LAB ID:	LAB NAME:
DATE:	ASSESSOR NAME:

TO-15 Checklist
Determination of VOCs in Air by GC-MS

Method Number:

SOP Number:

Revision Number:

SOP Date:

Personnel records observed:

Data records observed:

Checklist Category	Ref.	Y	N	NA	ELAP Code	Comment
Required Apparatus and Reagents - Sampling/Concentrator System	7.2.1				TO1000	
Does the lab have **Electronic Mass Flow Controllers** to maintain constant flows of purge, carrier, and sample gas, and to provide an analog output to monitor flow anomalies?	7.2.1.1				TO1001	
Does the autosampler system have a **Vacuum Pump**, capable of reducing the downstream pressure of the flow controller to provide the pressure differential necessary to maintain controlled flow rates?	7.2.1.2				TO1002	
Are the system's **Tubing and Fittings made of Stainless Steel** coated with fused silica to minimize active adsorption sites?	7.2.1.3				TO1003	
Does the lab have **Stainless Steel Cylinder Pressure Regulators** (standard, two-stage cylinder regulators with pressure gauges) for the Helium carrier gas?	7.2.1.4				TO1004	
Does the system include **Gas Purifiers** to remove organic impurities and moisture from gas streams?	7.2.1.5				TO1005	
Does the system include a **Six-port Gas Chromatographic Valve** for routing sample and carrier gas flows?	7.2.1.6				TO1006	
Does the system include an appropriate adsorbent trap **(Multisorbent Concentrator)** packed with an adsorbent having various retentive properties for trace gases?	7.2.1.7				TO1007	
Does the system include a **Cryogenic Concentrator** which facilitates the separation of compounds on the GC column?	7.2.1.8				TO1008	
Gas Chromatograph / Mass Spectrometric (GC/MS) System Requirements	7.2.2				TO1009	
Does the analytical system include a **GC** that is interfaced to a concentrator and have all required accessories including analytical columns and gases? All **GC carrier gas lines must be constructed from stainless steel or copper tubing**. Thread sealants made of Non-polytetrafluoroethylene (PTFE) or flow controllers with Buna-N rubber components **must not be used.**	7.2.2.1				TO1010	
Is the GC equipped with **Chromatographic Columns** that are recommended for the separation of target nonpolar compounds?	7.2.2.2				TO1011	
Does the system include either a linear quadruple or ion trap **Mass Spectrometer?** It must be capable of scanning from 35 to 300 amu and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 nag or less of p-bromofluorobenzene (BFB) is analyzed.	7.2.2.3				TO1012	
Is the **GC/MS Interface** constructed of all-glass, glass-lined, or fused silica-lined materials? Glass and fused silica should be deactivated.	7.2.2.4				TO1013	
Does the **Data System** (Computer) have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number? This type of plot is defined as a **Selected Ion**	7.2.2.5				TO1014	

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Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, software must be available that allows for the comparison of sample spectra with reference library spectra.						
Is the **Off-line Data Storage Device** capable of rapid recording and retrieval of data, and is it also suitable for long-term, off-line data storage?	**7.2.2.6**				**TO1015**	
Calibration System and Manifold Apparatus	**7.3**				**TO1016**	
Does the system have a **Calibration Manifold** used for the preparation of gaseous standards (either single compounds or multicomponent gases)?	**7.3.1**				**TO1017**	
Does the system have a **Humidifier** containing HPLC grade deionized water? Ideally, the HPLC water should be boiled prior to use in order to ensure VOC-free water.	**7.3.2**				**TO1018**	
Does the system have **Electronic Mass Flow Controllers** For the calibration gases?	**7.3.3**				**TO1019**	
Does the system have **Filters** inside the manifold for the collection of particulates? (Usually these are 47-mm, made of **Teflon®)**	**7.3.4**				**TO1020**	
Reagents	**7.4**				**TO1021**	
Does the lab have **Neat Gases or Manufacturer-Certified Gas Mixtures**?	**7.4.1**				**TO1022**	
Does the lab use **Ultra-high purity grade Helium** for the carrier gas in the GC?	**7.4.2**				**TO1023**	
Does the lab have **Liquid Nitrogen or Liquid Carbon Dioxide** used for cooling the secondary trap?	**7.4.3**				**TO1024**	
Does the lab use **HPLC grade Deionized Water** for the humidifier?	**7.4.4**				**TO1025**	
Canister Cleaning and Certification	**8.4.1**				**TO1026**	
Are all canisters clean and free of any contaminants before sample collection? Review the cleaning & certification documentation related to each canister by ID tag.	**8.4.1.1**				**TO1027**	
Are all canisters leak tested by pressurizing them to approximately 206 kPa (+30 psig) with humid zero air or zero N_2? Alternatively, are the canisters evacuated (to -30psig vacuum)? The initial pressure (or vacuum) is measured, and after 24 hours, the final pressure (or vacuum) is checked. To be acceptable, the reading should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.	**8.4.1.2**				**TO1028**	
Has any canister that has not tested clean at less than 0.2 ppbv of targeted VOCs not been used, or have the contaminants been noted in the cleaning/certification logbook?	**8.4.1.6**				**TO1029**	
As an option to the humid zero air cleaning procedures**, are the canisters heated in an isothermal oven** between 100º-105ºC during evacuation of the canister? At the end of the heating / evacuation cycle, the canisters are pressurized with humid zero air (or gaseous nitrogen) and analyzed by a	**8.4.1.8**				**TO1030**	

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GC/MS system after a minimum of 12 hrs of "aging."						
Cleaning of the Sampling System Components	**8.4.2**				**TO1031**	
Are sampling system components disassembled and cleaned before the sampler is assembled?	**8.4.2.1**				**TO1032**	
Are the parts then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100ºC for 12 to 24 hours?	**8.4.2.2**				**TO1033**	
Once the sampler is assembled**, is the entire system purged** with humid zero air for 24 hours?	**8.4.2.3**				**To1034**	
Zero Air Certification	**8.4.3**				**TO1035**	
Is the cleanliness of the sampling system determined by testing the sampler with humid zero air or **humidified zero nitrogen?** (N_2 is 100% free of VOCs and need not be passed through a chemical trap).	**8.4.3.1**				**TO1036**	
If the sampler passes the humid zero air test, is it then tested with **humid calibration gas standards** containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv)?	**8.4.3.3**				**To1037**	
Not all the target gases on the Title III list are available or compatible with compressed gas standards. In these cases, is sampler certification approached by a different means?	**8.4.5.1**				**TO1038**	
Preparation of Standards	**9.2**				**TO1039**	
Are the standard mixtures of target gases in high pressure cylinders **certified traceable to a NIST or EPA Certified Reference Materials?**	**9.2.1.1**				**TO1040**	
Are the "neat" standards that are used for making trace gas standards of high purity? Generally, a **purity of 98% or better** is commercially available	**9.2.1.2**				**TO1041**	
Does the lab retain the traceability certificates for the cylinders containing **primary stock standards?** (These are usually at concentrations from 100 ppbv to 1000 ppbv)	**9.2.1.3**				**TO1042**	
Does the lab have an **Instrument Performance Check Standard?** This is a prepared standard solution of BFB in Nitrogen or humidified zero air, at a concentration which will allow collection of 50 ng of BFB or less under the optimized concentration parameters.	**9.2.2.1**				**TO1043**	
Does the lab prepare **five working calibration standards** at the 2, 5, 10, 20, and 50 ppbv level **for each component**? A separate cal curve ranging from approximately 0.5 ppbv to 2 ppbv may be appropriate for labs with low-level capability.	**9.2.2.2**				**TO1044**	
Does the lab have an **Internal Standard Spiking Mixture:** A.) **Is an internal spiking mixture** containing bromo-chloromethane, chlorobenzene-d, and 1,4-difluorobenzene added to all samples and standards? B.) **Is the Int Std introduced into the trap** during the collection time for all calibration, blank, and sample analyses? Alternatively, ISTD containing BFB as the fourth component allows easy evaluation of BFB tune in any sample or blank, plus it allows using BFB as a retention time locking cmpd. C.) **Is the volume** of internal std spiking mixture added for	**9.2.2.3**				**TO1045** **TO1046**	

Checklist Category	Ref.	Y	N	NA	ELAP Code	Comment
each analysis **the same from run to run**?					**TO1047**	
Are the Standards prepared by dynamic dilution of the calibration stock standards with zero Nitrogen or humidified zero air, using mass flow controllers and a calibration manifold?	**9.2.3.1**				**TO1048**	
Does the lab have a **Standard Preparation Procedure** for use in High Pressure Cylinders? Usually, the standards are prepared as gas mixtures in a Dynamic Diluter. Gases are mixed in the diluter and then transferred to a cylinder to approx 20 psia or 5 psig. Alternatively, predetermined amounts of each neat standard compound are measured using a microliter or gastight syringe and injected into the cylinder.	**9.2.5**				**TO1050**	
Does the lab have a procedure for the Storage of Working Standards? **Working standards prepared in canisters may be stored for 30 days**.	**9.2.8**				**TO1052**	
Gas Chromatograph / Mass Spectrometric (GC/MS) Operating Conditions	**10**	**TO1053**				
Does the lab use the following **Analytical Sequence?** • Perform instrument performance check using BFB. • Initiate multi-point calibration or daily calibration checks. • Perform a laboratory method blank. • Complete this sequence for analysis of 20 field samples or each 24 hr period.	**10.3**				**TO1054**	
Does the lab use a BFB **Instrument Performance Check (IPC)?** The instrument performance check solution **must be analyzed initially and once per 24-hour time period** of operation	**10.4**				**TO1055**	
Does the **MS Tune pass the abundance criteria for BFB,** and retune the Mass Spec if BFB acceptance criteria aren't met?	**10.4.4**				**TO1056**	
Are the **Results of the BFB tuning** recorded and maintained as part of the instrumentation log?	**10.4.6**				**TO1057**	
Prior to the analysis of samples & blanks, but after the IPC criteria have been met, **is each GC/MS system calibrated at 5 concentrations that span the monitoring range of interest** (or is a daily CCV checked)? One of the calibration points from the initial calibration curve must be at the same concentration as the daily calibration standard (e.g., a midrange 10 ppbv).	**10.5**				**TO1058**	
Is each GC/MS system recalibrated following corrective action (e.g., ion source cleaning or repair, column replacement, etc.) which may change or affect the initial cal criteria, **or if the daily calibration acceptance criteria have not been met?**	**10.5.2**				**TO1059**	
Concentration of cal standards: A minimum of five concentration levels are needed to determine the instrument sensitivity and linearity. **Is one of the calibration levels near the detection level for the compounds of interest?**	**10.5.3**				**TO1060**	
Are Initial calibration acceptance criteria met (+/-30% D)	**10.5.6.2**				**TO1061**	

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before any field samples, blanks, or performance evaluation (PE) samples are analyzed?						
Prior to analyzing samples and blanks but after tuning criteria are met, **is the ICAL checked** by analyzing a daily cal std to ensure that the instrument is under control? The daily calibration standard should contain all the target compounds.	**10.6**				**TO1062**	
Is a check of the calibration curve performed once every 24 hours of run time on the GC/MS system?	**10.6.2**				**TO1063**	
Has the mid-level cal standard (10 ppbv) analyzed by the GC/MS system met the tuning and mass calibration Criteria?	**10.6.3**				**TO1064**	
Is the Relative Response Factor (RRF) calculated for each target Compound?	**10.6.4.1**				**TO1065**	
Percent Difference (%D): The percent difference in the daily RRF (24-hour) must be compared to the mean RRF in the most recent initial calibration. Does the lab calculate %D for each target compound?	**10.6.4.2**				**TO1066**	
Technical Acceptance Criteria: The %D for each target compound in a daily calibration sequence **must be within ± 30%** to proceed with analysis. Does the lab maintain a control chart showing %D values?	**10.6.5**				**TO1067**	
Blank Analyses: To monitor for possible laboratory contamination, **does the laboratory analyze a Method Blank at least once in a 24-hour analytical sequence**? All steps in the analytical procedure are performed on the blank using all items that would be used for a sample analysis.	**10.7**				**TO1068**	
Does the laboratory analyze a method blank after the calibration standard(s) and before any samples are analyzed?	**10.7.2**				**TO1069**	
Technical Acceptance Criteria for daily runs: • Is a blank canister analyzed daily? • Is the area response for each internal standard in the blank **within ± 40%** of the mean area response of the Internal Std in the most recent valid calibration? Also, is the retention time for each of the internal standards within ±0.33 minutes between the blank and the most recent valid calibration? • Does the blank NOT contain any target analyte at a concentration greater than its quantitation level (three times the MDL)? Also, does the blank NOT contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte?	**10.7.5**				**TO1070** **TO1071** **TO1072**	
Corrective Action: If the blanks do not meet the technical acceptance criteria, the analyst should consider the analytical system to be out of control. **If a blank is contaminated with an analyte, and that analyte is also found in associated samples, are those sample results "flagged" as possibly contaminated?**	**10.7.6**				**TO1073**	
If time remains in the 24-hour period in which an initial cal is	**10.8.2**				**TO1075**	

Checklist Category	Ref.	Y	N	NA	ELAP Code	Comment
performed, samples may be analyzed without analysis of a daily cal standard. **If time does not remain in the 24-hr period since the injection of the instrument performance check std** during which the initial calibration was performed, **are both the IPC standard and the daily calibration standard analyzed before sample analysis begins?**						
Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. **If secondary ion quantitation is performed, is the reason documented in the laboratory record book?**	**10.8.3.9**				**TO1076**	
Are Field samples analyzed only after the GC/MS system meets the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria?	**10.8.5.1**				**TO1077**	
Are the field samples analyzed along with a lab **Method Blank** that met the blank technical acceptance criteria?	**10.8.5.2**				**TO1078**	
Are all of the target analyte peaks in the samples **within the initial calibration range?**	**10.8.5.3**				**TO1079**	
Is the retention time for each internal standard **within ±0.33 minutes of the retention time** of the internal standard in the most recent valid calibration?	**10.8.5.4**				**TO1080**	
If the on-column **concentration of any compound in any sample exceeds the initial calibration range**, is an aliquot of the original sample diluted and reanalyzed?	**10.8.6**				**TO1081**	
Internal standard responses and retention times must be evaluated after data acquisition. If the retention time for any internal standard changes by more than **20 sec** from the latest daily (24-hour) calibration standard (or mean retention time over the initial calibration range), is the GC/MS system inspected and corrections made?	**10.8.6.1**				**TO1082**	
If the area response for any internal standard changes by more than ± 40% between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and corrections made as appropriate. **When corrections are made, are those samples reanalyzed?**	**10.8.6.2**				**TO1083**	
If, after reanalysis, the area responses or the RTs for all **internal standards** are inside the control limits, then the problem with the first analysis is considered to have been within the control of the Laboratory. **Does the lab submit only data from the analysis with SICPs within the limits?** This is considered the initial analysis and should be reported as such on all data deliverables.	**10.8.6.3**				**TO1084**	
Requirements for Demonstrating Method Acceptability for VOC Analysis from Canisters	**11.**	**TO1085**				
Does the lab meet the three performance criteria for an analytical system to qualify under Compendium Method TO-15? These criteria are: • **an MDL of no greater than 0.5 ppbv**, • **replicate precision within 25%,** and • **audit accuracy within 30%** for concentrations expected	**11.1.1**				**TO1086**	

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in contaminated ambient air (e.g. 0.5 to 25 ppbv).						
Is the procedure chosen to define the **method detection limit** the one specified in the Code of Federal Regulations (40 CFR 136 Appendix B)?	**11.2**				**TO1087**	
Is the measurement of replicate precision calculated according to the following procedure: The absolute value of the difference between replicate measurements of the sample divided by the average value, and expressed as a percentage (%RPD)?	**11.3**				**TO1088**	

APPENDIX B. COMMENT ON CANISTER CLEANING PROCEDURES

The canister cleaning procedures given in Section 8.4 require that canister pressure be reduced to <0.05mm Hg before the cleaning process is complete. Depending on the vacuum system design (diameter of connecting tubing, valve restrictions, etc.) and the placement of the vacuum gauge, the achievement of this value may take several hours. In any case, the pressure gauge should be placed near the canisters to determine pressure. The objective of requiring a low pressure evacuation during canister cleaning is to reduce contaminants. If canisters can be routinely certified (<0.2 ppbv for target compounds) while using a higher vacuum, then this criteria can be relaxed. However, the ultimate vacuum achieved during cleaning should always be <0.2mm Hg. Canister cleaning as described in Section 8.4 requires components with special features. The vacuum gauge must be capable of measuring 0.05mm Hg with less than a 20% error. The vacuum pump used for evacuating the canister must be noncontaminating while being capable of achieving the 0.05 mm Hg vacuum as monitored near the canisters. Thermoelectric vacuum gauges and turbomolecular drag pumps are typically being used for these two components.

An alternate to achieving the canister certification requirement of <0.2 ppbv for all target compounds is the criteria used in Compendium Method TO-12 that the total carbon count be <10ppbC. This check is less expensive and typically more exacting than the current certification requirement and can be used if proven to be equivalent to the original requirement. This equivalency must be established by comparing the total non-methane organic carbon (TNMOC) expressed in ppbC to the requirement that individual target compounds be <0.2 ppbv for a series of analytical runs.