State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

PAHs in Air by Method TO-13

Version 3.0

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Written by the Connecticut DEEP QA/QC Workgroup

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1.0	Posted on EPOC website	February 2006
2.0	Final version based on public comment	December 2006
3.0	Updates to reflect CAM method updates to improve consistency between different states.	May 2024

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Acronym List

ACRONYM	DEFINITION
BFB	Bromofluorobenzene
CASN	Chemical Abstracts Service Number
CCV	Continuing calibration verification
%D	Percent difference or percent drift
DEEP	CT Department of Energy and Environmental Protection
DF	Dilution factor
EP	Environmental professional
GC	Gas chromatograph
GC/MS	Gas chromatography/mass spectrometry
ICV	Initial calibration verification
In. Hg	Inches of mercury
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LLOQ	Lower Limit of Quantitation
MD	Matrix duplicate
NA	Not applicable
PAH	Polyaromatic hydrocarbons
PCB	Polychlorinated biphenyls
PFE	Pressured Fluid Extraction
PUF	Polyurethane foam
QA	Quality assurance
QC	Quality control
%R	Percent recovery
%RSD	Percent relative standard deviation
r/r^2	Correlation coefficient
RCP	Reasonable Confidence Protocols
RL	Reporting limit
RPD	Relative percent difference
RSR/RSRs	Remediation Standard Regulations
SIM	Selective ion monitoring
TCL	Target compound list
UCM	Unresolved complex mixture
UV	Ultraviolet
$\mu g/m^3$	micrograms per cubic meter
VOCs	Volatile organic compounds

1.0 Quality Assurance and Quality Control Requirements for TO-13

1.1 Overview of Method TO-13

Method TO-13 is a gas chromatography/mass spectrometry ("GC/MS") procedure used to determine polynuclear (or polyaromatic) organic compounds ("PAH"s) in air. This procedure requires an experienced GC/MS analyst familiar with sampling and analysis of PAHs using XAD-2® resin and polyurethane foam ("PUF") and the quality assurance/quality control ("QA/QC") requirements of the method. Although other detectors may be listed in the EPA method, the Connecticut Reasonable Confidence Protocols ("RCP"s) require the use of a mass spectrometer.

This method is generally applicable to the determination of PAHs involving three member rings or higher. Naphthalene, acenaphthylene, and acenaphthene have only ~35 percent recovery when using PUF alone as the collection medium. This procedure calls for using XAD-2® resin in conjunction with PUF to determine these important compounds. Nitro-PAHs have *not* been fully evaluated using this procedure; therefore, they are not included in this method.

All method references are to the latest published version of the method found in the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, published by the US EPA.

1.2 Summary of Method TO-13

Filters and sorbent cartridges (containing PUF and XAD-2®) are cleaned in solvents and vacuum dried. The filters and sorbent cartridges are stored in screw-capped jars wrapped in aluminum foil (or otherwise protected from light) before careful installation on the sampler.

Approximately 300 cubic meters (m^3) of air is drawn through the filter and sorbent cartridge using a high-volume flow rate air sampler or equivalent.

The amount of air sampled through the filter and sorbent cartridge is recorded, and the filter and cartridge are placed in an appropriately labeled container and shipped along with blank filter and sorbent cartridges to the analytical laboratory for analysis.

The filters and sorbent cartridge are extracted by Soxhlet extraction with appropriate solvent. The extract is concentrated by Kuderna-Danish (K-D) evaporator, or equivalent concentration hardware, followed by silica gel cleanup using column chromatography to remove potential interferences prior to analysis by GC/MS.

The extract is further concentrated by K-D evaporation, then analyzed by GC/MS. The analytical system is verified to be operating properly and calibrated using, at minimum, a five-point calibration.

A preliminary analysis of the continuing calibration verification ("CCV") is performed to check the system performance. If the CCV analysis fails to meet acceptance criteria, then take corrective measures and recalibrate the instrument.

The extracts are analyzed and used (along with the amount of air sampled) to calculate the concentration of PAHs in the air sample.

See Method TO-13 for additional publications and analytical approaches to determine PAHs in air.

Because of the relatively low levels of common PAHs in the environment, the methodology suggests the use of high volume (0.22 m^3/min) sampling technique to acquire sufficient sample for analysis. However, the volatility of certain PAHs prevents efficient collection on filter media alone. Consequently, this method utilizes both a filter and a backup sorbent cartridge, which provides for efficient collection of most PAHs involving three member rings or higher.

1.3 Method Interferences

1.3.1 Chemical Contaminants

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that result in discrete artifacts and/or elevated baselines in the detector profiles. These materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Glassware must be scrupulously cleaned. All glassware should be cleaned as soon as possible after use by rinsing with the last solvent used in it and then high-purity acetone and hexane. These rinses should be followed by detergent washing with hot water and rinsing with copious amounts of tap water and several portions of reagent water. The glassware should then be drained dry and heated in a muffle furnace at 400º C for four hours. Volumetric glassware must not be heated in a muffle furnace; rather it should be solvent rinsed with acetone and spectrographic grade hexane. After drying and rinsing, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Glassware should be stored inverted or capped with aluminum foil. *[Note: The glassware may be further cleaned by placing in a muffle furnace at 450º C for 8 hours to remove trace organics.]*

The use of high purity water, reagents, and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

Analysis of blanks provides information about the presence of contaminants. When potential interfering peaks or high levels of target compounds are detected in blanks, the laboratory should try and find the source of the contamination and eliminate it. **Subtracting blank concentrations from sample results is not permitted.** Any method blank exceedances should be fully documented in the laboratory report narrative.

1.3.2. Limitations

PAHs span a broad spectrum of vapor pressures (e.g., from 1.1 x 10^{-2} kPa for naphthalene to 2 x 10^{-13} kPa for coronene at 25º C). PAHs that are frequently found in ambient air are listed in provided in EPA Method TO-13. Those with vapor pressures above approximately 10^{-8} kPa will be present in the ambient air substantially distributed between the gas and particulate phases. This method will permit the collection of both phases.

Particulate-phase PAHs will tend to be lost from the particle filter during sampling due to volatilization. Therefore, separate analysis of the filter will not reflect the concentrations of the PAHs originally associated with particles, nor will analysis of the sorbent provide an accurate measure of the gas phase. Consequently, this method calls for *extraction of the filter and sorbent together* to permit accurate measurement of total PAH air concentrations.

Naphthalene, acenaphthylene, and acenaphthene possess relatively high vapor pressures and may not be efficiently trapped by this method when using PUF as the sorbent. The sampling efficiency for naphthalene has been determined to be about 35% for PUF. The user must use XAD-2® as the sorbent if these analytes are part of the target compound list (“TCL”).

1.3.3. Interferences

Matrix interferences may be caused by contaminants that are co-extracted from the sample. Additional clean-up by column chromatography may be required, see EPA Method TO-13.

During sample transport and analysis, heat, ozone, NO_2, and ultraviolet (“UV”) light may cause sample degradation. Incandescent or UV-shielded fluorescent lighting in the laboratory should be used during analysis.

The extent of interferences that may be encountered using GC/MS techniques has not been fully assessed. Although GC conditions described allow for unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere. The use of column chromatography for sample clean-up prior to GC analysis will eliminate most of these interferences. The analytical system must, however, be routinely demonstrated to be free of internal contaminants such as contaminated solvents, glassware, or other reagents which may lead to method interferences. A laboratory reagent blank should be analyzed for each reagent used to determine if reagents are contaminant-free.

Concern about sample degradation during sample transport and analysis was mentioned above. Heat, ozone, NO_2, and UV light also may cause sample degradation. These problems should be addressed as part of the user-prepared standard operating procedure (“SOP”) manual. Where possible, incandescent, or UV-shielded fluorescent lighting should be used during analysis. During transport, field samples should be shipped back to the laboratory chilled (~4° C) using ice.

1.4 Equipment and Supplies

Refer to the Sampling, Equipment and Materials, and Prep of PUF Sampling Cartridges Sections in EPA Method TO-13 for needed equipment and supplies. Note that the use of XAD-2 ® resin and a GC/MS system are required for sampling and analysis of PAHs. The amount of XAD-2® resin may not be decreased solely for the purpose of using automated solvent extraction (“ASE”) / Pressurized Fluid Extraction (“PFE”) – the resin bed and associated PUF plugs must fill the cartridge.

Field surrogates must be added to the cartridges prior to sampling. Alternate surrogate compounds (minimum of two) may be used.

Alternative XAD-2® cleanup procedures may be used as long as the method blank criteria listed in the Prep of PUF Sampling Cartridges Section in EPA Method TO-13 are met.

1.5 Sampling and Analysis

Chilled (≤4° C) samples are returned to the laboratory for analysis in the provided aluminum shipping container (containing the filter and sorbents). A "chain-of-custody" must be completed by the environmental professional (“EP”) when submitting samples to the laboratory.

If the time span between sample receipt and analysis is greater than 24-hours, then the sample must be kept refrigerated at ≤4° C. Minimize exposure of samples to fluorescent light. All samples should be extracted within one week (7 days) after sampling.

Follow the procedure outlined in the Assembly, Calibration, and Collection Using Sampling System Section of EPA Method TO-13. Document all calibration and sample collection data.

Samples should be extracted using the Soxhlet procedure. Sonication is not allowed. Follow the procedure outlined in the Sample Extraction, Concentration, and Cleanup Section of EPA Method TO-13. ASE/ PFE is allowed provided the entire amount of XAD-2® resin is extracted in one cartridge. The PUF may be extracted separately, and the extracts concentrated together.

Laboratory surrogates (minimum of two) must be added to the samples after receipt from the field and prior to extraction. Alternate compounds may be used.

The use of the silica gel column cleanup is optional. However, if silica gel cleanup is used, the extract must be put through the silica gel cleanup procedure as described in the Sample Extraction, Concentration, and Cleanup Section of EPA Method TO-13.

See Table 1A of this RCP method and the GC/MS Detection Section of Method TO-13 for specific QA/QC requirements.

The laboratory must use the internal standards listed in Method TO-13 for quantitation.

The mass spectrometer must be tuned using decafluorotriphenylphosphine ("DFTPP"). The suggested criteria are listed in Method TO-13. Alternative referenced criteria may be used, but all samples, standards, blanks, etc. must be analyzed under the same tune criteria.

The laboratory may utilize 1µl injections if the sensitivity required is still met.

1.6 Quality Control Requirements for Method TO-13

1.6.1 Reporting Limits/Lower Limits of Quantitation for Method TO-13

The reporting limit ("RL")/Lower Limit of Quantitation ("LLOQ") for a compound is dependent on the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for sample volume, sample introduction method, and any dilution factors, etc., as required. Table 1.0 lists approximate RL/LLOQs for air utilizing GC/MS.

Table 1.0: Typical Reporting Limits / Lower Limits of Quantitation[1]

Matrix	Typical Reporting Limit
Air	1 ng to 10,000 ng
[1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.	

Sample dilution or lower sample volume will also cause the RLs/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet the project Data Quality Objectives ("DQOs").

1.6.2 General Quality Control Requirements

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS instrumentation as a quantitative tool and skilled in the interpretation of chromatograms for PAHs.

Refer to the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air* for general quality control ("QC") requirements. These requirements ensure that each laboratory maintain a formal quality assurance ("QA") program and records to document the quality of all chromatographic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the GC/MS system operation may be found in the published method and include evaluation of calibrations and chromatographic performance of sample analyses, instrument QC and method performance requirements for the GC/MS system.

The minimum requirements for a formal QA program include an Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") to assess accuracy and matrix duplicates ("MD") to assess precision. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Blanks should not be used for MDs.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative method being used. These data must meet, or fall within, the performance standards as presented in Section 1.6 and Table 1A of this RCP. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform Method TO-13. The IDOC must include the following elements provided in Table 2.0:

Table 2.0: IDOC Requirements

QC Element	Performance Criteria
DFTPP Tuning	Method TO-13
Initial Calibration	Table 1A
Continuing Calibration	Table 1A
Method Blanks	Table 1A
Average Recovery	Table 1A
% Relative Standard Deviation	Table 1A
Surrogate Recovery	Table 1A
Internal Standards	Table 1A

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, and relative percent difference ("RPD") limits, and surrogate recovery limits. These limits must be equal to, or fall within, the limits specified in Table 1A.

1.6.3 Specific QA/QC Requirements and Performance Standards for the APH Method

Specific QA/QC requirements and performance standards for Method TO-13 are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the EP with "Reasonable Confidence" regarding the usability of analytical data to support environmental decisions. The concept of "Reasonable Confidence" is explained on the DEEP website.

While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally accept "Reasonable Confidence" data. To achieve "Reasonable Confidence" parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming analytical performance standards; and

3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

Table 1A: Specific QA/QC Requirements and Performance Standards for Method TO-13

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
PUF and XAD-2® Resin Cleaning and Certification	Assure cartridges are free from contamination	Per the Preparation of PUF Sampling Cartridge Section of the EPA Method TO-13. Alternative cleanup procedures may be used as long as method blank criteria are met.	NO Data kept on file in lab.	Do not use contaminated cartridges.	Reclean as necessary.
GC/MS Tunes with DFTPP	Inter-laboratory consistency and comparability	(1) Criteria listed in Method TO-13 (the same criteria must be used for all analyses). (2) Every 12 hours	NO	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration (“ICAL”)	Laboratory Analytical Accuracy	(1) Minimum of 5 standards. Standards must be prepared per Method TO-13. (2) Low standard must be ≤ RL/LLOQ. (3) %RSD ≤30%, r ≥0.99 (linear regression) or r^2 ≥0.99 (non-linear regression) for each target analyte. (4) If %RSD >30, linear or non-linear regression must be used. (5) Minimum RFs as per the TO-13 Method for lowest concentration standard and for average RF. (6) Must contain all target analytes. (7) Calibration must be performed under the same conditions as the samples. (8) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve, recoveries must be 70-130%. (9) RRT for each target compound and surrogate at each calibration level must be within ±0.06 RRT units of the mean RRT for the compound. (10) RT shift for each the IS at each calibration level must be within ±20.0 seconds compared to the mean RT over the initial calibration range for each internal standard.	NO	(1) Recalibrate as required by method. (2) 1 compound may fail RSD criteria as long as min RF criteria met and % RSD <50%. (3) If RSD >30% analyze additional aliquots of appropriate CALs to obtain an acceptable %RSD of RRFs over the entire concentration range, or take action to improve GC/MS performance	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification Standard ("ICV")	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Second source standard (3) Concentration level near mid-point of curve 4) Must contain all target analytes (5) Percent recoveries must be between 80 and 120%	NO	(1) Laboratories are allowed to have 20% of compounds out, as long as all compounds within recover 65-135% (2) Reanalyze ICV; if acceptable, no further action required. (3) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV	(1) Perform maintenance as needed, recalibrate. (2) Note non-conformances in laboratory report narrative.
Continuing Calibration Standard ("CCV")	Laboratory Analytical Accuracy	(1) Every 12 hrs prior to analysis of samples per Continuing Calibration Section of TO-13 Method. (2) %D between the measured RRF for each target/surrogate compound of the CAL 3 standard and the mean value calculated during initial calibration must be within 70-130%	NO	(1) Recalibrate as required by method. (2) Two compounds may fail %D criteria as long as min RF criteria met and %D <40%.	Report non-conforming compounds in laboratory report narrative.
Method Blanks ("MB")	Laboratory Contamination Evaluation	(1) Analyze with every batch or ≤ 20 field samples, whichever is more frequent. (2) All target compounds must be ≤ RL/LLOQ (not the MDL as specified in the method)	YES	Note non-conformances.	(1) Report non-conformances in laboratory report narrative. (2) All results for compounds present in method blank must be "B" flagged if detected in samples associated with the method blank.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample ("LCS")	Laboratory Method Accuracy	(1) Analyze with every batch or ≤ 20 field samples, whichever is more frequent. (2) Standard source should be different from the initial calibration source. (3) Concentration level near or at the mid-point of the initial calibration curve. 4) Must contain all target analytes (5) All target analytes spiked on the certified PUF cartridge must meet a percent recovery between 60-120% (6) Must meet criteria specified in the Method TO-13.	YES	(1) Reanalyze the LCS extract. (2) Locate & correct problem, reanalyze associated sample extracts.	Report non-conformances in laboratory report narrative.
Laboratory Control Sample ("LCSD")	Laboratory Method Accuracy & Precision	(1) Extracted with every batch or every ≤20 field 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 6-120%. (6) RPD must be ≤50%.	Yes	Report non-conformances in laboratory report narrative.	Report non-conformances in laboratory report narrative.
Surrogates	Accuracy in sample matrix.	(1) All surrogates must recover between 60-120%. (2) Field surrogates: Surrogates must be added to PUF cartridges prior to field deployment in accordance with Method TO-13. (3) Laboratory surrogates: Surrogates must be added to PUF cartridge prior to extraction accordance with Method TO-13.	YES	If surrogate out reanalyze to verify.	Note non-conformances in the laboratory report narrative. Note which surrogates are field and laboratory.
Internal Standards ("IS")	Laboratory Analytical Accuracy and Method Accuracy in Sample	(1) Laboratory must spike extracts with IS before analysis. (2) Must be prepared in accordance with Method TO-13. (3) Recovery must be 50 – 200% compared to recent CCV sample.	NO	(1) Evaluate the analytical system for malfunctions and correct (2) Reanalyze the sample	(1) Note non-conformances in laboratory report narrative (2) If reanalysis confirms matrix interference, report initial analysis and note in laboratory report narrative (3) If reanalysis meets criteria, report only compliant analysis

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Quantitation	N/A	(1) Quantified by IS method. The IS's used for target compounds are ones nearest the RT of a given analyte. (2) The RRF from daily continuing cal is used to calculate concentration of sample. Secondary ion quantitation is allowed ***only*** when there are sample interferences with primary ion. (3) Area of secondary ion cannot be substituted for area of a primary ion unless a RRF is calculated using the secondary ion. (4) A retention time window is calculated for each single component analyte and surrogate. Windows are established as ± RRT units of the retention time for analyte of mid-point of calibration curve of initial calibration or the continuing calibration standard.	N/A	N/A	Note any problems in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
General Reporting Issues	N/A	(1) The laboratory should report only concentrations detected above the sample specific RL/LLOQ. (2) Concentrations below the RL/LLOQ as "ND" with the reporting limit. (3) Dilutions: If diluted and undiluted analyses are performed, the lab 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.	N/A	N/A	(1) Qualification of results reported below the RL/LLOQ is required. (2) Performance of dilutions must be documented in the laboratory report narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory report laboratory report narrative. (3) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report laboratory report narrative. (4) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory report laboratory report narrative.

1.7 Analyte List for Method TO-13

The DEEP analyte list for Method TO-13 is presented in Table 1B. The compounds listed are readily analyzable by Method TO-13.

Additional PAH and other semi-volatile compounds may be determined by this procedure but have not been validated by the US EPA. It is up to the laboratory and/or EP to justify the inclusion of additional compounds. It is noted that polychlorinated biphenyls (“PCBs”) and certain pesticides may be collected using the same procedure as described in method TO-13. These compounds would require determination using an alternate analytical scheme such as gas chromatography coupled with an electron capture detector (“GC/ECD”).

Table 1B: Analyte List for Method TO-13

Analyte	**CAS No.**
Acenaphthene	83329
Acenaphthylene	208968
Anthracene	120127
Benzo(a)anthracene	56553
Benzo(a)pyrene	50328
Benzo(b)fluoranthene	205992
Benzo(g,h,i)perylene	191242
Benzo(k)fluoranthene	207089
Chrysene	218019
Dibenzo(a,h)anthracene	53703
Fluoranthene	206440
Fluorene	86737
Indeno(1,2,3-c,d)pyrene	193395
1-Methylnapthalene	90120
2-Methylnaphthalene	91576
Naphthalene	91203
Phenanthrene	85018
Pyrene	129000

1.7.1 Additional Reporting Requirements for the Method TO-13

While it is not necessary to request and report all the Method TO-13 analytes listed in Table 1B to obtain Reasonable Confidence status, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEEP strongly recommends that the full list of analytes be reported during the initial stages of a site investigation and/or at sites with an unknown or complicated history of chemical usage or storage.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.8 Routine Reporting Deliverables for Method TO-13

The following table (Table 3.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report these items in special circumstances.

Table 3.0: Report Deliverables for Method TO-13

Parameter	Deliverable	Comments
GC/MS Tunes	NO	Analysis cannot proceed without meeting tuning criteria.
Initial Calibration	NO	Note non-conformances in laboratory report narrative
Continuing Calibration	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive results above RL/LLOQ with "B" flag.
Lab Control Sample/Lab Control Sample Duplicate	YES	Note non-conformances in laboratory report narrative
Sample Duplicate	YES (If requested)	Note non-conformances in laboratory report narrative
Internal Standard Areas	NO	Note non-conformances in laboratory report narrative
General Reporting Issues	YES	Note non-conformances in laboratory report narrative
Identification and Quantitation	NO	Note non-conformances in laboratory report narrative
QA/QC Certification Form	YES	Signed by laboratory director or their designee
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory.

1.8.1 Additional Reporting Requirements for the Method TO-13

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as "ND" (Not detected at the specified RL/LLOQ). The RL/LLOQ for each compound in each sample must be listed on the report, based upon the lowest calibration standard, the exact sample volume, any dilution factors, percent moisture, etc.

- Compounds detected above the RL/LLOQ in blanks and found in samples, also above the reporting limit, shall be flagged with a "B" suffix (e.g., 25B).

- Report results for any library search compounds as estimated using a "J" suffix (e.g., 25J).

1.9 Sample Collection, Storage and Holding Times

Table 4.0 identifies the type of containers, preservation requirements, and holding times.

Table 4.0: Sample Containers, Storage, and Holding Times

Container	Storage	Holding Time
Aluminum shipping container wrapped in aluminum foil	Ship and short-term storage on ice ≤4 C Long-term storage Refrigerate at ≤4° C and in the dark	7 days from collection to extraction 40 days after extraction

1.10 Tentatively Identified Compounds

Due to the high levels of background contamination associated with the XAD-® resin and polyurethane foam, it is recommended that tentatively identifed compounds **NOT** be determined using this method.