Enclosure B to letter from EPA to Westates
dated 25 September 2003

EPA Comments on Westates' Quality Assurance Project Plan (QAPP)

Background

This enclosure provides comments on the Quality Assurance Project Plan (QAPP) which Westates submitted to EPA on May 30, 2003. We performed a detailed review of the QAPP and of the response to comments accompanying the QAPP.

General Items

COMMENT:
1. As for the CPT Plan, please submit the revised QAPP in both hard copy and electronic form (PDF is acceptable).

Also as for the CPT Plan, please indicate revisions in the text of the revised QAPP using annotations such as strike-out of removed text and red-lining of new text, along with a "clean" copy of the revised QAPP. Please also submit a response to comments to accompany the revised QAPP, providing detailed rationale and explanations in response to these comments, and indicating what portions of the QAPP were revised.

RESPONSE:
The revised QAPP information will be submitted in both hard copy and electronic form (PDF).

The revised QAPP will be submitted both as a clean copy and a "marked up" copy that will indicate all revisions. This letter is being submitted as the response to comments and will indicate after each comment what revisions were made.

COMMENT:
2. The QAPP was reviewed in terms of the guidance provided in the following documents:

"EPA Requirements for Quality Assurance Project Plans," (EPA QA/R-5, March 2001)

"Guidance for Quality Assurance Project Plans" (EPA QA/G-5, December, 2002)

"Guidance for the Data Quality Objectives Process" (EPA QA/G-4, August, 2000).

RESPONSE:
Noted.

COMMENT:
3. The QAPP was prepared following the older QAPP format in QAMS 005/80 (Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans) which was superseded by the more current "R-5" referenced above over ten years ago. However, the information provided in the QAPP

covers all relevant R-5 requirements, so this is not a problem. A number of clarifications or minor issues were identified and are noted below.

RESPONSE:
No revisions are being made based on this comment, however, the information above will be taken into account during the development of future QAPPs.

COMMENT:
4. Because of the large number of tests and associated quality control (QC) measures associated with them, a separate section on QA should be provided in the final test report.

RESPONSE:
The final test report will contain a separate section on QA. The plan presently includes an example test report outline, Figure 11-1, that list Section 5.0 Quality Assurance/Quality Control Results and includes several subsections.

Comments regarding the QAPP

COMMENT:
5. [Test Plan; Table 6-1, Anticipated Daily Schedule for Performance Test] The test schedule outlined in Table 6-1 is both ambitious and highly dependent on optimal operating conditions to execute. Generally access to sampling ports is physically constrained to relatively small platforms and a small number of ports, yet the sampling effort planned requires that several Modified Method 5 (MM5) Trains, or variants thereof, all be operational at the same time. Whether Method 0010, 0023A, 0061, 0026A, 0029, etc., a long probe and multiple impingers must be set up, leak tested, and run, many of them simultaneously. There are other non MM5 trains like the volatile Organic Sampling Train (VOST) which must be operational as well. In some cases there is only a half hour between runs. Even assuming that the contractor provides the number of personnel and equipment required to conduct the tests (for example, how will tests for Methods 1-4 be conducted on several ports at once?), it seems unlikely there would be sufficient room for the different teams and their probes to move about. The Test Plan, or Sampling Procedures part of the plan (Attachment A) should discuss how the different tests will be carried out simultaneously and what probability there is that the schedule in Table 6-1 can be met.

RESPONSE:
WCAI agrees that the testing schedule is aggressive. However, WCAI also feels that with the amount of data gathering being required (mostly to support the risk assessment activities) it is important to limit the time for each run as much as possible to ensure that operating conditions do not vary significantly from the beginning of the run to the end. WCAI's contractors have experience with numerous performance tests where a large number of sampling trains have been operated simultaneously. They have worked to develop the schedule shown in the test plan based on their previous experience with other facilities and with the WCAI stack and access platforms. In fact, WCAI plans to make several platform modifications (either permanently or using temporary scaffolding) to accommodate the needs of the testing crews and equipment.

If appropriately planned, each pair of sampling ports installed on the stack for isokinetic sampling can be used for two sampling trains. For example, the M0010-SV train for semivolatile organics and OC Pesticides and the M0010-P sampling train for PAHs and PCBs will each be operated for 180 minutes. It is planned that these two trains would share a single set of sampling ports (designated as "A" and "B", located in the same plane at 90 degree separation). Assuming that there are 6 sampling points on each

traverse, the M0010-SV train will start the run in port "A" a point 1, while the M0010-P train will start the run in port "B" at point 6. During the course of the run, the M0010-SV train will be moved into the stack for each new point, while the M0010-P train will be moved out of the stack for each new point. At the half-way point of the run (after 6 points have been sampled) the trains will each change ports and complete the second half of the run in the same sequence as was conducted during the first half.

If such a system is used, three sets of sampling ports (if properly spaced) can accommodate six isokinetic sampling trains.

Regarding the specific comment questioning the ability to conduct Methods 1-4 on several ports at once, this is common practice since each isokinetic sampling train incorporates a pitot tube, thermocouple, and impingers. Further, gas bags for Orsat analysis can be collected from the exhaust of each sampling train or a CEM can be used for O_2 and CO_2 determinations for multiple trains. Thus, Method 1 is conducted for each sample location (prior to the test) to determine the appropriate number of sampling points on a traverse. Method 2 is conducted on each isokinetic sampling train using the pitot tube and thermocouple attached to the probe (along with data from Methods 3 and 4). Method 3 is conducted from the Orsat bag from each isokinetic train or using the CEMS. Method 4 is conducted by determining the moisture gain in the impingers of each isokinetic train.

COMMENT:

6A. [Attachment A, Sampling Procedures; Table A-1, Spent Activated Carbon Sampling Procedure] It is recommended that volatile organic compound samples be placed in 40 mL VOA vials to reduce potential losses of volatile organic compounds (VOCs). Some losses will probably occur since the containers are not hermetically sealed, and if it is felt that the levels will be high enough, methanol preservation may be considered. See also comment 9 below.

RESPONSE:

We do not feel that it is advisable to attempt to follow SW-846 Method 5035 field preservation, section 2.2.2, for the collection of the spent activated carbon samples. The manipulation of the sample in the field to collect the sample and then transfer it to the preweighed sample container containing methanol while weighing the sample to insure the correct amount of sample versus methanol is collected without spilling the methanol, could also produce volatile compound loss. We suggest filling the sample container to the brim and tightly sealing it in a sample jar with a Teflon lined lid and keeping the sample chilled during transport to the laboratory. The field methanol preservation technique was developed for preserving solid samples however, it is not known how well methanol will "pull off" the volatile compounds from the carbon. We feel that methanol dispersion of the sample when performed, is better done in the laboratory under more controlled conditions as described in section 2.2.1 of this method.

It is also much easier to place the spent activated carbon into a wide-mouth jar than a standard 40-mL VOA vial.

COMMENT:

6B. [Table A-2, Spiking Material Sampling Procedure; Table A-3, Makeup Water Sampling Procedure] It is not clear what the purpose of the 40-mL VOA vial containers might be since their use is not discussed in the Procedure Summary.

RESPONSE:

Tables A-2 and A-3 have been changed to reflect which sample containers should be used for which

samples.

COMMENT:

7A. [Attachment B, Analytical Procedures; General] The various method descriptions are not clear with respect to when matrix spikes will be added. This should be clarified in situations where multiple impingers/filters will be collected. This comment is not relevant if only a blank spike of XAD-2 resin is planned.

RESPONSE:

Matrix spikes are typically added to the sample prior to extraction. The M0010 and M0023A trains specify in Table 5-2 that the matrix spike is a blank spiked resin for all analyses and that the condensate may be a matrix or method spike. The condensate sample would be spiked prior to extraction. There is no matrix spike for the tubes of the M0030 train and the condensate samples are spiked prior to analyses. M29 matrix spikes involve spiking a blank train sample. Matrix spikes are added to the train components of the separate halves prior to extraction. All other train and process samples are single component samples and are spiked prior to extraction or preparation in the laboratory.

COMMENT:

7B. Some of the methods are not clear with respect to the frequency with which spiking, blanks and other QC will take place. In some cases it specifies once per batch, but a batch is not defined. It is recommended QC samples be once per batch or per “x” number of samples.

RESPONSE:

Unless otherwise specified, a batch consist of a group of 20 or less samples which are prepared together following the same analytical protocol. Section 10.1 of the QAPP has been modified to include the definition of a batch.

COMMENT:

8. [Attachment B, Table B-6, Analysis of Volatile Organics in Solids, Semi-Solids and Liquids] It is indicated that spent carbon will be dispersed in methanol, “as appropriate.” It is not clear whether this will happen routinely or not. Once in methanol it is assumed, but not stated, that an aliquot would be withdrawn and injected into a standard purge and trap vessel, but none of this is discussed in the method description. Also, if samples will be placed in methanol, Westates may wish to consider preserving the samples in the field so that VOCs are not lost due to off gassing of the spent carbon.

RESPONSE:

Table B-6 has been revised to clarify how the samples will be handled.

COMMENT:

9. [Section 4.0, Organization of Personnel, Responsibilities, and Qualifications] The laboratory that will support the effort is not identified. Although this is not crucial to the project because the QAPP defines QC requirements prescriptively so that the quality system for the test is defined, the QAPP should optimally identify the laboratory that will be performing this support.

RESPONSE:
A specific laboratory has not yet been contracted for this project so no revision can be made at this time. When this information is available, it will be provided to the Agency.

COMMENT:
10. [Section 5.3.6, Stack Gas SVOCs, PAHs, OCPs, and PCBs] This section indicates that the sampling train will be spiked with isotopically labeled surrogate compounds. Although this is generally borne out in subsequent discussions, there is no provision for the spiking of organochlorine pesticide (OCP) surrogates prior to sampling, nor are the surrogates to be used in the OCP isotopically labeled (nor do they need to be) since a gas chromatographic method, rather than a gas chromatography/mass spectrometry (GC/MS) method will be used instead. The discrepancy in the text should be resolved in this section and other related parts of the QAPP (for example, Table 5-2).

RESPONSE:
No isotopically labeled surrogates for OCP will be spiked onto the XAD prior to sampling. This reference has been corrected in Section 5.3.6, Section 6.2.3.2, and Table 5-2.

COMMENT:
11. [General; Section 11.2, Data Validation] The QAPP makes reference to a number of different detection limits; seemingly more than necessary. There are method detection limits (MDLs), reliable detection limits (RDLs), Practical Quantitation Limits (PQLs), and Estimated Detection Limits (EDLs). It would be helpful if the QAPP could define a more limited number of measures, for example the MDL and a higher quantitation limit, or, at a minimum, provide concise definitions of each. Presently the plan defines some, but not all, of these terms. This means that lists, such as appear on page 60 of 72, which indicate the lab will report method detection limits and sample quantitation limits, are not clear in the context of PQLs, RDLs, and EDLs. We acknowledge that this plethora of detection limits is partly a result of different conventions in different EPA guidance documents, but the plan could simplify the terminology.

RESPONSE:
The plethora of detection limit terms is a result of different conventions in different EPA guidance and is needed to meet both the requirements of the test program and to give the laboratory some flexibility. Below is a listing of each detection limit referenced above and the definition of the term and how/why it is used in the document. A list of detection limit terms is added to the document as a revision of Section 5.3 by adding Subsection 5.3.1 Definitions.

MDL – method detection limit – The minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero. It is a statistical limit that is matrix dependent. The MDL is generally derived following SW-846 Chapter 1 Section 5.

PQL – practical quantitation limit – The lowest level that can be achieved reliably within specified limits of precision and accuracy during routine laboratory conditions. It is matrix dependent and is simply calculated as a multiple of the MDL. Each compound or element is assigned a multiplier that is contingent upon the behavior of the compound or element during analysis (generally 5 or 10).

RDL – reliable detection limit – A measurement required for risk assessment. It is similar to a PQL but is derived from the MDL by multiplying the MDL by 2.623. “Human Health Risk Assessment Protocol” U.S. EPA, Office of Solid Waste, July 1998.

EDL – estimated detection limit – This detection limit is used for isotope dilution methods only and is the detection limit that is reported for a target analyte that is not detected, or presents an analyte response that is less than 2.5 times the background level. EDLs are different for every sample.

The MDL is a required reporting level but it is not a concentration which can be assured to be achieved within specified limits of precision and accuracy during routine laboratory operating conditions. It is desired to know not only when an analyte is detected at a level where it can be stated with confidence that the analyte is present, but also when the analyte concentration can be reported as a reliable number and not an estimated value. To report results with this level of confidence the PQL or RDL is used depending on the use of the data and the laboratory's reporting capabilities.

COMMENT:

12. [Section 8.2.3, Digital Temperature Indicator] It is not clear how a mercury thermometer can be used to calibrate a digital thermometer up to 450°F.

RESPONSE:

Calibration of temperature sensors are described in Calibration Procedure 2e of the EPA document EPA/600/R-94/038c "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III Stationary Source-Specific Methods", September 1994.

COMMENT:

13. [Section 9.0, Analytical Procedures; Section 11.3.2, Reporting of Tentatively Identified Compounds] Section 9.0 is not clear with respect to the investigation of tentatively identified compounds (TICs). The text indicates that a library search will be performed for all SW-846 8260 and 8270 analyses. Usually such searches are based on the database of compounds in the instruments' database, which typically consists of 50,000+ compounds. A full library search is described later in Section 11.3.2. The two sections should be consistent and indicate TICs will be identified using the full scan of all database compounds.

RESPONSE:

Section 9.0 has been modified to indicate that a TIC will be identified using a full database scan as described in section 11.3.2.

COMMENT:

14A. [Section 9.0, Analytical Procedures; Table 9-1, Summary of Performance Test Analytical Procedures and Methods] The present plans call for the OCPs and semi-volatile organic compounds (SVOCs) to come from the same sampling runs. This means that after all the extracts from the Modified Method 5 train are combined that there must be sufficient extract to use for both analyses. The OCP extract will need to be solvent exchanged into hexane as a methylene chloride extract (or methylene chloride/acetone extract) and cannot be used for a method 8081 analysis. Assuming that the extract is split, this means that detection limits may be lowered as a result. Also Table 9-1 should indicate that the extract will be solvent exchanged.

RESPONSE:

Table 9-1 has been revised to indicate solvent exchange, as indicated in the method, of the OCP portion of the M0010 train extract. The procedure has been discussed with potential laboratory contractors, and

detection limit degradation is not expected to significantly impact the usability of the data. Planned preliminary testing prior to the formal performance test will be used to verify this supposition.

COMMENT:

14B. The organochlorine pesticide (OCP) analysis is likely to present considerable difficulties. XAD-2 resin often contains numerous impurities. Whereas gas chromatography/mass spectrometry (GC/MS) can possibly identify these impurities, or at least distinguish between them and target compounds, the GC/Electron Capture Detector (ECD) used in Method 8081 is not as discriminating. Please discuss how these difficulties will be addressed.

RESPONSE:

Focus Environmental, Inc. and its laboratory contractors have performed stack gas analysis for organochlorine pesticides in only a few cases. However, we have used the method described in the test plan and have not previously seen significant problems with Method 8081 when used for the organochlorine pesticides. Method 8081 provides several cleanup schemes within the method and in Method 3600 that may be utilized if chromatographic problems are expected due to the resin. All analysts performing this analysis should be experienced in GC techniques and in the interpretation of chromatograms. Additionally, a second column is used to confirm positive results for target compounds providing a second chance to determine if the peak is actually the target compound or an impurity with similar retention times. Typically impurities that are present are addressed through the use of blanks. There are several blanks utilized during the test including method blanks, a blank train, and reagent blanks including resin blanks. Since, for the OCP analysis, there is a specified list of compounds that are calibrated for and reported, the blanks can be evaluated to determine if there is either contamination of the media (filter or resin) or in the reagents that is reported as a target compound. Blank spikes are also utilized in this test program and would indicate if there is any difficulty in detecting target compounds in the presence of any impurities that may be found in the resin.

COMMENT:

15. [Section 11.3.5, Final Case Files] It is recommended that files be retained for at least five years, rather than three as indicated here.

RESPONSE:

This section has been revised to indicate files will be retained for five rather than three years.

COMMENT:

16. [Section 14.1.1, Field Audits] If a field audit will be conducted, it is recommended that it be held during the preliminary test. There are many steps involved before trains are set up (pre-spiking of materials, cleaning and leak testing, etc.), but there are also a large number of steps which must be taken during the collection of the samples themselves. An audit would be more effective if conducted while work was in progress. Then documentation of the earlier steps could be examined as well as the execution and documentation of activities during the tests themselves. A copy of the checklist referenced here should be included with the plan.

RESPONSE:

Field audits may be conducted while work is in progress but could also be conducted before or after work is completed. This is dependant on what is actually being audited. Prior to work being conducted, items such as equipment maintenance and SOPs could be audited, during actual work audits could be conducted for

conformance with the method or SOP, after the actual test/run is performed, audits could be conducted on records that were or should have been recorded. Section 14.1.1 has been revised to indicate that audits, if conducted for a particular activity may be conducted before, during of after the completion of the actual test being conducted. Since there are so many possible audits that may be conducted for a given test, the checklist has been left fairly vague to be able to be used or modified for several different cases. This checklist may be altered to fit the specific activity being audited. This checklist has been added as Figure 14-2.

Part of the reason for the planned preliminary test is to ensure that all procedures are followed or to identify if certain procedures need to be modified for this particular test.

COMMENT:

17A. [Table 5-1, Test Analytical Data Quality Objectives] A number of the precision objectives for stack gas samples are not clear. The precision objective is defined as a relative percent difference (RPD), but the statements suggest that data from all three test runs will be used. If this is the case, a relative standard deviation (RSD) should be calculated. The definition of RPD provided in the plan on page 9 of Table 5-1 [(highest value - lowest value)/average value] could be used for three results, but this is not felt to be appropriate if a RSD can be calculated. In some cases, a RPD will be calculated from a matrix spike/matrix spike duplicate (MS/MSD) analysis of spiked blank trains, which is appropriate. Table 5-2 should be evaluated to determine when RPD vs. RSD is appropriate and should also clarify that some of these calculations will take place with data from multiple runs.

RESPONSE:

Table 5-1 has been reviewed with regards to RPD vs. RSD and which is appropriate in each place. Changes to this table have been made where appropriate.

COMMENT:

17B. Some of the acceptance windows for relative percent difference for the spiked trains appear to be broader than one would expect (<50% RPD). A window more like <35% would appear to be tighter and easily achievable. This is especially true where the plan discusses a duplicate injection, such as for OCPs.

RESPONSE:

Acceptance windows for RPDs have been reviewed and some have been revised to reflect lower limits that should be achievable.

COMMENT:

18. [Table 5-2, Organic Surrogate Spike and Matrix Spike Recovery Limits] Please explain why no surrogate is identified to be added to the XAD-2 resin prior field use.

RESPONSE:

There are several surrogates listed in Table 5-2 that will be added to the XAD resin in both the M0023A and M0010 trains. Below is a list of these surrogates and the train/analysis with which they are associated.

$^{13}C_3$-labeled Naphthalene	M0010 SVOC

$^{37}Cl_4$-2,3,7,8-TCDD	M0023A PCDD/PCDF
$^{13}C_{12}$-2,3,4,7,8-PeCDF	M0023A PCDD/PCDF
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	M0023A PCDD/PCDF
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	M0023A PCDD/PCDF
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	M0023A PCDD/PCDF
$^{13}C_{12}$-2,4,4'-Tri-CB	M0010 PCB
$^{13}C_{12}$-2,3,3',5,5'-PeCB	M0010 PCB
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	M0010 PCB
d_{10}-Fluorene	M0010 PAH
d_{14}-Terphenyl	M0010 PAH

COMMENT:

19A. [Table 5-3, Estimated Stack Gas Detection Limits - Target Analytes] The footnotes to the table indicate that some detection limits are not known and so they are estimated, but all the compounds with these footnotes do not have any detection limits specified.

RESPONSE:

Estimated detection limits have been added to compounds that were footnoted as having estimated limits. Since none were available, the highest known detection limit was used for these compounds. It should be remembered that these are estimates and could change once a laboratory has been chosen.

COMMENT:

19B. There are several footnotes provided here which could not be located in Table 5-3.

RESPONSE:

The additional footnotes have been removed or revised in Table 5-3.

COMMENT:

20. [Section 6.2.3.8, Cascade Impactor for PSD] The third line is confusing. It is believed it should read, "...based on the flow of gas..." but this should be clarified.

RESPONSE:

A Cascade Impactor will no longer be used for the PSD analysis. This analysis will be performed by SIM on a smooth Method 5 filter. The text has been revised to indicate this change. Additionally Table A-15 from Attachment A has been replaced with a new table specific to this method.

COMMENT:

[General] The plan is quite lengthy, and combined with the associated Work Plan is very large. That fact notwithstanding, there are a number of minor editorial problems and a few misspellings throughout the document. Possibly a spell check and a grammar checker would find some of these problems so the document could be "cleaned up" to some degree. None of these problems affect the technical content.

RESPONSE:
Spell check and grammar checks have been performed on this document prior to being resubmitted. We hope that this clears up and editorial problems and misspellings.

COMMENT:
[General] The acronym "TOE" has not been defined.

RESPONSE:
TOE represents Total Organic Emissions. This acronym has been added to the list of acronyms following the table of contents in the QAPP and a reference has been included in Table 6-1 explaining the use of this acronym.