Title: Performance Monitoring Protocol (QA/QC) for HS-GC Systems (TX-38)

1. Introduction

This document addresses the performance monitoring procedure for headspace-gas chromatographic (HS-GC) systems. This is a type of analytical instrument which is used to analyze volatile chemicals that are often released from evidentiary samples. They must be maintained in such a way as to verify their ability to produce valid results, often for use within judicial proceedings.

2. Scope

This procedure is used to ensure that an instrument is in an operating condition such that samples can be analyzed accurately, reliably, and in a defensible manner. Quality assurance and quality control (QA/QC) within a forensic laboratory is vitally important and enables results to be trusted. Samples and actions taken to maintain instrumentation are included within this procedure and are related to normal routine casework conducted within the Toxicology Unit. Situations where parameters get changed, significant differences within instrumentation occur, or instrument malfunction can utilize performance monitoring protocols such as this one so as to alert users when instruments (or other components) do not perform as expected.

The procedure does not necessarily need to be updated when used with matrices not commonly encountered within the Toxicology Unit. Additional matrices can be used and chemicals other than those that have been previously validated can be used within this procedure given that proper QA/QC measures are utilized and decision criteria are met.

3. Principle

This procedure involves the daily evaluation of equipment for the use of analyzing samples for the possible presence of volatile compounds, (e.g., methanol, ethanol, acetone, isopropanol, and similar compounds). The term daily refers to each day the instrument is used for analysis of critical samples (e.g., casework). It can be used for either a headspace-gas chromatographic (HS-GC-FID) or headspace-gas chromatographic/mass spectrometric (HS-GC(FID/MS)) system. Samples are typically in aqueous environments, are sealed within air-tight vials, and are examined using headspace analysis-type techniques. Volatile components, when placed in closed heated environments, reach an equilibrium between their vapors and their solutions. Vapor phases are analyzed with appropriate chromatographic columns and detectors (e.g., HS-GC/FID, HS-GC(FID/MS), dual column technique). Volatile components of solutions are identified and, using other procedures, can be subsequently quantitated.

One system is comprised of a headspace autosampler, a gas chromatograph (GC) with two (2) columns, and a flame ionization detector (FID). Another system is comprised of a headspace autosampler, a gas chromatograph with two (2) columns, and two (2) detectors (FID and mass spectrometer (MS)). The headspace autosampler, also called an autoinjector, is a device used to sample the gas phase volatile analytes within a sealed vial. This sampling is transferred to the inlet of the GC and onto columns where volatile components are separated and sent to detectors. There are two columns in the GCs (front and

back) and each lead to their respective detectors. In one system both columns lead to FID detectors. In the other system one capillary column leads to an FID detector and the other column leads to a mass selective detector (MSD). This performance monitoring protocol is based upon manufacturer recommendations.

4. Specimens

Any aqueous sample may be suitable for this analysis. When available, 0.2 mL of specimen is typically used within this procedure. Biological specimens may be analyzed using this procedure, if necessary.

5. Equipment/Materials/Reagents

5.1 General laboratory glassware (e.g., Class A glassware)

5.2 Headspace autosampler vials (e.g., 20 mL) with appropriate seals/caps and crimper.

5.3 Pipettes (e.g., automatic pipettor-diluter), or equivalent

5.4 Vortex mixer

5.5 Headspace-gas chromatograph with flame ionization detectors (HS-GC(FID)), Shimadzu (or equivalent) or Headspace-gas chromatograph with mass spectral detector (HS-GC(FID/MS)), Agilent (or equivalent)

5.6 Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)

5.7 Capillary Column #1: (Rtx-BAC1 ; Restek 18003, 30m, 0.32mm i.d., 1.8 µm, or equivalent)

5.8 Capillary Column #2 (Rtx-BAC2 ; Restek 18002, 30m, 0.32mm i.d., 1.2 µm, or equivalent)

5.9 Deionized water (DIW, Millipore, or equivalent)

5.10 Ethanol (EtOH ; anhydrous 200 proof USP, or equivalent)

5.11 Methanol (MeOH ; HPLC grade, or equivalent)

5.12 Acetone (HPLC grade, or equivalent)

5.13 Isopropanol (IPA ; HPLC grade, or equivalent)

5.14 n-Propanol (NPA ; HPLC grade, or equivalent)

5.15 $Ethanol_{(aq)}$ (80 $mg/dL_{(aq)}$; Certified Reference Material (CRM) Standard, Cerilliant E-030, or equivalent)

5.16 Sodium Azide (NaN_3 ; Baker, or equivalent)

5.17 Volatile Mix Performance Solution (MeOH, EtOH, Acetone, IPA ; 0.025 $g\%_{(aq)}$; Certified Reference Material (CRM), Cerilliant (A-127), or equivalent)

6. PFTBA Tuning Solution (MSD)

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD. Under normal conditions, this should not need to be refilled.

7. Preparation of Performance Solutions

An externally-prepared ethanol certified reference material (CRM) solution at 0.080 g% $(w/v)_{(aq)}$ and one (1) externally-prepared volatile mixture CRM solution (0.025 g%) will be analyzed for QA/QC instrument evaluation purposes. If these CRM solutions are unavailable then the Lead Examiner (or higher) will be notified and, upon written approval, equivalent QA/QC performance (e.g., Internally/In-House prepared) solutions can be used. If substitutions are used then these will be documented within appropriate instrument logbooks.

With the exception of the negative control and blanks, externally-prepared solutions will primarily be used for QA/QC purposes. Comparable in-house solutions will be used when external solutions are not available. The volatile mix and ethanol solutions that are used within this procedure will be separate from those solutions used in casework analyses. They should be labeled 'for QA/QC purposes only' (or similar verbiage) and the same solutions will be used for all HS-GC instrument performance evaluations. When new solutions are created they will be verified against previous solutions to ensure quality and for verification purposes. All such information will be captured within appropriate logbooks.
A typical daily QA/QC sequence will include the following (not in any particular order):

a) Negative Control (with internal standard)

b) Externally-Prepared Volatile Mix [CRM] Performance Solution (0.025 g% ; MeOH, EtOH, Acetone, IPA)

c) Externally-Prepared Ethanol [CRM] Performance Solution (0.080 g%)

d) Water Blank (without internal standard)

New reagents, performance solutions, and other types of solutions will be analyzed and verified for quality (and documented) prior to being used and replacing solutions of different lots. Such new solutions will typically be analyzed along with older (previously verified) reagents/solutions so that they can be compared and verified for quality purposes as being acceptable for use. If lot numbers of replacement solutions do not change then some designation should be added to differentiate newer solutions.

Note-01: Additional solutions (other than what are listed) can be used, whether they be externally purchased or internally prepared. Any solutions used in place of the ones listed in this procedure will need both FSE2 and DD (or higher) approval prior to instrument use for casework.

Note-02: Lot numbers on labels for solutions/reagents internally generated will be of the format: month, day, year, analyst's initials, and a sequential number: {Example: 08112018MPR} If multiple lots are made on the same date by the same analyst then a sequential number will be added: {Example: 08112018MPR-01, 08112018MPR-02}

Note-03: Throughout the procedure different volumes/amounts can be used as long as the same final concentrations are obtained. If different volumes are used, however, such differences will be noted within instrument logbooks. The FSE2 (or higher) should be notified just to make sure nothing in the procedure will be negatively impacted.

7.1 If used, check balance using calibrated weights on the day solutions are prepared. Record appropriate weight-checks accordingly.

Control and Blank Solutions

7.2 Negative Control Performance Solution (0.000 g% (w/v) of MeOH, EtOH, Acetone, IPA)

7.2.1 Place known DIW into a 1000 mL volumetric flask containing 0.2 g of sodium azide.

7.2.2 Dilute to volume with DIW and mix well.

7.2.3 Transfer into an amber bottle (or equivalent) and properly label.

7.2.4 This solution will be tightly capped and stored refrigerated. It is stable for at least one (1) year from the date of its last verification.

Note: 04: Alternate variations of preparing this solution are acceptable (e.g., 250 mL of water with 50 mg of sodium azide).

Internal Standard (I.S.) Solutions:

7.3 n-Propanol [$NPA_{(aq)}$] Internal Standard (I.S.) Stock Solution (5.6 g% $(w/v)_{(aq)}$; 7.0 % $(v/v)_{(aq)}$)

7.3.1 Transfer 7.0 mL of n-propanol into a 100 mL volumetric flask, dilute to volume with DIW, and mix well. (Note: density of NPA is 0.803 g/mL).

7.3.2 Transfer to an amber bottle (or equivalent) and properly label.

7.3.3 This solution will be tightly capped and stored refrigerated. It is stable for at least one (1) year from the date of its last verification.

7.3.4 Store refrigerated.

7.4 n-Propanol [$NPA_{(aq)}$] Internal Standard Working Solution
($0.011 g\%\ (w/v)_{(aq)}$; $0.014\ \%\ (v/v)_{(aq)}$)

7.4.1 Transfer 4.0 mL of the $NPA_{(aq)}$ I.S. Stock Solution into a 2.0 L volumetric flask, dilute to volume with DIW, and mix well.

7.4.2 Transfer to an appropriate sealed container and properly label.

7.4.3 Label the vial appropriately.

7.4.4 This solution will be tightly capped and can be stored at room temperature. It is stable for at least one (1) year from the date of its last verification.

Note-05: The same internal standard solution used for casework can be used within this QA/QC procedure. Unlike the performance solutions, there is no need to have a separate internal standard solution just for QA/QC purposes.

Externally-Prepared Performance Solutions:

7.5 Externally-Prepared Volatile Mix [CRM] Performance Solution (0.025 g% (w/v))
(Volatile Mixture: $MeOH_{(aq)}$, $EtOH_{(aq)}$, $Acetone_{(aq)}$, $IPA_{(aq)}$)

7.5.1 This solution should initially be supplied as a 1.2 mL mixture consisting of 250 µg/mL concentrations of MeOH, EtOH, acetone, and isopropanol in water in ampules. No dilutions are necessary.

7.5.2 Transfer solutions into a properly labeled container and cap so as to ensure no volatile leakage after use.

7.5.3 Store refrigerated.

7.6 Externally-Prepared $Ethanol_{(aq)}$ [CRM] Performance Solution
($EtOH_{(aq)}$; $0.080\ g\%\ (w/v)_{(aq)}$)

7.6.1 This solution should initially be supplied as a 1.2 mL mixture consisting of 80 mg/dL concentrations of MeOH, EtOH, acetone, and isopropanol in water in ampules. No dilutions are necessary.

7.6.2 Transfer solutions into a properly labeled container and cap so as to ensure no volatile leakage after use.

7.6.3 Store refrigerated.

In-House (Internally-Prepared) Volatile Mix Performance Solution
(used only when External Solutions are not available):

7.7 In-House Volatile Mix Performance Solution
($MeOH_{(aq)}$, $EtOH_{(aq)}$, $Acetone_{(aq)}$, $IPA_{(aq)}$)
(0.010 g% (w/v)) ; (0.025 g% (w/v))

7.8 Performance Stock Solution (PSS ; 1.0 g% $(w/v)_{(aq)}$ each of MeOH, EtOH, Acetone, IPA)

7.8.1 Accurately weigh 5.000g (+/- 0.002g) of methanol, ethanol, acetone, and isopropanol. Quantitatively transfer into a 500 mL volumetric flask with DIW.
Add 0.1 g of sodium azide, dilute to volume with DIW, and mix well.

7.8.2 Transfer the PSS into an amber bottle (or equivalent) and properly label with lot number and other information (a properly capped volumetric flask with parafilm is considered equivalent).

7.8.3 This solution will be tightly capped to ensure no volatile leakage and will be stored refrigerated. It is stable for at least one (1) year from the date of its last verification.

7.9 In-House Volatile Mix Performance Solution (0.025 g% $(w/v)_{(aq)}$; 0.025 g/100 mL)

7.9.1 Using a volumetric pipette add 2.5 mL of the 1.0 g% (w/v) Performance Stock Solution (PSS) to a 100 mL volumetric flask, dilute to volume with DIW, and mix well.

7.9.2 Transfer into an amber bottle (or equivalent) and properly label with lot number and other information.

7.9.3 This solution will be tightly capped to ensure no volatile leakage and will be stored refrigerated. It is stable for at least one (1) year from the date of its last verification.

7.10 In House-Prepared $Ethanol_{(aq)}$ Performance Solution
($EtOH_{(aq)}$; 0.080 g% $(w/v)_{(aq)}$)

7.10.1 Accurately weigh 0.800g (+/- 0.002g) of ethanol and quantitatively transfer into a 1000 mL volumetric flask with DIW containing 0.2 g of sodium azide.
Dilute to volume with DIW and mix well.

7.10.2 Transfer to an amber bottle (or equivalent) and properly label.

7.10.3 This solution will be tightly capped to ensure no volatile leakage and will be stored refrigerated. It is stable for at least one (1) year from the date of its last verification.

8. Procedure

8.1 Record the remaining disk space from the instrument's hard drive. Do not use if less than 100 MB of disk space remain. If not recently performed, data backups should occur at the beginning of each month.

8.2 Check the time and date on both the computer (and instrument, if applicable) to ensure they are correct.

8.3 Record the line pressure of the helium supply (carrier gas). Record both the regulator and tank pressures. Change the tank if less than 100 psi remains.

8.4 Record hydrogen pressure and ensure water level is appropriate within generator. Add deionized water, if needed.

8.5 Ensure that the detectors are operating (i.e., FID is lit and perform an autotune on the MSD using ATUNE). Evaluate according to the Decision Criteria within this procedure. Save a print out of the autotune results and place within the appropriate instrument logbook.

8.6 Place all solutions within a room temperature environment for approximately ten (10) minutes.

8.7 Prepare or acquire necessary performance solutions.

8.7.1 Label vials appropriately.

8.7.2 Individually add 200 µL of sample and 1 mL of internal standard ($NPA_{(aq)}$) into separate autosampler vials and cap.

Note-06: While using the pipette/Diluter is preferred, manual pipetting is acceptable.

Note-07: Internally prepared performance solutions are acceptable if external solutions are unavailable.

Note-08: The QA/QC instrument performance must be evaluated on a daily basis and prior to samples being analyzed. If sequences continue into the next day, then the current sequence need not be interrupted. However, a new QA/QC evaluation will begin prior to a new sequence/batch being analyzed. Additional QA/QC analyses may be performed more frequently.

Setting-up Instrument with Samples and Analysis:

8.8 Ensure air and hydrogen are flowing and ensure detectors (flame ionization detector (FID) and/or mass spectrometer (MS) are operational).

8.9 Prepare the sequence table. Ensure lot number information accompanies each sample within the sequence.

8.10 Place the labeled headspace vials in the appropriate order within the instrument.

8.11 Save the sequence/Batch Table using the day's date and the analyst's initials (e.g., MPR-02262020).

8.12 Prior to analyzing samples:
Print the sequence list. Check that the physical placement of the headspace vials and the vial positions within the instrument's sequence list match. Once the check has been completed place an indication of the sample check (e.g., 'sequence checked' or 'sequence verified') on the sequence page along with Analyst's initials and date.

8.13 Ensure there's a printout of the instrument method in the logbook. Evaluate that method printout with the one which is ready to be used in the sequence to ensure that all parameters are correct.

8.14 Begin the sequence, analyze the samples, and evaluate the data.

8.15 Place the sequence and relevant data printouts into the appropriate logbook.

8.16 Update the logbook with results of the instrument evaluation. Notify the lead Examiner (and higher) if anything fails that can't be fixed.
Record all problems and resolutions within the appropriate logbook.

9. Instrumentation

Instrumental Parameters:

The following are the typical operating parameters for the instrument used in this procedure. With approval from the Lead Examiner and Deputy Director (or higher), the instrument conditions may be modified to adjust or improve the procedure. Documentation of such changes must be included within the appropriate logbook so that any instrumental parameter change can be identified.
The appendix contains an abbreviated version of the procedure. The checklist can be used by analysts. Any changes within the instrumental parameters, if listed on the checklist, will be reflected on the checklist by the analyst filling it out and making appropriate corrections.

HS-GC(FID):
The following parameters can be used. Some values may change due to slight variability (e.g., flow rates). Significant differences must be approved (FSE2 and higher) prior to changing.

AOC-5000	Parameters		AOC-5000	Parameters
Cycle	HS-inj		**Syringe Temperature**	75 C
Syringe	2.5 mL-HS		Fill Speed	500 µL/sec
Sample Volume	850 µL		Pull Up Delay	500 msec
Incubation Temperature	50 C		Inject To	GC Injector 1
Incubation Time	4 min.		Injection Speed	1 mL/sec
Agitation Speed	300 rpm		Pre-Injection Delay	500 msec
Agitation On	3 sec.		Post-Injection Delay	0 msec
Agitation Off	1 sec.		Syringe Flushing	25 sec.
Oven Temp	55°C		**Injection Port SPL1**	
Sample Line Temp	150°C		Injection Mode	Split
Transfer Line Temp	150°C		Carrier Gas	He
Shaking Level	1		Flow Control Mode	Velocity
Multi Injection Count	1		Pressure	136.6 kPa
Multi Injection Count	1		Total Flow	35.0 mL/min.
Pressurizing Gas Pressure	50.0 kPa		Column Flow	5.00 mL/min.
Equilibrating Time	15.00 min.		Linear Velocity	65.1 cm/sec.
Pressurizing Time	0.15 min.		Purge Flow	10.0 mL/min
Pressure Equilib. Time	0.35 min.		Split Ratio	4.0
Load Time	0.50 min.		Injection Volume (mL)	850 µL
Autosampler (HS-20)	**Parameters**		**Autosampler (HS-20)**	**Parameters**
Load Equilib. Time	0.25 min.		Needle Check	Yes
Injection Time	0.50 min.		Action Leak Check Error	Continue
Needle Flush Time	0.50 min.		Action - No vial on tray	Skip
GC Cycle Time	4.51 min.			
Check GC Ready	On		**Column Oven**	
Ext. GC Ready Check Limit	10 min.		Initial Temperature	50.0°C
Check System Ready	On		Equilibration Time	0.0 min
Ext. Sys. Ready Check Limit	45 min.		GC Run Time	4 min
Analysis Mode	Constant		Ramp Rate	0°C/min
			Final Temp.	50.0
			Hold Time	4.00

* or equivalent

HS-GC(FID) (Cont'd):

Column Information	FID 1	FID 2
Column Names	**Rtx-BAC1**	**Rtx-BAC2**
Column Cat. #	18003	18002
Film Thickness	1.80 µm	1.20 µm
Column Length	30.0 m	30.0 m
Inner Diameter	0.32 mm	0.32 mm
Column Max Temp	260°C	260°C
Detector	**FID 1**	**FID 2**
Temperature	260°C	260°C
Sampling Rate	40 msec.	40 msec.
Stop Time	4.00 min	4.00 min
Delay Time	0.00 min	0.00 min
Subtract Detector	None	None
Makeup Gas	He	He
Makeup Flow	30.0 mL/min	30.0 mL/min
H2 Flow	40.0 mL/min	40.0 mL/min
Air Flow	400.0 mL/min	400.0 mL/min

HS-GC(FID/MS):
The following parameters can be used. Some values may change due to slight variability (e.g., flow rates). Significant differences must be approved by Unit Lead (and higher) prior to changing.

Autosampler	Parameters		Autosampler	Parameters
GC Cycle Time	6.5 min.		Sample Vial Penetration	15 mm
Sample Volume	0.5 mL		Sample Vial Penet. Speed	50 mm/sec
Incubation Time	5 min.		Sample Aspiration Rate	12 mL/min
Incubation Time Increment	0 min.		Sample Post Asp. Delay	1 sec.
Heat Agitator	On		Inlet Penetration Depth	45 mm
Incubation Temperature	50 C		Inlet Penet. Speed	50 mm/sec
Heat Syringe	On		Pre-Inject Time Delay	0.5 sec.
Pre-Injection Flush Time	5 sec.		Inject Flow Rate	10 mL/min.
Agitator Speed	250 rpm		Post Injection Delay	0.5 sec
Agitator On Time	5 sec.		Flush Time	10 sec.
Agitator Off Time	5 sec.		Continuous Flush	Off
GC	**Parameters**		**Injector**	**Parameters**
Run Time	6.5 min.		Mode	SPLIT
Post Run Time	0 min.		Split Ratio	10:1
Oven Equilibration Time	0 min.		Split Flow	46.049 mL/min.
Max. Oven Temperature	250 C		Injector Heater	250 C
Initial Temperature	47 C		Injector Pressure	9.5 psi
Hold Time	6.5 min.		Injector Total Flow	53.654 mL/min.
Post Run Temperature	100 C		Septum Purge Flow	3 mL/min.
			Gas Saver	On
Transfer Line	**Parameters**			
Temperature	200 C			

HS-GC(FID/MS) (Cont'd):

Column Information	Agilent	Agilent
Column Names	DB-BAC-1 Ultra Inert (Col. #2)	DB-BAC-2 Ultra Inert (Col. #3)
Column Cat. #	123-9334UI	123-9434UI
Film Thickness	1.8 µm	1.2 µm
Column Length	30 m	30 m
Inner Diameter	320 µm	320 µm
Column Max Temp	260°C	260°C
Pressure (initial)	7.9 psi	7.9 psi
Pressure (post run)	3.8589	3.8589 psi
Flow	2.6857 mL/min.	1.5733 mL/min.
Ave. Velocity	59.637 cm/sec.	27.599 cm/sec.
Holdup Time	0.8384 min.	1.8117 min.
Detector	**MS**	**FID (Front Detector)**
Temperature (Source)	230 C	250 C
Temperature (Quad)	150 C	–
Sampling Rate	–	20 Hz (Signal #1)
Makeup Gas	–	He
Makeup Flow	–	25 mL/min.
H_2 Flow	–	30 mL/min.
Air Flow	–	400 mL/min.
Ion Source	EI	–
Fixed Electron Energy	70 eV	–
Solvent Delay	1 min.	–
Gain Factor	2	–
EM Saver	False	–
Acquisition Type	Scan	–
Start Mass	29 m/z	–
End Mass	200 m/z	–
Threshold	0	–
Scan Speed	781 (N=3) Scan Speed	–

10. Decision Criteria

The following criteria are used as guidelines in determining the acceptability of the data produced in this procedure. Retention time (chromatographic characteristic), peak shape, and signal-to-noise (aka: integrated peaks) are used as the basis for peak detection. In most cases all of the criteria below will be met in order to ensure instrument operability for casework.

10.1 Chromatography:

Chromatographic peaks will possess good chromatographic quality (i.e., Gaussian peak shape, reasonable peak width, distinguishing signal-to-noise). In order for a chromatographic peak to be deemed acceptable it will compare favorably to corresponding chromatographic peaks from the previous day's successful QA/QC evaluation.

10.1.1 Retention Time (RT):

Retention times of chromatographic peaks of interest should compare favorably (i.e., be within ±0.1 minute) to retention times of related peaks from the previous day's successful QA/QC evaluation. The relative retention times (RRTs) of each of the analytes within the performance mix solution (methanol, ethanol, acetone, and isopropanol) to the internal standard (NPA) can be compared. These values should not change from the previous day's RRTs by more than one percent (1%). If they do change by more than 1% then contact an FSE2 (or higher) for guidance. Decisions as to how to proceed can be noted within appropriate log sheets.

10.1.2 Signal-to-Noise Ratio (SNR):

To justify the existence of a chromatographic peak, the peak's baseline signal-to-noise ratio (SNR) based on height should equal or exceed 3.

10.1.3 Acceptability of Chromatographic Data:

Chromatographic peaks must be present for all four (4) analytes within the 0.025 g% Volatile Mix [CRM] Performance Solution. The relative areas for each component (MeOH, EtOH, IPA, Acetone) should be greater than 0.05, 0.10, 0.5, 1.5, respectively. The relative area for the 0.080 g% ethanol chromatographic peak (from mass spectral data) should be greater than 0.50. Using the 'Percent Report' function can assist with obtaining this information.

Note-09: Relative areas for analytes are analyte chromatographic peak areas divided by the internal standard peak area. These areas can either me automatically or manually integrated.

10.2 Mass Spectrometry (if applicable)

In order for the instrument to be ready for use with casework, correct mass spectra for each analyte must be present. Mass spectra, along with their ratios, will compare favorably to mass spectra from the previous day's successful QA/QC evaluation (or to acceptable library reference spectra).

10.2.1 Mass Spectral Tune:

Verify the results of the tune. Compare the results of the tune to previous tune results from successful QA/QC evaluations. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures. The following are typical ATUNE values for the MSD:

10.2.1.1 PFTBA Tune: Mass ±0.4 for m/z 69, 219, and 502

10.2.1.2 Peak width (Pw50): 0.45–0.65

10.2.1.3 Isotope Ratios:
69 (0.5%–1.5%)
219 (2%–8%)
502 (5%–15%)

10.2.1.4 Relative abundance:
69 equal to 100 %
219 greater than 30 %
502 greater than 1 %

10.2.2 Mass Spectra:

No significant peaks of analytes not expected within QA/QC solutions will be present. Significant peaks are considered peaks which have been automatically integrated and/or which negatively affect the quality of the results.

11. Safety

This procedure is carried out in a laboratory environment and standard safety procedures appropriate for such an environment will be utilized, including gloves, safety glasses, and protective clothing (e.g., lab coat). Biological specimens will be handled using universal precautions and will be treated as biohazardous. Potentially contaminated items and surfaces will be cleaned prior to use.

12. References

Manufacturer's Instrument Manuals

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Appendix:
QA/QC Procedure for HS-GC(FID) and/or HS-GC(FID)/MS)

The following is an abbreviated version of this procedure and a pdf-version can be used.
This form may be used at the bench level by the analyst performing the procedure.

This form can be placed in the instrument logbook along with data printouts. When lot numbers to solutions change this form can be used to document such changes and can be placed in front of subsequent printouts to identify which reagents were used in subsequent QA/QC evaluations.

Neg. Ctrl. Performance Sol'n $[volatile\text{-}free]_{(aq)}$	**Lot:**
Volatile Mix Performance $Soln_{(aq)}$ Externally Prepared / In-House	**Volatile Mix Perf. Sol'n: 0.025 g% Lot:**
Ethanol Performance $Solution_{(aq)}$ Externally Prepared / In-House	**Ethanol: 0.080 g% Lot:**
n-Propanol Internal Standard Sol'n	**Lot:**
Other:	**Type: Lot:** **Type: Lot:** **Type: Lot:** **Type: Lot:** **Type: Lot:**

Procedure:

	For Blood or Urine (or Other Matrix) Specimens
Place samples at room temperature.	
Check hard drive disk space. Ensure data backup (monthly). Check time/date.	
Check gases (He, H_2) and fluid levels.	
Ensure detectors are operating. Autotune Mass Spectrometer (if applicable).	
Prepare or acquire solutions (e.g., Three – Neg. Ctrl., Vol. Perf. Mix, EtOH)	
Prepare automatic Pipettor/Dilutor and prime (if using)	
Prepare samples (i.e., 1 mL of I.S.(n-$propanol_{(aq)}$) + 200 µL sample)	
Cap and label vials appropriately	
Between-sample rinse (if automatic Pipettor/Dilutor is used)	
Create sequence within instrument software, save, and print. Verify vial positions, document and initial/date.	
Ensure methods and parameters are correct and print copy – initial/date.	
Analyze samples	
Evaluate data based on Decision Criteria	
Tune File data acceptable	
Relative Retention Times (RRT) Not Greater Than 1% from Previous Day's RRTs	
Rel. Area's for 0.025 g% Vol. Mix At Least: 0.05, 0.20, 3.0, 0.7 (MeOH, EtOH, Acetone, IPA) and 0.60 for EtOH	

Rev. #	History
1	New document
2	Updated 10.1.2 and added 10.1.3 section which included updated criteria for peak acceptability based on relative areas. Updated Note-09. Section 10.2.1.3 - Isotope Ratios: "210" changed to "219." Before section 8.8 added "and Analysis." Within section 7.4 made a calculation correction. Updated section 8.13 – 8.15 to not require printout of method and other minor changes.