46) Toluene ( )

Ret. Time 4.92 min., Extract & Integrate from 4.42 to 5.42 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 92.00			*** METH DEFAULT ***
Q1 91.00	168.60	30.0	*** METH DEFAULT ***
Q2 65.00	18.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	83022
2		2.000	354336
4		4.000	756770
10		10.000	1943415
20		20.000	4210818
40		40.000	8504001
80		80.000	11884977

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

47) trans-1,3-Dichloropropene ( )

Ret. Time 4.74 min., Extract & Integrate from 4.24 to 5.24 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	31.80	30.0	*** METH DEFAULT ***
Q2 39.00	47.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	5090
.5		0.500	19741
2		2.000	92096
4		4.000	211830
10		10.000	564509
20		20.000	1432926
40		40.000	3248349
80		80.000	6473680

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

48) 4-Methyl-2-pentanone ( )

Ret. Time 4.57 min., Extract & Integrate from 4.07 to 5.07 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 58.00			*** METH DEFAULT ***
Q1 43.00	284.60	30.0	*** METH DEFAULT ***
Q2 85.00	34.20	30.0	*** METH DEFAULT ***

Q3 100.00 26.60 30.0 *** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		2.500	14865
2		10.000	70616
4		20.000	151811
10		50.000	411846
20		100.000	1005852
40		200.000	2442832
80		400.000	4889324

Qualifier Peak Analysis ON
Curve Fit: Linear

---

49) Ethyl Methacrylate ( )

Ret. Time 5.02 min., Extract & Integrate from 4.52 to 5.52 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 69.00			*** METH DEFAULT ***
Q1 41.00	92.70	30.0	*** METH DEFAULT ***
Q2 99.00	13.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	-1
2		2.000	70276
4		4.000	154911
10		10.000	433140
20		20.000	1044298
40		40.000	2410765
80		80.000	4865111

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

50) 1,1,2-Trichloroethane ( )

Ret. Time 4.81 min., Extract & Integrate from 4.31 to 5.31 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 97.00	115.50	30.0	*** METH DEFAULT ***
Q2 85.00	65.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	11757
2		2.000	51008
4		4.000	107221
10		10.000	267330
20		20.000	603003
40		40.000	1325551
80		80.000	2787104

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

51) 1,3-Dichloropropane ( )

Ret. Time 4.95 min., Extract & Integrate from 4.45 to 5.45 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 76.00			*** METH DEFAULT ***
Q1 78.00	33.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29346
2	2.000	123101
4	4.000	255428
10	10.000	631020
20	20.000	1469382
40	40.000	3177709
80	80.000	6409200

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

52) Tetrachloroethene ( )

Ret. Time 5.31 min., Extract & Integrate from 4.81 to 5.81 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 164.00			*** METH DEFAULT ***
Q1 129.00	95.00	30.0	*** METH DEFAULT ***
Q2 131.00	92.20	30.0	*** METH DEFAULT ***
Q3 166.00	126.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	30549
2	2.000	128565
4	4.000	304078
10	10.000	780279
20	20.000	1836799
40	40.000	4283658
80	80.000	7511298

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

53) 2-Hexanone ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 43.00			*** METH DEFAULT ***
Q1 58.00	52.30	30.0	*** METH DEFAULT ***
Q2 57.00	17.90	30.0	*** METH DEFAULT ***
Q3 100.00	9.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

.5	2.500	27011
2	10.000	131644
4	20.000	289106
10	50.000	787212
20	100.000	1851402
40	200.000	4236312
80	400.000	7539620

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

54) Dibromochloromethane ( )

Ret. Time 5.07 min., Extract & Integrate from 4.57 to 5.57 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 129.00			*** METH DEFAULT ***
Q1 127.00	77.20	30.0	*** METH DEFAULT ***
Q2 131.00	24.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	3077
.5	0.500	12991
2	2.000	61375
4	4.000	131396
10	10.000	356406
20	20.000	846178
40	40.000	1973175
80	80.000	4217462

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

55) 1,2-Dibromoethane ( )

Ret. Time 5.19 min., Extract & Integrate from 4.69 to 5.69 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 107.00			*** METH DEFAULT ***
Q1 109.00	93.60	30.0	*** METH DEFAULT ***
Q2 188.00	2.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	12919
2	2.000	55661
4	4.000	117401
10	10.000	305324
20	20.000	715174
40	40.000	1556303
80	80.000	3255634

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

56) Chlorobenzene ( )

Ret. Time 5.65 min., Extract & Integrate from 5.15 to 6.15 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 112.00			*** METH DEFAULT ***
Q1 77.00	79.10	30.0	*** METH DEFAULT ***
Q2 114.00	32.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	90122
2	2.000	365313
4	4.000	785507
10	10.000	1970078
20	20.000	4313073
40	40.000	8541466
80	80.000	11704384

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

57) 1,1,1,2-Tetrachloroethane ( )

Ret. Time 5.62 min., Extract & Integrate from 5.12 to 6.12 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 131.00			*** METH DEFAULT ***
Q1 133.00	94.80	30.0	*** METH DEFAULT ***
Q2 119.00	63.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	20673
2	2.000	90159
4	4.000	202233
10	10.000	535512
20	20.000	1280134
40	40.000	2935922
80	80.000	5725099

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

58) Ethylbenzene ( )

Ret. Time 5.77 min., Extract & Integrate from 5.27 to 6.27 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 106.00	31.40	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	161160
2	2.000	685677
4	4.000	1508279
10	10.000	3770498
20	20.000	7873752
40	40.000	11149258

80 80.000 -1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

59) Xylene P,M ( )

Ret. Time 5.87 min., Extract & Integrate from 5.37 to 6.37 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	202.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	1.000	114013
2	4.000	485527
4	8.000	1060558
10	20.000	2717851
20	40.000	5873938
40	80.000	11076742
80	160.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

60) Xylene O ( )

Ret. Time 6.06 min., Extract & Integrate from 5.56 to 6.56 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 106.00			*** METH DEFAULT ***
Q1 91.00	214.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	54337
2	2.000	231464
4	4.000	493290
10	10.000	1264666
20	20.000	2791658
40	40.000	5774262
80	80.000	8905597

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

61) Styrene ( )

Ret. Time 6.03 min., Extract & Integrate from 5.53 to 6.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 104.00			*** METH DEFAULT ***
Q1 78.00	38.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	

.5	0.500	73630
2	2.000	330195
4	4.000	711583
10	10.000	1891192
20	20.000	4203942
40	40.000	8324207
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

62) Bromoform ( )

Ret. Time 5.89 min., Extract & Integrate from 5.39 to 6.39 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 173.00			*** METH DEFAULT ***
Q1 173.00	48.30	30.0	*** METH DEFAULT ***
Q2 254.00	7.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	5553
2	2.000	28532
4	4.000	63305
10	10.000	180189
20	20.000	441185
40	40.000	1058296
80	80.000	2064692

Qualifier Peak Analysis ON
Curve Fit: Linear

---

63) Isopropylbenzene ( )

Ret. Time 6.25 min., Extract & Integrate from 5.75 to 6.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	28.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	147350
2	2.000	638325
4	4.000	1394619
10	10.000	3535968
20	20.000	7420548
40	40.000	-1
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

64) Bromofluorobenzene (SURR) ( )

Ret. Time 6.25 min., Extract & Integrate from 5.75 to 6.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 95.00			*** METH DEFAULT ***
Q1 174.00	41.90	30.0	*** METH DEFAULT ***
Q2 176.00	40.00	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	5.000	556575
2	5.000	605642
4	5.000	585388
10	5.000	613660
20	5.000	659987
40	5.000	704906
80	5.000	705556

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

65) 1,2,3-Trichloropropane ( )

Ret. Time 6.13 min., Extract & Integrate from 5.63 to 6.63 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 77.00	32.00	30.0	*** METH DEFAULT ***
Q2 110.00	34.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	12150
2	2.000	50809
4	4.000	111182
10	10.000	303078
20	20.000	619808
40	40.000	1455200
80	80.000	2604221

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

66) Bromobenzene ( )

Ret. Time 6.34 min., Extract & Integrate from 5.84 to 6.84 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 156.00			*** METH DEFAULT ***
Q1 77.00	222.40	30.0	*** METH DEFAULT ***
Q2 158.00	96.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	29166
2	2.000	117524
4	4.000	246605
10	10.000	630757
20	20.000	1421789
40	40.000	3025896

80 80.000 5597158

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

67) 1,1,2,2-Tetrachloroethane ( )

Ret. Time 6.05 min., Extract & Integrate from 5.55 to 6.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 83.00			*** METH DEFAULT ***
Q1 131.00	6.90	30.0	*** METH DEFAULT ***
Q2 85.00	63.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	0.100	3844
.5	0.500	15382
2	2.000	66017
4	4.000	136992
10	10.000	358859
20	20.000	830291
40	40.000	1791803
80	80.000	3090820

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

68) trans-1,4-dichloro-2-Butene ( )

Ret. Time 5.99 min., Extract & Integrate from 5.49 to 6.49 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 89.00			*** METH DEFAULT ***
Q1 77.00	74.80	30.0	*** METH DEFAULT ***
Q2 75.00	228.30	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	638
2	2.000	4512
4	4.000	10708
10	10.000	32385
20	20.000	84356
40	40.000	226788
80	80.000	439064

Qualifier Peak Analysis ON
Curve Fit: Linear

---

69) n-Propylbenzene ( )

Ret. Time 6.45 min., Extract & Integrate from 5.95 to 6.95 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 120.00	18.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	159408
2		2.000	752792
4		4.000	1500131
10		10.000	3970324
20		20.000	8194754
40		40.000	11233565
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

70) 2-Chlorotoluene ( )

Ret. Time 6.49 min., Extract & Integrate from 5.99 to 6.99 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	36.40	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	100962
2		2.000	368309
4		4.000	920999
10		10.000	2313968
20		20.000	4582811
40		40.000	8952763
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

71) 4-Chlorotoluene ( )

Ret. Time 6.52 min., Extract & Integrate from 6.02 to 7.02 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 126.00	24.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	96491
2		2.000	411583
4		4.000	864866
10		10.000	2208525
20		20.000	4938537
40		40.000	9466229
80		80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

72) 1,3,5-Trimethylbenzene ( )

Ret. Time 6.60 min., Extract & Integrate from 6.10 to 7.10 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	48.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	107563
2	2.000	464083
4	4.000	1035966
10	10.000	2682494
20	20.000	5779808
40	40.000	8844332
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

73) Tert-Butylbenzene ( )

Ret. Time 6.72 min., Extract & Integrate from 6.22 to 7.22 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 91.00	66.00	30.0	*** METH DEFAULT ***
Q2 134.00	25.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	92303
2	2.000	400197
4	4.000	886626
10	10.000	2344875
20	20.000	5153945
40	40.000	8603110
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

74) Pentachloroethane ( )

Ret. Time 6.61 min., Extract & Integrate from 6.11 to 7.11 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 165.00			*** METH DEFAULT ***
Q1 167.00	129.80	30.0	*** METH DEFAULT ***
Q2 117.00	112.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	8301
2	2.000	34243
4	4.000	66818
10	10.000	181308
20	20.000	411621
40	40.000	790841

80	80.000	1473353

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

75) 1,2,4-Trimethylbenzene ( )

Ret. Time 6.78 min., Extract & Integrate from 6.28 to 7.28 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 120.00	49.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	102812
2	2.000	452143
4	4.000	977137
10	10.000	2540050
20	20.000	5450487
40	40.000	9864930
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

76) sec-Butylbenzene ( )

Ret. Time 6.82 min., Extract & Integrate from 6.32 to 7.32 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 105.00			*** METH DEFAULT ***
Q1 134.00	20.60	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	144115
2	2.000	612799
4	4.000	1345547
10	10.000	3492656
20	20.000	7462943
40	40.000	10181768
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

77) 1,3 Dichlorobenzene ( )

Ret. Time 6.84 min., Extract & Integrate from 6.34 to 7.34 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	42.00	30.0	*** METH DEFAULT ***
Q2 148.00	63.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response

.1	not used for this compound		
.5	0.500	55013	
2	2.000	215324	
4	4.000	466146	
10	10.000	1251420	
20	20.000	2580851	
40	40.000	5690137	
80	80.000	9083215	

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

78) 4-Isopropyltoluene ( )

Ret. Time 6.92 min., Extract & Integrate from 6.42 to 7.42 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 119.00			*** METH DEFAULT ***
Q1 134.00	27.50	30.0	*** METH DEFAULT ***
Q2 91.00	22.90	30.0	*** METH DEFAULT ***

Lvl	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	110802
2	2.000	489433
4	4.000	1077882
10	10.000	2772073
20	20.000	5947033
40	40.000	8693769
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

79) 1,4 Dichlorobenzene ( )

Ret. Time 6.87 min., Extract & Integrate from 6.37 to 7.37 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	45.00	30.0	*** METH DEFAULT ***
Q2 148.00	63.10	30.0	*** METH DEFAULT ***

Lvl	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	52504
2	2.000	210511
4	4.000	446875
10	10.000	1119457
20	20.000	2616981
40	40.000	5337468
80	80.000	9010711

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

80) Hexachloroethane ( )

Ret. Time 7.28 min., Extract & Integrate from 6.78 to 7.78 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 201.00			*** METH DEFAULT ***
Q1 166.00	115.50	30.0	*** METH DEFAULT ***
Q2 117.00	171.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	9264
2	2.000	42836
4	4.000	103881
10	10.000	312878
20	20.000	766128
40	40.000	1844662
80	80.000	3345618

Qualifier Peak Analysis ON
Curve Fit: Linear

---

81) n-Butylbenzene ( )

Ret. Time 7.10 min., Extract & Integrate from 6.60 to 7.60 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 91.00			*** METH DEFAULT ***
Q1 92.00	55.10	30.0	*** METH DEFAULT ***
Q2 134.00	23.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	101116
2	2.000	433773
4	4.000	992649
10	10.000	2536325
20	20.000	5408499
40	40.000	8720395
80	80.000	-1

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

82) 1,2-Dichlorobenzene-d4 ( )

Ret. Time 7.03 min., Extract & Integrate from 6.53 to 7.53 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 152.15			*** METH DEFAULT ***
Q1 150.15	163.20	30.0	*** METH DEFAULT ***
Q2 115.15	66.50	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	5.000	348724
2	5.000	377760
4	5.000	361903
10	5.000	386518

20	5.000	403921
40	5.000	422550
80	5.000	436092

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

83) 1,2 Dichlorobenzene ( )

Ret. Time 7.04 min., Extract & Integrate from 6.54 to 7.54 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 146.00			*** METH DEFAULT ***
Q1 111.00	45.20	30.0	*** METH DEFAULT ***
Q2 148.00	64.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	43588
2	2.000	175301
4	4.000	368649
10	10.000	949377
20	20.000	2057969
40	40.000	4447623
80	80.000	7504978

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

84) 1,2-Dibromo-3-Chloropropane ( )

Ret. Time 7.25 min., Extract & Integrate from 6.75 to 7.75 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 75.00			*** METH DEFAULT ***
Q1 155.00	49.80	30.0	*** METH DEFAULT ***
Q2 157.00	62.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	1126
2	2.000	6299
4	4.000	13345
10	10.000	38267
20	20.000	82780
40	40.000	185095
80	80.000	376237

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

85) 1,2,4-Trichlorobenzene ( )

Ret. Time 7.93 min., Extract & Integrate from 7.43 to 8.43 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***

Q1	182.00	95.30	30.0	*** METH DEFAULT ***
Q2	145.00	28.00	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	22255
2		2.000	91716
4		4.000	196945
10		10.000	524338
20		20.000	1161093
40		40.000	2630837
80		80.000	5813290

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

86) Hexachlorobutadiene ( )

Ret. Time 8.09 min., Extract & Integrate from 7.59 to 8.59 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 225.00			*** METH DEFAULT ***
Q1 223.00	63.70	30.0	*** METH DEFAULT ***
Q2 227.00	63.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		0.100	4451
.5		0.500	14881
2		2.000	64952
4		4.000	144414
10		10.000	374262
20		20.000	816664
40		40.000	1894579
80		80.000	4344336

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

87) Naphthalene ( )

Ret. Time 8.05 min., Extract & Integrate from 7.55 to 8.55 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 128.00			*** METH DEFAULT ***

Lvl	ID	Conc (ug/l)	Response
.1		not used for this compound	
.5		0.500	28047
2		2.000	126064
4		4.000	282471
10		10.000	780602
20		20.000	1749514
40		40.000	3917418
80		80.000	8089070

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

88) 1,2,3-Trichlorobenzene ( )

Ret. Time 8.15 min., Extract & Integrate from 7.65 to 8.65 min.

Signal	Rel Resp.	Pct. Unc.(abs)	Integration
Tgt 180.00			*** METH DEFAULT ***
Q1 182.00	95.50	30.0	*** METH DEFAULT ***
Q2 145.00	37.20	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ug/l)	Response
.1	not used for this compound	
.5	0.500	18230
2	2.000	76131
4	4.000	161446
10	10.000	420837
20	20.000	946961
40	40.000	2195208
80	80.000	4897589

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

END OF DATA ANALYSIS PARAMETERS

Mon Dec 17 14:51:23 2007

ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 60_8081

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE (EPA METHOD 608.3, SM 6630C and SW-846 METHOD 8081B)

APPROVED BY:

Operations Manager	Date 2/8/18
QA Manager	Date 1/24/18
Laboratory Director	Date 2/8/18

Organochlorine Pesticides by Gas Chromatography: Capillary Column Technique (EPA METHOD 608.3, SM 6630C and SW-846 METHOD 8081B)

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentrations of various organochlorine pesticides, in extracts from wastewater, ground water, surface water, sludge, soil, and solid matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD).

1.2 Several cleanup/fractionation schemes are provided in this procedure. Any compound is a potential method interference when it is not a target analyte.

1.3 Compound identification (single component compounds) based on single column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This procedure describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS is also recommended as a confirmation technique if sensitivity permits.

1.4 This procedure has reporting limits of 0.1 µg/L for aqueous samples and 5 µg/Kg for solid and waste samples. Higher reporting levels will result from reduced sample volumes and/or low percent solids. Lower detection limits are achievable by further concentration of sample extract upon request.

1.5 This method is performance-based. It may be modified to improve performance (e.g., to overcome interferences or improve the accuracy of results) provided all performance requirements are met. Examples of allowed method modifications for 608.3 are described at 40 CFR 136.6.

2.0 METHOD SUMMARY

2.1 A measured volume or weight of sample (approximately 1 L for aqueous samples and 2-30 g for soils) is extracted using the appropriate sample extraction technique.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using the separatory funnel technique (SOP 50_3510C). Solid samples are extracted with methylene chloride/acetone using the Soxhlet extraction (SOP 50_3540) or the microwave extraction method (SOP 50_3546). The extracts for liquid and solid samples are exchanged into hexane before analysis. Samples may also be extracted using a disk-based solid-phase extraction (SPE), providing such a method meets requisite QC criteria.

2.3 A variety of cleanup steps may be applied to the extract, depending on the nature of the co-extracted matrix interferences and the target analytes. After cleanup, the

extract is injected into a gas chromatograph with a wide-bore fused silica capillary column and electron capture detector (GC/ECD) for analysis.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan SOP 90_0001 in conjunction with the Safety orientation

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDSs for the concentrated chemicals used in the laboratory are kept on file in a central location that is available for all employees to review.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 Liter borosilicate glass jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4° C. Two liters should be provided so samples can be re-extracted when necessary.

4.2 Use potassium iodide starch paper to check each water sample for residual chlorine. If chlorine is detected, add sodium thiosulfate to remove the chlorine. Add 1-mL 10% sodium thiosulfate solution per liter. Addition of sodium thiosulfate solution to the sample container may be performed in the laboratory or prior to field use. For EPA method 608.3 if the sample is being analyzed for Aldrin, sodium thiosulfate should be added to remove residual chlorine when residual chlorine is present. For method 8081 if any residual chlorine is detected the sample must be treated with sodium thiosulfate. Since most 608.3 samples are analyzed for Aldrin each sample is checked for residual chlorine with the potassium iodide starch paper. The paper will react to free Iodine/Chlorine and peroxides in solution. Lower levels react with strip at 5 - 10 ppm. Chlorine reacts immediately. Initial reactions show a slight blue color, while higher concentrations turn the strip from dark blue to purple. Upper limits for chlorine are between 400 to 450 ppm. If the paper indicates the presence of chlorine sodium thiosulfate is added. If no chlorine is detected no sodium thiosulfate is added.

4.3 Aqueous samples must be extracted within 7 days from date sampled. When analyzing samples for EPA method 608.3, samples that are not between pH 5-9 ***must*** be extracted within 72 hours. If the samples will not be extracted within 72 hours of collection, the sample should be adjusted to ~~a pH range of 5.0-9.0 with~~ sodium hydroxide solution or sulfuric acid. Record the volume of acid or base used.

4.4 Soil/sediment samples are collected in 4–8 ounce jars with Teflon lined caps. The samples are stored in the dark at 4°C. Thirty grams of sample is required for extraction and ten grams is required to determine the percent solids. One hundred grams should be provided so samples can be re-extracted when necessary.

4.5 Soil/Sediment samples must be extracted within 14 days of date sampled.

4.6 All extracts are stored in 10ml Teflon capped vials in the extract storage refrigerator located in the Organic Prep lab. These extracts are stored at 4°C and must be analyzed within 40 days of date extracted.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Sources of interference in this method can be grouped into three broad categories: 1) contaminated solvents, reagents or sample processing hardware; 2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and 3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents, and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned as soon as possible after use by rinsing with the last solvent used. Detergent washing with hot water, and rinses with tap water should follow this. See SOP 50_0001.

5.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. SOP 50_3660 is suggested for removal of sulfur.

5.5 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Florisil (SOP 50_3620) cleanup eliminates co-eluting chlorophenols.

6.0 EQUIPMENT/APPARATUS

6.1 **Gas chromatograph**: an analytical system complete with gas chromatograph suitable for on-column and splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD) and DOS based PC system interfaced to the GC with HP Chemstation/ EnviroQuant software.

6.1.1 **Wide-bore Columns**:

6.1.1.1 Column 1 (RTX-CL Pesticide) 30 m x 0.53-mm ID fused silica capillary column, 0.50 um film thickness.

6.1.1.2 Column 2 (RTX-CL Pesticide II) 30m-x 0.53-mm ID fused silica capillary column, 0.42 um film thickness.

6.1.1.3 RTX CL Pesticide I and II 0.32mm ID columns may also be used on secondary instruments.

6.1.2 **Miscellaneous Instrument Parts**:

6.1.2.1 Single Gooseneck glass liners from either HP or Restek.

6.1.2.2 Gold Seals and Washers (HP 05971-27305)

6.1.2.3 Graphite Ferrules (0.53mm.)

6.1.2.4 O-rings (HP #5180-4182)

6.1.2.5 HP ceramic tiles for cutting columns.

6.2 **10 uL** glass bore Injector **Syringe**.

6.3 Class A **Volumetric flasks,** various sizes.

6.4 **Sample vials**: glass with Teflon-lined crimp tops, 2.0ml.

6.5 **Hamilton microliter syringes** – 10 µl, 25 µl, 100 µl, 500 µl, and 1000 µl

6.6 **Data system**:

6.6.1 **Computers:** Computer systems are networked to a Windows 2012 R2 server. Daily backups to disk are done at 3:45 AM. Full backups are performed on Saturday and differential backups Sunday through Friday. We keep 14 disk backups on disk. Full disk backups are copied to tape on Sundays. We keep the weekly tapes for 4 weeks, the monthly tapes for 4 months, the quarterly tapes for 4 quarters, and the yearly tapes for 10 years.

6.6.2 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to the electron capture detectors and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. The software is capable of integrating the abundance in any EICP between specified times. Current version for SVOA GC6, GC7 and GC11: G1701DA Version D.00.00.38.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: All reagents should be reagent or pesticide grade for this analysis. Unless otherwise indicated all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1 **Solvents**:

7.1.1.1 **n-Hexane**
7.1.1.2 **Methylene Chloride**
7.1.1.3 **Acetone**
7.1.1.4 **Isooctane (2,2,4-trimethylpentane)**

7.2 **Standards**: Store all standard solutions (stock, working, and matrix spiking), with the exception of primary standards, at 0-6°C in the dark. Primary standards are stored at room temperature to prevent crystallization and consequent loss of analytes from solution, as recommended by the manufacturer. Studies have determined that there is no noticeable loss of analytes by evaporation during normal storage periods.

7.2.1 **Primary Standards**: Expiration dates of unopened/opened primary standards are as stated in SOP 50.0006 or as stated by the manufacturer, whichever is earlier. Copies of the certificates of analysis are in a binder in the SVOA Dept. These certificates detail the compounds in each of the mixes. Certificates are kept on file in the laboratory. The following are typical stock standard solutions purchased as certified solutions. Other vendors may be used.

Primary Standard	Manufacturer	Catalog No.	Concentration
Pesticide Mix	AccuStandard	M-8081-SC	1000 µg/ml
Hexachlorobenzene	AccuStandard	APP-9-112	100 µg/ml
PEM Mix	AccuStandard	M-8081-DS	200 µg/ml
TCX/DCB Pest/PCB Surrogate	Accustandard	CLP-032-Rpak	200 mg/L
Toxaphene	Accustandard	P-093S	100 µg/ml
Chlordane	Accustandard	AS-0089	1000 µg/ml

Alachlor Standard	Restek	32204	1000 µg/ ml
Secondary Standard	**Manufacturer**	**Catalog No.**	**Concentration**
Pesticide Second Source	Ultra Scientific	PPM-808C	1000 µg/ml
Hexachlorobenzene	Ultra Scientific	EPA-1125	1000 µg/ml
Toxaphene	Restek	32071	5000 µg/ ml
Chlordane	Ultra Scientific	PP -150	100 µg/ ml
Alachlor Solution	Ultra Scientific	EPA-1068	5000 µg/ ml

7.2.2 **Primary Stock standard** is used to prepare the single-component Pesticide working standards. 50 µl of the primary pesticide mix standard, 250 µl of the primary surrogate standard, 50µl of the Alachlor Standard and 500 µl of the Hexachlorobenzene standard sre added to a 25 ml volumetric flask with the Hamilton syringes and diluted to the 25 ml mark with Hexane. The final concentration for each analyte and surrogate is 2000 ng/ml. ***The standard has an expiration date of one year from preparation or the earliest expiration date of the primary standards (whichever is earliest).***

7.2.3 **Pesticide Working Standards**

7.2.3.1 **Pesticide Initial Calibration Working Standards** are prepared by dilution of the primary stock standards with Hexane. Standards are prepared in volumetric flasks. Working standards are stored in the 40 ml amber vials for a maximum of six months. The lowest concentration level is at a concentration near but above the method detection limit. The concentrations should correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. The following table details the concentrations (ng/ml) of each analyte at each individual level.

Target Analyte	Volume Stock (7.2.2)	Initial Conc. (ng/ml)	Final Volume (ml)	Final Concentration
Level 1	12.5 µl	2000	25.0	1 ng/ml
Level 2	62.5 µl	2000	25.0	5 ng/ml
Level 3	125 µl	2000	25.0	10 ng/ml
Level 4	750 µl	2000	50.0	30 ng/ml
Level 5	1500 µl	2000	50.0	60 ng/ml
Level 6	1000 µl	2000	25.0	80 ng/ml
Level 7	1250 µl	2000	25.0	100 ng/ml

7.2.3.1.1 All levels are prepared using Hamilton syringes and hexane as the diluent. All analytes and surrogates are at the same concentration, see list of analytes in 8.5.8.4. All standards are measured with a 1000 µl graduated syringe

unless otherwise noted. All levels are prepared from dilutions of the stock standard.

7.2.3.2 **Pesticide Continuing Calibration Verification Standard (CCV)**: This is the Level 4 or 6 standard as prepared in 7.2.3.1.

7.2.4 **Pesticide Second Source Standards (ICV/LCS/MS)**

7.2.4.1 **Pesticide Second Source Stock standard**: This stock standard is prepared at a concentration of 2000 ng/ml. Fifty (50) µl of the second source pesticide mix from Ultra, 50 µl of the Hexachloro benzene second source, 250 µl of the surrogate mix and 10 µl of the second source Alachlor mix are added to a 25 ml volumetric flask and diluted to the 25 ml mark with Hexane. ***The standard has an expiration date of one year from preparation or the earliest expiration date of the primary standards (whichever is earliest).***

7.2.4.2 **Pesticide Second Source Initial Calibration Verification (ICV) Working Standard** is prepared at a concentration of 50 ng/ml. 1.25 ml of the second source stock standard (7.2.4.1) is diluted to 50 ml in a 50 ml vol. flask. The standard is transferred to a 40 ml vial.

7.2.5 **Toxaphene and Chlordane Standards:** Separate standards are prepared for each multi-component target analyte.

7.2.5.1 The **Primary Toxaphene Stock Standard** is prepared at a concentration of 5000 ng/ml for Toxaphene and using hexane as the diluent. 500 µl of the primary Toxaphene Mix from Accustandard, measured with a 1.0 ml graduated syringe, and 4 µl of the primary surrogate mix, measured with a 10 µl graduated syringe, are diluted to 10 ml in a 10 ml volumetric flask. (Surrogate at 80 µg/L.)

7.2.5.2 The **Toxaphene Working Standard** is prepared from the Primary Toxaphene stock standard (7.2.5.1). Only a single point is needed unless otherwise specified: Method 608.3 requires a minimum of three levels (50 ug/L, 500 ug/L and 1000 ug/L) using a 1000 µl graduated syringe with hexane as diluent.

Level	Initial Conc. (ng/ml)	Amount Added (µl)	Final Vol. (µl)	Final Conc. Toxaphene (ng/ml)	Final Conc. Surrogate (ng/ml)
1	5000	500	1000	2500	40

7.2.5.3 Prepare the **Toxaphene Second Source Stock Standard** at 2500 ng/ml from a 100 µg/ml solution (prepared by diluting 0.2 ml of 5000 µg/ml (Restek) to 10 ml with hexane).

7.2.5.4 Prepare the **Toxaphene Second Source Working Standard** at 2500 ng/ml from a 100 µg/ml solution (prepared by diluting 0.2 ml of 5000 µg/ml (Restek) to 10 ml with hexane); dilute 250 µl of the 100 µg/ml solution and 2 µl of surrogate to a final volume of 10 ml, with hexane.

7.1.5.5 The **Chlordane Stock Standard** is prepared at a concentration of 10 µg/ml using hexane as the diluent. 100 µl of the primary Chlordane Mix from Accustandard (100 µg/ml), measured with a 1000 µl graduated syringe, and 50.0 µl of the primary surrogate mix, measured with a 100 µl graduated syringe, are diluted to 10 ml in a 10 ml volumetric flask. (Surrogate at 1.0 µg/ml.)

7.1.5.6 The **Chlordane Working Standards** are prepared at five levels from the Chlordane stock standard (7.2.5.3). All levels are prepared using a 1000 µl graduated syringe and diluted with hexane.

Level	Initial Conc. (ppm)	Amount Added	Final Volume (ml)	Final Conc. Chlordane (ng/ml)	Final Concen. Surrogate (ng/ml)
1	10	625 (µl)	25	250	25

7.1.5.7 **The Chlordane Second Source Standard (ICV)** is prepared at 250 ng/ml using hexane as the diluent. 125 µl of the Ultra Scientific Chlordane Mix measured with a 500 µl graduated syringe and 12.5 µl of the primary surrogate mix, measured with a 25 µl graduated syringe, are diluted to 50 ml in a 50 ml volumetric flask.

7.1.6 **Surrogate/Matrix Spike Solution**: Prepared according to the following chart.

Analyte	Initial Conc.	Amount	Final Volume	Final Conc.
TCX/DCB (Surrogate)	200 ug/ml	500 µL[3]	200 ml[1]	0.5 ug/ml
Pesticide Mix (Ultra) Hexachlorobenzene (Ultra)	1000 ug/ml	50 µl[2]	100 ml	0.5 ug/ml
Alachlor (Ultra)	5000 ug/ml	10 µl[4]	100 ml	0.5 ug/ml

[1] Volumetric glassware size (e.g., final vol. of 200 ml made up in a 200 ml vol. flask).
[2] 100 µl syringe is used. [3] 1000 µl syringe is used. [4] 25 µl syringe is used.

8 PROCEDURE

8.1 **Extraction**:

Water samples are extracted at a neutral pH with methylene chloride using separatory funnel extraction (SOP 50_3510). Extract solid samples with methylene chloride-acetone (1:1) using Soxhlet extraction (SOP 50_3540) or microwave extraction (SOP 50_3546) procedures.

8.2 **Cleanup/Fractionation**:

8.2.5 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section:

8.2.5.5 Perform Florisil Cleanup (SOP 50_3620) to separate pesticide residues from other chlorinated hydrocarbons (polar compounds).

8.2.5.6 Perform Sulfur Cleanup (SOP 50_3660) to remove elemental sulfur, which interferes with electron capture gas chromatography of certain pesticides.

8.2.5.7 Perform Gel Permeation Cleanup (SOP 50_3640) to separate pesticide residues from other high molecular weight hydrocarbons and sulfur compounds. **Currently inactive.**

8.2.5.8 Perform Carboprep Cleanup to remove mid- to high non-polar compounds.

8.3 **Instrument Maintenance**: See Section 19.0 for instrument maintenance and troubleshooting instructions.

8.4 **Instrument Set Up**:

8.5.1 Operating Conditions: Set the chromatographic system to the following operating conditions:

Injector temperature =	225 °C
Detector temperature =	325 °C
Oven temperature Program:	
Initial temperature =	120 °C
Initial time =	1.00 min
Ramp rate =	11.0°C/min to 300°C.
Final time =	8.5 min
Carrier gas =	Helium at 0.99999 purity.
Constant pressure =	5.0 psi

8.6 **Initial Calibration (single component analytes):**

8.6.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.6.2 All acceptance criteria for initial and continuing calibration apply to both the primary and secondary columns.

8.6.3 **Priming the Column**: Once the chromatographic system operating conditions have been established, calibration may begin. Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed or de-activated by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Several analytes, including Aldrin, may be observed in the injection immediately following this system priming. Always run an acceptable solvent blank prior to running any standards or samples.

Loading the instrument: All standards and samples are transferred with disposable pipettes into 2 ml target vials designed to fit the HP auto-sampler. The target vials are labeled with the ID of the standard or sample using a fine point marker. The tray on the auto-sampler is numbered 1-99. A single column connects the inlet to a glass y splitter that is connected to two different columns that each go to a different detector. This allows each standard and sample to be injected once, split into two different columns and detected on two different detectors.

The PEM standard is placed in slot 1. The 7-level standards are placed in slots 2-8. This is the typical setup for the calibration standards. Five points minimum are required for 8081. Three points minimum are required for method 608.3. Vials can be placed in different slots as long as the slot number is written in the logbook.

8.6.4 **Log Book**: All samples set up on the instrument must be entered into the run logbook prior to sample analysis. Run logs are Excel spreadsheets stored on the network. The logbook must be filled out completely with the date, vial number (slot number), computer file number, method number, ESS lab ID, and the initials of the analyst setting up the instrument.

8.6.4.1 Date includes the day, month, and year.

8.6.4.2 Vial number: This field ***must*** be filled in for each entry.

8.6.4.3 Computer file ID is an abbreviation of the path and file associated with a particular vial number. (e.g. vial #3 analyzed on 3/12/98 has a computer file ID of 031298003)

8.6.4.4 The ESS Lab ID includes the ID of the standards, samples and all QC samples.

8.6.4.5 Initials are signed by the analyst setting up the instrument.

8.6.4.6 The Comment section is a summary of calibration results, dilution information, and any unusual observations. (Examples would include: carry over information into the sample, retention time shift, injection time, calibration standard is less than 15% recovery, sample needs dilution.)

8.6.4.7 The Method section is the method in the chromatographic software used to operate the instrument.

8.6.5 At the beginning of each 12-hour shift a **Performance Evaluation Mixture** (PEM) is run to check for degradation problems. The PEM must also be analyzed before the initial calibration. DDT and Endrin easily degrade in the injection port when the injection port or front of the column is dirty. When the PEM is injected, the degradation products of 4,4'- DDT (4,4'-DDE and 4,4'-DDD) are analyzed along with the degradation products of Endrin (Endrin aldehyde and Endrin Ketone). If the degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration. Corrective action includes the following:

8.6.5.1 Maintaining the injection port. DDT and Endrin breakdown can be minimized by thoroughly cleaning the injection port. Particulates on the surface of the injection port or liner can contribute to the Endrin breakdown.

8.6.5.2 High Endrin breakdown can also occur while using a y-split, if particles get trapped in the y-split taper replace y-split.

8.6.5.3 A poorly cut column can contribute to the DDT breakdown. Check the column to ensure the end is not jagged.

8.6.6 The percent breakdown is calculated as shown in section 9.4.

8.6.7 After the PEM mix has been analyzed and is within the required QC criteria, the analysis of the 7 level calibration standards may begin. All 7-level standards are injected like the sample using the instrument set-up

criteria in section 8.4. The standards are injected from the lowest to the highest standard (Pesticide Level 1 – Level 7).

8.6.8 **Starting a run with Chemstation Software**: The GC is controlled through Chemstation. These methods are set up with the instrument setup information from section 8.4. All of the operating parameters are saved in Chemstation under method file ID. The PT8081 method operates the front and rear injectors. These methods are saved in the C:\HPCHEM\5\METHOD\ directory. A copy of the Chemstation method is in Attachment A. To run the instrument:

8.6.8.1 Open the **Chemstation** icon.

8.6.8.2 From the Chemstation Menu select **Sequence**.

8.6.8.3 In **Initial Setup** type the name of the directory where data will be stored. For the pesticide analysis, all data is stored in a directory called GE for GC3 and GH for GC6 followed by the date. (E.g. if samples were set up to run on 9/12/05 on GC3 then the directory would be called GE091205)

8.6.8.4 In **Analytical Sequence** type in the vial numbers that correspond to when the front and rear injectors will start and stop analyzing.

8.6.8.5 In the **Sample List** type in the ID's of the samples and standards next to the corresponding vial numbers.

8.6.8.6 Select **Save** to save the newly created sequence. Save the sequence with the day created or the ID. (e.g.: A sequence saved on Monday is saved as Mon.s. Sequences do not have to be saved and can be easily recreated. Saving a sequence can save time if for any reason the analytical sequence needs to be restarted due to an instrument or computer malfunction.

8.6.8.7 Start the analysis by selecting **Run Analytical Sequence**.

8.6.8.8 A Chemstation data file will be created for each sample and standard. These files will be stored in the directory C:\HPCHEM\1\data.

8.6.9 **Reviewing the Initial Calibration Data:** All GC Chemstation Data is reduced with the EnviroQuant software. (Refer to EnviroQuant Operator's Manual).

8.6.9.1 Setting up an EnviroQuant Method: All pesticide methods are set up as documented in Attachment A. With each new calibration, the

response and absolute retention times are updated. The absolute retention times are taken from Pesticide Level 4 (50 ppb).

8.6.10 Updating a New Initial Calibration:

8.6.10.1 Load any pesticide method.

8.6.10.2 Save the method under the date that the standards and samples were set up to run. Methods are saved with the designator 8081 followed by two letters that are sequentially increased for each new calibration generated. (e.g: 8081AA, 8081AB, 8081AC ...) ***Note that the ID of this new method is recorded in the Method column of the Run Logbook.***

8.6.10.3.1 Convert and link all Chemstation data files to EnviroQuant files.

8.6.10.4 Load the Pesticide Level-4 standard file. Go into QEDIT and correctly integrate all of the pesticides. The following is the order of elution for the individual analytes:

Elution Order RTX-CL Pest.	**Elution Order** RTX-CL Pest. II	**Compound**	**Elution Order** RTX-CL Pest.	**Elution Order** RTX-CL Pest. II	**Compound**
1	1	TMX (Surr.)	14	13	Endosulfan I
2	2	Hexachlorobenzene	15	15	Dieldrin
3	3	a-BHC	16	16	Endrin
4	4	g-BHC	17	17	4,4'-DDD
5	5	b-BHC	18	18	Endosulfan II
6	6	d-BHC	19	19	4,4'-DDT
7	7	Heptachlor	20	20	Endrin Aldehyde
8	9	Aldrin	21	22	Methoxychlor
9	8	Alachlor	22	21	Endosulfan Sulfate
10	10	Heptachlor Epoxide	23	23	Endrin Ketone
11	11	g-chlordane	24	24	DCB (Surr.)
12	12	a-chlordane			
13	14	4,4'-DDE			

8.6.10.5 Go into the initial calibration and update the absolute retention times and responses.

8.6.10.6 For Levels 1,2,3,5, 6, and 7 update only the responses. ***After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound from each calibration standard's printout***

corresponds to the area count listed in the calibration method in Enviroquant

8.6.10.7 The software is set up to calculate the average response factor from the 7-level curve (Section 9.0). The percent relative standard deviation of the response factor in each of the 7-levels must be less than 20% and a minimum of 5 points must be used. (Note: the requirement for method 608.3 is 20% RSD and a minimum of 3 points can be used), (SM 6630C is 20%RSD). If any of the pesticides are outside of this criterion, check the data setup in the EnviroQuant software and review the integration of each of the 7-levels. If manual integrations need to be made, update responses again.

8.6.10.8 When updating responses, all peaks in each level should fall within the retention time windows established according to section 8.6. If the peaks have drifted outside of the retention time windows, then the initial calibration is not acceptable. This peak drift is usually due to a carrier gas leak or clog in a gas line. Most leaks come from the injection port. To correct a leak, start by tightening all connections, including the column nut, gold seal, and insert retainer nut. If this doesn't work, go to section 19.0 and replace the ferrule, the septa, and the o-ring. In some cases, the gooseneck liner will get clogged with a piece of septa. Inspect the liner for pieces of septa. If the problem persists, consult with supervisor or a service representative. Once the problem has been corrected, reanalyze the entire initial calibration.

8.6.10.9 For those analytes of interest that continue to be greater than 20% RSD (20% for 608.3, 20% for SM), use a linear calibration or non-linear calibration (quadratic regression). The linear calibration is a regression equation that does not pass through zero. See Calculations in section 9.0. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where,

y= instrument response (peak area)
a= Slope
x= Concentration
b= intercept

The regression calculation will generate a coefficient of determination that is a measure of the 'accuracy of fit' of the regression line to the data. In order to be used for quantitative purposes, the calibration curve must have a correlation

coefficient of $r \geq 0.995$ or coefficient of determination of $r^2 \geq 0.99$. A minimum of five consecutive points are needed for average response or linear regression. A minimum of six consecutive points are needed for quadratic regression. The analyst should select the regression order that introduces the least calibration error into the quantitation. *The maximum allowable %RSD for each target analyte is indicated below Refer to* ***Attachments B, C and E*** *for further details.*

8.6.10.10.1 Document all maintenance performed in the instrument log.

8.6.10.10.2 Once all integrations are correct, the %RSD is less than 20%, $r \geq 0.995$ or $r^2 \geq 0.99$ and all standards are within the RT windows, save the method.

8.6.10.10.3 After the initial calibration has been generated, the analyst must create a copy of the 10 ppb standard and quantitate the file with the new calibration. Review of the analyte recoveries in the standard will demonstrate the appropriateness of the calibration curve.

8.6.10.10.4 Analysis of a second source calibration verification standard is performed immediately after initial calibration (7.2.4.2). Acceptance criterion is 80-120% with no exceptions.

8.7 Retention Time Windows

8.7.1 Retention time windows are central to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC external standard methods. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

8.7.2 Determining Retention Time Windows:

8.7.2.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions are optimized for the target analytes and surrogates in the sample matrix to be analyzed.

8.7.2.2 Make three injections of the mid-range CCV single component standard mixtures and multi-component analytes (Toxaphene and Chlordane) over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

8.7.2.3 Record the retention time in minutes for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, calculate the mean and standard deviation of three to five peaks used for calibration or identification.

8.7.2.4 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then use a default standard deviation of 0.01 minutes. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

8.7.2.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the mid-range CCV (Level 4 standard) at the beginning of each analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

8.7.2.6 Absolute retention time windows must be calculated for each analyte and surrogate on each chromatographic column and instrument used.

8.7.2.7 New retention time windows must be established when a new GC column is installed or when GC column has been shortened during maintenance.

8.7.2.8 Surrogates are added to each sample, blank, QC sample and calibration standard. Surrogate retention times in the calibration standards are useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window for standards, the analyst is advised to determine the cause and correct the problem before continuing analyses.

8.8 **Sample Analysis**: Once the initial calibration has passed all of the quality control criteria, sample analysis may begin. All samples and standards must be run under the same conditions as the initial calibration.

8.8.1 **Sequence of Analysis:** At the beginning of each 12-hour shift, the PEM must be successfully analyzed. Samples cannot be analyzed more than 12 hours after the first PEM standard is injected and no more than 20 samples can be analyzed within the 12 hour period. Immediately after the PEM and prior to sample analysis the initial calibration must be verified with the mid-range CCV (Level 4 working standard). Samples are immediately analyzed after the CCV. Samples include the method blank, blank spikes, samples, matrix spikes, and matrix spike duplicates. The CCV alternates between the Pesticide Level 2 and 4 standards.

NOTE: 1) ***Analysis of a second source calibration verification standard is performed immediately after initial calibration in place of the CCV.*** 2) ***It is not acceptable practice to group QC samples together and/or to analyze QC samples on one instrument and their associated samples on another instrument. Analyst must try to analyze batch QC samples, as capacity allows, along with their associated field samples.***

8.8.2 **Loading calibration verification standards and samples**: See section 8.5.4.

8.8.3 **Log Book entries**: See section 8.5.5.

8.8.4 **Running Continuing Calibration Standards and Samples with Chemstation Software:** See section 8.5.8

8.8.5 **Reviewing Continuing Calibration Standard and Sample data:** All GC Chemstation data is reduced with the Enviroquant Software. (Refer to Hewlett Packard's EnviroQuant Operators Manual)

8.8.5.1.1 Update the Method with Continuing Calibration standards:

8.8.5.1.2 Open the Enviroquant Icon on the computer connected to the instrument that ran the samples and standards.

8.8.5.1.3 Load the method with the initial calibration data that corresponds to the samples and standards analyzed. See section 8.5.9.11. Usually this is the last method entered into the logbook.

8.8.5.1.4 Convert all Chemstation files to Enviroquant Files

8.8.5.1.5 Load the CCV standard that ran just prior to the first sample set up in the analytical sequence. Go into QEDIT and correctly integrate all of the pesticides. Go into the initial calibration and update the absolute retention times

only. **Do not update responses!** The order of the pesticide elution for each of the columns is listed in section 8.5.10.4. The CCV standard should only have 22 predominant peaks. If there are more or less than 22 peaks then refer to the following corrective action and reanalyze all standards.

Problem	Corrective Action
Wrong standard injected	Check sequence setup section 8.5.4. Make sure standards are correctly placed on tray.
Contaminated standard	Inject another aliquot of standard or remake standard.
Standard prepared incorrectly	Remake standard
Dirty injection port	Perform maintenance. Refer to section 19.0

8.8.5.1.6 Save the updated method.

8.8.6 Reviewing the Continuing Calibration Standards:

8.8.6.1.1 Use the method updated in section 8.7.5.1.5 to analyze (calculate and generate report) the calibration standards which ran before and after the samples of interest.

8.8.6.1.2 If the retention times have shifted outside the retention time window, see section 8.6. If there is high breakdown, perform the maintenance in section 19.0.

8.8.6.1.3 The CCV standards must be injected at intervals of not less than once every 12 hours or 20 samples and at the end of the analysis sequence. Note: every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded. Concentration levels must alternate between low and high concentration standards (equivalent to second and fourth levels in calibration curve). See calculations in section 9.0. Acceptance criteria are as follows:

Routine Analysis, EPA 608.3	Percent difference/drift $\leq$ 20% for all reported analytes
MCP CAM	-Percent difference/drift $\leq$ 20% for all reported analytes
SM6630C	Percent difference/drift <15% for all reported analytes

8.8.6.1.4 The breakdown in the PEM standard must meet criteria in Section 8.5.5. All of the retention times must also be within the established Retention Time windows (see

section 8.6). When these criteria are exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. (See section 19.0) If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

8.8.7 Sample injection may continue for as long as the calibration verification standards and standards interspersed within the samples meet the QC requirements. The sequence ends when the standard that follows a set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

8.8.8 When analyzing samples for multiple component pesticides (Chlordane and Toxaphene), any hits must be verified with a mid-level standard analyzed within 12 hours of the sample. The calibration factors of these analytes must be less than 20% difference when compared to the initial calibration. (<20%D for 608.3 and SM6630C) The retention times must also be within the established RT windows.

8.9 Sample Data Reduction:

8.9.1 All samples bracketed within acceptable calibration standards are analyzed by the same method used to analyze the standards. Through EnviroQuant, a quantitation report and chromatogram is generated for each sample.

8.9.2 The EnviroQuant software will tentatively identify a single component pesticide when a peak from a sample extract falls within the daily retention time window.

8.9.3 The analyst must carefully review the chromatograms, in QEdit, to ensure that all peaks were identified correctly.

8.9.3.1 Each blank, blank spike, blank spike duplicate, sample, matrix spike, and matrix spike duplicate should have surrogates identified. If the surrogate has not been identified, go into Qedit and integrate the peak in the surrogate retention time window. In some cases, surrogates cannot be identified due to interferences from unknown components that elute at the same time as the surrogate (see section 11.0 for out of criteria corrective action). This must be noted in the case narrative. If the surrogates are outside the retention time window, then the retention times have shifted. If this

happens, the sample must be re-analyzed. Oily samples can cause a retention time shift. These samples will need to be diluted at least 3x prior to analysis.

8.9.3.2 The analyst must also review the baseline on all the sample and standard chromatograms. A baseline rise will result from contamination of the injection port and column. When the baseline interferes with sample and standard analysis, re-analyze samples and standards. Attempt to isolate the sample or samples that contaminated the system. Perform cleanup procedure or dilute these samples, if possible, before re-analysis.

8.9.3.3 Late eluting compounds can carryover from sample to sample or sample to standard. Large unresolved peaks in a sample are a good indication of carryover. If carryover occurs, first identify the sample that is causing the carryover. Rerun samples with carryover contamination. Run instrument blanks, consisting of clean hexane, after the sample that carried over until carry over is eliminated. In cases when carry over is too great, the sample may need to be cleaned up further or diluted.

8.9.4 The on-column concentration of each analyte in the sample is provided on the quantitation report. The concentration is in ng/ml. The concentration is calculated from the average response factor or line equation from the initial calibration, which ran prior to analysis.

8.9.5 To quantitate a pesticide analyte, the concentration must be greater than or equal to the MDL. If the analyte concentration is below the MDL then it cannot be reported as detected.

8.9.6 If the analyte concentration is above the MDL and below the level 7 concentration standard, then the result is tentative and must be confirmed.

8.9.7 If the analyte concentration is above the level 7 concentration, then dilute the sample to bring the concentration of the analyte to within the level 1 and level 7 range.

8.9.8 Confirm pesticide by performing the same analysis on a different column. If the sample was initially analyzed on an RTX-CL1 Pesticide column and there was an analyte detected above the method detection limit, the sample must be confirmed on an RTX-CL2 Pesticide Column. The reverse also holds true.

8.9.9 Analyte results are considered confirmed when there is a peak within the retention time window for the analyte on both the primary and confirmation columns **and** both results are above the MDL.

8.9.10 The final result is determined by comparing the initial analysis with the confirmation analysis.

8.9.10.1 When an analyte result is within calibration range, the primary column result is compared to the confirmation result. If the RPD< 40%, then the higher result is reported (EPA 608 requires lower result to be reported in all cases). If the RPD > 40%, the analyst must evaluate the chromatogram for co-eluting peaks. If no co-elution is detected and all other QC is within criteria, report the higher result. If co-elution was present, the analyst is to report the result without co-elution and an explanation is to be provided in the case narrative.

8.9.10.2 If the confirmation results are at a concentration greater than the level 7 standard, then a dilution must be made and analyzed. The criterion stated in 8.8.10.1 is applied to the two results.

8.9.10.3 ESS Laboratory will, upon request, report analytes results to the MDL. Analyte results between the MDL and RL have a greater uncertainty in concentration than results within the calibration range and will be J-flagged as estimated.

8.9.11 Identification of mixtures (i.e., Toxaphene and Chlordane) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s). Quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using external calibration procedures.

8.10 Quantitation of Multiple Component Analytes:

8.10.1 Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handing Chlordane and Toxaphene.

8.10.2 Initial calibration for routine analysis will consist of a one point calibration at the mid point of the calibration curve.

8.10.3 Chlordane is a technical mixture of at least 11 major components and 30 or more minor components. *Trans*- and *cis*-Chlordane are the two major components of technical Chlordane. However, the exact percentage of each in the technical material is not completely defined,

and is not consistent from batch to batch. *NOTE: ESS Laboratory calibrates for both alpha- and gamma-chlordane when analyzing pesticides. Both compounds are present in technical chlordane. Therefore, if neither compound is present in the sample extract the laboratory has proof that the sample is non-detect for technical chlordane and will not recalibrate the sample file with the technical chlordane method.*

8.10.3.1 The GC pattern of a Chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents from the technical Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

8.10.3.2 Whenever possible, if Chlordane residue does not resemble technical Chlordane, the analyst should quantitate the peaks of alpha -Chlordane, gamma-Chlordane, and Heptachlor separately against the appropriate reference materials, and report the individual residues.

8.10.3.3 When the GC pattern of the residue resembles that of technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using 3-5 major peaks. The peaks are quantitated individually and the average concentration is reported as the result.

8.10.4 When the GC patterns resemble Toxaphene, the analyst will quantitate the chromatogram using the average of five peaks (EPA method 8081B, Section 11.6.1.3.3 allows for the use of a subset of 4–6 peaks) in the sample chromatogram, which must be compared to that of the standard to ensure that all of the major components in the standard are present is the sample. Otherwise, the sample concentration may be significantly underestimated. The use of 4 to 6 peaks provides results that agree well with the total peak area approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT.

9 CALCULATIONS

9.1 Calculate pesticide soils as follows:

$$\text{Pesticide Result ug/Kg} = \frac{\text{(Concentration of Peak ppb)(Extract Volume ml)(Dilution)}}{}$$

(% Solid/100)(Weight of Sample Measured g)

NOTE: For multi-component pesticides (Chlordane and Toxaphene) use average concentration of 3-5 peaks for Chlordane and total area for Toxaphene.

9.2 Calculate pesticide aqueous as follows:

$$\text{Pesticide Result ug/L} = \frac{\text{(Concentration of Peak ppb)(Extract Volume in ml)(Dilution)}}{\text{Volume of Sample in ml}}$$

NOTE: For multi-component pesticides (Chlordane and Toxaphene) use average concentration of 3-5 peaks for Chlordane and total area for Toxaphene.

9.3 Calculate pesticide matrix spike recoveries as follows:

9.3.1 Matrix spike added is calculated as follows:

$$\text{Matrix Spike Added} = \frac{\text{(Concentration of Spike)(Volume of Spike Added)}}{\text{Initial Volume}}$$

NOTE: Initial volume can be in liters or grams. If it is a soil, make sure to multiply initial volume by % solids/100.

9.3.2 Matrix spike result is calculated as follows:

9.3.2.1 (Refer to Section 9.1, 9.2, 9.3 and 9.4.)

9.3.3 Matrix spike % recovery is calculated as follows:

$$\text{\% Recovery} = \frac{\text{Matrix Spike Result –sample result}}{\text{Matrix Spike Added}} \times 100$$

9.4 Calculate percent breakdown as follows (for DDT and Endrin):

$$\text{\% Breakdown for 4,4'-DDT} = \frac{\text{Total DDT Degradation Peak Area (DDE + DDD)}}{\text{Peak Area (DDT + DDE + DDD)}} \times 100\%$$

$$\text{\% Breakdown Endrin} = \frac{\text{sum of degradation peak area (Aldehyde + Ketone)}}{\text{sum of all peak area (Endrin +Aldehyde + Ketone)}} \times 100\%$$

9.5 For surrogate recoveries, % RSD and all other calculations done by the computer's software refer to Hewlett Packard © 1992 printed in USA 11/92 Part No. HP G1032-90020.

9.6 Calibration Factor (CF)

CF = Peak Area / Mass of Compound Injected

9.7 Percent Relative Standard Deviation:

% RSD = (SD / Average CF) x 100%

Where: SD = standard deviation

9.8 Percent Difference:

% D = ((CFv – CF ave) / CF ave) x 100%

Where:
CFv = Calibration Factor of the verification standard.
CF ave = The mean calibration factor from the initial calibration.

9.9 Percent Drift:

$$\% \text{ Drift} = \frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}} \times 100\%$$

10 QUALITY ASSURANCE/QUALITY CONTROL

10.1 Immediately after the initial calibration, a second source standard (ICV – see table in Section 7.2.1) is analyzed. This standard is prepared at the Level 4 concentration. The percent recovery of the second source must be 80-120%. If the percent recovery is outside criterion, then corrective action must be taken. Perform instrument maintenance and/or prepare a new second source standard. If the second analysis of the ICV is not within criterion, then a new calibration curve must be generated. **Sample analysis cannot begin until a valid second source has been analyzed.**

10.2 **Accuracy and Precision**: All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:

Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution must be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve. In most cases the blank spike or matrix spike solution is used.

Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

QC Sample Analysis: The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation: Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation: Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$\%RSD = (s / \bar{x})\ 100\ \%$

Where:
s = Standard Deviation of a finite number of values. On a scientific calculator use the **σxn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision: Report Accuracy and Precision data with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:
Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable
Sample Fractionated by: If Applicable Sample Analyzed by:
Accuracy: Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results: The percent recoveries should be between 50-130% and the %RSD should be less than 30%. If any of the accuracy and precision results do not fall within the criteria then re-prep and re-analyze all QC samples only for those analytes that were not within criteria.

10.3 A Method Blank is extracted with each 20 samples or analytical batch. The concentration of analytes in the method blank should be less than the MRL (see

Attachment B for Method 608.3; MRL is the Level 1 standard). If the concentration of any analyte exceeds the MRL or 608.3 requirements, then proceed to Section 11.0 for corrective action.

10.4 Run a 7-point initial calibration curve (Method 8081 allows 5 point minimum, Method 608.3 allows a minimum of 3 points), using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria are ≤ 20%.

10.5 A Blank spike and Blank spike duplicate (BS/BSD) are analyzed with each batch of 20 or less samples. The acceptance criterion for the BS/BSD is 40-140% Recovery, 20% RPD for waters and 30% RPD of the certified value for soils. ***See Attachment B for 608.3 blank spike acceptance criteria and see Attachment E for MADEP MCP CAM requirements.*** If the BS/BSD is not within limits, see section 11.0 for corrective action. Sporadic Marginal Exceedance number is as follows:

10.6 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits of 30-150%. Both surrogates on both columns must be within criterion. ***See Attachment E for MADEP MCP CAM requirements.***

10.6.1 If the same surrogate is outside limits on both columns, re-extract the sample.

10.6.2 If both surrogates are outside limits on one column only, then re-analyze the sample extract.

10.6.3 If a surrogate is diluted below the lowest calibration standard, no corrective action is necessary.

10.6.4 If re-extraction or reanalysis yields similar results, the laboratory must note this in the project narrative.

10.6.5 If re-extraction or reanalysis is performed within hold time and produce results within criterion, then only results from re-extraction or reanalysis needs to be reported.

10.6.6 For MADEP, if re-extraction or reanalysis is performed outside hold time and produce results within criterion, then both results from re-extraction or reanalysis should be reported.

10.6.7 Note all exceedances in project narrative.

10.7 Continuing calibration verification (CCV) must be performed at the beginning and end of each analytical sequence, see 8.7.5.2.3 for frequency/level of CCV. The CCV

uses the Pesticide Level 2 and 4 standards, alternated throughout the analytical sequence.

10.8 Acceptance criteria is a follows:

Routine Analysis EPA 608.3	Percent difference/drift $\leq$20% for all reported analytes
MCP CAM	-Percent difference/drift $\leq$20% for all reported analytes
SM 6630C	Percent difference/drift $\leq$ 15% for all reported analytes

10.8.1 When a CCV is out of this acceptance window, the laboratory should stop analyses and take the following corrective action:

10.8.1.1 When criteria are exceeded then remake and re-analyze CCV If corrective action does not produce a second consecutive (immediate) CCV within acceptance limits, then the analyst may demonstrate the initial calibration is valid by analyzing two consecutive CCVs at two concentrations. If CCV criteria are not acceptable, then a new initial calibration must be performed. ***All samples analyzed after the last acceptable CCV must be re-analyzed except for CCVs that are exceeded high, samples with results that are non-detect may be reported. It has been shown that results would have been detected***

10.8.1.2 When performing analysis for MA MCP, any analyte outside of criteria in the CCV must be noted in the project narrative

10.9 The breakdown of DDT and Endrin must be measured before standards and samples are analyzed at the beginning of each 12-hour shift. Injector maintenance and re-calibration must be performed if the breakdown is greater than 15% for either compound (CAM). See section 8.5.6 for details. For calculation refer to Section 9.0.

10.10 A matrix spike and matrix spike duplicate (MS/MSD) are analyzed with every batch of 20 samples. The acceptance limits 40-140% Recovery and $\pm$ 30% RPD. ***See Attachment B for Method 608.3 requirements and Attachment E for MADEP MCP CAM requirements.*** If the blank spike (BS/BSD) results are acceptable and the matrix spike/matrix spike duplicate results are outside of QC limits, note exception in case narrative.

10.11 Control charts are generated quarterly for the BS (Soil and Water)

10.12 MDLs are determined in reagent water and Ottawa sand/sodium sulfate and verified annually. See SOP 110_0013 for complete MDL study instructions. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

11 DATA VALIDATION

11.1 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges by completing the SVOA Analysis Map (see Section 11.3) for GC/ECD Chlorinated Pesticides. The only exceptions made to ranges would be the following:

11.1.1 For MS/MSD, the RPD should be ±30% (***see Attachment E for MADEP MCP CAM requirements***). However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be 40-140%. ***See Attachment B for Method 608.3 and Attachment E for MADEP MCP CAM requirement)***. If the matrix spike is outside limits, check the BS/BSD. If the BS/BSD is within limits, matrix interferences are present and should be noted in the narrative.

11.1.3 Analytical batches with Method blanks above the MRL will be re-prepped and re-analyzed with the following exceptions:

11.1.3.1 Samples that are at least twenty times higher than the method blank (10 x for 608.3) may be reported.

11.1.3.2 When the method blank is less than one-third of the regulatory compliance limit associated with the analyte the method blank would be acceptable.

11.1.3.3 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.3.4 Any results that are reported with method blank contamination must be B-flagged.

11.1.4 For the BS/BSD, the %Recovery should be 40-140%. ***See Attachment B for Method 608.3 and Attachment E for MADEP MCP CAM requirements***. If the BS/BSD is outside this criteria, the analytical batch will be re-extracted and re-analyzed with the following exceptions:

11.1.4.1 For BS/BSD >140%, ***See Attachment E for MADEP MCP CAM requirements***. Samples with results below the MRL may be reported. It has been shown that the results above MRL would have been detected.

11.1.4.2 For BS/BSDs < 45% samples with results above a regulatory limit may be reported.

11.1.4.3 In some instances there may be insufficient sample to re-extract. The client is to be contacted and the results, if reported, are to be reported as estimated values when this occurs.

11.1.4.4 Any samples that are reported with invalid BS/BSD data must have a notation in the case narrative.

11.2 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.3 All data is reviewed for accuracy by a second analyst. Results of this review are noted on the Run Log.

12 REFERENCES

12.1 SW846, Third Edition, Updates III and IV, Methods 8081A and B. .

12.2 HP GC EnviroQuant User's Guide, HPG1045A

12.3 HP Environmental Data Analysis User's Guide HPG0032C

12.4 HP 6890 GC and HP 5890 Series II Operations Manuals

12.5 Standard Methods, 22nd Edition, Method No 6630 C.

12.6 Massachusetts DEP WSC-CAM V B.

12.7 EPA Method 608.3 (2016)

12.8 TNI Standard: Volume 1, Module 2 and Volume 1, Module 4.

12.9 40 CFR Part 136 - Guidelines Establishing Test Procedures for the Analysis of Pollutants

13 ATTACHMENTS

13.1 Attachment A – Deleted

13.2 Attachment B – Specific QC Requirements for EPA Method 608.3

13.3 Attachment C – Summary of Method Quality Objectives.

13.4 Attachment D – Chemstation method.

13.5 Attachment E – Specific QC Requirements for Mass MCP WSC-CAM-V B.

14 POLLUTION PREVENTION and WASTE MANAGEMENT

14.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

15 METHOD PERFORMANCE

15.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 50-130% Recovery and %RSD of $\leq 30\%$.

15.2 The precision and accuracy data in Table 1 were developed using the Soxtherm extraction method. Values are in ug/L.

16 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data

Compound	Spk	Avg	%Rec	%RSD	Compound	Spk	Avg	%Rec	%RSD
4,4'-DDD	50	55.177	110.4	5.7	Endosulfan Sulfate	50	52.684	116.6	5.6
4,4'-DDE	50	56.231	112.5	6.5	Endrin Aldehyde	50	51.402	105.4	4.5
4,4'-DDT	50	62.526	125.1	6.0	Endrin Ketone	50	52.191	113.7	6.9
alpha-BHC	50	50.965	106.6	7.6	gamma-BHC (Lindane)	50	52.584	102.8	5.4
alpha-Chlordane	50	60.291	101.9	9.4	gamma-Chlordane	50	58.635	104.4	4.4
beta-BHC (1)	50	53.577	120.6	6.5	Heptachlor	50	54.870	105.2	8.5
Decachlorobiphenyl	50	34.056	107.2	6.2	Heptachlor Epoxide	50	54.761	117.3	7.2
delta-BHC	50	52.437	68.1	3.1	Hexachlorobenzene	50	47.598	109.7	7.8
Dieldrin	50	57.653	104.9	6.7	Methoxychlor	50	59.114	109.5	7.0
Endosulfan I	50	58.857	115.3	6.3	Tetrachloro-m-xylen	50	41.102	95.2	7.9
Endosulfan II	50	58.316	117.7	7.3					

17 DEFINITIONS

17.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

17.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

17.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

17.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

17.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

17.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 2 to 3 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

17.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

17.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

17.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

17.10 **Matrix Duplicate**: An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

17.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

17.12 **Matrix Spike Duplicates**: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

17.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

17.14 **Method Detection Limit (MDL)**: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

17.15 **Organic-Free Reagent Water**: All references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

17.16 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

18 PERSONNEL QUALIFICATIONS

18.1 Analysts who perform this analysis must have a working knowledge of quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

18.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

19 TROUBLESHOOTING

19.1 **Instrument Maintenance**: The following procedure is performed when the instrument is initially set up or when a continuing calibration has failed the QC criteria.

19.1.1 Set the GC system to room temperature.

19.1.2 Turn off oven.

19.1.3 Remove column by unscrewing the column in the injection port.

19.1.4 Remove septum nut and septa. Discard septa.

19.1.5 Remove the insert retainer nut. This will expose the O-ring and glass liner. Using a set of tweezers, remove O-ring and liner. If O-ring is not distorted then set aside for later use. Otherwise, replace O-ring. Remove the glass liner. Rinse liner with methanol and scrub with a cotton swab. If the liner is visibly stained, then replace with a new one.

19.1.6 With cotton swab dipped in methanol, clean the injection port and retainer nut.

19.1.7 Remove the gold seal nut located on the bottom of the injection port. With a cotton swab and methanol, clean the gold seal.

19.1.8 Replace all parts in the following order:

19.1.8.1 Gold seal nut. Hand tighten and 1/4 turn with wrench.

19.1.8.2 Insert clean or new glass liner.

19.1.8.3 Place O-ring over liner. Slide O-ring over and down the liner until it fits snug against the injection port.

19.1.8.4 Replace insert retainer nut.

19.1.8.5 Place a new green septa into insert retainer nut.

19.1.8.6 Replace septum nut. **Only hand tighten!**

19.1.8.7 Slide column nut and a new graphite ferrule over column.

19.1.8.8 Using a ceramic tile, cut 3-6 inches off the column. The cut must be square with no jagged edges.

19.1.8.9 Connect column to injection port by inserting 3 mm of column into the injection port and hand tighten column nut then adding 1/4 turn with a wrench.

19.1.9 Make sure all gases are flowing. (Measure flows with bubble meter.) The flow should be between 5 and 6 ml/min.

19.1.10 Turn on injection port temperature.

19.1.11 Set oven temperature to 120 °C and allow the system to stabilize. Bake out the oven at 300°C for two hours. Reset back to 120°C.

19.2 Record all maintenance in the instrument's maintenance logbook.

20 DATA MANAGEMENT AND RECORDS

20.1 Data Management - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

20.2 Records – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for ten years from last use (10 years for drinking water is mandatory). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

ATTACHMENT B - Specific Requirements for Method 608.3

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Instrument set up. Each time the ICV or CCV cannot meet criteria.	• Minimum of 3 standards and contains all analytes • Low standard ≤ MRL • RSD≤20%,(*r≥0.995opt.)*, $r^2 \geq 0.92$	• No allowance. Perform maintenance and recalibrate.
ICV – second source verification standard	Immediately following initial calibration.	• %Rec = 80-120% (official method states 85-125%) • Must contain all target analytes. • No allowances	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is within acceptable criteria then calibration is accepted, otherwise recalibrate. • Report exceedance in narrative
CCV	Prior to sample analysis, every 12 hours and every 10 field samples and at the end of each analytical sequence.	• Concentration level near midpoint of curve • Must contain all target analytes. • Percent difference or percent drift must be ≤ 20%	• If criteria are exceeded then remake and re-analyze CCV. If second consecutive CCV is within criteria then calibration is verified, otherwise re-calibrate system and re-analyze any sample analyzed after invalid CCV.
Method Blank	One per analytical batch of 20 or fewer samples.	Matrix specific. If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample in the batch, whichever is greatest, analysis of samples must be halted and samples in the batch must be re-extracted and the extracts reanalyzed.	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate	Prior to sample analysis, every 12 hours and every 20 field samples and at the end of each analytical sequence.	• Prepared using standard source different than used for initial calibration • Concentration level should be between low and mid-level standard	• Report exceedance in the project narrative. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report

		• Must contain all single component analytes • Matrix specific • Percent recoveries per Table 4 of Method 608.3. (45-140%)	just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Matrix Spike/ Matrix Spike duplicate	Prior to sample analysis, every 12 hours and every 10 field samples and at the end of each analytical sequence.	• Prepared using the same source as the blank spike • Concentration between low and mid-level standard • Must contain all single component analytes • Matrix specific • Percent recoveries per Table 4 of Method 608. (45-140%)	• Check BS/BSD, if recoveries are acceptable then note exceedance in project narrative.

COLUMN 1
Capillary Column
Model Number: RESTEK 11140
clp pest .53
Max temperature: 330 'C
Nominal length: 30.0 m
Nominal diameter: 530.00 um
Nominal film thickness: 0.50 um
Mode: constant pressure
Pressure: 2.83 psi
Nominal initial flow: 2.7 mL/min
Average velocity: 24 cm/sec
Inlet: Front Inlet
Outlet: Front Detector
Outlet pressure: ambient

COLUMN 2
Capillary Column
Model Number: RESTEK 11340
RESTEK CLPESTICIDE II 0.53
Max temperature: 330 'C
Nominal length: 30.0 m
Nominal diameter: 530.00 um
Nominal film thickness: 0.42 um
Mode: constant pressure
Pressure: 3.00 psi
Nominal initial flow: 2.8 mL/min
Average velocity: 26 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

FRONT DETECTOR (μECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 60.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

BACK DETECTOR (μECD)
Temperature: 300 'C (On)
Mode: Constant makeup flow
Makeup flow: 60.0 mL/min (On)
Makeup Gas Type: Nitrogen
Electrometer: On

SIGNAL 1
Data rate: 20 Hz
Type: front detector
Save Data: On
Start Save Time: 3.00 min
Stop Save Time: 30.00 min
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: back detector
Save Data: On
Start Save Time: 3.00 min
Stop Save Time: 30.00 min
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
Derive from front detector

COLUMN COMP 2
Derive from back detector

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	1	
Sample Pumps	5	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
PostInj Solvent A Washes	2	
PostInj Solvent B Washes	2	
Viscosity Delay	0	seconds
Plunger Speed	Fast	
PreInjection Dwell	0.00	minutes
PostInjection Dwell	0.00	minutes

Back Injector:

Method: Q:\SVOA\GC3_GE\METHODS\8081DQ.MTH Present GC-3 method.

Method Information

pesticide method 8081

Run Time Checklist

Pre-Run Program:	none
Name:	
Parameter:	pes
Data Acquisition:	On
Use Barcode Labels:	Off
Data Analysis:	On
Sig. 2 Mth:	none
Post-Run Program:	none
Name:	
Parameter:	

Injector Information

Injection Source:	Auto
Injection Location:	Front
Front:	
Sample Washes:	2
Sample Pumps:	2
Sample Volume:	2 stops
Viscosity Delay:	2 sec.
Solvent A Washes:	3
Solvent B Washes:	3
On-Column:	No

Purge A/B:

	Init Value	On Time (Min.)	Off Time (Min.)
A (Valve 3)	Off	1.00	0.00
B (Valve 4)	On	0.00	0.00

A - Splitless Injection:	No
B - Splitless Injection:	No

Temperature Information

Zone Temperatures:

	Set point
Inl. A	200 C.
Inl. B	Off
Det. A	300 C.
Det. B	300 C.
Aux.	Off

Oven Parameters:	
Oven Equib. Time:	0.20 Min.
Oven Max:	320 C.
Oven	On
Cryo	Off

Method: Q:\SVOA\GC3_GE\METHODS\8081DQ.MTH

Ambient:	25 C.
Cryo Blast	Off

Oven Program:

	Set Point
Initial Temp:	120 C.
Initial Time:	1.00 Min.

Level	Rate (C./Min.)	Final Temp. (C.)	Final Time. (Min.)
1	12.0	300	9.00
2(A)	0.00		
3(B)			

Next Run Time: 25.00 Min.

Signal Information

Save Data: Both

Signal 1

Source:	Det. A
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	4.00 Min.
Stop Data:	25.00 Min.

Signal 2

Source:	Det. B
Peak Width:	0.053 Min.
Data Rate:	5.000 Hz.
Start Data:	4.00 Min.
Stop Data:	25.00 Min.

Valves/Relays Information

Initial Setpoints:
5890 Valves:

Valve 1:	Off
Valve 2:	Off
Valve 3 (Purge A):	Off
Valve 4 (Purge B):	On

Detector Information

Detector	Type	State
A	ECD	On
B	ECD	On

Timed Events:

Events:	Value:	Time:
Signal Switch	OFF	0.00
Signal Switch	ON	0.00

Inlet A Pressure Program Information

Constant Flow: Off

Pressure Program:

	Setpoint
Initial Pres.:	20.0 psi
Initial Time:	1.00 min.

Level	Rate (psi/min.)	Final Pres.(psi)	Final Time (min)
1	99.00	2.6	40.00
2(A)	0.00		
3(B)			

Total Program Time: 41.18

GC Pressure Units: psi

Fixed Values:

Column Length (m):	30.00
Column Diameter (mm):	0.530
Gas:	He
Vacuum Comp:	Off

Packed Column Information:

Equation: ---

Calibration Parameters:

	Pres. (psi)	Flow (ml/min)
1	---	---
2	---	---
3	---	---

Last pressure calibration: ---

Packed Column Flow Setting (ml/min): 0.0

Sequence Recalibration Table

Cal. Line	Cal. Level	Update Response Factor	Update Retention Times	Recalib Interval

Signal Plot Information

Signal	Attn. (2^)	Offset (%)	Time (Min.)
1	0	10	10
2	0	10	10

Integration Events

Events:	Value:	Time:
Initial Area Reject	1	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	0	INITIAL

Report Specification

Destination: Report to Screen
Based on: Area
Calculations: ESTD
Printer Output: None
Report Header: None

Graphics Options

Title: Vertical
Include:
Axes Units: On
Peak Names: On
Retention Times: On
Baselines: On
Tick Marks: On
Peak Labels Font: Default 12

Calibration Settings

Title:
GC3

Reference window: 0.500 minutes
Non-reference window: 0.500 minutes
Units of amount: ng/ul
Multiplier: 1.0
RF uncal peaks: 1.0
ISTD# to adjust uncal peaks: 0
Sample Amount: 0.0

Sample ISTD Information

No Sample ISTD Amounts

Multilevel Information

Fit: Linear
Origin: Force

Calibration Table Empty

ATTACHMENT E

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B).	No	NA	NA	NA
Endrin/DDT Breakdown	Laboratory Analytical Accuracy	(1) Before samples are analyzed and at the beginning of each 12-hour shift. (2) % Breakdown must be $\leq$15 and must be evaluated using peak areas.	Yes	(1) If DDT breakdown >20%, reject nondetect results for 4,4'-DDT. (2) If endrin breakdown >20%, reject nondetect results for endrin.	Perform injection port maintenance. Re-calibrate, if required.	Report exceedances (% breakdown >15%) and associated samples in laboratory narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be $\leq$RL. (4) %RSD $\leq$20, r $\geq$0.99 (linear regression), or r^2 $\geq$0.99 (non-linear regression) for each single-component pesticide. (5) If %RSD >20, linear or non-linear regression must be used. (6) Must contain all single-component	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value[3], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, or r^2 <0.99) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		pesticides. (7) Multi-component analytes: Analysis of a single standard at expected mid-point of calibration range. (8) Calibration must be performed under the same conditions as the samples. (9) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.			when recalculated using the final calibration curve.	
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all single-component pesticides. (5) Percent recoveries must be between 80-120% for each target analyte.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 80-120% for any analyte, report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (NOTE: if internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required). (2) Concentration level must alternate between low and high concentration standards (equivalent to second and fourth levels in calibration curve). (3) Must contain all single-component pesticides.	No	NA	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Renalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing	Report non-conforming compounds (%D >20) and associated samples in laboratory narrative.

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(4) Multi-component analytes must be verified with a one-point standard within 12 hours of being detected in a sample. (5) %D must be ≤20 for each target analyte. (6) Verify that all analytes fall within retention time windows. (7) Area count of internal standard in continuing calibration must be within ±50% of the average area count in the associated initial calibration.			calibration exhibited high response and associated pesticides were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
						laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all single-component pesticides.[1] (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable LCS results, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all single-component pesticides.[1]	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. 2) If recovery is outside of 40-140% for any

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for target analytes. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			(2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	analyte or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable LCS results, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all single-component pesticides.[1] (5) Percent recoveries between 30 – 150%. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 2 surrogates, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: TCMX and DCB (2) Percent recoveries must be between 30-150% for both surrogates on both columns.	Yes (report surrogate recoveries from both columns)	Recovery <10%; affects all nondetect results in affected sample.	If the same surrogate is outside of limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and pesticides were detected in the sample. NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., < 10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	(1) Report recoveries outside of 30-150% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Internal Standards (optional)	Laboratory Analytical Accuracy and Method Accuracy in	(1) Minimum of 1. Recommended internal standard: DCB (2) Area counts in samples must be between 50 – 200% of the area counts	No	Recovery <20%; affects all nondetect results quantitated using	If internal standard is outside of limits, reanalyze sample unless chromatographic	(1) Report nonconformances in laboratory narrative. Include actual recovery

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
	Sample Matrix	in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.		affected internal standard in associated sample.	interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of pesticides under most circumstances. Regardless if peak area or peak height is used, the same method used for quantitation of samples must also be used for	NA	If RPD >100 for single-component pesticides, reject positive result for affected pesticide.	If the RPD between the dual column results is >100 for single-component pesticides or >500 for multi-component pesticides,	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		calibration standards. (2) The laboratory must use the average calibration factor, response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each single-component pesticide. (3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[4]		If RPD >500 for multi-component pesticide, reject positive result for affected pesticide.	reanalyze the sample on dilution. Both analyses must be reported. Alternatively, additional sample cleanup techniques may be warranted.	exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. **NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must employ a cleanup technique to reduce the presence of sulfur interference.** (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not

Table V B-1: Specific QC Requirements and Performance Standards for Chlorinated Pesticides (SW-846 8081B) Using WSC-CAM-V B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		standards. (4) Refer to Appendix V B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.				preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (4) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

[1]Refer to Section 1.6 for guidance regarding the inclusion of multi-component pesticides in LCSs and MS/MSDs.
[2]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.
[3]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.
[4]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

ULTRA SCIENTIFIC
Analytical Solutions

Appendix B

Certificate of Analysis

OC-05077-80

Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 1 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

This Certified Reference Material (RM) was manufactured and verified in accordance with ULTRA's ISO 9001 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
acenaphthene	000083-32-9	29697-41	1002 ± 5 µg/mL
acenaphthylene	000208-96-8	ER030707-01	1000 ± 5 µg/mL
anthracene	000120-12-7	33383-91	1002 ± 5 µg/mL
benz[a]anthracene	000056-55-3	ER121707-01	1002 ± 5 µg/mL
benzo[b]fluoranthene	000205-99-2	ER022008-02	1002 ± 5 µg/mL
benzo[k]fluoranthene	000207-08-9	34750-27	1002 ± 5 µg/mL
benzo[ghi]perylene	000191-24-2	ER041205-01	1002 ± 5 µg/mL
benzo[a]pyrene	000050-32-8	ER050707-01	1002 ± 5 µg/mL
carbazole	000086-74-8	CEE-III-129	1002 ± 5 µg/mL
chrysene	000218-01-9	ER081006-02	1002 ± 5 µg/mL
dibenz[a,h]anthracene	000053-70-3	ER091206-01	1002 ± 5 µg/mL
fluoranthene	000206-44-0	LB50809	1000 ± 5 µg/mL
fluorene	000086-73-7	04003CK	1002 ± 5 µg/mL
indeno[1,2,3-cd]pyrene	000193-39-5	ER082107-02	1002 ± 5 µg/mL
naphthalene	000091-20-3	14205KB	1001 ± 5 µg/mL
phenanthrene	000085-01-8	07419ME	1002 ± 5 µg/mL
pyrene	000129-00-0	01216PE	1002 ± 5 µg/mL
azobenzene	000103-33-3	04606DF	1003 ± 5 µg/mL
4-chloroaniline	000106-47-8	01310MW	1001 ± 5 µg/mL
2-chloronaphthalene	000091-58-7	FIE01	1001 ± 5 µg/mL

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

ACCREDITED

ISO 17025 Accredited A2LA Cert. No. 0851-01

ISO 9001:2000 Registered TUV USA, Inc. Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 295-2330
www.ultrasci.com

William J. Leary
Quality Assurance Manager

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 2 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

Analyte	CAS#	Analyte Lot	True Value
4-chloro-3-methylphenol	000059-50-7	061657	1000 ± 5 µg/mL
dibenzofuran	000132-64-9	480477	1001 ± 5 µg/mL
1,4-dichlorobenzene	000106-46-7	22628EB	1003 ± 5 µg/mL
2,4-dichlorophenol	000120-83-2	05729EZ	1001 ± 5 µg/mL
2-methyl-4,6-dinitrophenol	000534-52-1	168-60A	1003 ± 5 µg/mL
2,4-dinitrophenol	000051-28-5	12310CU	1000 ± 5 µg/mL
2,4-dinitrotoluene	000121-14-2	18219TA	1000 ± 5 µg/mL
2,6-dinitrotoluene	000606-20-2	08328CR	1003 ± 5 µg/mL
hexachlorobenzene	000118-74-1	02927MC	1002 ± 5 µg/mL
hexachloroethane	000067-72-1	12604HB	1004 ± 5 µg/mL
pentachlorophenol	000087-86-5	06324ED	1002 ± 5 µg/mL
2-nitrophenol	000088-75-5	N960045	1003 ± 5 µg/mL
4-nitrophenol	000100-02-7	LO1OL	1003 ± 5 µg/mL
2-nitroaniline	000088-74-4	13201TU	1004 ± 5 µg/mL
3-nitroaniline	000099-09-2	03020DL	1002 ± 5 µg/mL
4-nitroaniline	000100-01-6	3926DK	1002 ± 5 µg/mL
2,4,5-trichlorophenol	000095-95-4	07521CO	1004 ± 5 µg/mL
2,4,6-trichlorophenol	000088-06-2	07509ME	1004 ± 5 µg/mL
bis(2-chloroethyl) ether	000111-44-4	BC-013187	1004 ± 5 µg/mL
bis(2-chloroethoxy)methane	000111-91-1	381-90A	1002 ± 5 µg/mL
bis(2-ethylhexyl) phthalate	000117-81-7	D-23230	1002 ± 5 µg/mL
4-bromophenyl phenyl ether	000101-55-3	05819CO	1002 ± 5 µg/mL
butyl benzyl phthalate	000085-68-7	05613ED	1003 ± 5 µg/mL
4-chlorophenyl phenyl ether	007005-72-3	28314CA	1004 ± 5 µg/mL

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

WASHED

A2LA ACCREDITED

ISO 17025 Accredited A2LA Cert. No. 0851-01

ISO 9001:2000 Registered TUV USA, Inc. Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 295-2330
www.ultrasci.com

William J. Leary
Quality Assurance Manager

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product Number: SVM-8270 **Page:** 3 of 3

Lot Number: CG-0219 **Lot Issue Date:** Jan-2010 **Expiration Date:** Feb-2011

Analyte	CAS#	Analyte Lot	True Value
2-chlorophenol	000095-57-8	09106BQ	1000 ± 5 µg/mL
di-n-butyl phthalate	000084-74-2	AC-011687	1001 ± 5 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	1003 ± 5 µg/mL
1,3-dichlorobenzene	000541-73-1	05902LZ	1003 ± 5 µg/mL
diethyl phthalate	000084-66-2	09615KD	1001 ± 5 µg/mL
2,4-dimethylphenol	000105-67-9	14822JF	1001 ± 5 µg/mL
dimethyl phthalate	000131-11-3	D-44220	1000 ± 5 µg/mL
di-n-octyl phthalate	000117-84-0	0001418030	1000 ± 5 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	1002 ± 5 µg/mL
hexachlorocyclopentadiene	000077-47-4	03907AU	1000 ± 5 µg/mL
isophorone	000078-59-1	10830MQ	1002 ± 5 µg/mL
2-methylnaphthalene	000091-57-6	15416DA	1002 ± 5 µg/mL
nitrobenzene	000098-95-3	DI-10604DI	1001 ± 5 µg/mL
N-nitrosodimethylamine	000062-75-9	12070JS	1002 ± 5 µg/mL
N-nitrosodi-n-propylamine	000621-64-7	FIM01	1004 ± 5 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	1002 ± 5 µg/mL
o-cresol	000095-48-7	09416AA	1001 ± 5 µg/mL
p-cresol	000106-44-5	09410PI	1003 ± 5 µg/mL
bis(2-chloroisopropyl) ether	000108-60-1	PR-16041	1003 ± 5 µg/mL
phenol	000108-95-2	1373574	1004 ± 5 µg/mL

Matrix: methylene chloride/benzene (3:1)

ULTRA uses balances calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001, and calibrated Class A glassware in the manufacturing of these standards.

MASTER

A2LA ACCREDITED

ISO 17025 Accredited A2LA Cert. No. 0851-01

ISO 9001:2000 Registered TUV USA, Inc. Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 295-2330
www.ultrasci.com

William J. Leary
Quality Assurance Manager

9K29100,101

Certificate of Analysis

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 563626 **Lot No.:** A066327
Description : Custom Appendix IX Mix #2
Expiration Date[1]**:** September 2010 **Storage:** Freezer
Handling: Sonicate ampule 15 minutes prior to opening

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	% Uncertainty [4] (95% C.L.; K=2)
1	1,4-Dioxane	123-91-1	99%	1,000.000 ug/ml	+/-0.64 %
2	Ethyl methacrylate	97-63-2	99%	1,000.000 ug/ml	+/-0.64 %
3	Methyl methanesulfonate	66-27-3	99%	1,000.000 ug/ml	+/-0.64 %
4	Ethyl methanesulfonate	62-50-0	99%	1,000.000 ug/ml	+/-0.64 %
5	Benzaldehyde	100-52-7	99%	1,000.000 ug/ml	+/-0.64 %
6	Pentachloroethane	76-01-7	98%	1,000.090 ug/ml	+/-0.64 %
7	Acetophenone	98-86-2	99%	1,000.000 ug/ml	+/-0.64 %
8	2,6-Dichlorophenol	87-65-0	99%	1,000.000 ug/ml	+/-0.64 %
9	Hexachloropropene	1888-71-7	99%	1,000.000 ug/ml	+/-0.64 %
10	epsilon-Caprolactam	105-60-2	99%	1,000.000 ug/ml	+/-0.64 %
11	Isosafrole (cis & trans)	120-58-1	99%	1,000.000 ug/ml	+/-0.64 %
12	1,2,4,5-Tetrachlorobenzene	95-94-3	99%	1,000.000 ug/ml	+/-0.64 %
13	Safrole	94-59-7	99%	1,000.000 ug/ml	+/-0.64 %
14	Biphenyl	92-52-4	99%	1,000.000 ug/ml	+/-0.64 %
15	1-Chloronaphthalene	90-13-1	86%	1,000.180 ug/ml	+/-0.64 %
16	Diphenyl ether	101-84-8	99%	1,000.000 ug/ml	+/-0.64 %
17	1,4-Naphthoquinone	130-15-4	99%	1,000.000 ug/ml	+/-0.64 %
18	Pentachlorobenzene	608-93-5	99%	1,000.000 ug/ml	+/-0.64 %
19	Diallate (cis and trans)	2303-16-4	99%	1,000.000 ug/ml	+/-0.64 %
20	Phenacetin	62-44-2	99%	1,000.000 ug/ml	+/-0.64 %
21	Atrazine	1912-24-9	99%	1,000.000 ug/ml	+/-0.64 %
22	Pentachloronitrobenzene (quintozene)	82-68-8	99%	1,000.000 ug/ml	+/-0.64 %
23	Pronamide (Propyzamide)	23950-58-5	98%	1,000.090 ug/ml	+/-0.64 %
24	4-Nitroquinoline-N-oxide	56-57-5	----%	1,000.000 ug/ml	+/-0.64 %
25	Isodrin	465-73-6	98%	1,000.090 ug/ml	+/-0.64 %
26	Aramite	140-57-8	----%	1,000.000 ug/ml	+/-0.64 %
27	Chlorobenzilate	510-15-6	99%	1,000.000 ug/ml	+/-0.64 %
28	Kepone	143-50-0	99%	1,000.000 ug/ml	+/-0.64 %
29	7,12-Dimethylbenz(a)anthracene	57-97-6	99%	1,000.000 ug/ml	+/-0.64 %
30	3-Methylcholanthrene	56-49-5	99%	1,000.000 ug/ml	+/-0.64 %
31	Dibenz(a,j)acridine	224-42-0	99%	1,000.000 ug/ml	+/-0.64 %

9K24098,99

Certificate of Analysis

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31850 **Lot No.:** A070659

Description : 8270 MegaMix

Expiration Date[1]**:** April 2011 **Storage:** Freezer

Handling: Sonicate ampule 15 minutes prior to opening

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	% Uncertainty [4] (95% C.L.; K=2)
1	N-Nitrosodimethylamine	62-75-9	99%	1,000.000 ug/ml	+/-0.60 %
2	Pyridine	110-86-1	99%	1,000.000 ug/ml	+/-0.60 %
3	Aniline	62-53-3	99%	1,000.000 ug/ml	+/-0.60 %
4	Phenol	108-95-2	99%	1,000.000 ug/ml	+/-0.60 %
5	Bis(2-chloroethyl)ether	111-44-4	99%	1,000.000 ug/ml	+/-0.60 %
6	2-Chlorophenol	95-57-8	99%	1,000.000 ug/ml	+/-0.60 %
7	1,3-Dichlorobenzene	541-73-1	99%	1,000.000 ug/ml	+/-0.60 %
8	1,4-Dichlorobenzene	106-46-7	99%	1,000.000 ug/ml	+/-0.60 %
9	Benzyl alcohol	100-51-6	99%	1,000.000 ug/ml	+/-0.60 %
10	1,2-Dichlorobenzene	95-50-1	99%	1,000.000 ug/ml	+/-0.60 %
11	2-Methylphenol (o-cresol)	95-48-7	99%	1,000.000 ug/ml	+/-0.60 %
12	Bis(2-chloroisopropyl)ether	108-60-1	99%	1,000.000 ug/ml	+/-0.60 %
13	4-Methylphenol (p-cresol)	106-44-5	99%	500.000 ug/ml	+/-0.60 %
14	3-Methylphenol (m-cresol)	108-39-4	99%	500.000 ug/ml	+/-0.60 %
15	N-Nitroso-di-n-propylamine	621-64-7	99%	1,000.000 ug/ml	+/-0.60 %
16	Hexachloroethane	67-72-1	99%	1,000.000 ug/ml	+/-0.60 %
17	Nitrobenzene	98-95-3	99%	1,000.000 ug/ml	+/-0.60 %
18	Isophorone	78-59-1	99%	1,000.000 ug/ml	+/-0.60 %
19	2-Nitrophenol	88-75-5	99%	1,000.000 ug/ml	+/-0.60 %
20	2,4-Dimethylphenol	105-67-9	99%	1,000.000 ug/ml	+/-0.60 %
21	Bis(2-chloroethoxy)methane	111-91-1	99%	1,000.000 ug/ml	+/-0.60 %
22	2,4-Dichlorophenol	120-83-2	99%	1,000.000 ug/ml	+/-0.60 %
23	1,2,4-Trichlorobenzene	120-82-1	97%	999.973 ug/ml	+/-0.60 %
24	4-Chloroaniline	106-47-8	99%	1,000.000 ug/ml	+/-0.60 %
25	Naphthalene	91-20-3	99%	1,000.000 ug/ml	+/-0.60 %
26	Hexachlorobutadiene	87-68-3	98%	1,000.029 ug/ml	+/-0.60 %
27	4-Chloro-3-methylphenol	59-50-7	99%	1,000.000 ug/ml	+/-0.60 %
28	2-Methylnaphthalene	91-57-6	97%	1,000.070 ug/ml	+/-0.60 %
29	1-Methylnaphthalene	90-12-0	99%	1,000.000 ug/ml	+/-0.60 %
30	Hexachlorocyclopentadiene	77-47-4	99%	1,000.000 ug/ml	+/-0.60 %
31	2,4,6-Trichlorophenol	88-06-2	99%	1,000.000 ug/ml	+/-0.60 %
32	2,4,5-Trichlorophenol	95-95-4	99%	1,000.000 ug/ml	+/-0.60 %
33	2-Chloronaphthalene	91-58-7	99%	1,000.000 ug/ml	+/-0.60 %
34	2-Nitroaniline	88-74-4	99%	1,000.000 ug/ml	+/-0.58 %
35	1,4-Dinitrobenzene	100-25-4	99%	1,000.000 ug/ml	+/-0.60 %
36	Acenaphthylene	208-96-8	99%	1,000.000 ug/ml	+/-0.60 %
37	1,3-Dinitrobenzene	99-65-0	99%	1,000.000 ug/ml	+/-0.60 %
38	Dimethylphthalate	131-11-3	99%	1,000.000 ug/ml	+/-0.60 %
39	2,6-Dinitrotoluene	606-20-2	99%	1,000.000 ug/ml	+/-0.60 %
40	1,2-Dinitrobenzene	528-29-0	99%	1,000.000 ug/ml	+/-0.60 %
41	Acenaphthene	83-32-9	99%	1,000.000 ug/ml	+/-0.60 %
42	3-Nitroaniline	99-09-2	99%	1,000.000 ug/ml	+/-0.58 %
43	2,4-Dinitrophenol	51-28-5	97%	1,000.009 ug/ml	+/-0.60 %
44	Dibenzofuran	132-64-9	99%	1,000.000 ug/ml	+/-0.60 %
45	2,4-Dinitrotoluene	121-14-2	99%	1,000.000 ug/ml	+/-0.60 %
46	4-Nitrophenol	100-02-7	99%	1,000.000 ug/ml	+/-0.60 %

9K24095, 97

Certificate of Analysis

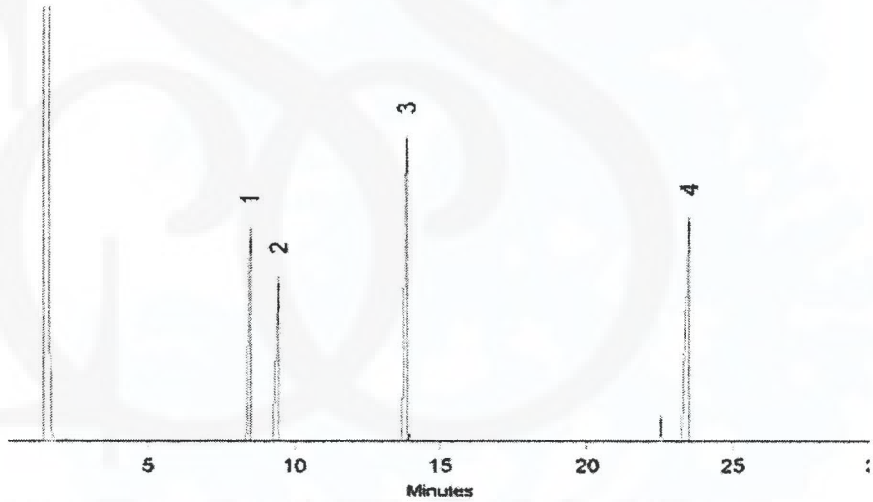
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31082 **Lot No.:** A062485
Description : B/N Surrogate Standard Mix (3/90)
Expiration Date[1]**:** September 2011 **Storage:** Refrigerate

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	1,2-Dichlorobenzene-d4	2199-69-1	99%	5,000.000 ug/ml	+/-0.03 %
2	Nitrobenzene-d5	4165-60-0	99%	5,000.000 ug/ml	+/-0.03 %
3	2-Fluorobiphenyl	321-60-8	99%	5,000.000 ug/ml	+/-0.03 %
4	p-Terphenyl-d14	1718-51-0	97%	4,999.962 ug/ml	+/-0.03 %
Solvent:	Methylene Chloride	75-09-2	99%		

Column: 30m x .25mm x .5um
Rtx-5 (cat.#10238)
Carrier Gas: hydrogen @ 40cm/sec.
Temp. Program: 100°C to 330°C
@ 20°C/min. (hold 5 min.)
Inj. Temp: 250°C
Det. Temp: 300°C
Det. Type: FID



Diane Shaffer
Diane Shaffer - QA Analyst

Balance 1128353505

Manufactured under Restek's ISO 9001:2000
Registered Quality System
Certificate #FM80397

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, OSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Tech Tip:

Due to the limited solubility of p-terphenyl-d14 in methanol, we do not recommend that this mixture be diluted in methanol.

9K24094, 96

Certificate of Analysis

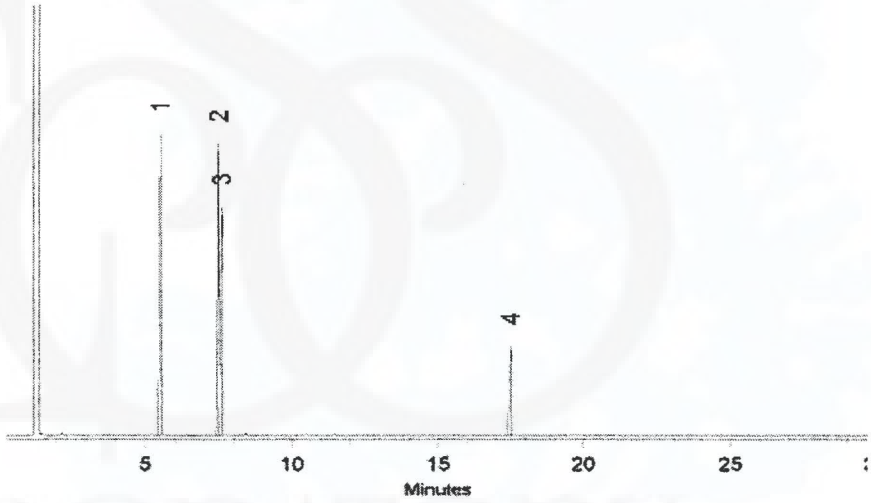
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 31083 **Lot No.:** A061440
Description : Acid Surrogate Standard Mix (3/90)
Expiration Date[1]**:** January 2013 **Storage:** Refrigerate

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	2-Fluorophenol	367-12-4	99%	7,500.100 ug/ml	+/-0.03 %
2	Phenol-d6	13127-88-3	99%	7,502.200 ug/ml	+/-0.03 %
3	2-Chlorophenol-d4	93951-73-6	99%	7,500.900 ug/ml	+/-0.03 %
4	2,4,6-Tribromophenol	118-79-6	99%	7,500.000 ug/ml	+/-0.03 %
Solvent:	Methanol	67-56-1	99%		

Column: 30m x .25mm x .25um Rtx-5 (cat.#10223)
Carrier Gas: hydrogen @ 40cm/sec.
Temp. Program: 40°C (hold 2 min.) to 330°C @ 10°C/min. (hold 10 min.)
Inj. Temp: 250°C
Det. Temp: 300°C
Det. Type: FID



Diane Shaffer
Diane Shaffer - QA Analyst

Balance 1128360905

Manufactured under Restek's ISO 9001:2000 Registered Quality System Certificate #FM80397

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

UK2402,73,74,76-93

125 Market Street
New Haven, CT 06513
USA

AccuStandard, Inc.

Tel (203)786-5290
Fax (203)786-5287
Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: DRH-006S
DESCRIPTION: Proposed DEP(MA) - PAH Mix
LOT: 209101293
SOLVENT: CH2Cl2

EXPIRATION: Oct 29, 2019

See reverse for additional certification information.

Component	CAS #	Purity % (GC/MS)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acenaphthene	83-32-9	100	1005	1005
Acenaphthylene	208-96-8	99.1	1000	991
Anthracene	120-12-7	100	1001	1001
Benz(a)anthracene	56-55-3	99.6	1000	996
Benzo(a)pyrene	50-32-8	98.9	1002	991
Benzo(b)fluoranthene	205-99-2	100	1004	1004
Benzo(g,h,i)perylene	191-24-2	99.6	1002	998
Benzo(k)fluoranthene	207-08-9	99.5	1000	995
Chrysene	218-01-9	100	1005	1005
Dibenz(a,h)anthracene	53-70-3	99.5	1006	1001
Fluoranthene	206-44-0	99.9	1001	1000
Fluorene	86-73-7	98.1	1007	988
Indeno(1,2,3-cd)pyrene	193-39-5	98.2	1006	988
2-Methylnaphthalene	91-57-6	100	1004	1004
Naphthalene	91-20-3	99.8	1004	1002
Phenanthrene	85-01-8	98.9	1002	991
Pyrene	129-00-0	98.6	1006	992

17 Components

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±2% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc. or -01, -02, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

Certificate of Analysis

9K24069

DESCRIPTION: Pentachlorophenol

CATALOG NO.: 48692 MFG DATE: Sep-2008

LOT NO.: LB61534 EXPIRATION DATE: Sep-2011

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3) CONCENTRATION		STD DEV	SUPELCO LOT NO
PENTACHLOROPHENOL	87-86-5	98.9	500.5	507.0	+/-	11.92	LB37346

(1) Determined by capillary GC-FID, unless otherwise noted.

(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

TOPLEVEL PARAMETERS

(GC/MS-1)

Method Information For: C:\HPCHEM\1\METHODS\SV1NB.M

Method Sections To Run:

Appendix C

Save Copy of Method With Data
Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
THIS METHOD IS USED TO ANALYZE FOR BASE-NEUTRAL AND ACID EXTRACTABLE COMPOUNDS USING A CAPILLARY COLUMN. THIS METHOD IS USING TCL SOW 88 LIST. 625 AND 8270 ARE APPLICABLE 02/95.

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information

Inlet : GC
Tune File : DFTPP.U
Acquisition Mode : Scan

MS Information

Solvent Delay : 0.75 min

EM Absolute : False
EMV Offset : 0.0
Resulting Voltage : 1835.3

[Scan Parameters]

Low Mass : 35
High Mass : 500
Threshold : 500
Sampling # : 2 A/D Samples 4
Plot 1 low mass : 35
Plot 1 high mass: 500

[Real Time Plot Parameters]

Time Window : 25 min
Iconize Real Time Display : False
Plot 1 type : Extracted ion
Scale minimum : 0
Scale maximum : 400000
Plot 2 type : No plot

GC Inlet Information

[Inlet A Temperature Program Information]

Oven Track : Off
Inlet A Off

[Ir B Temperature Program Information]

Oven Track : Off
Initial Temp. : 275 C
Initial Time : 480.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Inlet A Pressure Program Information]

Constant Flow : On 7.1 psi at 40 C
Pressure Units : psi

[Inlet A Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 8.0 psi
Flow : 1.1 ml/min
Linear velocity : 37.5 cm/sec
Split flow : 1 ml/min
Split ratio : 0.9

[Inlet B Pressure Program Information]

Constant Flow : Off
Initial Pres. : 7.1 psi
Initial Time : 0.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	99.00	36.0	0.70
2	99.00	7.1	0.00
3	0		

Total Program Time: 1.28 min
Pressure Units : psi

[Inlet B Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 7.1 psi
Flow : 0.9 ml/min
Linear velocity : 33.9 cm/sec
Split flow : 1 ml/min
Split ratio : 1.2

[Auxiliary Channel C Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)

1 0
Total Program Time: 480.00 min

[Auxiliary Channel D Information]

Cor t:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level Rate(psi/min) Final Pres.(psi) Final Time (min)
1 0
Total Program Time: 480.00 min

[Auxiliary Channel E Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level Rate(psi/min) Final Pres.(psi) Final Time (min)
1 0
Total Program Time: 480.00 min

[Auxiliary Channel F Information]

Comment:

Pressure Program:
In l Pres. : 0.0 psi
Ini l Time : 480.00 min

Level Rate(psi/min) Final Pres.(psi) Final Time (min)
1 0
Total Program Time: 480.00 min

GC Temperature Information
-- ---------- ----------

[GC Zone Temperatures]

Inj. A : 250 C Off
Inj. B : 275 C
Det. A : 50 C Off
Det. B : 300 C
Aux. : 280 C Off

[Oven Parameters]

Oven Equib Time : 0.20 min
Oven Max : 325 C
Oven : On
Cryo : Off
Ambient : 25 C
Cryo Blast : Off

[O Program]

Initial Temp. : 60 C
Initial Time : 2.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	35.00	130	0.00
2	12.00	325	3.75
3	0.00		

Next Run Time : 24.00 min

Injector Information
--- ---

Injection Source : Auto
Injection Location : Front

Sample Washes : 1
Sample Pumps : 4
Sample Volume : 1 stop(s)
Viscosity Delay : 1 sec
Solvent A Washes : 4
Solvent B Washes : 2
On Column : No

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	On	0.15	0.00
B	On	0.15	0.00

END OF ACQUISITION PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\SV1NB.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: Yes
Printer: No
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
C: ABASE\NBS75K.L	0
C:\DATABASE\PAH.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Ger. te Report During Run Method: No

Quantitative Report Settings

Report Type: Detailed

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

ELEMENT ID: 0603035(SOIL) 0603036(AQUEOUS)
Calibration Last Updated: Fri Apr 07 09:13:18 2006

Reference Window: 5.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD)

Ret. Time 3.86 min., Extract & Integrate from 3.36 to 4.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	115.00	58.70	30.0	*** METH DEFAULT ***
Q2	150.00	289.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response
5		40.000	442167
10		40.000	372682
50		40.000	381573
80		40.000	339990
120		40.000	337070
160		40.000	306591
200		40.000	356509
25		40.000	437292
CC		40.000	499862

Qualifier Peak Analysis ON ISTD conc: 40.000 ng/uL
Curve Fit: Avg. RF

---

2) N-Nitrosodimethylamine ( )

Ret. Time 0.85 min., Extract & Integrate from 0.35 to 1.35 min.

Sig		Rel Resp.	Pct. Unc.(abs)	Integration
Tg	74.05			*** METH DEFAULT ***
Q1	42.10	53.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response

TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\SV2KC.M

Method Sections To Run:

(ave Copy of Method With Data
(, Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Appendix C

Method Comments:
8270

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS (MS2)

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

7673 Injector

ront Injector:
No parameters specified

Back Injector:

Sample Washes	2
Sample Pumps	2
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	1 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	275 C
Detector A:	Off	50 C
Detector B:	On	300 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.10 minutes
Oven Max:	350 C
ven State:	On
ryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	60 C
Initial Time:	1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	35.0	130	0.00
2(A)	12.0	325	4.75
3(B)	0.0	50	1.00

Next Run Time: 24.00 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	0.0 psi
Initial Time:	650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length:	30.00 m
Column Diameter:	0.530 mm
Gas:	He
Vacuum Compensation:	Off

Inlet B:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	7.0 psi
Initial Time:	0.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	99.00	36.0	0.50
2(A)	99.00	7.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 1.09 minutes

Column Length:	30.00 m
Column Diameter:	0.250 mm
Gas:	He
Vacuum Compensation:	On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\PAH2DR.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Lib. y to Search	Minimum Quality
nbs75k.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: Yes
Printer: Yes
File: No

Generate Report During Run Method: No

LL PAH ELEMENT ID 0604005
Calibration Last Updated: Thu Apr 06 15:54:57 2006

Reference Window: 0.50 Minutes
Non-Reference Window: 0.50 Minutes
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD TR)

Ret. Time 4.21 min., Extract & Integrate from 4.06 to 4.36 min.

Signal		Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	115.00	51.80	30.0	*** METH DEFAULT ***
Q2	150.00	153.70	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response
0.2		2.000	5361
0.4		2.000	5216
1.0		2.000	4812
2.0		2.000	10368
5.0		2.000	5671
8.0		2.000	5719
CC		2.000	6400
0.1		2.000	5740

Qualifier Peak Analysis ON ISTD conc: 2.000 ng/uL
Curve Fit: Avg. RF

---

2) 1,2 Dichlorobenzene-d4(SURR) ( )

Ret Time 4.41 min., Extract & Integrate from 3.91 to 4.91 min.

Signal		Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	150.00	173.80	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response
0.2		0.200	588
0.4		0.400	1046
1.0		1.000	2405
2.0		2.000	10416
5.0		5.000	13918
8.0		8.000	23368
CC		1.000	-1
0.1		0.100	384

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Naphthalene-d8 (ISTD TR)

Ret. Time 5.67 min., Extract & Integrate from 5.52 to 5.82 min.

Signal		Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	136.00			*** METH DEFAULT ***
Q1	68.00	7.10	30.0	*** METH DEFAULT ***

Lvl	Conc (ng/uL)	Response
0.2	2.000	13794
0.4	2.000	14266
1.0	2.000	13382

SVOA

ESS LABORATORY MS5 RUN LOG

Appendix D

DATE	6/25/2014	**Work orders reported:**			
ANALYST	IBM				
METHOD	SV5CV	**MATRIX**	**SEQ ID**	**CAL ID**	
Internal Std:	4E06161	SOIL	CXF0327	1406003	
Tune Method:	DFTPP	AQ		1406004	
METHOD (2)	SV5CV	**MATRIX**	**SEQ ID**	**CAL ID**	
Internal Std:	4E06161	SOIL	CXF0333	1406003	
Tune Method:	DFTPP	AQ	CXF0352	1406004	

VIAL #	SAMPLE ID	METHOD	FILE ID	M	STD ID/COMMENTS	BATCH ID	MATRIX	RW	RPT	IMP BY
1	CXF0327-TUN1	DFTPP	SV549316	1	4F03143			IBM	X	
2	CXF0327-CCV1	SV5A	SV549317	1	4F23093			IBM	X	
3	1406538-01	SV5A	SV549318	1	0.05/0.5,0.05/0.5=100X	CF42313	S	IBM	X	X
4	1406538-02	SV5A	SV549319	1	0.05/0.5,0.05/0.5=100X	CF42313	S	IBM	X	X
5	1406538-03	SV5A	SV549320	1	0.05/0.5,0.05/0.5=100X	CF42313	S	IBM	X	X
6	1406538-06	SV5A	SV549321	1	0.05/0.5,0.05/0.5=100X	CF42313	S	IBM	X	X
IDLE										
1	CXF0333-TUN1	DFTPP	SV549322	1	4F03143			IBM	X	
2	CXF0333-CCV1	SV5A	SV549323	1	4F23093			IBM	X	
3	CF42420-BLK1	SV5A	SV549324	1		CF42420	A/S	IBM	X	
4	CF42420-BS1	SV5A	SV549325	1		CF42420	A/S	IBM	X	
5	CF42420-BSD1	SV5A	SV549326	1		CF42420	A/S	IBM	X	
6	CF42424-BLK2	SV5A	SV549327	1	TCLP	CF42424	S	IBM	X	X
7	1406522-01	SV5A	SV549328	1	TCLP	CF42424	S	IBM	X	X
8	CF42424-MS1	SV5A	SV549329	1	TCLP	CF42424	S	IBM	X	X
9	1406519-01	SV5A	SV549330	1		CF42420	A	IBM	X	X
10	CF42517-BLK1	SV5A	SV549331	1		CF42517	S	IBM	X	
11	CF42517-BS1	SV5A	SV549332	1		CF42517	S	IBM	X	
12	CF42517-BSD1	SV5A	SV549333	1		CF42517	S	IBM	X	
13	CF42420-MS1	SV5A	SV549334	1		CF42420	A	IBM	X	X
14	1406519-02	SV5A	SV549335	1		CF42420	A	IBM	X	X
15	1406519-03	SV5A	SV549336	1		CF42420	A	IBM	X	X
16	1406519-04	SV5A	SV549337	1		CF42420	A	IBM	X	X
17	1406519-05	SV5A	SV549338	1		CF42420	A	IBM	X	X
18	1406538-07	SV5A	SV549339	1		CF42517	S	IBM	X	X
19	1406582-01	SV5A	SV549340	1		CF42517	S	IBM	X	X
20	CF42517-MS1	SV5A	SV549341	1		CF42517	S	IBM	X	X
21	CF42517-MSD1	SV5A	SV549342	1		CF42517	S	IBM	X	X

QUADS:	**BENZ ACID, 24-DNP, PCP**
SCV:	**HCCPD 68%, BENZIDINE 50%**
CCAL0327:	**NNDMA 60%, PYRIDINE 67%, HCCPD 78%**
CCAL0333:	**NNDMA 61%, PYRIDINE 70%, HCCPD 75%, BENZIDINE 75%**
Comments:	

Review (includes a check and review of manual integrations)

ESS LABORATORY
INSTRUMENT MAINTENANCE LOGBOOK

START DATE: 05/30/2012
REVIEWED BY / DATE:

DATE	INSTRUMENT	REASON FOR MAINTENANCE	SERVICE PERFORMED	COMMENTS	INIT.

60.0057

Table II B-1 Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
GC/MS Tunes with DFTPP	Inter-laboratory Consistency & Comparability	(1) Criteria for DFTPP listed in Table 3 of SW-846 8270D (the same criteria must be used for all analyses). (2) Every 12 hours prior to sample analysis. (3) DDT breakdown must be evaluated and must be <20%. (4) Pentachlorophenol and benzidine peak tailing must be evaluated. Peak tailing factor must be <2 for benzidine and pentachlorophenol. NOTE: Pentachlorophenol tailing must be evaluated when analyzing for acid SVOCs and benzidine tailing must be evaluated when analyzing for base-neutral SVOCs. NOTE: Tune must be performed in full scan mode for SIM analyses.	No	NA	Perform instrument/injection port maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing factor exceedances in laboratory narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used).	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if >10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. (3) If recalculated	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
		(3) Low standard must be ≤RL. (4) %RSD ≤20, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8270D for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples. (9) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (10) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards.			concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value[2], or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	(i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target analytes. (5) Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes(**) which must	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, including "difficult" analytes(**), report non-conforming compounds in laboratory narrative.

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		exhibit percent recoveries between 40-160%.				
Continuing Calibration	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) %D must be ≤20 for each target analyte. (5) Minimum RFs as per Table 4 of SW-846 8270D. (6) Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration standard.	No	RF <0.05; affects nondetect results for affected analyte in all samples analyzed under this continuing calibration.	(1) Recalibrate if >20% of all target analytes or >15% of analytes from a particular class (base-neutral or acid) exceed %D criteria. (2) If internal standard is outside of criteria, locate source of problem and reanalyze the continuing calibration. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D <40.	Report non-conforming compounds (%D >20 or minimum RF not met) and associated samples in laboratory narrative.
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL except for common laboratory contaminants (phthalates) which must be <5x the RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; reextract and reanalyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
						time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix-specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes(**) which must exhibit percent recoveries between 15-140% (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes(**), report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of

Table II B-1 Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
						holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target analytes. (4) Matrix-specific (e.g., water, soil). (5) Percent recoveries must be between 40-140% for the base-neutral compounds and between 30-130% for the acid compounds except for "difficult" analytes(**) which must exhibit percent recoveries between 15-140%. (6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140% for base-neutral compounds and >130% for acid compounds), reextraction is not required if affected compounds were not detected in associated samples.	(1) if sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any base-neutral compound or 30-130% for any acid compound, including "difficult" analytes(**) or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

Table II B-1 Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries between 40 -140% for the base-neutral compounds and between 30-130% for the acid compounds. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral surrogates and 3 acid surrogates, at retention times across GC run. Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol NOTE: For SIM analyses, surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs and use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in solid matrices must be between 30-130% for all surrogates. Percent recoveries in water matrices must be between 30-130% for base-neutral surrogates and 15-110% for acid surrogates.	Yes	Recovery <10%; affects all nondetect SVOC results associated with the surrogate compound class (base-neutral or acid) in affected sample.	If two or more surrogates for any one class (base-neutral or acid) are outside of limits or if any one surrogate recovers at <10%: (1) Re-extract the sample if surrogate recoveries are low. (2) Re-extract the sample if surrogate recoveries are high and associated SVOCs were detected in the sample. Re-extraction is not required if one of the following exceptions applies: (a) If surrogate recoveries are high and target	(1) Report recoveries outside of acceptance limits in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the

Table II B-1 Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
					analytes are not detected in sample, re-extraction is not required. (b) Re-extraction is not required if obvious interference present (e.g., UCM). If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%), re-analyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	holding time and yields acceptable surrogate recoveries, the laboratory must report results of both extractions. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 6 at retention times across GC run. NOTE: For SIM analyses, the number of internal standards used will be dependent on the analytes of interest. Internal standards must elute in close proximity to the analytes of interest. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If one or more internal standards are outside of limits, reanalyze sample unless obvious interference present (e.g., UCM). NOTE: If obvious interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is performed within holding

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
						time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each analyte. (3) The internal standard used for quantitation must be the one nearest the retention time of the subject analyte. (4) Results must be reported with 2 or more "significant figures" if ≥RL. If reporting values below the RL, report with 1 or more "significant figures".[3]	NA	NA	NA	NA

Table II B-1: Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350	Required Corrective Action	Required Analytical Response Action
Identification	NA	Refer to SW-846 8270D, Section 11.6.	NA	NA	NA	NA
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Refer to Section 3.3, TICs by GC/MS, for guidance. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (5) Refer to Appendix II B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (3) TICs will be evaluated at the discretion of the data user consistent with the guidelines presented in Appendix II B–3. (4) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (5) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the

Table II B-1. Specific QC Requirements and Performance Standards for SVOCs (SW-846 8270D) Using WSC-CAM-II B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
						laboratory narrative. (6) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

** Potentially "difficult" analytes include: 4-chloroaniline, 4-nitrophenol, phenol, 2,4-dinitrophenol

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

CPSC Phthalate Analysis:

Application: Samples that are prepared and extracted according to the SOP 50_CPSC_Phthalates are analyzed using this procedure. This procedure describes standards, instrument operating conditions, calculations and reporting.

Standards:

Internal Standard: Acenaphthene-D10 (Purchased fro Sigma Aldrich at 200ppm) 10ul of this standard is added to all samples and standards (10 ul for every ml) for a final concentration of 2ppm.

Dibutyl Phthalate ($C_{16}H_{22}O_4$, DBP), Single Component, CAS No. 84-74-2. Analytical grade

Di-(2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$, DEHP), Single Component, CAS No. 117-81-7. Analytical grade

Benzyl Butyl Phthalate ($C_{19}H_{20}O_4$, BBP), Single Component, CAS No. 85-68-7. Analytical grade

Di-n-octyl phthalate ($C_{24}H_{38}O_4$, DnOP), Single Component , CAS No. 117-84-0. Analytical grade

Diisononyl phthalate ($C_{26}H_{42}O_4$, DINP), Multi Component, CAS No. 28553-12-0/68515-48-0. Analytical grade

Diisodecyl phthalate ($C_{28}H_{46}O_4$, DIDP), Multi Component , CAS No. 26761-40-0/68515-49-1. Analytical grade

Calibration standards are prepared as follows:

Single component analytes: These analytes are in the 8270 Low Level Stock solution at 20ppm. Serial dilutions are made for the calibration standards at comcentrations of 0.05, 0.1,0.5, 1.0, 2.0, and 5.0ppm.

Multi Component analytes. A separate standard is prepared for each multi-component analyte at 10,000ppm(0.5g/50mls). These are diluted to create calibration standards at 1,5,10,50 and 100ppm.

Blank Spikes: Prepared by adding 0.5mls of the SVOA MS spiking solution. At a final volume of 50ml this will produce on column concentrations at 1ppm.

CRM (Certified Reference Material) : Use the second source standard from the SVOA 8270 method. Diluted the 20ppm stock standard (1:20) for the 1ppm CRM standard. Add the appropriate amount of IS.

GC-MS Operating Procedures and Quality Control Measures
A GC-MS system with an automatic injector is suggested for the sample analysis.

The following GC conditions are used (Table 1):

Table 1. GC Conditions Column	HP-5MS; 30m x .22 mm ID x 0.25 µm
Initial Flow Mode	1 ml/min, constant flow (He gas)
Injection Mode	Pulsed splitless
Injection Amount	1 µl
Injection Port Temp	275° C
Pulse Pressure & Time	35 psi, 0.5 min
Purge Flow & Time	20 ml/min, 2 min
Solvent Delay	5 minutes
Initial Oven Temp, Hold Time	50° C, 1 min
Ramp Temp 1, Plateau	30° C/min, 280° C
Ramp Temp 2, Plateau	15° C/min, 310° C
Final Hold Time	4 minutes

Samples are analyzed using the Select Ion Monitoring (SIM) program listed in Table 2. Scan for corresponding ions of each compound listed in a particular stage (e.g., set SIM Stage 3 to scan for 149, 167, 261, 279, 293, and 307 *m/z*). Note that retention times and optimal *m/z* scan values may differ between instruments. For many instruments it will be possible to program the detector to scan for the various ions in the different stages within a single run.

Table 2. SIM Settings **Retention Time (min)**	**Corresponding Ions (*m/z*)**	
Scan Stage 1:	*5 - 9.5 minutes*	
Acenaphthene-D10 (Internal Standard)	7.93 – 7.99	**188**
DBP	8.52 – 8.57	**149**, *167, 205, 223*
Scan Stage 2:	*9.5 - 10.8 minutes*	
BBP	9.84 – 9.93	*91.1,* ***149****, 206*
DEHP	10.42 – 10.49	***149****, 167, 279*
Scan Stage 3:	*10.8 – End*	
DnOP	11.15 – 11.24	***149****, 167, 261, 279*
DINP	10.90 – 12.1	***149****, 167, 293*
DIDP	11.20 – 13.00	***149****, 167, 307*

Analysis

1. Prepare at least four calibration standards for each of the six phthalates of interest (in the range of 0.5 to 10 µg/ml), along with one calibration blank (methylene chloride). Each calibration standard should have an internal standard concentration of 2 µg/ml.
2. Prior to analysis run the 8270 TUNE standard. Spectral ratios must be within criteria to ensure the MS is functioning properly.
3. Analyze standards and blank with the GC-MS. Qualitatively analyze the results to ensure proper retention times and no contamination.
4. Integrate the peak area from valley to valley (the time range listed in Table 2) for each standard. Compounds scanned in Stages 1 ,2 and 3 are integrated using their quantitative ions (in **bold**).
5. Construct a calibration curve from normalized phthalate signals. Normalization is performed by dividing the integrated phthalate signal area by the integrated internal standard signal area.
6. Analyze a CRM to ensure a proper calibration. The analyzed value should be within ±10% of the expected value. If not, prepare new standards and re-run calibration.
7. Analyze the QC standards and all samples. Analyze a CRM if time has passed since the last calibration check. Add the appropriate amount of IS to samples and QC.
8. Quantitate results. If the results are out of the calibration range, return to step 5 of the phthalate extraction method (perform another dilution to get results in calibration range).

Calculations and Results

Results can be reported as follows:

Percentage [Phthalate] = % Phthalate (w/w) = [(C x V x D) / (W x 1000)] x 100

Where

C = Concentration of phthalate in GC-MS sample (in µg/ml)

V = Total volume of THF and hexanes added from steps 2 and 3 of phthalate extraction method

D = Dilution factor from step 5 of phthalate extraction method

W = Weight of sample collected (in mg)

Repeat calculation for each phthalate present in sample

1,4-Dioxane Analysis using Isotope Dilution

Application: Samples are prepared and extracted in accordance with SW-846 methods 3510 or 3520 and analyzed using modified SW-846 method 8270 GC/MS-SIM. 1,4-Dioxane-d8 is added to the samples prior to extraction. This isotopically labeled compound functions both as internal standard and as surrogate for the analyte of interest and serves to correct the variability associated with extraction of the target analyte using the specified extraction procedure. Macro preparation methods cited above may be adjusted for micro quantities per SW846 Chapter Two providing QC acceptance criteria are met.

Standards:

Internal Standard / Surrogate: 1,4-Dioxane-d8 (purchased from Absolute Standards, Cat. # 92785, @10,000 ppm), diluted to 1000 ppm in DCM.

Procedure:

GC-MS Operating Procedures and Quality Control Measures
A GC-MS system with an automatic injector is suggested for the sample analysis.
The following GC conditions are used (Table 1):

Table 1. GC Conditions Column	HP-5MS; 30m x .22 mm ID x 0.25 µm
Initial Flow Mode	1 ml/min, constant flow (He gas)
Injection Mode	Pulsed splitless
Injection Amount	1 µl
Injection Port Temp	275° C
Pulse Pressure & Time	35 psi, 0.5 min
Purge Flow & Time	20 ml/min, 2 min
Solvent Delay	2 minutes
Initial Oven Temp, Hold Time	50° C, 1 min
Ramp Temp 1, Plateau	30° C/min, 280° C
Ramp Temp 2, Plateau	15° C/min, 310° C
Final Hold Time	4 minutes

Samples are analyzed using the Select Ion Monitoring (SIM) program listed in Table 2. Scan for corresponding ions of each compound listed in a particular stage (e.g., set SIM Stage 3 to scan for 149, 167, 261, 279, 293, and 307 *m/z*). Note that retention times and optimal *m/z* scan values may differ between instruments. For many instruments it will be possible to program the detector to scan for the various ions in the different stages within a single run.

Alternative Extraction Procedure: Micro-extraction by 3580A

35 mL of sample are placed into a 40 mL VOA vial and spiked with 17.5 uL of a 1000 ppm solution of 1,4 Dioxane-d8. For QC, use DI water. Spike blank spikes and matrix spike samples with 175 uL of a 100 ppm solution of 1,4-Dioxane in Acetone.

Add approximately 6 g of muffled Sodium Sulfate and exactly 3.5 mL of Methylene Chloride. Cap the vial tightly and shake vigorously for at least 2 minutes.
Centrifuge the samples first and then turn them upside down and let them settle for a few minutes (the centrifugation keeps the insoluble residue of Sodium Sulfate from caking together on the septum). Then, carefully remove 1.0 mL of the extract through the septum, using a syringe. Spike with regular 8270 IS solution at 20 ppm and analyze.

Appendix J: Method 522 solid phase extraction of 1,4-Dioxane

Equipment/Apparatus: Resprep 6ml EPA METHOD 521 cartridge catalogue #26032

Vacuum manifold with at least 6 positions for cartridges.

PTFE sample transfer tubing.

Vacuum pump with 1000-2000 ml water trap.

Amber Glass bottles for QC samples.

40ml sample vials, target vials.

O&G Glass funnels with glass wool and 3g or less anhydrous sodium sulfate.
Turbovap II concentration workstation: Concentration Tubes (Part# 45817) 200ml.

Procedure: Wet each cartridge bed using three solvents and the vacuum apparatus to pull solvents through each cartridge. Rinse with two cartridge volumes of Dichloromethane(DCM), then two volumes of Methanol, and finally two volumes DI water. Do not vacuum dry the cartridges, leave a small amount of DI water in the cartridge. Attach PTFE transfer tubing to the cartridges. Measure 500ml of DI water for each QC sample and 500ml of analytical sample into amber bottles. Spike the QC samples (Blank, Blank Spike, Blank Spike Dup) and samples with 2.5ul Dioxane surrogate. Spike 50ul Dioxane Matrix spike into appropriate samples.

Place the sample ends of the PTFE transfer tubes in the QC/Sample bottles. Turn on Vacuum pump to start extracting the samples. Empty the Vacuum pump water trap into the shake-out waste drum every so often to avoid sample water from getting into the pump. Once all the samples have been filtered through the cartridges, remove the transfer tubes from the cartridges and air dry using the vacuum for 1 minute. Remove any water from the manifold and use Kimtech wipes to dry the collection tips of the manifold. Label and place appropriate 40ml glass vials into their correct position in the vacuum manifold.

Add half the cartridge volume of DCM to each cartridge and allow the DCM to soak/elute. Once the DCM has eluted into the cartridge add another half volume of DCM. Briefly use the vacuum to initiate drop wise elution of the DCM (for less than 5 seconds). Add four more half volumes of DCM to each cartridge, for a total of three volumes (approximately 9 ml DCM). Use the vacuum pump to get the last of the DCM from the cartridges. Filter each sample through the O&G funnels into Turbovap concentration tubes and concentrate each sample to 0.5ml Final volume.

ESS Laboratory
Division of Thielsch Engineering
Cranston, RI

SOP NO. 30_6010
INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY
(SW 846 METHOD 6010C/ EPA METHOD 200.7/MCP-CAM IIIA)

APPROVED BY:

Operations Manager	Date 6/24/14
QA Manager	Date 6/23/14
Laboratory Director	Date 6/6/14

MASTER

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (SW 846 METHOD 6010C/ EPA METHOD 200.7/MCP-CAM IIIA)

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled argon plasma analysis (ICP) determines trace elements, including metals, in solution. All matrices, including ground water, aqueous samples, TCLP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis, with the possible exception of drinking water.

1.2 Elements for which Methods 6010C/200.7 are applicable are listed in Table 3. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Table 3 provide typical reporting limits for clean aqueous samples.

2.0 METHOD SUMMARY

2.1 Prior to analysis, samples must be solubilized or digested using EPA sample preparation methods 3005A or EPA 200.7 (SOP 30_3005 addresses these methods) for aqueous matrices and method 3050B (SOP 30_3050B) for solids.

2.2 When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis; however, it is ESS Laboratory policy to digest all non-potable water samples.

2.3 Method 6010C describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and a photosensitive device monitors the intensities of the lines. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 5.0 should also be recognized and appropriate corrections made.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been

performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_0001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 Material Safety Data Sheets for the chemicals used in the laboratory are kept on file in each department and are available for all employees to review.

3.4 The laboratory employee should review proper emergency response to spills or injury prior to attempting this procedure. Employees must know the location of spill kits, eyewashes, showers, and fire fighting equipment. Employees must also have knowledge of disaster evacuation routes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Prior to analysis, all aqueous samples are to be field preserved with nitric acid to a pH of less than two. If necessary, SDWA and CWA samples are to be adjusted with nitric acid prior to digestion to pH<2 and sample(s) then held for 24 hours until the pH has stabilized at pH<2, (applicable to EPA Method 2007, per EPA MUR 2012).

4.2 After digestion, samples are stored in specimen containers or Hot Block Tubes.

4.3 Hold time for aqueous preserved samples is 180 days from day of sampling for all metals except mercury. The hold time for samples to be analyzed for mercury is 28 days. Samples prepared and/or analyzed after this date are to be flagged as estimated values.

4.4 Prior to use, all glassware will be soaked in a 10% HNO_3 bath for at least 15 minutes and rinsed a minimum of three times with ICP solution (refer to SOP 30_0001). The HNO_3 bath is checked for contamination on a weekly basis and recorded in the batch log. When aqueous samples are digested by the Hot Block procedure plastic ware is used, so this step may be omitted.

4.5 All results for solid samples are to be corrected for a dry weight determination at 105°C.

4.6 For dissolved metals analysis, a non-preserved sample must be filtered through a 0.45 µm filter within 24 hours of collection and preserved to a pH of less than 2.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or

recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Computer correcting the raw data after monitoring and measuring the interfering element can compensate for spectral overlap. Unresolved overlap requires selection of an alternative wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

5.1.1 Analysts must verify the absence of spectral interferences from an element in a sample for which there is no instrument detection channel. Laboratory standard wavelengths are listed in Table 3.

5.1.2 Element specific interference is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Cd is to be determined (at 226.502 nm) in a sample containing approximately 10 mg/L of Fe. 100 mg/L of Fe would yield a false signal for Cd equivalent to approximately 0.007 mg/L. Therefore, the presence of 10 mg/L of Fe would result in a false signal for Cd equivalent to approximately 1 ppb. The interference correction factors should be determined annually. The possibility of interferences other than those determined does exist. The analyst should be aware of these interferences when conducting analyses.

5.1.3 Generally, interferences are discernable if they produced peaks, or background shifts, corresponding to 2% to 5% of the peaks generated by analyte concentrations.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. This SOP uses yttrium as an internal standard to minimize this effect. Differences in solution volatility can also cause inaccuracies when organic solvents are involved. If physical interferences are present, they must be reduced by diluting the sample. A problem that can occur with high dissolved solids is salt build-up at the top of the nebulizer, which affects aerosol flow rate and causes instrumental drift. Changing the nebulizer and removing salt build-up at the tip of the torch sample injector can be used as a measure to control salt build-up. Control of the argon flow rate does improve instrument performance.

5.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

6.0 EQUIPMENT/APPARATUS

6.1 **Inductively coupled argon plasma emission spectrometer with background correction.**

6.1.1 Perkin Elmer 4300DV Serial #077N1032302, using an AS-91 Autosampler and a Polyscience Chiller, Serial # G54802.

6.2 **Gases**:

6.2.1 Liquid **Argon** gas supply.

6.2.2 Liquid **Nitrogen** gas supply.

6.3 DELETED

6.4 **Class A volumetric flasks**, 50 ml, 100 ml and 250 ml

6.5 **Variable transfer pipettes**, 1.0 to 5.0 ml and 0.1 to 1.0 ml calibrated according to SOP 110.0005. Also, 0.01 ml to 0.1 ml micropipettes

6.6 T**emperature adjustable hot plate and hot blocks,** capable of maintaining a temperature of 95^{O}C.

6.7 **Data system**:

6.7.1 **Computers:** The Metals laboratory has one ICP-AES system analyzing per Method 200.7/6010C. System is a Lenovo ThinkCenter M Series computer with a Windows 7 operating system.. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec to tapes. As the system acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto a dedicated hard-drive.

6.7.2 **Software:** ICP WINLAB32 with revision (3.1.0).

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: reagent grade or better chemicals are used in all tests. If the purity of a reagent is in question, it is analyzed for contamination.

7.1.1 **Hydrochloric acid (conc.)**, HCl: Trace Grade.

7.1.2 **Hydrochloric acid (1:1)**, HCl: Add 500 ml concentrated HCl to 400 ml water and dilute to 1 liter in an appropriate beaker. Never add water to acid.

7.1.3 **Nitric acid (conc.)**, HNO_3: Trace Grade.

7.1.4 **Nitric acid (1:1)**, HNO3: Add 500 ml concentrated HNO_3 to 400 ml water and dilute to 1 liter in an appropriate beaker. Never add water to acid.

7.1.5 **De-Ionized Water** (DI): The deionized water is prepared using the equipment described in SOP 110.0003 The conductivity must also be checked and recorded according to SOP 110.0003. The lab supervisor needs to be notified if the conductivity is > 0.056 µmhos/cm. At this point, the supervisor will arrange for the cartridges to be replaced, if deemed necessary.

7.1.6 **ICP solution**: Any type of diluting (primary standards or samples) is done using ICP solution. ICP Solution is prepared by adding 400 ml of nitric acid and 600ml of hydrochloric acid to 5.0 liters of DI water in a 20 liter carboy and bringing up to 20 liters with DI water.

7.1.7 **Ghost Wipe**: Environmental Express Part No. 4210.

7.2 **Standards**:

7.2.1 **Primary Stock Solutions:** Solutions are stored at room temperature and not used beyond the manufacturer's expiration date. See manufactures certificate of analysis for concentrations.

Standard	Vendor	Elements
Mixed Standard 1	VHG	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn
ICCV (see Section 7.2.2.6.)	VHG (another lot) or CPI	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Ti, Tl, V, Zn

7.2.2 **Working standard solutions** are used to calibrate the instruments. They are prepared daily. Initial analyte concentrations are obtained from the bottle label or the vendor's certificate of analysis. Calibration concentrations may be viewed and/or adjusted in the calibration page of the method editor window (refer to ICP Winlab Software Guide).

7.2.2.1 **CAL 1**: ICP Solution (see 7.1.6)

7.2.2.2 **CAL 2**: Add 0.2 ml of Mixed Standard 1 (see 7.2.1) to a 200 ml volumetric flask and dilute to the mark with ICP solution.

7.1.2.3 **CAL 3**: Add 1.0 ml of Mixed Standard 1 (see 7.2.1) to a 200 ml volumetric flask and dilute to the mark with ICP solution. This

standard is also used as the **CCV** (continuing calibration verification) and as the **ICV** (initial calibration verification; see 7.2.2.5).

7.2.2.4 **CAL 4**: Add 1.0 ml of Mixed Standard 1 (see 7.2.1) to a 100 ml volumetric flask and dilute to the mark with ICP solution.

7.2.2.5 **ICV**: See **CAL 3** (see 7.2.2.3).

ICP Standard concentration (mg/L)

Elements	Std 1	Std 2	Std 3	Std 4	Std 5
Be	0.01	0.05	0.10	0.20	0.50
Ag, Cd	0.05	0.25	0.50	1.00	2.50
As, B, Ba, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Sn, Ti, Tl, V, Zn	0.10	0.50	1.00	2.00	5.00
Se	0.20	1.00	2.00	4.00	10.00
Al, Fe	0.50	2.50	5.00	10.00	25.00
Ca, Mg	1.0	5.0	10.0	20.0	50.0
K, Na	5.0	25.0	50.0	100.0	250.0

NOTE: Standards 4 and 5 are used primarily for Ba, Pb, Zn but may also be used for other metals as long as all quality control criteria are met for the element.)

7.2.2.6 The **instrument check standard** (SCV1) is used to verify the instrument calibration. It is prepared by adding 0.5 ml of ICCV (see 7.2.1) to a 100 ml volumetric flask and diluting to the mark with ICP solution.

7.2.2.7 The **interference check solutions** contain known concentrations of interfering elements, and are run at the beginning of each analytical run in order to test correction factors.

7.2.2.7.1 **IFA** is prepared by adding 12.5 ml of Inorganic Venture's CLP-ICS-A to a 250 ml volumetric flask and diluting to the mark with ICP solution.

7.2.2.7.2 **IFB** is prepared by adding 12.5 ml of Inorganic Venture's CLPP-ICS-A and 1.25 ml of CLPP-ICS-B in a 250 ml volumetric flask and diluting to the mark with ICP solution.

7.2.2.8 Three **CRL Standards** (at the lower limit of quantitation) are used to determine the detection limits below the lowest point of the calibration curve. Three solutions are prepared to cover the range of reporting limits.

7.2.2.8.1 **CRL1** is prepared by adding 25 ml of Standard 1 (see 7.2.2.1) to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.8.2 **CRL2** is prepared by adding 1 ml of Standard 3 (see 7.2.2.3 to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.8.3 **CRL3** is prepared by adding 0.5 ml of Standard 3 (see 7.2.2.3 to a 50 ml centrifuge tube and diluting to the 50 ml mark with ICP solution.

7.2.2.9 Low Level CCV (MCP & SW846-6010C)

7.2.2.9.1 Identical to CRL Standards (see § 7.2.2.8)

7.3 **Two types of blanks** are required for the analysis. The calibration blank is used in establishing the analytical curve, and as an initial and periodic check for contamination throughout the analytical run. The method blank ID is created through the Element LIMS and is linked to the Batch ID. For example, BG52701-BLK1 where B = Batch, G = Month, 5 = Year, 27 = Date, 01 = Batch Number.

7.3.1 The **calibration blank** (ICB1, CCB) is prepared using ICP solution. (See 7.1.6).

7.3.2 The **method blank** must contain all the reagents and in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.4 **Internal standard** is added to all standards and samples automatically, by the instrument. A 5 ppm yttrium standard is used for the 4300DV . It is prepared by adding 5 ml of a 1000 ppm standard (purchased from Perkin Elmer – Cat# PE#N9300167) to a 1000 ml volumetric flask and diluting to the mark with ICP solution. Expiration of 5 ppm Yttrium standard is determined by the manufacturer and is recorded on the container label. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. Recovery criteria is 70-130%; if not met, sample is rerun.

7.5 The **Manganese X, Y optimization standard** (1.0 ppm) is used to fine tune the X, Y adjustment of the torch. Prepared by adding 0.25 ml of 1000 ppm Manganese Standard (SCP Science Cat# 140-051-251) to a 250 ml volumetric flask and diluting to the mark with ICP solution. Expiration of 1 ppm Manganese standard is determined by the manufacturer and is recorded on the container label.

8.0 PROCEDURE

8.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples that have been pre-filtered and acidified do not need acid digestion as long as the samples and standards are matrix matched and the dissolved solids are < 0.2% w/v. Solubilization and digestion procedures are presented in Sample Preparation Method SOP 30_3005A and 30_3050B. ***NOTE: It is ESS Laboratory's policy to digest all dissolved samples.***

8.2 Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 15 minutes of operation prior to calibration).

8.2.1 Operating Conditions: The analyst should follow the instructions provided by the instrument manufacturer. For operation with organic solvents, use of the auxiliary argon inlet is recommended, as is solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each analyte line on that particular instrument. All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

8.3 Optimize and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Step 7.2.2 and the manganese optimization standard described in Step 7.5.

8.3.1 Double click **Winlab 32** Icon. Wait for the status check to be completed for the instruments and accessories.

8.3.2 Select Method.

8.3.3 Click on **Plasma** Icon on tool bar. Turn the virtual switch to the **on** position or press **F9** to automatically ignite the plasma.

8.3.4 After the plasma has ignited, allow the instrument to stabilize for 30 minutes before calibration.

8.3.5 Place centrifuge tube containing 1.0 ppm Mn solution on the auto-sampler tray. Press F10 and select appropriate location or click on **Analysis** column above the tool bar. Choose **auto-sampler Go To location** and enter the appropriate auto-sampler location to move the arm to the Mn solution. Click on **tools** and choose **spectrometer control**. Click on **AlignView**. The optimization should be reported in the Mn Profile Logbook (See Attachment A).

8.3.6 Flush the system with the calibration blank (Step 7.3.1) between each standard or as the manufacturer recommends. Press F10 and select **Wash**. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve should consist of a blank and a minimum of three standards.

8.3.6.1 Click on **File**. Choose **New, Sample Info File**. Enter batch ID number, which is the date, followed by x for the 3100 instrument only, and then a through z depending on the file number for the day. (i.e. 092798xa – date, 3100 instrument, first file.)

8.3.6.2 Click on **ESS setup of 4300DV** Icon. The starting location is always 9 (1-8 reserved for standard and QC). For **sample number range**, adjust the end indicator to equal the number of samples being analyzed. Enter the sample IDs in the **Sample ID** column.

8.3.6.3 For duplicates and spikes, click on the **Matrix Check Samples** box to the right of the duplicate or matrix spike ID.

8.3.6.3.1 For duplicates, select option **Duplicate** and make sure that the reference sample number corresponds to the original sample. Click **OK**.

8.3.6.4 There are currently two methods being used on the ICP, **EVERYTHINGX** and **EVERYTHING-DV**. For both methods, click on **Analyze QC's before**. In the cell at the top of the column, enter 1, 4,6-9,2,3,5,6 or select 00Daily Cal from open Sample Info File to calibrate before running samples.

8.3.6.5 For both methods, arrow down 10 cells and enter **after 5,6**. Repeat this for every ten cells. This will ensure that the CCV and CCB will run every 10 samples.

8.3.6.6 At the end of the run, enter **5,6,2,3**. This will finish off all QC that is required to run at the end of an analysis. These QC numbers are all defined in the method. QC will run before the wash step.

8.3.6.7 Click on **File, Save As, and Sample Info File**; enter same file # as batch ID.

8.3.6.8 Click on **AUTO** Icon. On the setup screen, click on **Method** Cell to choose the appropriate method.

8.3.6.8.1 Make certain that the **SIF ID** is correct in the SIF cell.

8.4.7.10.2 Open **Results Data Set Name** under Q:\Metals\Results ICP#\Results and assign the appropriate ID to the data set. The ID is the Batch ID with 'ad' added to the end.

8.4.7.10.3 Make sure that the **Save Data** and **Print Log** option boxes are selected.

8.4.7.10.4 Click on **Analyze tab**. Click on **Print List** at bottom of analyze window and print this out. Click **Analyze All** to start the run.

8.5 Before beginning the sample run, reanalyze the mixed calibration standard 3 as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 10% for 6010B or 5% for 200.7. If they do, follow the recommendations of the instrument manufacturer to correct for this condition and then recalibrate.

8.6 Analyze the instrument check standard (see 7.2.2.6) and the calibration blank (see 7.3.1). Analyze CRL – 1, 2, 3 and Standard 2 (low level quantitation standard), then the IFB and IFA solutions (see 7.2.2.7.1 and 7.2.2.5.2) immediately before the first sample analysis. Re-analyze the continuing calibration standard (Cal 3, see 7.2.2.3) after every ten samples and at the end of the run.

8.7 Below is a typical analytical sequence for a batch of twenty samples:

- Standard Blank (S0)
- Calibration Standard(s)
- Re-analyzed Calibration Standard 3; Recovery ± 10% (± 5% for 200.7)
- Instrument Check Standard (SCV1); Recovery ± 10%
- Calibration Blank (ICB) <MDL
- CRL1 ±30% (DoD QSM Recovery ±20%)
- CRL2 ±30% (DoD QSM Recovery ±20%)
- CRL3 ±30% (DoD QSM Recovery ±20%)
- Standard 2 ±30% (DoD QSM ±20%)
- Interference Check Sample (IFA) ± MRL
- Interference Check Sample (IFB) Recovery ± 20%
- Method blank- BLK
- Blank spike- BS
- Blank spike duplicate- BSD
- Sample 1
- Matrix duplicate
- Matrix spike
- Serial dilution
- Post digestion spike
- Samples 2-3
- CCV
- CCB

- Samples 4-11
- Matrix duplicate
- Matrix spike
- CCV
- CCB
- Serial dilution
- Post digestion spike
- Samples 12-19
- CCV
- CCB
- CRL1 ±30% (DoD QSM Recovery ±20%)
- CRL2 ±30% (DoD QSM Recovery ±20%)
- CRL3 ±30% (DoD QSM Recovery ±20%)
- Standard 2 ±30% (DoD QSM Recovery ±20%)
- Sample 20
- CCV
- CCB
- Interference Check Sample (IFA) ± MRL (Conn. RCP only)
- Interference Check Sample (IFB) Recovery ± 20% (Conn. RCP only)

8.8 All sample information is recorded in the run logbook (see Attachment B) and typed into the sample info file (SIF) on the ICP. The run log is used to load the auto-sampler tray. The sample information printout is compared to the run log for accuracy. A second level review is required to ensure that all samples ae labeled correctly and put into the correct positions on the autosampler. The second level analyst must initial under the date on the sequence logbook file; if analyst is absent at the start of the run then the analyst removing tubes can do the second level check at the end of the run.

8.9

9.0 CALCULATIONS - Note: The instrument printout is in mg/L.

9.2 All results should be reported with up to three significant figures.

9.3 The following calculation for **aqueous samples** will provide results in mg/L:

$$\text{Final conc.} = \frac{\text{mg/l (raw)} \times \text{dilution factor} \times \text{final volume}}{\text{Initial volume}}$$

9.4 The following calculation for **solid/soil samples** will provide results in mg/kg dry weight:

$$\text{Final conc.} = \frac{\text{mg/l (raw)} \times \text{dilution factor} \times \text{final volume (ml)}}{\text{Initial weight (g)} \times (\%\text{solids}/100)}$$

9.5 When reporting results, include date analyzed and initials of analyst on summary sheets and actual runs.

9.6 Hardness. The preferred method for determining hardness is to compute it from the results of separate determinations of calcium and magnesium (SM 2340B):

Hardness, mg equivalent $CaCO_3$ /L = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

9.7 The following calculation for **wipe samples** will provide results in µg/Sample:

Final conc. = mg/l (raw) x dilution factor x final volume (ml)

9.8 Lead calculation for Paint Chips

$$\text{Percent Lead} = \frac{E\ \text{mg/l (Pb)} \times D \times V\ \text{(ml)} \times 100\%}{M\ \text{(g)}}$$

Where: E = Concentration of lead in extract
D = Dilution factor
V = Volume of extract (usually 50 ml)
M = Mass of paint chips

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.2 All quality control data should be maintained and available for easy reference or inspection. A summary of general method quality objectives may be found in Table 4.

10.3 The upper limit of the Linear Dynamic Range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The Linear Dynamic Range should be determined by analyzing a minimum of three consecutively higher standard concentrations of the analyte until the observed analyte concentration is no more than ± 10% outside the stated concentration of the standard. Determined LDRs must be documented and kept on file. The analysis from the resulting data establishes the LDR, which may be used for the analysis of samples. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined. Record is filed in Directory Q:\Quality\QA\LDR.

10.4 Instrument detection limits (IDL) are calculated by running the calibration blank seven consecutive times. Calculate the standard deviation and multiply by 3 (or 3.14) to determine the IDL. IDL shall be ≤LOD. The IDL must be determined after initial set-up and after any significant change. Record is filed in Directory Q:\Quality\QA\IDL.

10.4 Calibration for the ICP consists of a blank and a minimum of three standards. The correlation coefficient (R) for each element calibration curve must be ≥ 0.995 (≥ 0.998 for 6010C and MCP; r^2=0.99 per DoD).

10.5 Initial and Periodic Method QC Demonstrations: The procedures specified in Section 10.3.1 through 10.3.2 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.5.2 Accuracy and Precision: To demonstrate initial laboratory capability, analyze a minimum of four initial calibration verification standards. The mean concentration must be within ± 10 % of the stated values. (± 5% for 200.7)

10.5.3 Method Detection Limits (MDL) for Individual Analytes: MDLs must be determined for each analyte/matrix/instrument combination at initial set-up and thereafter annually. See SOP 110_0013 for specific instruction on MDL determination.

10.6 Dilute and reanalyze samples that are more concentrated than the linear range study or use an alternate, less sensitive line for which quality control data is already established. **For DoD, dilute and re-analyze samples that are more concentrate than the upper calibration range.**

10.7 It is recommended that whenever a new or unusual sample matrix is encountered, or per preparatory batch at a minimum, a series of tests is performed prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 10.7.1 and 10.7.2 will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.7.1 Serial dilution **(required by DoD QSM; conditionally required by MCP – see Attachment D)**: If the analyte concentration is sufficiently high (50x the instrumental detection limit after dilution), an analysis of a 1+4 (5x) dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

10.7.1.1 Prepare the serial dilution by diluting the sample with ICP solution. If the sample was diluted to bring the result within the linear dynamic range, then the serial dilution must be performed on that dilution (Ex. Sample result based on 5x dilution, then serial dilution must be 25x for the applicable metals.)

10.7.2 Post digestion spike addition **(not required by MCP)**: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% (75-125% for method 6010B) of the known value. The spike addition should produce a minimum level of 20 times and a maximum of 100

times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

10.7.2.1 Measure out 5 ml of prepared sample into a centrifuge tube. Add 0.05 ml of mixed standard 1 (see 7.2.1), then bring to 10 ml with sample. Mix and set up to run after the serial dilution. The post digestion spike is performed on that dilution that the sample result was obtained.

10.8 Check the instrument standardization by analyzing check standards as follows:

10.8.1 Verify calibration initially after calibration (200.7), every 10 samples and at the end of the analytical run, using a calibration blank (Step 7.3.1) and a continuing calibration check standard (Section 7.2.2.3).

10.8.1.1 The results of the check standard (see 7.2.2.3) should agree within 10% of the expected value (±5% initially for 200.7).

10.8.1.2 . See section 11.0 for corrective action for out of criteria results.

10.8.1.3 Method 6010C requires that a low level ICV and low level CCV be run, meeting an accuracy criteria of 30% **(20% per DoD QSM)** .

10.8.1.4 The results of the calibration blank should be no greater than the MRL **(≤½LOQ per DoD QSM)**. See section 11.0 for corrective action for out of criteria results.

10.8.2 Verify the interelement and background correction factors at the beginning and end of each analytical run (***the end interelement check is only required for Conn. RCP***). Do this by analyzing the interference check solution (Step 7.2.2.5). Results should be within ±20% for analytes found in the IFB and ± MRL, or **< the LOD if DoD QSM criteria need to be met**, for analytes not found in the IFA (verified trace impurity from one of the spiked analytes). If outside of limits, stop analysis and recheck background corrector points and/or interelement correction factors. Re-analysis interelement check solutions. Samples may not be analyzed without a valid interference check.

10.8.3 Replicate samples are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent.

10.8.3.1 The relative percent difference between replicate determinations is to be calculated as follows:

$$RPD = \frac{D_1 - D_2 \times 100}{(D_1 + D_2)/2}$$

Where:

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

10.8.3.2 A control limit of ±20% RPD shall be used for aqueous samples that are ≥5x the MRL and ±MRL for when <5x MRL. A control limit of ±35% RPD shall be used for soil/sediment samples that are ≥5x the MRL and ±2xMRL for when <5x MRL. ***Refer to Attachment D for DoD requirements and Attachment E for 6010C/MCP requirements.*** See section 11.0 for corrective action for out of criteria results.

10.8.4 Spiked samples are to be analyzed at a frequency of 10% or per analytical batch, whichever is more frequent.

10.8.4.1 The spiked sample recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{SSR-SR}{SA} \times 100$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added to Sample

10.8.4.2 A control limit of 25% shall be used for all sample matrices. See Table 2 for DoD control limits, sporadic marginal exceedance limits are same as for blank spike (section 10.10.2) See section 11.0 for corrective action for out of criteria results.

10.9 Employ a minimum of one method blank per sample batch to determine if contamination is occurring during sample preparation. The method blank must contain all the reagents and in the same volumes as used in the processing of the samples. It must be carried through the complete procedure as the sample solution used for analysis.

10.9.1.1 The value for the method blank should be less than the MRL (**≤ ½ the LOQ in the case of DoD**). See section 11.0 for corrective action for out of criteria results.

10.10 At least one BS/BSD must be analyzed for each batch of samples. These should be prepared and analyzed in the same manner as the samples (see SOP 30_3005 and 30_3050B.

10.10.1 The BS/BSD recovery range must be within the range of 80-120% (85-115% for 200.7), ***see Attachment D for DoD blank spike recovery acceptance criteria.*** The RPD must be ≤20% for aqueous and ≤ 30% for soils, ***DoD %RPD <20% for all matrices***.

10.10.2 If the BS or BSD falls outside of this range, see section 11.0 for corrective action for out of criteria results.

10.11 Method Reporting Limit Standards (CRLs) are run at the beginning of every analytical run. Results should agree to ± 30% (**± 20% if DoD QSM Criteria are to be met**) at the LOQ. If not, review blank data for contamination.

10.12 BS/BSDs are plotted in Element to determine if in-house limits are within default limits.

11 DATA VALIDATION

11.7 Data validation will be accomplished by reviewing all of the quality control parameters and assuring that they are within recommended ranges and recording any deviations in the ICP Tray Sequence Logbooks (Attachments B-1 and B-2). The only exceptions made to ranges would be the following:

11.1.1 For duplicates, ±20% RPD shall be used for aqueous samples that are ≥5x the MRL and ±MRL for when <5x MRL. A control limit of ±35% RPD shall be used for soil/sediment samples that are ≥5x the MRL and ±2xMRL for when <5x MRL. ***When analyzing DoD samples %RPD ≤ 20%.*** However, there are cases where duplicates may not work. If this is the case, inform client in narrative concerning sample non-homogeneity.

11.1.2 For matrix spikes, the % Recovery should be ±25%. If the matrix spike is >30%, check the LCS. If the LCS is within limits, matrix interferences are present and must be noted in the narrative. ***See Table 2 for DoD MS control limits.*** If the matrix spike is <30% and non-detected results were found re-digest sample and MS to confirm matrix interference(for **DoD** work, perform Method of Standard additions, see Addendum 1).

11.1.3 PDS and Serial dilution (section 10.7) are used to test the presence of matrix affect when the matrix spike or duplicate is outside criteria. When the PDS or SD is used, their failure is to be reported in the projective narrative as a confirmation of matrix affect.

11.1.4 For the BS/BSD, the % Recovery must be within 80-120% (85-115% for 200.7). In the case of DoD samples, LCS limits specified in the **DoD QSM Appendix C** (Attach D) shall be used for batch control unless project specific criteria exist. Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard

deviations. Marginal exceedances are not allowed for those analytes determined by a project to be target analytes (i.e. "risk drivers") without project specific approval. For analytes that are not listed in the **DoD QSM Appendix C** control limits tables, a laboratory shall use their in-house control limits for batch control and data reporting.

11.1.5 If the BS or BSD is outside criterion, then re-prep and re-analyze samples with the following exception: ***For high BS/BSDs, those samples that are non-detects may be reported.***

11.1.6 Analytical batches with Method blanks above the MRL **(≤ ½ the LOQ in the case of DoD)** will be re-prepped and re-analyzed with the following exceptions:

11.1.6.1 Samples that are that are at least twenty times higher than the method blank may be reported.

11.1.6.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.1.6.3 If the analyte is found in the method blank above the MRL but is not in any of the associated samples, no corrective action is needed.

11.1.6.4 Any results that are reported with method blank contamination must be B-flagged.

11.1.7 All unusual observations and method deviations will be noted in the narrative accompanying the data report presented to the client.

11.2 All unusual observations and method deviations will be noted in the Comments section of the Tray Sequence Logbook (Attachment B) and will be included in the project narrative accompanying the data report presented to the client.

12.0 REFERENCES

12.1 SW846 Method 6010C, Inductively Coupled Plasma – Atomic Emission Spectrometry; third edition, Update III and SW846 On-line, respectively.

12.2 ICP Winlab Software Guide, Perkin Elmer. Part No. 0993-8966.

12.3 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma- Atomic Emission Spectrometry, Method 200.7, EPA/600/R-04/111 May 1994.

12.4 Environmental Express procedure for Lead Analysis with the Ghost Wipe, per HUD Guidelines for Lead in Dust Wipes, Appendix A-5.0 and NIOSH Standard 7082.

12.5 TNI Standard: Volume 1, Module 2 and Volume 1, Module 4.

12.6 DoD Quality Systems Manual, Revision 5, July 2013.

12.7 MCP WSC-CAM Section IIIA.

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 85-115% Recovery and %RSD of ≤ 20%.

14.2 The precision and accuracy data in Table 1 are typical for aqueous metals analysis.

15.0 TABLES, DIAGRAMS, ATTACHMENTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 2/14/2005

Compound Name	Spk	Avg	%Rec	%RSD	Compound Name	Spk	Avg	%Rec	%RSD
Silver	0.25	0.2409	96.4	3	Manganese	0.5	0.4863	97.3	4
Aluminum	2.5	2.5089	100.4	4	Molybdenum	0.5	0.4919	98.4	4
Arsenic	0.5	0.4615	92.3	5	Sodium	25	24.0496	96.2	4
Boron	0.5	0.4843	96.9	4	Nickel	0.5	0.4991	99.8	4
Barium	0.5	0.4803	96.1	4	Lead	0.5	0.4856	97.1	4
Beryllium	0.05	0.0489	97.8	4	Antimony	0.5	0.4652	93.0	3
Calcium	5	4.8144	96.3	4	Selenium	1	0.9435	94.3	4
Cadmium	0.25	0.2258	90.3	4	Tin	0.5	0.4809	96.2	4
Cobalt	0.5	0.4838	96.8	4	Strontinum	0.05	0.0490	98.0	4
Chromium	0.5	0.4878	97.6	4	Titanium	0.5	0.4845	96.9	4
Copper	0.5	0.5010	100.2	4	Thallium	0.5	0.4727	94.5	5
Iron	2.5	2.4523	98.1	4	Vanadium	0.5	0.5030	100.6	4
Potassium	25	22.0020	88.0	4	Zinc	0.5	0.4614	92.3	4
Mangnesium	5	4.7553	95.1	4					

Attachments:

Table 2 DELETED

Table 3 Instrument Wavelengths and Limits

Table 4 General Method Quality Objectives

Attachment 1 Method of Standard Additions

Attachment A-1 DELETED

Attachment A-2 DELETED

Attachment B-1 DELETED

Attachment B-2 ICP III Tray Sequence Logbook

Attachment C DELETED

Attachment D DoD QSM Version 5 Method Quality Objectives.

Attachment E MCP WSC-CAM Section IIIA Method Quality Objectives.

16.0 DEFINITIONS

16.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

16.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

16.3 **Blank spike (BS, LCS)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When it is necessary to test for lead, per HUD specifications, the LCS is prepared by spiking a Ghost Wipe (ASTM E 1792, current lot) with an appropriate amount of ICV solution.

16.4 **Matrix spike (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

16.5 **Matrix spike duplicate (MSD)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MSD is analyzed exactly like a sample, and its purpose is to determine the precision of the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MSD corrected for background concentrations.

16.6 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

16.7 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

16.8 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of BS or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

16.9 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP 80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING/MAINTENANCE

18.1 **Instrument Maintenance**:

18.1.1 **Daily:**

- Inspect water level in re-circulating water chiller and inspect for leaks.
- Inspect peristaltic pump tubing, replace if worn.

- Inspect RF coil for excess condensation, wipe down to avoid arcing. Also, the coil should be cool to the touch, which indicates that cooling water is circulating through system.
- Inspect purge window and clean as needed.

18.1.2 **Monthly**

- Check ventilation filters clean as needed.

18.1.3 **Bi-annually**

- Replace chiller water.
- Vacuum ICP air vents

18.2 Record all maintenance in the instrument's maintenance logbook.

18.3 Extraordinary problems may require assistance from Perkin-Elmer Corporation. ESS Laboratory maintains a service contract with this vendor.

19.0 Data Management And Records

19.1 **Data Management** - ESS Laboratory's utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

TABLE 3

INSTRUMENT WAVELENGTHS AND LIMITS

Detection Element	Wavelength(nm)
Aluminum	237.313
Antimony	206.836
Arsenic	188.979
Barium	233.527
Beryllium	313.107
Boron	182.528
Cadmium	228.802
Calcium	315.886
Chromium	267.716
Cobalt	228.616
Copper	324.752
Iron	238.204
Iron	302.107
Lead	220.353
Magnesium	279.077
Manganese	257.610
Molybdenum	202.031
Nickel	231.604
Potassium	766.491
Selenium	196.026
Silver	328.068
Sodium	330.237
Thallium	190.801
Tin	189.927
Titanium	337.279
Vanadium	292.402
Zinc	213.857

The wavelengths listed are utilized because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see step 5.1).

Table 4
Summary of Method Quality Objectives for Method 200.7/6010B
Metals by Inductively Coupled Plasma

QC Element	Frequency	Criteria	Corrective Action
Initial Calibration	Daily following optimization of ICP and prior sample analysis.	• Minimum of blank and three standards. • Low standard at MRL • R ≥0.995 (Do not force through zero for LR)	• No allowance. Perform maintenance and recalibrate.
ICV	Immediately following daily initial calibration.	• %Rec = 90-110%, %RPD < 5%. • Use separate source from initial calibration standards.	• If criteria exceeded, remake and re-analyze ICV. If second consecutive ICV is within criteria then calibration is accepted, otherwise recalibrate.
Low Level Calibration Standard (CRI)	Daily after initial calibration to support MRL.	• Only used if low standard is not at the MRL. • %Recovery 70-130%. • MRL is at level of last successfully passed CRL.	• If no CRL passes, MRL at concentration in lowest calibration standard.
CCV	After calibration, every 10 samples and at end of analytical run.	• Concentration level near midpoint of curve • Same source as calibration standard. • %Rec = 90-110%, %RPD ≤5% (200.7 first CCV 95-105%)	• If criteria are exceeded, then remake and re-analyze CCV. If second consecutive CCV is within criteria, then calibration is verified, otherwise re-calibrate system and re-analyze any sample since the last valid CCV.
Continuing Calibration Blank	After calibration, every 10 samples and at end of analytical run.	• Must be matrix-matched (same acid concentration as standards and QC samples.) • Analytes < MRL	• Re-calibrate and re-analyze all samples since last valid CCB.
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL	• Report exceedance in the project narrative. • Samples that are non-detect may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged.
Blank spike (BS)	One per analytical batch of 20 or fewer samples.	• Use standard source different than used for initial calibration and Matrix specific • Concentration level should be between low and mid-level standard • Percent recoveries 80-120%. (85-115% for 200.7)	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply.

		• DoD % Recoveries are listed in QSM Version 5, Appendix C	
Blank spike duplicate (BSD)	One per analytical batch of 20 or fewer samples.	• Prepared as above • Percent recoveries same as above • %RPD ≤ 20% aqueous and ≤ 30% soil.	• Report exceedance in the project narrative. • If LCS is biased high and sample is non-detect, then may report sample result. • Re-digest and re-analyze if the above exceptions do not apply.
Matrix Spike	One per analytical batch of 10 or fewer samples	• Prepared using the same source as the blank spike • Concentration level should be between low and mid-level standard • Percent recoveries between 75-125%. • DoD % Recoveries are listed in QSM Version 5, Appendix C	• Report exceedance in the project narrative. • Laboratories are expected to develop in-house control limits per each media. Control limits should fall within default limits.
Matrix duplicate	One per analytical batch of 10 or fewer samples	• **Aqueous**: Relative percent difference is ±20% for samples > 5x MRL and ±MRL for samples <5x the MRL. • **Soil**: Relative percent difference is ±35% for samples > 5x MRL and ±2xMRL for samples <5x the MRL.	• Note exceedance in project narrative. • If MS %Recovery is >30% and LCS is in control, no corrective action is required. If %Recovery is <30% and non-detect results found perform PDS.
Serial Dilution	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Result of 5x dilution must be ±10% of undiluted sample when result is ≥25x MRL.	• Narrate serial dilution QC out of control. May be indication of matrix affect.
Post Digestion Spike (PDS)	One per analytical batch of 10 or fewer samples, performed on Dup/Spk sample.	• Perform PDS if Serial dilution is not in control or if sample is < 25x the MRL. • Percent recovery must be 85-115% (75-125% for 6010B).	• Narrate PDS out of control if MS is also out of control. May be indication of matrix affect.
Linear Dynamic Range	Semi-annually	• Use successively higher concentrated standards against calibration curve until a standard is reached that is >10% difference from true value. Upper LDR is 90% of the highest standard within criteria.	• N/A
Instrument Detection Limit	Quarterly	• Analyze ten replicates of the calibration blank.	• N/A

v

ATTACHMENT I

SOP Modification

Method of Standard Additions

SOP(s) Modified: Inductively Coupled Plasma-Atomic Emission Spectroscopy (SW 846 Method 6010B/ EPA Method 200.7

Modification Objective: This addendum defines when the method of standard additions must be performed and how to perform the procedure.

Method Summary: The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

For DoD/Navy projects, the method of standard additions (MSA) shall be used when the matrix spike or matrix spike duplicate is outside control limits and the failure is confirmed by the serial dilution/post digestion spike test. MSA calculation will only be applied to the metal(s) that failed.

MSA Procedure: The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume Vx of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals (performed by the ICP). The unknown sample concentration C_X is calculated:

$$C_X = \frac{S_B \times V_S \times C_S}{(S_A - S_B) \times V_x}$$

Where S_A and S_B are the analytical signals of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample.

Since ESS Laboratory performs a post digestion spike (PDS) on each sample it has chosen for the matrix spike the results from the spiked and unspiked aliquots will be used in the MSA calculation.

Example: MS result for Titanum was 56.8% and the PDS was at 69%. MSA calculated as follows:

$V_x = 10$ ml $V_S = 0.05$ ml $C_S = 100$ mg/L

Signal S_A (spiked aliquot) = 109644
Signal S_B (unspiked aliquot) = 19801

$$C_X = \frac{(19801 \times 0.05 \text{ ml} \times 100 \text{ mg/L})}{(109644 - 19801) \times 10 \text{ ml}} = 0.11 \text{ mg/L}$$

ATTACHMENT B-2

ESS LABORATORY
ICP III TRAY SEQUENCE LOGBOOK

#	SAMPLE	#	SAMPLE	#	SAMPLE	#	SAMPLE
1	STD 1:	31		61		91	
2	STD 2:	32		62		92	
3	STD 3:	33		63		93	
4	STD 4:	34		64		94	
5	SCV1:	35		65		95	
6	CRL 1:	36		66		96	
7	CRL 2:	37		67		97	
8	CRL 3:	38		68		98	
9		39		69		99	
10		40		70		100	
11		41		71		101	
12		42		72		102	
13		43		73		103	
14		44		74		104	
15		45		75		105	IFB:
16		46		76		106	IFA:
17		47		77			
18		48		78			
19		49		79			
20		50		80			
21		51		81			
22		52		82			
23		53		83			
24		54		84			
25		55		85			
26		56		86			
27		57		87			
28		58		88			
29		59		89			
30		60		90			

Internal Standard

ID:_____

SIF:_____

RDS:_____

_____

METHOD:_____

ANALYST:_____

DATE:_____

Comments:_____

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. * If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.

* Attached

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table – 8. Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

ANALYTE	AQ LCL	AQ UCL	SOIL LCL	SOIL UCL
Aluminum	86	115	74	119
Antimony	88	113	79	114
Arsenic	87	113	82	111
Barium	88	113	83	113
Beryllium	89	112	83	113
Boron	85	113	72	114
Cadmium	88	113	82	113
Calcium	87	113	81	116
Chromium	90	113	85	113
Cobalt	89	114	85	112
Copper	86	114	81	117
Iron	87	115	81	112
Lead	86	113	81	112
Magnesium	85	113	78	115
Manganese	90	114	84	114
Molybdenum	89	113	82	116
Nickel	88	113	83	113
Potassium	86	114	81	116
Selenium	83	114	78	111
Silver	84	115	82	112
Sodium	87	115	83	118
Strontium	90	113	83	114
Thallium	85	114	83	111
Tin	88	115	80	120
Titanium	91	111	83	114
Zinc	87	115	82	113

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency (IDP)	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedures in Section 9.4 of SW-846 6010C and the applicable preparation method (SW-846 3000 series).	No	NA	Refer to Section 9.4 of SW-846 6010C, the applicable preparation method requirements in SW-846 3000 series methods, and Section 1.1.2 of this protocol.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered groundwaters) and solid samples must be prepared (digested) prior to analysis. See Appendix III A-4 for preparation method references.	No	NA	NA	NA
Linear Dynamic Range (LDR)	Laboratory Analytical Accuracy	(1) Frequency: check LDR every 6 months (Section 10.4 of SW-846 6010C). (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a minimum of 3 different concentration standards across the range. See SW-846 Method 6010C for details.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency: Following profiling and optimization of ICP; daily prior to sample analysis. (2) Minimum calibration blank plus one calibration standard for each target analyte or a multi-point curve. (3) Linear regression with correlation coefficient $r \geq 0.998$; non-linear regression may be used if $r^2 \geq 0.998$.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6010C.	Suspend all analyses until initial calibration meets criteria.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency: Immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes.	No	NA	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(5) Percent recoveries must be between 90-110% for each target analyte.				
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Immediately after ICV. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.
Low-Level Calibration Verification (LLCV)	Laboratory Analytical Sensitivity (verify low-end of calibration range / verify RL)	(1) Frequency: Daily prior to sample analysis if initial calibration did not contain a low-level standard at the RL for each target analyte. If initial calibration includes the RL as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL for all target analytes. (4) Percent recoveries must be 70-130% for all target analytes.	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are ≤10x RL in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes are >10x RL in associated field samples, include explanation in laboratory narrative; no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL in the associated field samples.
Interference Check Standards (ICSA and ICSAB)	Laboratory Analytical Accuracy (checks background points and interelement interference corrections on instrument)	(1) Frequency: Daily prior to sample analysis. (2) ICSA and ICSAB must contain known amounts of interfering analytes (see SW-846 6010C). (3) Percent recoveries must be 80-120% for all target analytes. (4) Non-spiked analytes in the ICSA must be <2x RL.	No	NA	(1) Reanalyze ICSA/AB; if acceptable, no further action required. (2) If ICSA/AB is still outside of criteria, adjust interference corrections, background corrections, and/or linear ranges, as needed and reanalyze ICSA/AB. (3) Recalibrate and reanalyze all samples since last compliant ICSA/AB.	Suspend all analyses until ICSA/AB meet criteria. If automatic (computerized) corrections for background and IECs are not used during analysis, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency: Every 10 samples and at the end of the analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte.	No	NA	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies. (3) If recovery is high (>110%) and all associated sample results are non-detected, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Frequency: Every 10 samples following CCV and at the end of the analytical run. (2) Prepared using same concentration of acids as calibration standards. (3) Target analytes must be <RL.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of contaminant in CCB is >RL but all associated sample results are either non-detected or >10x concentration in CCB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Method Blank (MB)	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be digested with the samples using the same preparation method as the samples. (3) Target analytes must be <RL.	Yes	NA	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, redigest and reanalyze MB and all associated field samples in batch – unless (3) applies. (3) If concentration of contaminant in MB is >RL but all associated sample results are either non-detected or >10x concentration in MB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Frequency: One per digestion batch of ≤20 field samples. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: affected analytes in associated samples may be rejected.	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for same analyte, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery exceedances in laboratory narrative.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
LCS Duplicate (LCSD)	Laboratory Analytical Accuracy & Precision	(1) Frequency: One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels must be same as LCS. (4) Must contain all target analytes; analyze immediately following LCS. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.	Yes ONLY if no MD	Same as above for LCS for recovery evaluation	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for same analyte, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory narrative.
Matrix Spike (MS) Project-Specific	Method Accuracy in Sample Matrix	(1) Solid Samples (Soil/Sediment) Frequency: One per 20 field samples per matrix; designated by data user on COC or at project set-up. Aqueous Samples Frequency: One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for affected metal in all associated samples.	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in-control, no corrective action is required. (3) If MS recovery is <30% and associated with non-detected results, redigest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS exceedances in laboratory narrative. If redigested due to recoveries <30%, report both sets of sample/MS data.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010C) Using WSC-CAM-III A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Matrix Duplicate (MD) Project-Specific	Method Precision in Sample Matrix	(1) Frequency: One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for each target analyte must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.
Dilution Test	Accuracy in Sample Matrix	(1) Frequency: One per ≤20 field samples per matrix; only if project-specific MS requested and analyte concentration is >50x RL. (2) Perform 5x serial dilution on same sample used for MS/MD. (3) %D of the Sample & Dilution results for target analytes at levels >50x RL must be ±10% for all matrices.	Yes ONLY if project-specific MS requested by data user	NA	Narrate.	Report exceedances in laboratory narrative.
General Reporting Issues	NA	(1) Non-detected values must be reported with the sample-specific RL for each target analyte using all preparation/dilution factors. (2) The laboratory must only report values ≥ the sample-specific RL; optionally, values below the sample-specific RL can be reported as estimated, if requested. (see SW-846 Method 6010C, Section 10.3.3). The laboratory must report results for samples and blanks in a consistent manner. (3) Sample concentrations that exceed the LDR must be diluted and reanalyzed to fall within the linear dynamic range. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific RL. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target analytes, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not preserved properly or are not received with an

HERITAGE
CONSULTANTS, LLC

INTEGRATED HISTORIC PRESERVATION PLANNING

November 29, 2017

Ms. Susan Moberg
Director or Energy and Environmental Services
Vanasse Hangen Brustlin
1 Cedar Street, Suite 400
Providence, RI 02903

RE: Addendum to *Phase IA Cultural Resources Assessment Survey of the Proposed Tobacco Valley Solar Project in Simsbury, Connecticut;* Historical Research Concerning Martin Luther King, Jr., and Possible Associations with the Proposed Project Area

This document represents an addendum to the prepared and submitted report entitled *Phase IA Cultural Resources Assessment Survey of the Proposed Tobacco Valley Solar Project in Simsbury, Connecticut* (Heritage Consultants, LLC 2017). The purpose of the current research effort is to collect and assess the available evidence concerning the possibility that during 1944 and 1947, Martin Luther King Jr., may have worked on property that coincides with the project area associated with the proposed Tobacco Valley Solar Project in Simsbury, Connecticut, and specifically within either of the two tobacco barns situated immediately to the north of Hoskins Road. This research was conducted between November 17, 2017 and November 26, 2017.

During this research, representatives from Heritage Consultants, LLC (Heritage) visited the Simsbury Town Hall and the Connecticut Valley Agricultural Museum in an effort to recover any documentation related to Martin Luther King, Jr. The Simsbury Historical Society also was contacted to set up an appointment to look through their collections; however, their staff has failed to respond to the request to date. Finally, Heritage accessed the online resources of Stanford University's Martin Luther King, Jr. Research and Education Institute, and contacted a representative of the Stanford collection who has not yet responded to a request for additional information about correspondences associated with Martin Luther King Jr. (see below).

As mentioned above, the primary goal of this research was to determine whether any definitive associations with Martin Luther King, Jr., and the proposed project site and/or barns contained therein could be made. In doing so, Heritage also endeavored to ascertain where Martin Luther King, Jr. stayed while working in Simsbury in the hope that such information may shed light on his possible association with the proposed project site. The two possible locations for his residence during the summers of 1944 and 1947 are the extant boarding house on the southern side of Hoskins Road and a boarding house that was once situated near the intersection of Firetown Road and Barndoor Hills Road; the latter was burned down by the Town of Simsbury's Fire Department in the 1980s (see below). The Hoskins Road boarding house is located on the opposite side of Hoskins Road from the barns mentioned above and well to the east of the project parcel. The other boarding house was located over a mile away to the west (see Figure 1). These boarding houses were spatially associated with a large complex of tobacco fields, barns, and other buildings. Overall, the evidence seems to indicate that Martin Luther King, Jr. resided at the Firetown Road dormitory well to the west of the project area, which according to local tradition is the one that the Morehouse College students stayed at when they were in town. Although it may be interpreted that residents of these boarding houses worked primarily in the nearest fields and buildings, specific evidence of such practices is lacking. The

only clear evidence about where Marin Luther King, Jr. was assigned to work was in the kitchen of the dormitory during part of 1944 (see below); otherwise, there is no available information about the location of his outdoor work assignments.

During the current research, representatives of Heritage Consultants visited the Town of Simsbury Town Clerk's office and reviewed land records that confirmed that during the period in question, the proposed project parcels were owned by Cullman Bros., Inc. (later Culbro, Inc.) (Simsbury Land Records, Vol. 146, Pg. 60 and Vol. 80, Pg. 441). This research also resulted in the collection of copies of property survey maps prepared in 1963, when the company was selling off parts of its land (Town Clerk Maps #1073, 1074, 1075, and 1076). The information on these maps has been compiled in Figure 1, which shows the large area of land the company owned near the project site and the location of the buildings that existed at the time. This included the boarding house on Hoskins Road; the one that stood near the intersection of Firetown Road and Barndoor Hills Road is not labeled as such. This may be either because the surveyors who compiled the maps made different decisions about what to label, or because it was no longer being used as a boarding house by 1963. Nonetheless, this information makes the relative locations of these boarding houses, as well as the then-surviving barns and other buildings, clear.

As mentioned above, Heritage's representatives also reviewed materials made available online by Stanford University's Martin Luther King, Jr. Research and Education Institute. This resulted in the identification of transcripts of five letters that Martin Luther King, Jr. wrote in 1944 while working on the Cullman property in Simsbury (before he started attending Morehouse College as a freshman). In the first three letters, dated June 11, June 15, and June 18, 1944, he reported to his family that he was working in the boarding house kitchen and serving as a volunteer Sunday prayer leader. The fourth letter, dated August 5, 1944, seems to suggest he may have been working outdoors, as he referred to very long hours and losing money due to heavy rains keeping them from work. It is important to note that he said in the letter that "we" were losing money; he might have been speaking of his fellow workers generally. The letters are particularly instructive in that Martin Luther King, Jr. seemed eager to emphasize that he was working in the kitchen in June (perhaps it was a higher-status job). The fifth letter, dated August 30, 1944, makes no mention of work. From these letters, then, it can be concluded that in June 1944, Martin Luther King, Jr. was working in the kitchen of one of the boarding houses, not in the fields or barns, and that in August he *may* have been working in the fields or barns, but there is nothing to indicate which fields or barns. A request to Stanford University's Martin Luther King, Jr. Research and Education Institute for any additional 1944 letters or any 1947 letters is pending.

Heritage representatives also visited the Connecticut Valley Agricultural Museum (formerly the Luddy/Taylor Connecticut Valley Tobacco Museum) (CVAM), which holds an extensive collection of Cullman Bros./Culbro materials. These materials include a collection of employee identification cards that recorded to which field each worker was assigned. Unfortunately, these records were kept only up until 1939, and therefore failed to shed any light on Martin Luther King, Jr. during 1944 and 1947. CVAM also curates scrapbooks of "publicity" (newspaper articles and other items) related to Cullman Bros./Culbro that were collected for many years, including 1947, but not 1944. A review of the 1947 volume indicates that the musical activities of the Southern college students that year included a concert at the *Hartford Times* building in Hartford, but does not mention any of the singers by name. None of the collected materials mention where any of the groups of workers (ranging from the African American college students to Jamaicans to white high school girls from Florida) were assigned to live or work.

Heritage representatives also reviewed a 2001 archaeological report that included an appendix concerning potential Martin Luther King, Jr. connections in Simsbury (Banks and Lavin 2001). In that report, Banks and Lavin duplicated a 1991 *Hartford Courant* article that reported that Martin Luther King, Jr. lived in a wooden workers' dormitory off Firetown Road," and a local bank's newsletter from 1997 that contained an article about his stay in town that likewise also asserted that he "lived in a wooden dormitory off Firetown

Road." Banks and Lavin also spoke to local historian Pamela McDonald who, in contrast to *the Hartford Courant* article, who told them she believed Martin Luther King, Jr. stayed at the Hoskins Road dormitory because he belonged to a Morehouse College singing group that performed in Hartford, and the Hoskins Road dormitory residents were the ones involved with that. McDonald's assertion cannot be verified because she offered no source as to why she knew this was singing group's residence. Thus, Banks' and Lavin's report offers conflicting information about where Martin Luther King, Jr. resided in 1944 and 1947, and offered with no convincing basis for choosing one option over the other. Finally, none of the material in Banks' and Lavin's 2001 report makes any reference in which fields or barns Martin Luther King may have worked might have worked in while being employed by Cullman Bros./Culbro.

In contrast, an online article prepared by the Simsbury Historical Society is very definite that the dormitory Martin Luther King, Jr. lived in during 1944 was "on Firetown Road near Barndoor Hills." Importantly, the article also asserts that he worked in the fields adjacent to the Firetown Road dormitory. Regarding his 1947 stay, the Simsbury Historical Society article offers no information about his residence (Simsbury Historical n.d.). Similarly, an online article written by Dawn Byron Hutchins at *Connecticut Explored* specifies "A Morehouse dormitory was built on the Simsbury/Granby border in 1936; Martin Luther King Jr. spent the summers of 1944 and 1947 there" (Hutchins 2011). Based on the reference to the closeness of the Simsbury/Granby border, this article appears to be refencing the Firetown Road dormitory, which is closer to the identified town boundary than the dormitory along Hoskins Road well to the south. The article does not cite any specific source for this detailed information about the "Morehouse dormitory." It is credible, however, that local informants would remember this dormitory as the one where the Morehouse College students stayed, thus leading to the various reports that it was the Firetown Road dorm that Martin Luther King, Jr. occupied. Again, however, there is no specific information in these sources about where he might have worked, only about where he lived.

According to a 2012 news article by Clennon L. King, the (volunteer) Simsbury Fire Department's website at the time featured photographs of the burning dormitory and referred to the building as "the Morehouse Dormitory," which similarly suggests that the college connection was well-known to local residents. This fire was a controlled burn undertaken as a firefighting exercise in 1984 (King 2012). The news article did not provide a link to the website, and the current version of the Simsbury Fire Department's website no longer contains the information. Historical works about tobacco-growing in Connecticut mention the temporary importation of Southern African American college students and the participation of Martin Luther King, Jr. in that program, but it provides no specifics about his residence or work space. These resources also refer to workers being moved around the companies' lands by truck or bus as the status of the crops required (Dunlap 2016, Harris 2005, O'Gorman 2002, Hall and Harvey 1995).

In sum, it seems clear that the information about Martin Luther King, Jr. staying at the Firetown Road dormitory is based on local memory. The weight of the historical evidence that could be discovered suggests any connection of Martin Luther King, Jr. to the dormitory on Hoskins Road is likely in error. In addition, in the absence of available work records or more specific information from his letters, it cannot be said for sure what work Martin Luther King, Jr. did while in Simsbury or where other than his duties the dormitory kitchen in June 1944. The only concrete fact that can be drawn from the available historical sources is that Martin Luther King, Jr., resided on Cullman Bros. land. In the absence of new information, where on those thousands of acres of land and numerous barns he worked remains unknown. Thus, an association between Martin Luther King, Jr. and the proposed project site, or any barns within it, could be made.

Banks, Marcus and Lucianne Lavin
2001 *Phase I Archaeological Reconnaissance Survey of the Proposed Cell Phone Tower Site at 54 Floydville Road in East Granby, Connecticut.* CHPC #1041. Seymour, CT: American Cultural Specialists LLC; Hartford, CT: Connecticut State Historic Preservation Office.

Connecticut Valley Agricultural Museum
Var. Windsor, Connecticut: CVAM.

Dunlap, Brianna E.
2016 *Connecticut Valley Tobacco*. Charleston, South Caroline: The History Press.

Hall, Robert L. and Harvey, Michael M., eds.
1995 *Making a Living: The Work Experience of African-Americans in New England: Selected Readings*. Boston: New England Foundation for the Humanities.

Harris, Katherine J.
2005 *An Historical Perspective on African American and West Indian Tobacco Workers*. Simsbury, Connecticut: Simsbury Historical Society; Hartford, Connecticut: Connecticut Historic Preservation and Museum Division.

Hutchins, Dawn Byron
2011 "Laboring in the Shade." *Connecticut Explored*. Accessed November 17, 2017. https://connecticuthistory.org/laboring-in-the-shade/

King, Clennon L.
2012 "Obscure Simsbury Martin Luther King Landmark Delights Neighbors." Published January 16, 2012. Accessed November 27, 2017. http://www.registercitizen.com/news/article/Obscure-Simsbury-Martin-Luther-King-landmark-12050113.php

Martin Luther King, Jr. Research and Education Institute
Var. Martin Luther King, Jr. Papers Project. Stanford: Stanford University. Accessed November 24, 2017. https://kinginstitute.stanford.edu/king-papers/about-papers-project

O'Gorman, James F.
2002 *Connecticut Valley Vernacular: The Vanishing Landscape and Architecture of the New England Tobacco Fields*. Philadelphia: University of Pennsylvania Press.

Simsbury Historical Society
n.d. "Martin Luther King: His Time in Simsbury, Connecticut." Simsbury Historical Society website. Accessed November 17, 2017. http://www.simsburyhistory.org/SimsHistory/mlking.html

Simsbury, Town of
Var. Records of the Simsbury Town Clerk. Simsbury, Connecticut: Office of the Town Clerk.

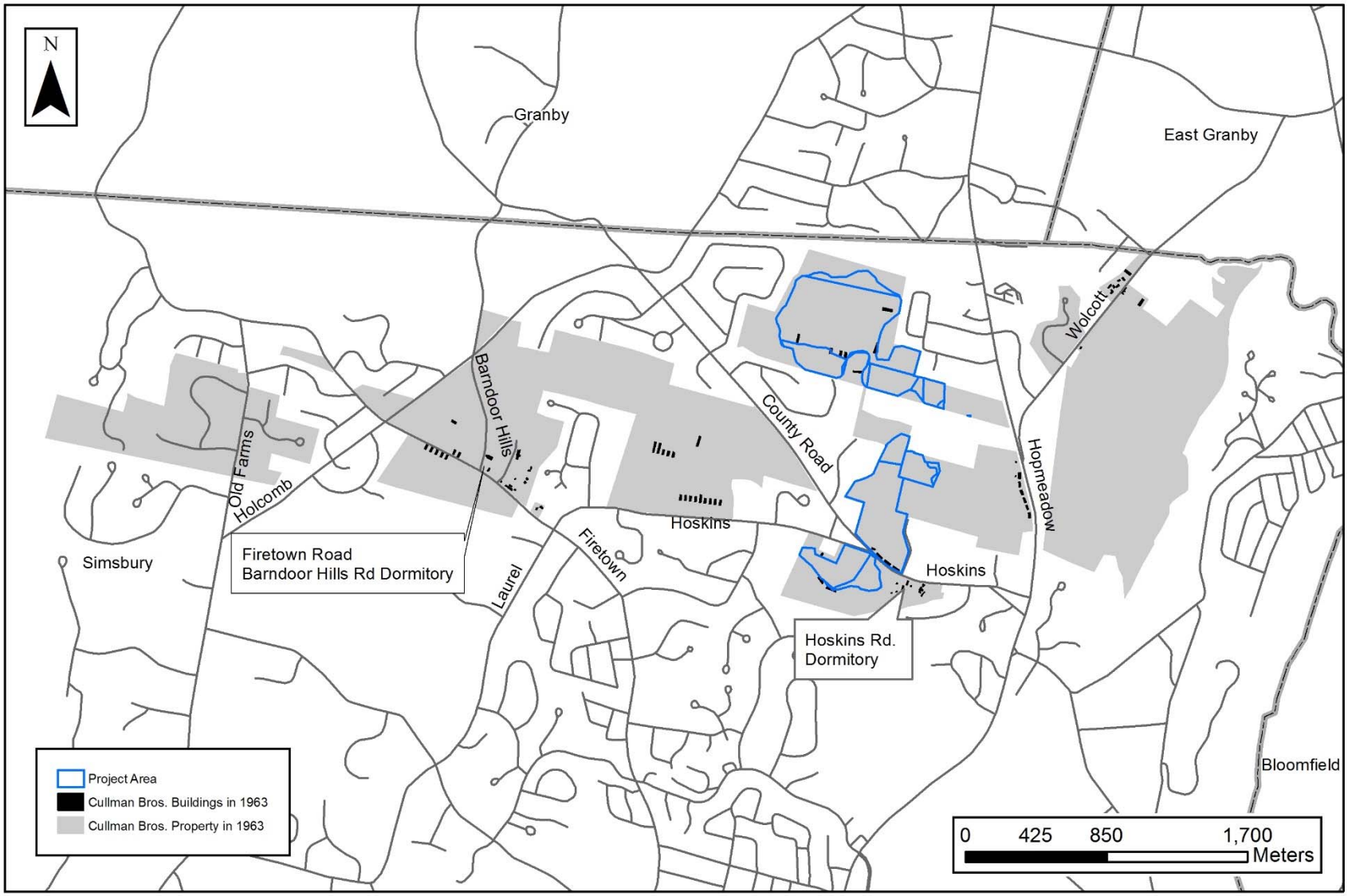


Figure 1. Digital map showing the locations of Cullman Brothers, Inc., lands and buildings in 1963 (map compiled from information on file at the Simsbury Town Hall).

OPTION AGREEMENT

This Agreement effective the ___ day of September, 2018, by and between **DWW SOLAR II, LLC,** a Deleware limited liability company, hereinafter referred to as "Optionor", and **THE TOWN OF SIMSBURY**, a Connecticut municipality, hereinafter referred to as "Optionee".

WHEREAS, Optionor is the developer of a 26.4 MW AC solar photovoltaic array proposed to be located in the Hoskins Road area in Simsbury, Connecticut ("the Project"), the site of which is comprised of several parcels of land (the "Property"), which is more fully described on Exhibit A.

WHEREAS, Optionor and Optionee executed a settlement agreement ("Settlement") concerning the Project in connection with an administrative appeal docketed with the Connecticut Superior Court as *Town of Simsbury v. Connecticut Siting Council et al.,* Docket No. HHB-CV18-6042321-S.

WHEREAS, in the Settlement, along with additional consideration, Optionor agreed to grant an option to Optionee to purchase the Property for One Dollar ($1.00) within six (6) months of Optionee's receipt of notice from Optionor that Optionor has ceased use of the Property for the purpose of converting sunlight into electricity or ninety-nine (99) years from the date of the execution of the Settlement, whichever is earlier.

NOW, THEREFORE, in consideration of the promises exchanged pursuant to the terms of this Agreement, and for other good and valuable consideration, the sufficiency of which the Parties acknowledge, the Parties agree as follows:

1. **Grant of Option** In consideration of the payment of **ONE DOLLAR ($1.00)**, and other valuable consideration made to the Optionor by the Optionee, the receipt of which is hereby acknowledged by Optionor, the Optionor does hereby grant and give to the Optionee, and Optionee accepts, the right and option to purchase the Property (the "Option"), upon the terms and conditions hereinafter set forth.

2. **Term of Option** The Option shall expire six (6) months from the date the Optionee receives notice from the Optionor that it has ceased use of the Property for the purpose of converting sunlight into electricity or ninety-nine (99) years from the date of the execution the Settlement.

3. **Exercise of Option** The Option may be exercised as specified in Section 2 hereof by Optionee in accordance with the procedures and at the addresses set forth in Section 5 hereof. The notice of exercise of the Option shall be in writing, signed by the Optionee and in substantially the following form:

"The Town of Simsbury ("Optionee"), acting herein by _____, its _____, hereunto duly authorized, hereby notifies you that it exercises its Option to purchase for ONE DOLLAR ($1.00) the Property described in a certain Option effective September __, 2018, from you to Optionee. Optionee ratifies and confirms all provisions of the Option. Optionee represents that it has had no dealings or negotiations with any broker or agent in connection with the Property which are the subject of this Option."

The date of the exercise of the Option shall be the date notice of the exercise of the Option by Optionee is given to Optionor by any of the methods provided in Section 5 hereof. Optionor represents that it has had no dealings or negotiations with any broker or agent in connection with the Property which are the subject to this Option.

4. **Closing Date and Conveyance** In the event that this Option is exercised by Optionee, the sale and purchase pursuant to the terms of the Option shall be consummated and closed (hereinafter referred to as the "Closing") by the Optionor and Optionee within thirty (30) days of the date Optionee exercised the Option. The Property is to be conveyed by a good and sufficient warranty deed executed by Optionor and running to Optionee. Said deed shall convey record and marketable title to the Property, free and clear of all liens, judgments, encroachments, easements, federal tax liens, state tax liens, mechanics liens, whether inchoate or perfected, including but not limited to encroachments and rights or claimed rights of third parties, or other similar encumbrances or restrictions on the use of the Property, except: (a) provisions of existing building and zoning laws and (b) such taxes for the then current year as are not due and payable on the date of the delivery of such deed.

5. **Notices** All notices to be given Optionor shall be in writing and delivered by hand, or by reputable overnight courier or by certified mail, return receipt requested, addressed as follows:

To the Optionor at: with a copy to:

ADDRESS

or at such other addresses as the Optionor shall from time to time designate by written notice to the Optionee by certified mail, return receipt requested. All notices to be given to Optionee shall be in writing and delivered by hand or by reputable overnight courier or by certified mail, return receipt requested, addressed as follows:

If to Optionee:	with a copy to:
Town of Simsbury 933 Hopmeadow Street Simsbury, CT 06070 Attention: Town Manager	Updike, Kelly & Spellacy, P.C. 100 Pearl Street, 17th Floor Hartford, CT 06103 Attention: Robert DeCrescenzo

or at such other address as the Optionee shall from time to time designate by written notice to the Optionor.

6. **Place of Closing** The closing shall take place at the law offices of Updike, Kelly & Spellacy, P.C., 100 Pearl Street, 17th Floor, Hartford, Connecticut 06103, or at such other addresses as the parties shall mutually designate.

7. **Recordation** This Option may be recorded in the land records at the expense of the Optionee. The Optionee shall execute and deliver to the Optionor a release in recordable form in the event this Option is terminated by Optionee prior to the end of the option period.

8. **Due Diligence** Optionor agrees to provide Optionee and Optionee's duly authorized agents and consultants with reasonable access to the Property to enable Optionee to inspect and examine the Property, take measurements, perform tests and for any other legitimate purpose. Optionee shall promptly repair any damage to the Property caused by such access and to restore the Property to a condition similar to that prior to the performance of any such inspection and examination and agrees to indemnify, defend and hold Optionor harmless from and against any and all claims, demands, actions, causes of action or other liabilities (including, without limitation, reasonable attorney's fees and disbursements) caused by Optionee, its agent and consultants in connection with such access. From such inspections and prior to the exercise of any Option and, if any Option is exercised, for the Due Diligence Period, Optionee shall be entitled to: (i) obtain an engineering inspection and report including, without limitation, with respect to the availability of utilities with respect to the Property and the physical condition of the buildings and all improvements including, without limitation, the roof, structure, mechanical and utility systems, disclosing no conditions with respect to the Property which are unsatisfactory to Optionee; (ii) obtain a title report and survey of the Property disclosing no title exceptions or conditions with respect to the Property which are unsatisfactory to Optionee; and (iii) review all loans and tenancies relating to the Property.

9. **Miscellaneous**

a. The captions of this Option are for convenience and reference only and shall not be deemed or construed to bind, modify, increase or decrease the terms and conditions of this Option, or any interpretation or construction thereof. Any reference in this Option to the singular or to any gender shall similarly apply to the plural or to every other gender if and when the sense requires.

b. The terms and conditions contained in this Option shall apply to and be binding upon the parties herein and their respective successors, heirs, executors, administrators and assigns.

c. This Option and any and all exhibits annexed hereto and made a part of this Option constitutes the entire agreement of the parties, and any and all other or prior agreements, representations or warranties are hereby terminated, cancelled and agreed to be void and of no force or effect. No change, amendment, deletion or addition to this Option shall be effective unless in writing and signed by the parties.

{signature pages follow}

In Testimony Whereof, we have hereunto set out hands and seals, and to a duplicate instrument of the same tenor and date, at _____, Connecticut, on the day and year first above mentioned.

Signed, Sealed and
Delivered in the
Presence of:

OPTIONOR:
DWW SOLAR II, LLC

_____ by:_____L.S.

Duly Authorized

_____

STATE OF CONNECTICUT}
} ss.
COUNTY OF }

On this ____ day of _____, 2018, before me, the undersigned officer, personally appeared _____, [position], known to me (or satisfactorily proven) to be the person whose name is subscribed to the within Instrument and acknowledged that he executed the same as his free act and deed and the free act and deed of the limited liability company for the purposes therein contained.

In Witness Whereof, I hereunto set my hand and official seal.

_____
Commissioner of the Superior Court
Notary Public
My Commission Expires:

OPTIONEE:
TOWN OF SIMSBURY

_____ By:_____.

Duly Authorized

_____

STATE OF CONNECTICUT}
} ss.
COUNTY OF }

On this ____ day of _____, 2018, before me, the undersigned officer, personally appeared _____ [position], known to me (or satisfactorily proven) to be the person whose name is subscribed to the within instrument and acknowledged that he executed the same as his free act and the free act and deed of the corporation for the purposes therein contained.

In Witness Whereof, I hereunto set my hand and official seal.

_____
Commissioner of the Superior Court
Notary Public
My Commission Expires:

Record and Return to:

_____
_____
_____
Attn: _____

QUIT CLAIM DEED

TO ALL PEOPLE TO WHOM THESE PRESENTS SHALL COME, GREETING:

KNOW YE, THAT DWW SOLAR II, LLC, a Delaware limited liability company with an address at c/o D.E. Shaw & Co. L.P., 1166 Avenue of the Americas, 9th Floor, New York, New York 10036 (herein designated as the "**Grantor**"),

for the consideration of **ONE DOLLAR ($1.00)** and other good and valuable consideration received to the Grantor's full satisfaction of **THE TOWN OF SIMSBURY, CONNECTICUT**, with an address at c/o Town Hall, 933 Hopmeadow Street, Simsbury, Connecticut 06070 (herein designated as the "**Grantee**"),

does hereby give, grant, bargain, sell and convey unto said Grantee and unto the Grantee's successors and assigns forever, with QUIT CLAIM COVENANTS, all of Grantor's interest in and to the real property commonly known as **_____ Hoskins Road, Town of Simsbury, County of Hartford and State of Connecticut** on an "AS-IS", "WHERE-IS" basis and without any representation or warranty, express or implied, of any kind, which real property is more particularly set forth and described on the attached **Exhibit A** (the "**Premises**").

No further text on this page – signature page follows

IN WITNESS WHEREOF, the Grantor has hereunto set its hand and seal this _____ day of _____, 20____.

Signed, Sealed and Delivered
in the Presence of:

GRANTOR:

DWW SOLAR II, LLC,
a Delaware limited liability company

_____

_____

By: _____
Name:
Title:

STATE OF _____)
) ss:
COUNTY OF _____)

On this the ___ day of _____, 20___, before me, the undersigned officer, personally appeared _____, who acknowledged himself/herself to be the _____ of **DWW SOLAR II, LLC**, a Delaware limited liability company, and that he/she as such officer, having been authorized so to do, executed the foregoing instrument for the purposes therein contained and for and on behalf of such limited liability company by signing the name of the limited liability company by himself/herself as such officer.

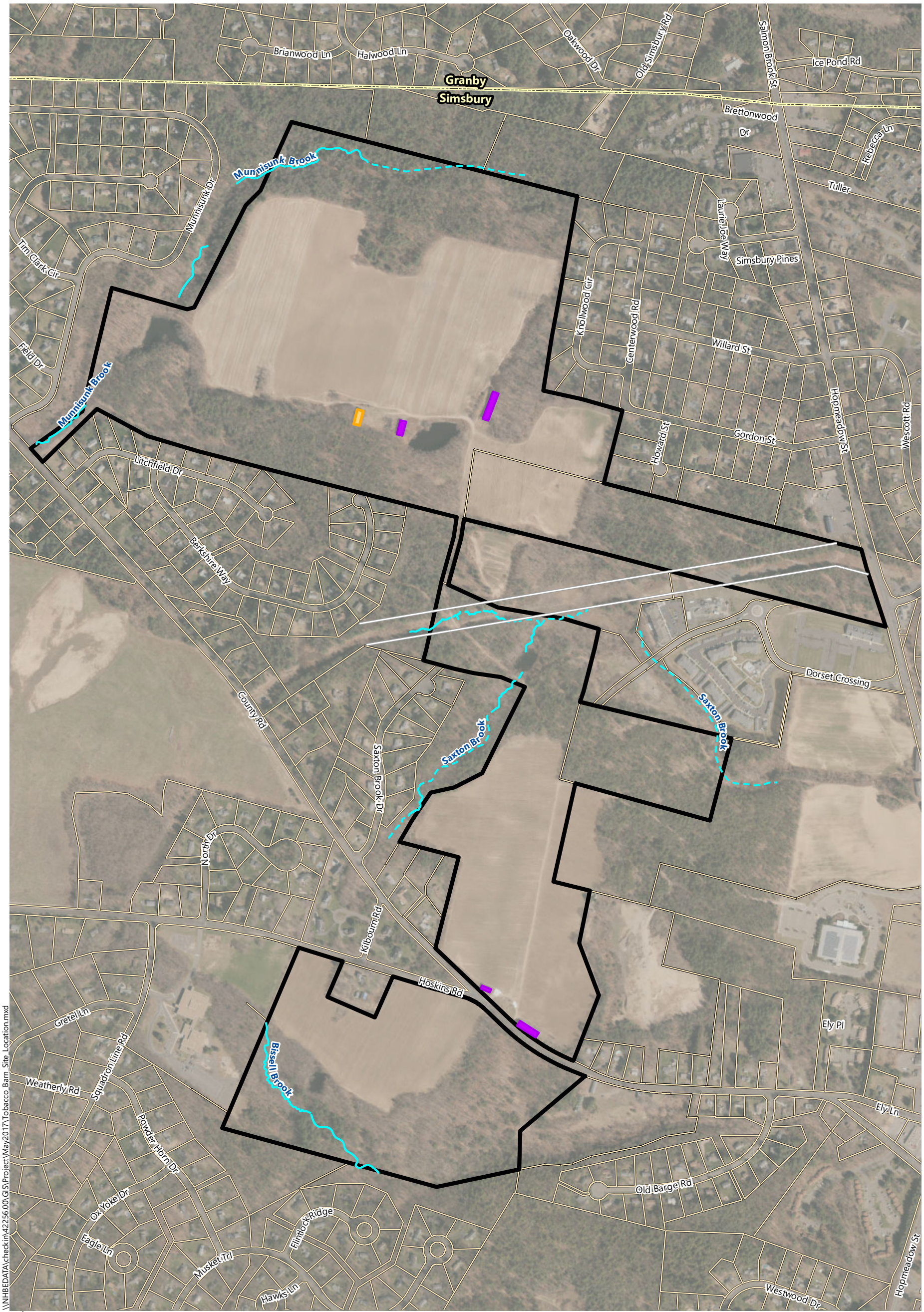
IN WITNESS WHEREOF, I hereunto set my hand.

_____
Commissioner of the Superior Court
Notary Public
My Commission Expires:

EXHIBIT A

[Legal Description]

March 20, 2018



\\NHBEDATA\checkin\42256.00\GIS\Project\May2017\Tobacco_Barn_Site_Location.mxd

0 325 650 1300 Feet

- Property Boundary
- Adjacent Parcels
- Town Boundary
- Existing Eversource ROW
- Stream
- Approximate Stream
- Barn to be Retained
- Barn to be Removed

Tobacco Valley Solar | Simsbury, Connecticut

Tobacco Barn Location Map

Source: VHB, CTDEEP, ESRI

Environmental Design & Research,
Landscape Architecture, Engineering & Environmental Services, D.P.C.

217 Montgomery Street, Suite 1000, Syracuse, New York 13202
P. 315.471.0688 • F. 315.471.1061 • www.edrdpc.com

July 27, 2018

DWW Solar II LLC
c/o Ms. Aileen Kenney
56 Exchange Terrace Suite 300
Providence, RI 02903

RE: Tobacco Valley Solar Farm
EDR Project No. 17057

Dear Ms. Kenney:

The following is a summary of the proposed visual screening plan for the Tobacco Valley Solar Project (the Project) completed by Environmental Design & Research, Landscape Architecture, Engineering & Environmental Services, D.P.C. (EDR) on behalf of DWW Solar II, LLC (DWW). We believe the plan is responsive to the comments provided by Mr. James Rabbitt Director of Planning and Development at the Town of Simsbury during a meeting between Mr. Rabbitt, Gordon Perkins or EDR and Susan Moberg of VHB in Simsbury on April 6, 2018.

The attached plan sheets last revised 7/27/2018, 10 sheets depict the proposed visual screening plan. In general, the screening plan has been designed to minimize and mitigate perceived visual impacts of the Project by increasing setbacks and adding proposed plantings/screening techniques.

Area 2A – Hoskins Road and County Road

In this location the screening plan proposes to retain the two existing barns and construct a 2-foot to 3-foot berm with an evergreen and deciduous planting buffer between the barns. Additionally, a linear street tree arrangement will be planted along Hoskins Road. In earlier screening concepts, this buffer was made up of Crab Apple trees, in an effort to provide visual interest and reduce the potential for conflict with overhead wires. Mr. Rabbitt noted in the April 6 meeting, that the Town would prefer the use of Thundercloud Cherry Plum, but our subsequent research revealed that this plant may become invasive, is susceptible to disease, and may have a short life span. Alternatives to this tree could include Serviceberry and Crusader Hawthorn. Both are native species that would provide year-round visual interest and wildlife habitat.

Deciduous trees are proposed to be installed at 1 ½ inch caliper and evergreen trees will be between 5-7 feet tall at the time of installation. Trees of this size are ideal for reducing transplanting stress, increased survival rates, and better establishment in new soils.

The berm will consist of an undulating landform between the two existing barns on Hoskins Road. This berm will not conflict with the existing grades around the barns or substantially modify site drainage patterns. Additionally, a low cedar post fence will follow the foot of the berm to provide a human scale element closer to Hoskins Road (Sheets 1 and 2 of 10).

Area 2B – Hoskins Road

In this location, DWW is proposing in-fill planting to reinforce the existing, healthy stand of White Pine which currently provides partial screening of the Project site. This will include a continuation of the street tree plantings found in Area 2A, as well as additional evergreen and deciduous trees and shrubs to provide year-round visual interest and screening of the Project (Sheet 3 of 10). A berm is not deemed necessary at this location because of the considerable difference in elevation between the road and the site which already provides an obstruction of views into the site for westbound vehicles. Mr. Rabbitt concurred with this assessment regarding the berm.

Area 4 – County Road

At Mr. Rabbitt's request, the plan includes the installation of a residential-scale entrance to the solar facility. This will include 3 small sections of cedar fence (Sheet 10 of 10) with integrated plantings of Northern Bayberry and Red Chokeberry, which are small shrubs (5-10 feet) that will provide year-round visual interest. Additionally, a gate (examples on Sheet 10 of 10), will be installed approximately one vehicle length from the road to provide a safe pull off from County Road for entering/exiting vehicles.

Additionally, the existing hedgerow along County Road and along the abutting properties will be reinforced with a mix of evergreen and deciduous trees to increase the screening between viewers and the Project (Sheet 4 and 5 of 10).

Areas 5, 6, and 7 – Howard and Gordon Streets and Knollwood Circle

Adjacent to these neighborhoods, the plan incorporates a dense planting scheme which, in time, will provide a visual buffer between the Project and abutting residents. This will include native evergreen and deciduous species that will provide wildlife and cover, visual screening, and year-round visual interest (Sheet 6 of 10). Larger species such as Colorado Blue Spruce and Eastern Red Cedar will provide a backdrop to smaller species such as Northern Bayberry, Fragrant Sumac, Mountain Laurel, and Red Chokeberry. Additionally, deciduous trees such as Swamp Oak and Service Berry and Crusade Hawthorn will add variety and habitat to the planted buffer.

Area 8 – County Road and Litchfield Drive

This location includes the Project entry and construction access road. As described above at Area 4, at this location the plan incorporates a residential scale entrance and gate to soften the look of the entry road. Additionally, during construction, a temporary 5-6-foot-tall construction screen will be installed between the access road and the abutting properties as shown on Sheet 1. Examples of the temporary fence are provided on Sheet 10. The existing vegetative buffer along the site access road will be evaluated to determine the need for additional evergreen tree plantings to minimize lines of sight toward the proposed Project from the adjacent residents on Litchfield Drive.

It is important to note that the traffic during construction will be split between the temporary entrance on Hoskins Road and this entrance. Additionally, construction is expected to occur over a period of 6-8 months, so any potential construction-related visual impacts will be of relatively short duration.

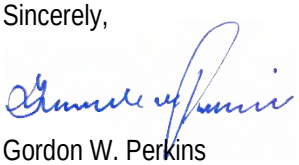
Area 9 – Berkshire Way

In this location, there is a 200-foot existing vegetated buffer which will be left intact throughout the duration of the Project construction and operation. The clearing of vegetation in this area does not include grubbing (the removal of

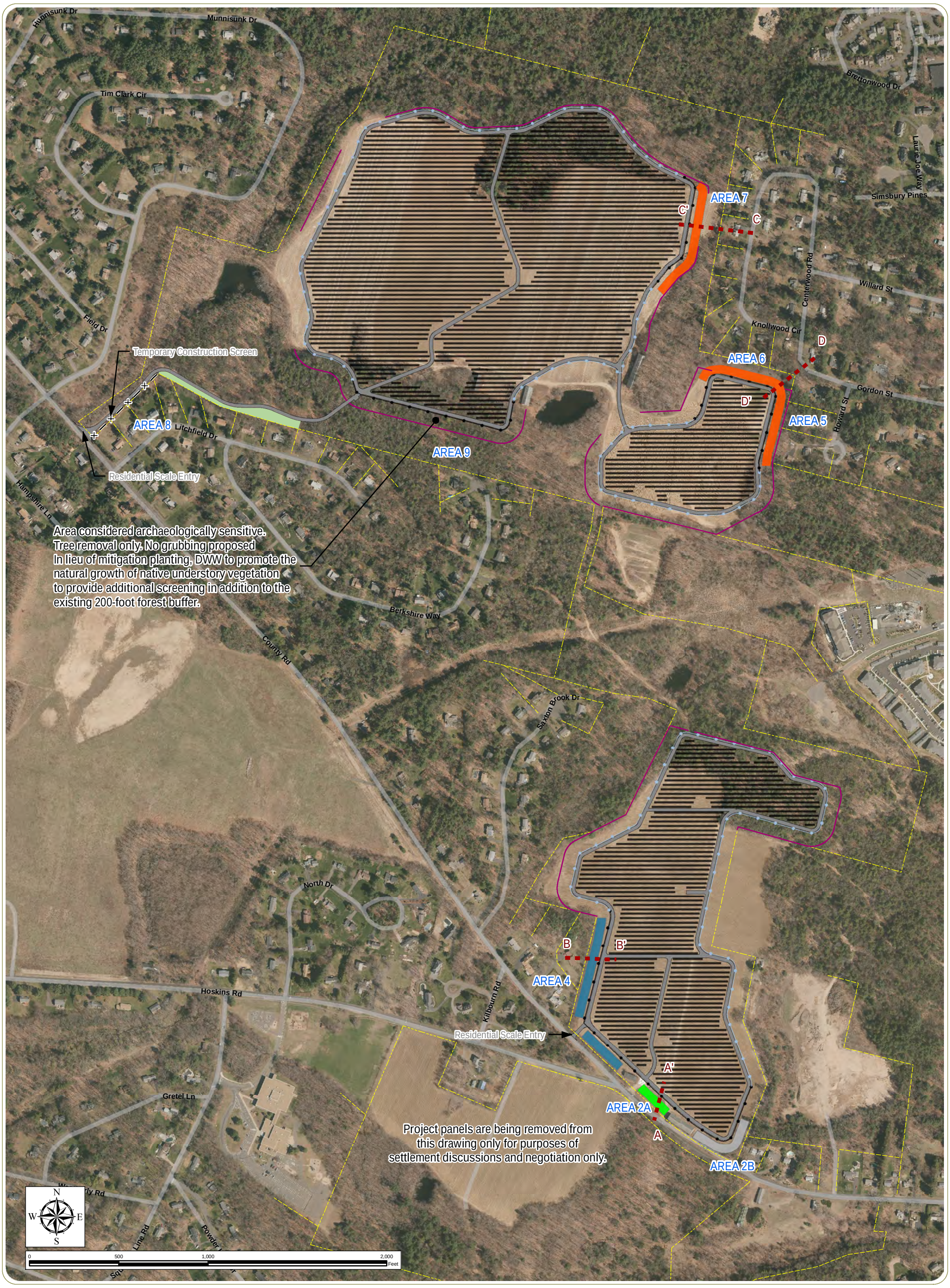
stumps) due to the potential archeological sensitivity of this location. For the same reasons, trees and shrubs cannot be planted in this location since this soil disturbance may impact the potential archaeological resources. However, it can be anticipated that opportunistic understory growth will take advantage of the increased light exposure resulting from canopy removal. The screening plan incorporates management of this understory growth to selectively promote native species of shrubs, which will provide an additional level of Project screening. Off-site mitigation at the two closest abutting properties on Berkshire Way may ultimately be a more effective visual screen as previously discussed with abutters.

As recommended by the Town for use adjacent to the above-described mitigation plantings, the plan includes black vinyl coated chain link fence (rather than galvanized fencing) for required perimeter security fencing. Examples of the use of this material at an existing solar facility in upstate New York are provided on Sheet 10.

Sincerely,



Gordon W. Perkins
Senior Project Manager/Visualization Specialist



Tobacco Valley Solar Project

Town of Simsbury, Hartford County, Connecticut

Mitigation Modules

Sheet 1 of 10

Notes: 1. Basemap: 2017 CT Aerial Photos. 2. This map was generated in ArcMap on July 27, 2018.
3. This is a color graphic. Reproduction in grayscale may misrepresent the data.

Mitigation Areas:
- Area 2A
- Area 2B
- Area 4
- Areas 5, 6, & 7
- Area 8 (Infill Planting Where Needed)

- Vegetation Clearing Limit
- Line of Sight
- Property Line

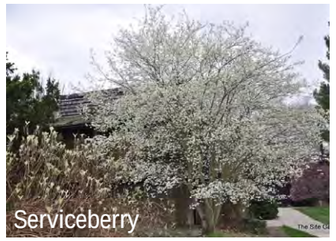
- Access Road
- Black Vinyl-Coated Chain Link Fence
- Galvanized Chain Link Fence
- Proposed Solar Panels

Original Date: 4/6/2018
Revision Date: 7/27/2018
EDR Project Number: 17057

www.edrdpc.com

PLANT LIST

KEY	BOTANICAL NAME	COMMON NAME	PLANTING SIZE	TYPE	SPACING	MATURE HT.	MATURE WIDTH
	AMELANCHIER CANADENSIS	SERVICEBERRY	6-7' HT.	B&B	AS SHOWN	25' - 30'	15' - 20'
	CRATAEGUS CRUS-GALLI INERMIS 'CRUSADER'	CRUSADER HAWTHORN	1-¼" CAL.	B&B	AS SHOWN	15' - 20'	12' - 15'
	PICEA PUNGENS	COLORADO SPRUCE	6 - 7' HT.	B&B	AS SHOWN	30' - 60'	10' - 20'
	THUJA PLICATA 'GREEN GIANT'	GREEN GIANT WESTERN RED CEDAR	5 - 6' HT.	B&B	AS SHOWN	50' - 70'	15' - 25'
	JUNIPERUS VIRGINIANA	EASTERN RED CEDAR	5 - 6' HT.	B&B	10' ON-CENTER	25' - 35'	10' - 12'
	KALMIA LATIFOLIA 'KEEPSAKE'	KEEPSAKE MOUNTAIN LAUREL	30 - 36" HT.	B&B	5' ON-CENTER	4' - 5'	5' - 6'
	ILEX GLABRA	INKBERRY	18 - 24" HT.	B&B	6' ON-CENTER	5' - 8'	5' - 6'
	ARONIA ARBUTIFOLIA	RED CHOKEBERRY	18 - 24" HT.	B&B	4' ON-CENTER	6' - 10'	3' -5'


Serviceberry


Eastern Red Cedar


Crusader Hawthorn


Mountain Laurel


Colorado Spruce

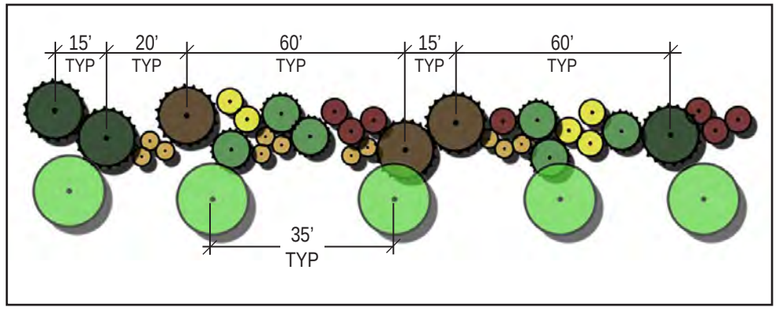

Inkberry


Green Giant Arborvitae


Red Chokeberry

AREA 2A - PLANTING MODULE LAYOUT

1. DIMENSIONS ARE APPROXIMATE AND MAY NEED TO BE FIELD ADJUSTED BASED ON EXISTING CONDITIONS.
2. REFER TO PLANTING SCHEDULE FOR SHRUB SPACING.



Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 2 of 10

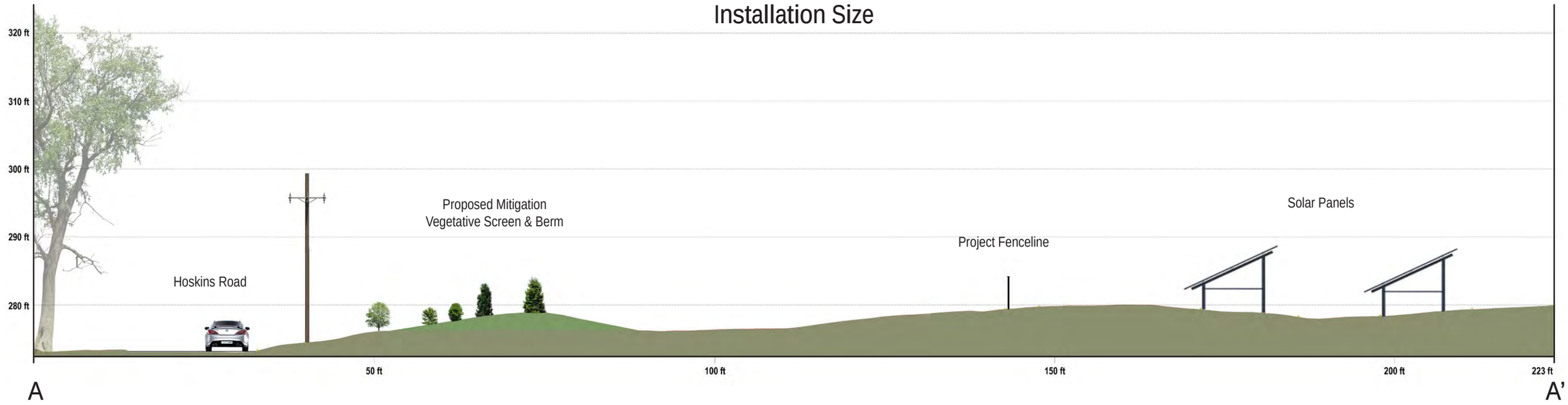
50 25 0 25 50 100
Graphic Scale (in feet)

North

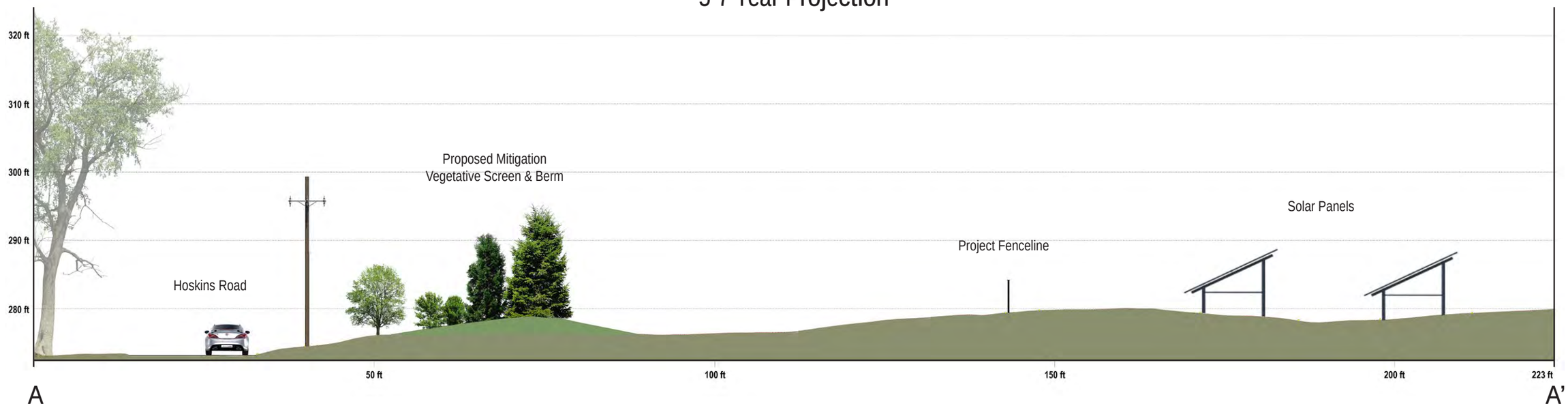
Area 2A - Proposed Planting Module

Original Date: 4/6/2018
Revision Date:7/27/2018
EDR Project Number: 17057

Installation Size



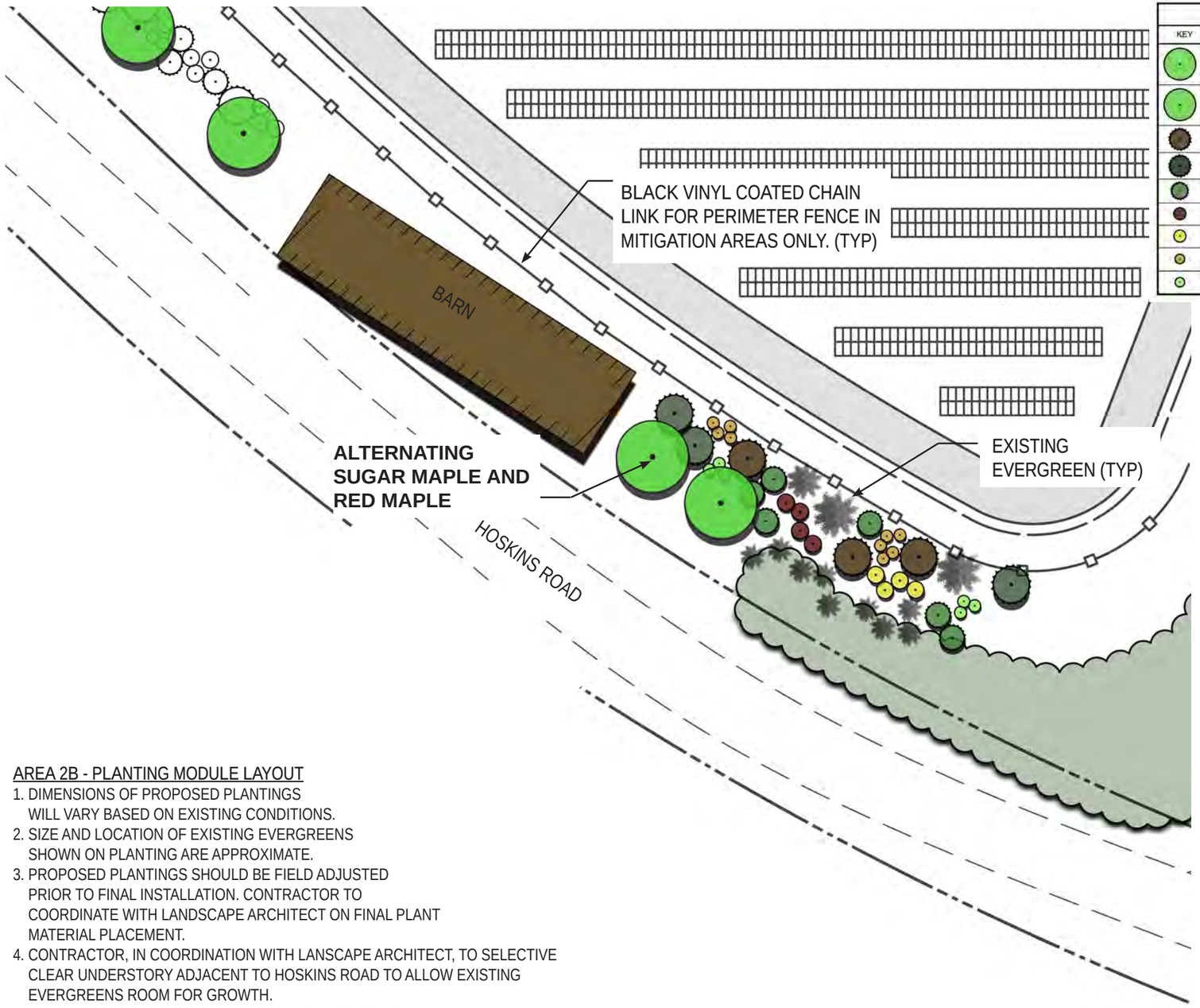
5-7 Year Projection



Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 3 of 10

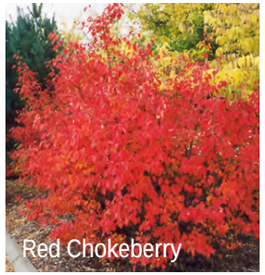
Area 2A - Proposed Planting Module

Original Date: 4/6/2018
Revision Date: 4/25/2018
EDR Project Number: 17057



PLANT LIST

KEY	BOTANICAL NAME	COMMON NAME	PLANTING SIZE	TYPE	SPACING	MATURE HT.	MATURE WIDTH
	ACER RUBRUM OCTOBER GLORY	RED MAPLE	3" CAL.	B&B	AS SHOWN	40' - 50'	35' - 40'
	ACER SACCHARUM 'LEGACY'	SUGAR MAPLE	3" CAL.	B&B	AS SHOWN	40' - 50'	35' - 40'
	PICEA PUNGENS	COLORADO SPRUCE	6 - 7' HT.	B&B	AS SHOWN	30' - 60'	10' - 20'
	THUJA PLICATA 'GREEN GIANT'	GREEN GIANT WESTERN RED CEDAR	5 - 6' HT.	B&B	AS SHOWN	50' - 70'	15' - 25'
	JUNIPERUS VIRGINIANA	EASTERN RED CEDAR	5 - 6' HT.	B&B	10' ON-CENTER	25' - 35'	10' - 12'
	KALMIA LATIFOLIA 'KEEPSAKE'	KEEPSAKE MOUNTAIN LAUREL	30 - 36" HT.	B&B	5' ON-CENTER	4' - 5'	5' - 6'
	ILEX GLABRA	INKBERRY	18 - 24" HT.	B&B	6' ON-CENTER	5' - 8'	5' - 8'
	ARONIA ARBUTIFOLIA	RED CHOKEBERRY	18 - 24" HT.	B&B	4' ON-CENTER	6' - 10'	3' - 5'
	RHUS AROMATICA	FRAGRANT SUMAC	18 - 24" HT.	B&B	6' ON-CENTER	2' - 6'	6' - 10'



AREA 2B - PLANTING MODULE LAYOUT

1. DIMENSIONS OF PROPOSED PLANTINGS WILL VARY BASED ON EXISTING CONDITIONS.
2. SIZE AND LOCATION OF EXISTING EVERGREENS SHOWN ON PLANTING ARE APPROXIMATE.
3. PROPOSED PLANTINGS SHOULD BE FIELD ADJUSTED PRIOR TO FINAL INSTALLATION. CONTRACTOR TO COORDINATE WITH LANDSCAPE ARCHITECT ON FINAL PLANT MATERIAL PLACEMENT.
4. CONTRACTOR, IN COORDINATION WITH LANSCAPE ARCHITECT, TO SELECTIVE CLEAR UNDERSTORY ADJACENT TO HOSKINS ROAD TO ALLOW EXISTING EVERGREENS ROOM FOR GROWTH.
5. REFER TO PLANTING SCHEDULE FOR SHRUB SPACING.

Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 4 of 10

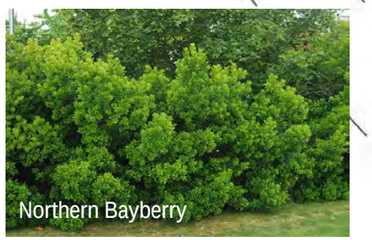
50 25 0 25 50 100
Graphic Scale (in feet)

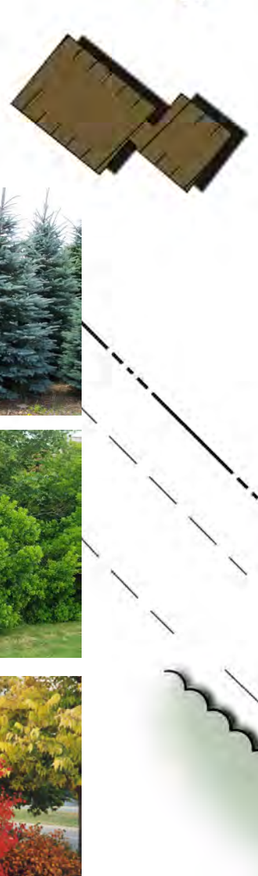
North

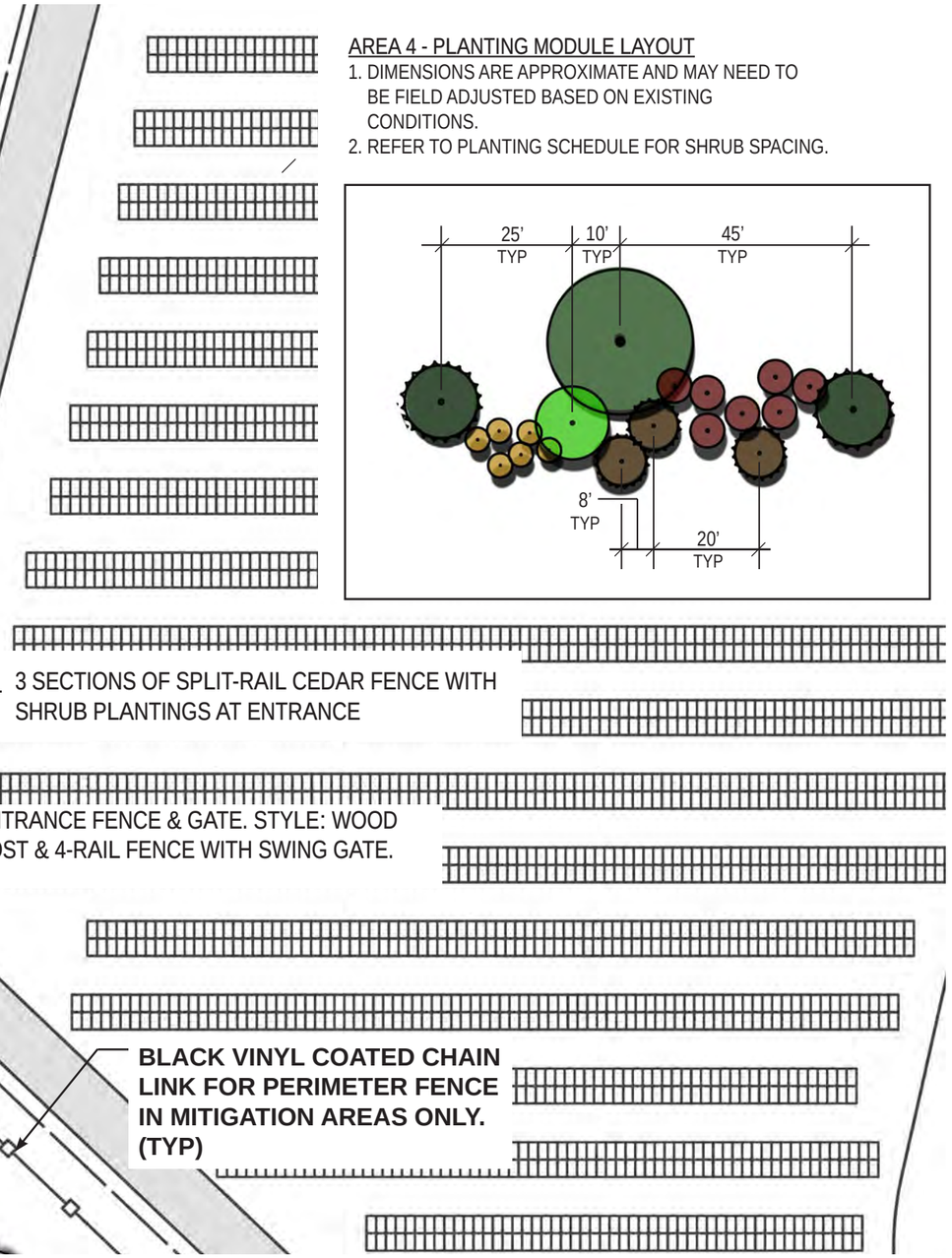
Area 2B - Proposed Planting Module

Original Date: 4/6/2018
Revision Date:7/27/2018
EDR Project Number: 17057

PLANT LIST							
KEY	BOTANICAL NAME	COMMON NAME	PLANTING SIZE	TYPE	SPACING	MATURE HT.	MATURE WIDTH
	QUERCUS COCCINEA	SCARLET OAK	1½" CAL.	B&B	AS SHOWN	50' - 70'	40' - 50'
	OSTRYA VIRGINIANA	EASTERN HOPHORNBEAM	1½" CAL.	B&B	AS SHOWN	25' - 40'	20' - 30'
	PINUS STROBUS	EASTERN WHITE PINE	6 - 7' HT.	B&B	AS SHOWN	50' - 80'	20' - 40'
	PICEA PUNGENS	COLORADO SPRUCE	6 - 7' HT.	B&B	AS SHOWN	30' - 80'	10' - 20'
	MORELLA (MYRICA) PENSYLVANICA	NORTHERN BAYBERRY	30 - 36" HT.	B&B	8' ON-CENTER	5' - 10'	5' - 10'
	ARONIA ARBUTIFOLIA	RED CHOKEBERRY	18 - 24" HT.	B&B	4' ON-CENTER	6' - 10'	3' -5'







AREA 4 - PLANTING MODULE LAYOUT

1. DIMENSIONS ARE APPROXIMATE AND MAY NEED TO BE FIELD ADJUSTED BASED ON EXISTING CONDITIONS.
2. REFER TO PLANTING SCHEDULE FOR SHRUB SPACING.

Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 5 of 10

50 25 0 25 50 100
Graphic Scale (in feet)

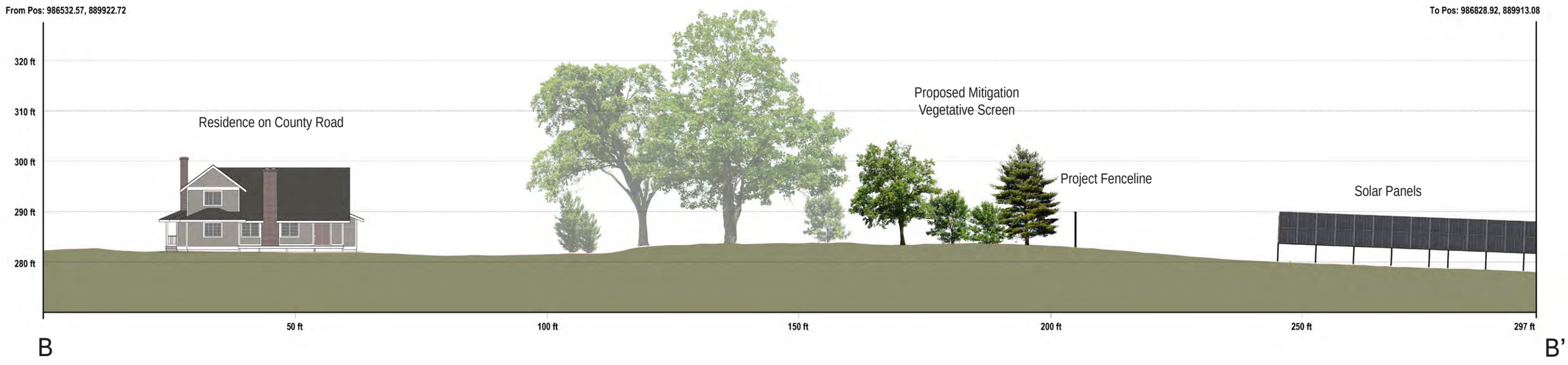
North

Area 4 - Proposed Planting Module

Original Date: 4/6/2018
Revision Date:7/27/2018
EDR Project Number: 17057

Installation Size

5-7 Year Projection



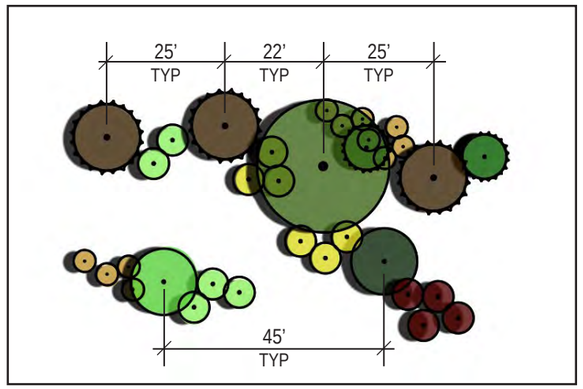
Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans

Area 4 - Proposed Planting Module

Original Date: 4/6/2018
Revision Date: 4/25/2018
EDR Project Number: 17057

AREA 5,6 & 7 - PLANTING MODULE LAYOUT

1. DIMENSIONS ARE APPROXIMATE AND MAY NEED TO BE FIELD ADJUSTED BASED ON EXISTING CONDITIONS.
2. REFER TO PLANTING SCHEDULE FOR SHRUB SPACING.

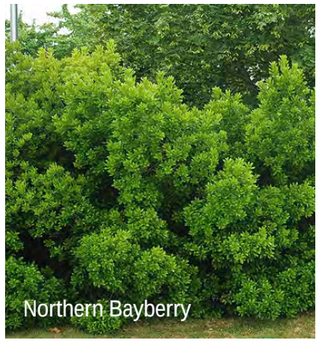


PLANT LIST

KEY	BOTANICAL NAME	COMMON NAME	PLANTING SIZE	TYPE	SPACING	MATURE HT.	MATURE WIDTH
	QUERCUS BICOLOR	SWAMP WHITE OAK	2½" CAL.	B&B	AS SHOWN	50' - 60'	50' - 60'
	AMELANCHIER CANADENSIS	SERVICEBERRY	6-7' HT.	B&B	AS SHOWN	25' - 30'	15' - 20'
	CRATAEGUS CRUS-GALLI INERMIS CRUSADER	CRUSADER HAWTHORN	1¾" CAL.	B&B	AS SHOWN	15' - 20'	12' - 15'
	PICEA PUNGENS	COLORADO SPRUCE	6 - 7' HT.	B&B	AS SHOWN	30' - 60'	10' - 20'
	JUNIPERUS VIRGINIANA	EASTERN RED CEDAR	5 - 6' HT.	B&B	10' ON-CENTER	25' - 35'	10' - 12'
	KALMIA LATIFOLIA 'KEEPSAKE'	KEEPSAKE MOUNTAIN LAUREL	30 - 36" HT.	B&B	5' ON-CENTER	4' - 5'	5' - 6'
	MORELLA (MYRICA) PENSYLVANICA	NORTHERN BAYBERRY	30 - 36" HT.	B&B	8' ON-CENTER	5' - 10'	5' - 10'
	RHUS AROMATICA	FRAGRANT SUMAC	18 - 24" HT.	B&B	6' ON-CENTER	2' - 6'	6' - 10'
	ARONIA ARBUTIFOLIA	RED CHOKEBERRY	18 - 24" HT.	B&B	4' ON-CENTER	6' - 10'	3' -5'


Swamp Oak


Colorado Spruce


Northern Bayberry


Serviceberry


Eastern Red Cedar

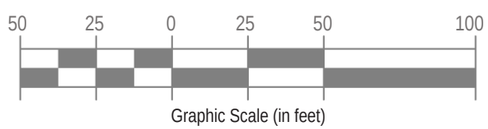

Fragrant Sumac


Crusade Hawthorn


Mountain Laurel


Red Chokeberry

Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 7 of 10

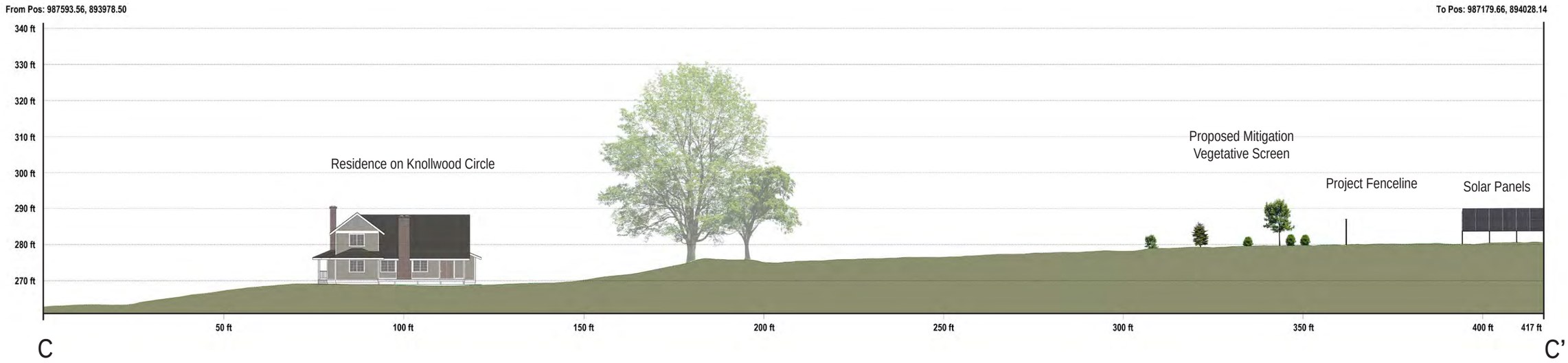



North

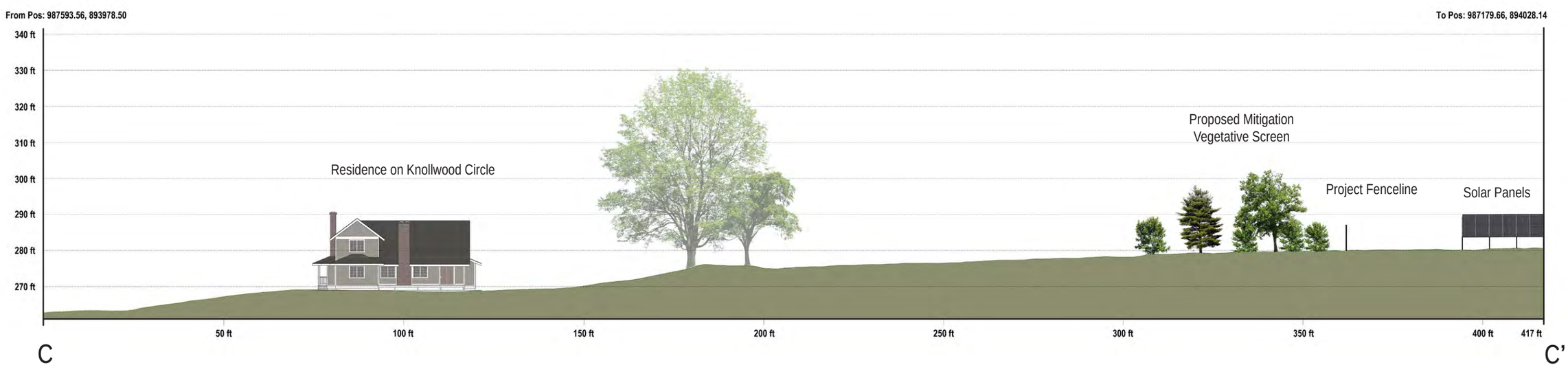
Area 5,6 & 7 - Proposed Planting Module

Original Date: 4/6/2018
Revision Date:7/27/2018
EDR Project Number: 17057

Installation Size



5-7 Year Projection

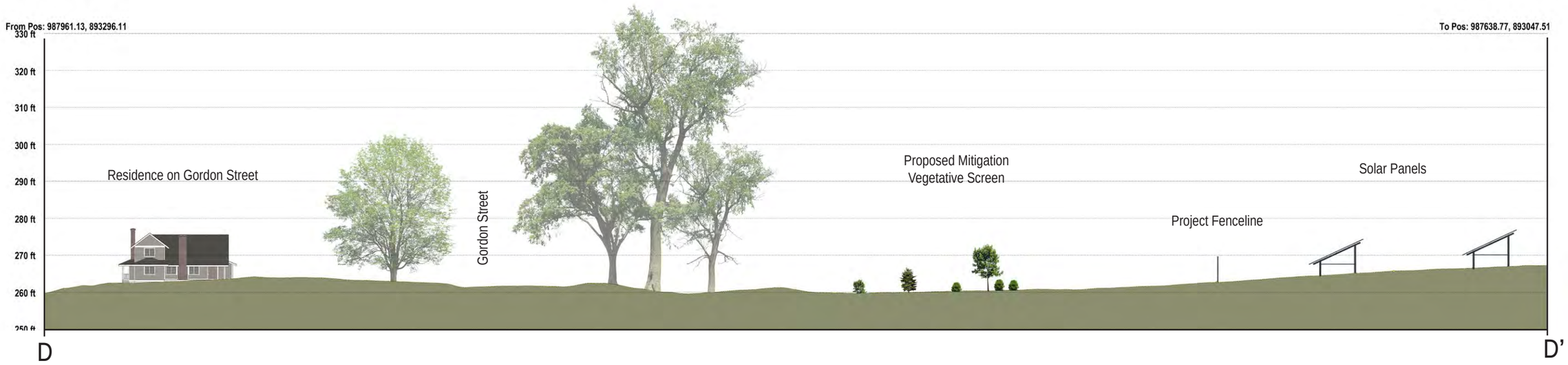


Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 8 of 10

Area 7 - Proposed Planting Module

Original Date: 4/6/2018
Revision Date: 4/25/2018
EDR Project Number: 17057

Installation Size



5-7 Year Projection

Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 9 of 10

Area 5 & 6 - Proposed Planting Module

Original Date: 4/6/2018
Revision Date: 4/25/2018
EDR Project Number: 17057

Residential Scale Gate (Areas 4 & 8)

Entry - Roadside Treatment (Areas 4 & 8)

Residential Scale Gate (Areas 4 & 8)

Residential Scale Gate (Areas 4 & 8)

Existing Solar Site Mitigation - Year 3

Existing Solar Site Mitigation - Year 3

Existing Solar Site Mitigation - Year 3

Existing Solar Site Mitigation - Year 3

Black Vinyl Coated Fence on Existing Solar Site

Black Vinyl Coated Fence on Existing Solar Site

Mowed Walking Path

Temporary Construction Screen (Area 8)

Tobacco Valley Solar
Town of Simsbury, Hartford County, Connecticut
Preliminary Planting Mitigation Plans
Sheet 10 of 10

Precedent Mitigation Material Examples

Original Date: 4/6/2018
Revision Date: 4/25/2018
EDR Project Number: 17057

Deepwater Wind Settlement Agreement

SEPTEMBER 12, 2018

Agenda

- Background
- Settlement Agreement
- Next Steps

Background

- In 2016, Connecticut, Rhode Island and Massachusetts issued a joint Clean Energy RFP for renewable energy projects of 20 megawatts or greater; Deepwater Wind's solar project, sited in Simsbury, was selected

- Under Connecticut state law, projects that would generate more than 1 megawatt of energy are permitted through the CT Siting Council and are not subject to local zoning and land use requirements

- In June 2017, the Town secured party status with the Siting Council; DWW held a public hearing in Simsbury on September 12, 2017, and the Town actively participated in the Siting Council proceedings in November 2017

Background (continued)

- On December 21, 2017, the CT Siting Council ruled that no Certificate of Environmental Compatibility and Public Need is required for the project

- On January 22, 2018, the Board of Selectmen voted unanimously to appeal the Siting Council's ruling in order to retain the right to appeal the decision at a later time if the Town's concerns were not adequately addressed through negotiations with DWW

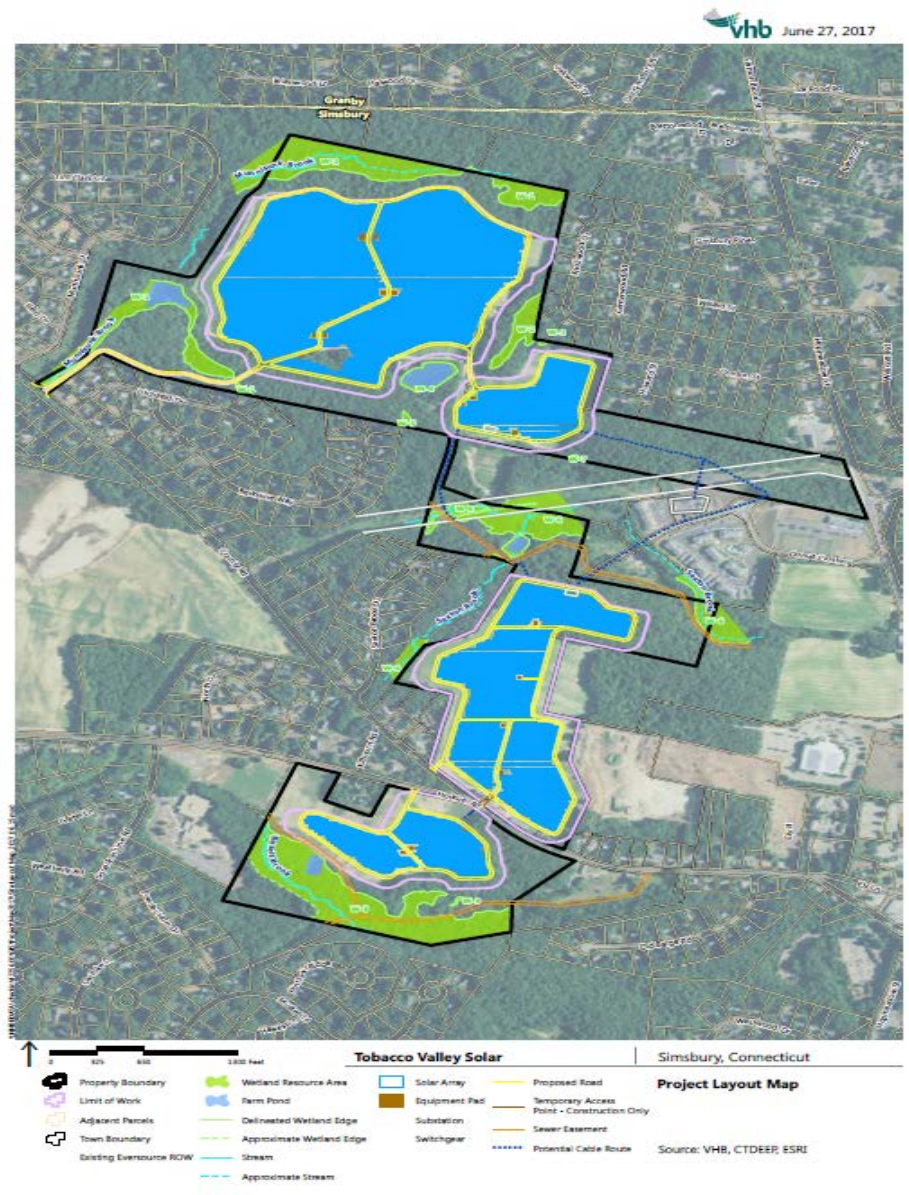
Settlement Agreement

The Town had two primary objectives:

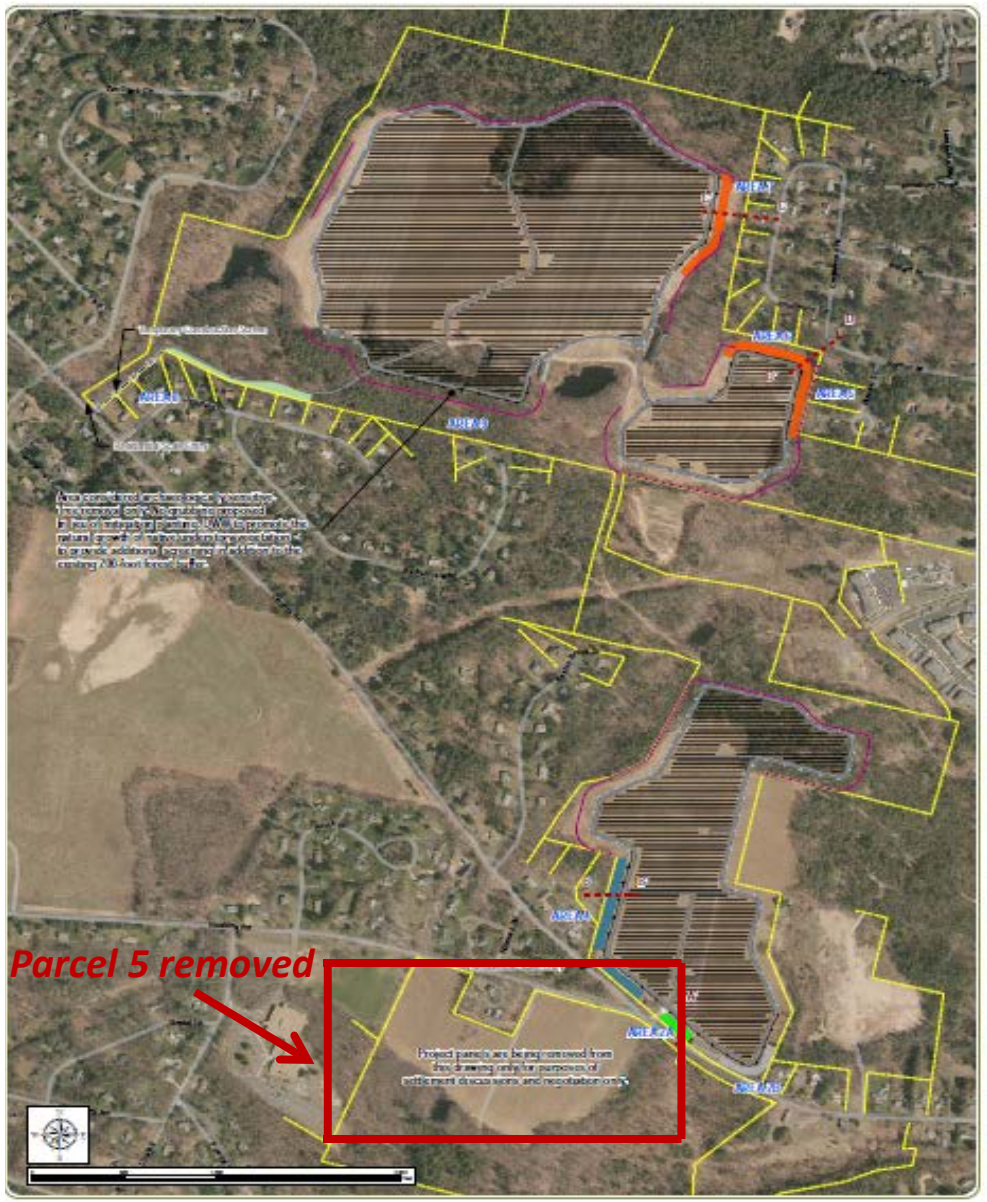
1. Protect public health and safety
   - Soil and water quality

2. Minimize the impact to the neighboring community
   - No development on Parcel 5 (south of Hoskins)
   - Appropriate screening and landscaping, including preservation of barns
   - Decommissioning process
   - Guaranteed option to purchase

Approved Plan



Amended Plan



Soil Management Plan

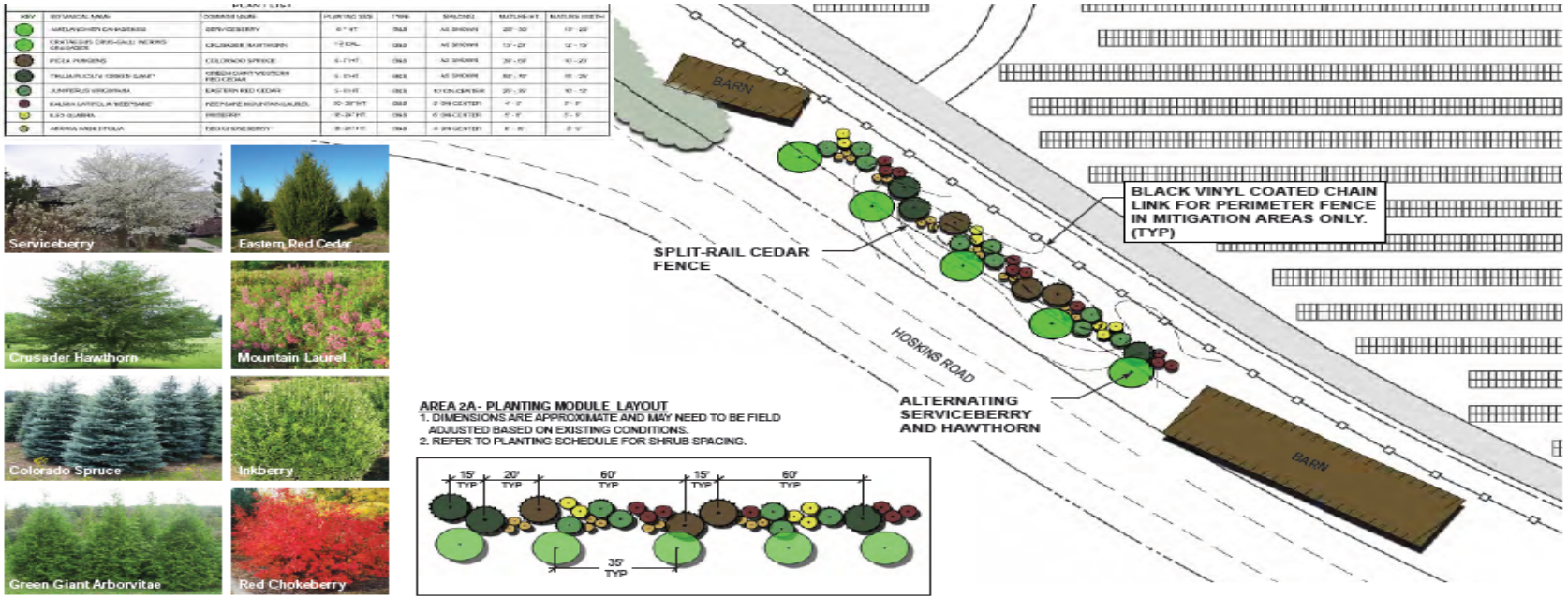
- During excavation activities, contractor will be required to comply with the following:
  - Testing of materials potentially requiring special management (farm dump or buried debris) encountered during excavation. Coordinate with the contractor and consultant for this.
- Comply with requirements of the following:
  - CTDEEP General Permit for Discharge of storm water during construction (including water quality testing or discharge)
  - CTDEEP 2002 Erosion and Sediment Control Guidelines
  - Dust Control
  - Classification of soils for onsite reuses
  - Compliance with Health and Safety Plan

Water Testing

- DWW will perform a well receptor survey which will identify wells providing potable water within a 500 ft radius of the project
- DWW will test wells of home owners that grant permission within the area identified in the well receptor survey for the following compounds: EDB, DBCP and 123TCP by EPA Method 504.1 or Method 524.3, VOCs by EPA Method 524.2, chlorinates pesticides by EPA Method 8081, and the metals arsenic and copper by EPA Method 6010C.
- Testing of wells (with home owner's permission) would occur prior to start and finish of construction activities
  - Results from the testing will be shared with The Town of Simsbury and homeowners
  - Home owners are responsible to notify CTDEEP when result exceed the MDLs
- Town has committed to pay up to $25,000 toward survey and testing

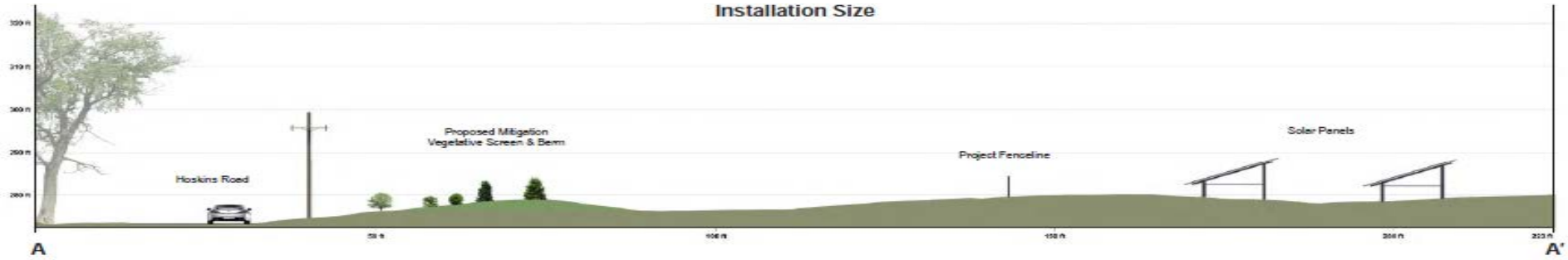
Visual Screening

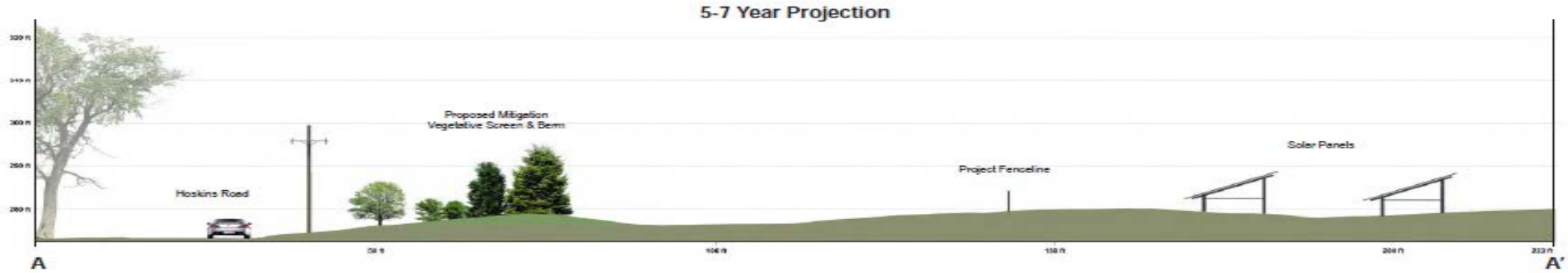
DWW has developed a robust screening plan from the initial submission. Methods such as native plantings, berms, split rail fencing and coating of interior fencing will be implemented in order to minimize the visual impact of the development.



Visual Screening (continued)

Visual simulations have been prepared to illustrate how the methods of screening will look at the time of installation and in years 5-7. Two existing Barns along Hoskins Road will be retained and maintained.





Next Steps

- Town and DWW will execute Release and Settlement Agreement and Option to Purchase Agreement
- DWW will submit its development and management plan to the CT Siting Council for approval
- Local and state agency permitting process
- Construction
  - Approximately 18 months
  - Water testing
  - Soil management

Next Steps

- Long-term
  - Decommissioning
  - Option to Purchase Land