2022-S-0006 Standard Practice for Gas Chromatography Electron Ionization Mass Spectrometry Analysis of Ignitable Liquids

Ignitable Liquids, Explosives & Gunshot Residue Subcommittee
Chemistry: Trace Evidence
Organization of Scientific Area Committees (OSAC) for Forensic Science

Draft OSAC Proposed Standard

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Standard Practice for
Gas Chromatography Electron Ionization Mass Spectrometry Analysis of Ignitable Liquids

1. Scope

1.1 This practice covers the instrumental analysis of ignitable liquids, as well as extracts from fire debris samples, by gas chromatography-electron ionization mass spectrometry (GC-MS).

1.2 This practice describes performance criteria for use during initial GC-MS method development and optimization, data evaluation and acceptance criteria, quality assurance and quality control considerations, and limitations.

1.3 *This standard is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practice E2917), and demonstrated proficiency to perform forensic casework.*

1.4 *The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.*

1.5 *This standard does not purport to address all of the possible safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*[1]:

E1386 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

E1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples

[1] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal

E1413 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration onto an Adsorbent Tube

E(CLASS) Classification for Ignitable Liquids Encountered in Forensic Fire Debris Analysis

E(INTRP) Test Method for Interpretation of Gas Chromatography-Electron Ionization Mass Spectrometry Data for the Identification of Ignitable Liquid Classes in Forensic Fire Debris Analysis

E1732 Terminology Relating to Forensic Science

E2154 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction (SPME)

E2451 Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples

WK72631 (E2549 Revision) Practice for Validation and Verification of Analytical Methods for Forensic Science Service Providers Performing Forensic Chemistry Analysis

E2881 Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry

E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

E3189 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Static Headspace Concentration onto an Adsorbent Tube

E3197 Terminology Relating to Examination of Fire Debris

E3245 Guide for Systematic Approach to the Extraction, Analysis, and Classification of Ignitable Liquids and Ignitable Liquid Residues in Fire Debris Samples

E3255 Practice for Quality Assurance of Forensic Science Service Providers Performing Forensic Chemical Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms that can assist in interpreting this Practice, refer to Terminology E1732, Terminology E3197, and Classification E(CLASS).

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 **analytical batch**, *n*—a set of samples that are analyzed together on the same instrument; also known as a sample sequence

3.2.2 **carryover**, *n*—material left over from a previous sample that results in signal that is observed in the data of a subsequent sample

3.2.3 **GC-MS method**, *n*—the complete set of instrument parameters utilized in the analysis of a test sample.

3.3 *Abbreviations*:

3.3.1 **C#** – total number of carbon atoms associated with the substitutions on a base molecule; for example, C3 alkyl benzenes include n-propyl benzene, 3-ethyl toluene, 1,2,4-trimethyl benzene, etc.

4. Significance and Use

4.1 This Practice is useful for generating GC-MS data from ignitable liquids and extracts from samples suspected to contain ignitable liquid residues. Refer to Guide E3245 for perspective on the use of GC-MS within the framework of ignitable liquids analysis. Refer to Practices E1386, E1388, E1412, E1413, E2154, and E3189 for sample preparation procedures. Refer to Test Method E(INTRP) for interpretation of data generated using this Practice.

4.2 This Practice is intended to be used in conjunction with quality assurance procedures covered in Practice E3255.

5. Apparatus

5.1 Gas Chromatograph (GC)—A chromatograph capable of using capillary columns and being interfaced to a mass spectrometer.

5.1.1 Sample Inlet System—A sample inlet system that can be operated in either split or splitless mode with capillary columns.

5.1.2 GC Oven—A column oven capable of reproducible temperature program operation in the range from at least 35 to 300 °C.

5.1.3 Column—A capillary, bonded phase, methylsiloxane or phenylmethylsiloxane column or equivalent.

5.1.3.1 Alternate column stationary phases can be used for targeted analyses. As an example, screening for oxygenated compounds could be improved by use of a more polar column than those listed above.

5.2 Mass Spectrometer (MS)—A mass selective detector capable of acquiring mass spectra from m/z 10 to m/z 400 with unit resolution or better.

5.3 Data Station—A computerized data station capable of performing the following functions in conjunction with the GC-MS, either through its operating system or by user programming:

5.3.1 Recording instrument parameters used during sample runs.

5.3.2 Storing time sequenced mass spectral data from sample runs.

5.3.3 Retrieving and displaying sample data files.

5.3.4 Generating extracted ion profiles.

5.3.5 Retrieving a specified mass spectrum from a data file and comparing it against one or more mass spectral libraries.

5.3.6 Preparing data presentations in electronic or hard copy format.

5.4 Mass Spectral Library—One or more searchable databases containing mass spectra of individual chemical compounds.

5.5 Sample introduction devices.

5.5.1 For liquid samples, a syringe capable of introducing a sample size appropriate for the volume of the inlet upon expansion of the injected solvent. Typical liquid sample sizes are in the range from 0.1 to 10 microliters.

5.5.2 For vapor samples, a vapor-tight syringe capable of introducing a sample size appropriate for the volume of the inlet. Typical vapor sample sizes are in the range from 0.5 to 5 milliliters.

5.5.3 For some adsorbent tubes (see Practices E1413 and E3189) and for SPME fibers (see Practice E2154), a system capable of desorbing the trapped volatile compounds and introducing them into the GC-MS.

6. Chemicals, Reagents, and Reference Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals or better. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[2] Other grades can be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Solvent/Diluent—Use carbon disulfide, diethyl ether, pentane, or other solvent that does not interfere with the analysis. It is generally desirable to use a solvent whose volatility greatly exceeds that of most expected solutes to facilitate sample concentration by evaporation, if necessary. The polarity of the solvent should also be considered.

6.3 Carrier Gas—Use helium or hydrogen of purity 99.995% or higher.

6.4 Test Mixture—Use a test mixture that is suited to the GC-MS method being utilized. More than one test mixture can be used.

6.4.1 The composition of a general-purpose ignitable liquids test mixture at minimum consists of the following components diluted in solvent:

6.4.1.1 A mixture of alkanes (at least five normal alkanes ranging from n-hexane to n-eicosane) and aromatic compounds that cover the approximate volatility and polarity range of the ignitable liquid classes for which the GC-MS method was developed, and

6.4.1.2 At least one pair of closely-eluting compounds, such as isomers or closely-eluting oxygenates, to facilitate the evaluation of the adequacy of the resolution of the GC-MS method.

6.4.2 The composition of a targeted test mixture consists of compounds that cover the volatility range and composition of interest.

6.4.3 Oxygenated compounds can be added to the general-purpose ignitable liquids test mixture or prepared as a separate targeted test mixture.

6.4.4 Examples of suitable test mixtures are listed in Table 1. Refer to Test Method E2881 for information about test mixtures for use with vegetable-oil based (VOB) products.

[2] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Table 1: Examples of possible test mixtures.

General Purpose Test Mixture with Oxygenated Compounds	General Purpose Test Mixture without Oxygenated Compounds	Targeted Test Mixture for Oxygenated Compounds
Ethanol	N-alkanes (C_8-C_{20})	Ethanol
Acetone	Methylcyclohexane	Acetone
2-Propanol (Isopropanol)	1-methyl-2-ethylbenzene	2-Propanol (Isopropanol)
2-Butanone	1-methyl-3-ethylbenzene	2-Butanone (Methyl ethyl ketone)
Even-numbered n-alkanes (C_6-C_{20})	1,3,5-trimethylbenzene	
Toluene	1-methyl-4-ethylbenzene	
p-xylene		
1-methyl-2-ethylbenzene		
1-methyl-3-ethylbenzene		
1,2,4-trimethylbenzene		

6.5 Reference Ignitable Liquids—Maintain reference ignitable liquids for the various ignitable liquid classes defined in Table 1 of Practice E(CLASS).

6.5.1 Reference ignitable liquids can be obtained from commercial or retail sources. Certified ignitable liquid standards are not required.

6.5.2 Assess reference ignitable liquids in accordance with Appendix 2 of Practice E3255 prior to use.

7. Instrument Maintenance

7.1 Establish and follow an instrument maintenance routine that takes into consideration the recommendations of the instrument manufacturer and the overall use of the equipment.

7.1.1 Continually evaluate data features to determine if maintenance is necessary to optimize performance. Data features that can indicate a need for maintenance include changes in retention time, peak abundance, peak shape, or the presence of unexplainable or extraneous peaks.

7.2 Clean syringes thoroughly with a suitable solvent (see Section 6.2) between injections to minimize the potential for carryover.

7.2.1 Analyzing a solvent blank between samples is recommended but is not required if studies performed in accordance with Annex A1 demonstrate that the cleaning procedure prevents carryover.(**1**)[3]

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

8. GC-MS Method Development: Performance Criteria and Optimization

8.1 Any GC-MS method can be utilized for ignitable liquids analysis, provided it meets the following performance criteria:

8.1.1 *All components of interest are eluted*.

8.1.1.1 For a general-purpose ignitable liquids GC-MS method, demonstrate that, at a minimum, light, medium, and heavy range components are present in the total ion chromatogram (TIC).

NOTE: If light oxygenated compounds (e.g. methanol, ethanol, acetone) are not included in a general purpose ignitable liquids GC-MS method, develop a separate targeted GC-MS method for the analysis of these compounds.

8.1.1.2 For a targeted GC-MS method, demonstrate that the compounds of interest are present in the TIC.

8.1.2 *Peak shapes are approximately Gaussian with narrow widths*.

8.1.2.1 Gaussian (i.e. symmetrical "bell-shaped") peaks are ideal; however, some asymmetry and tailing is normal with temperature gradient programs.(**2**, **3**)

8.1.2.2 Some factors that can affect peak width are column specifications, column efficiency, temperature program(s), GC flow rate, and sample concentration.(**4**, **5**)

NOTE: Column efficiency is the relationship between a peak's retention time and its width.(**5**) Optimum column efficiency for practical performance in ignitable liquids analysis is that which results in peaks with the shortest retention time that allows for the desired resolution.

8.1.3 *Peak resolution is sufficient for the recognition of patterns associated with the components of interest, or identification of targeted components.* Due to the number of similar compounds present in ignitable liquids, baseline resolution of all compounds is not necessary.

8.1.3.1 For a general-purpose ignitable liquids GC-MS method, at a minimum, demonstrate that peak resolution in the C3 and C4 alkyl benzene groups in gasoline, and

peak resolution in the normal alkanes pattern in diesel fuel, is sufficient for recognition of key diagnostic features, as discussed in E(CLASS).

8.1.3.2 Adequate peak resolution can usually be achieved through the selection of appropriate column specifications and gas chromatographic method parameters.

8.1.4 *Retention times of components of interest are sufficiently reproducible.*

8.1.4.1 For a general-purpose ignitable liquids GC-MS method, at a minimum, demonstrate that peaks in the light range (e.g. toluene), medium range (e.g. 1,2,4-trimethylbenzene), and heavy range (e.g. n-hexadecane) have sufficiently reproducible retention times (e.g. no more than the greater value of either ±1% or ±0.1 minute (**6**) deviation from the mean value from 10 repetitions performed during the method development process).

8.1.4.2 For a targeted GC-MS method, at a minimum, demonstrate that the peak for one compound of interest has sufficiently reproducible retention times (e.g. no more than the greater value of either ±1% or ±0.1 minute (**6**) deviation from the mean value from 10 repetitions performed during the method development process).

8.1.5 *Mass spectra are collected over one or more mass-to-charge (m/z) ranges which encompass the relevant ions used for identification of the components of interest.*

8.1.5.1 For a general-purpose ignitable liquids GC-MS method, start scans at a m/z value no greater than 33 for the lower limit, and scan to an upper limit at or greater than a m/z value of 400.

NOTE: If light oxygenated compounds (e.g. methanol, ethanol, acetone) are included in a general-purpose ignitable liquids GC-MS method, start scans at a lower m/z value (e.g. 10).

8.1.6 *Relevant ions in the mass spectra of the components of interest are of sufficient intensity for identification of the components, either by computer library search or by comparison with reference mass spectra, in accordance with Test Method E(INTRP).*

8.1.6.1 Contributions from extraneous ions are minimized in the mass spectra. Some sources of extraneous ions are column bleed, carrier gas impurities, detector noise, cleanliness of the mass selective detector, and poor vacuum performance.

8.1.6.2 Parameters that can affect the intensity of ions in the mass spectra include the mass threshold, analog-to-digital (A/D) samples, tune method (e.g. auto tune, standard spectra tune, etc.), and tune parameters.

8.2 Optimize GC-MS methods for sensitivity, to reduce the potential for carryover, and for run time efficiency while also meeting the performance criteria in 8.1.

8.2.1 Sensitivity can be optimized by adjusting injection, inlet, and detector parameters.

8.2.2 Reducing the potential for carryover can be optimized by adjusting injector parameters or syringe wash parameters, or both, and by running blanks.

8.2.3 Run time efficiency (i.e. minimization of total run time that satisfies performance criteria) can be optimized by adjusting temperature, flow, and column parameters.

8.3 Examples of GC-MS method parameters for the analysis of samples suspected to contain ignitable liquids are provided in Appendix 1.

9. Sample Preparation, Analysis, and Preservation

9.1 Prepare samples in accordance with Section 9 of Guide E3245 in conjunction with one or more of Practices E1386, E1388, E1412, E1413, E2154, or E3189, as appropriate.

9.2 Analyze the sample by GC-MS.

9.2.1 Use an appropriate GC-MS method that has been developed and optimized for use in ignitable liquids analysis (see Section 8) and validated in accordance with Practice E2549.

9.2.2 Use an appropriate sample introduction device (see Section 5.5) to deliver the sample to the GC-MS.

9.3 When analysis is complete, preserve and store samples according to Practice E2451.

10. Data Evaluation and Acceptance Criteria

10.1 For all sample types, verify that the sample was properly introduced to the GC-MS, that the instrument performed correctly, and that data were recorded.

10.1.1 Confirm that a TIC and mass spectra were recorded.

10.1.2 Evaluate chromatographic features of the TIC.

10.1.2.1 Confirm that peaks are present in the TIC as appropriate for the sample and GC-MS method utilized.

NOTE: A blank sample run using a GC-MS method with a solvent delay might not contain any peaks.

10.1.2.2 Confirm that most peak shapes are approximately Gaussian with narrow widths (see also Section 8.1.2).

10.1.2.3 Confirm that the baseline is stable (e.g. with respect to drift, noise, or spikes).

10.2 For blanks, also confirm that there are no peaks present in the TIC that correspond to compounds of interest that would interfere with the ability to interpret the data for the presence of ignitable liquids, in accordance with Test Method E(INTRP).

10.3 For test mixtures, also confirm that retention times for selected peaks are within the greater value of either ±1% or ±0.1 minute (**6**) of the retention times established during development and optimization from previously established values for the GC-MS method used.

10.3.1 A sudden shift of retention times or decrease in abundances could indicate that instrument maintenance is necessary.

10.3.2 If maintenance is conducted that would reasonably be expected to change retention times (e.g. column trimming or column change) or abundances (e.g. ion source cleaning), run the test mixture a minimum of three times to determine the new retention times or abundances to be used for comparison.

10.4 Reject data from samples that do not satisfy the evaluation criteria as follows:

10.4.1 For questioned samples, use the criteria in Section 10.1.

10.4.2 For blanks, use the criteria in Sections 10.1 and 10.2.

10.4.3 For test mixtures, use the criteria in Sections 10.1 and 10.3.

10.5 Samples that produce rejected data can be reanalyzed. Additional sample treatment, such as dilution of overconcentrated samples, can be utilized as needed prior to reanalysis.

10.6 Maintain records of rejected data, any additional sample treatment utilized, and any reanalysis performed as a portion of the casework documentation.

11. Quality Assurance and Quality Control

11.1 Validate or verify GC-MS instruments and GC-MS methods according to Practice WK72631 and Practice E3255 in order to demonstrate their suitability for use in ignitable liquids analysis prior to use on unknown samples.

11.1.1 Maintain records of all validations and verifications in accordance with Practice WK72631 and Practice E3255.

11.2 Tune the mass spectrometer in accordance with manufacturer's recommendations in order to ensure correct calibration of mass to charge ratios and to optimize mass spectrometer system performance.

11.2.1 Tune using perfluorotributylamine (PFTBA), or another generally accepted tuning compound in accordance with manufacturer's recommendations.

11.2.2 Evaluate tune results against acceptance criteria established in accordance with manufacturer's recommendations.

11.2.3 Maintain tuning records as a portion of either the quality control documentation or the casework documentation.

11.3 Analyze at least one process blank per analytical batch in order to demonstrate that materials such as solvents, adsorption media, glassware, and other sample processing hardware are free from contaminants that can cause interferences.

11.3.1 Use the type of blank appropriate for the phase (i.e. vapor or liquid) of the questioned sample to be analyzed and, if applicable, to the method used to isolate the questioned sample from the original fire debris material (see Practices E1388, E1412, E3189, E1413, E2154, and E1386).

11.3.2 Evaluate the data from blanks according to Sections 10.1 and 10.2.

11.3.3 Maintain data from blanks as a portion of the casework documentation.

11.4 Analyze at least one test mixture at the beginning of each analytical batch in order to demonstrate proper instrument performance with respect to the components of interest.

11.4.1 Use a test mixture that is appropriate for the GC-MS method being performed (see Section 6.4).

11.4.2 Evaluate the data from test mixtures to confirm proper instrument performance according to Sections 10.1 and 10.3.

11.4.3 Maintain data from test mixtures as a portion of the casework documentation or the quality control documentation, or both.

11.5 Maintain data from reference ignitable liquids that are to be used for the interpretation of questioned samples and that have been analyzed using the same GC-MS methods as the questioned samples.

12. Limitations

12.1 In general, analysis of ignitable liquids by GC-MS is subject to limitations, including the following:

12.1.1 Co-eluting compounds can result in no identification or misidentification. However, with appropriate GC-MS method development (see Section 8) and the use of closely eluting compounds in the test mixture (see Section 6.4) the possibility of co-eluting compounds in ignitable liquids analysis can be minimized. Refer to Test Method E(INTRP) for interpretation of data suspected to contain co-eluting compounds arising from substrate interferences.

12.1.2 Compounds outside of the approximate volatility and polarity range for which a specific GC-MS method was developed and optimized might not be observed in the data, or might not give rise to data that are suitable for comparisons. However, the use of one or more GC-MS methods developed and optimized to cover the light through heavy range components of ignitable liquids (see Section 8) can be used to minimize this limitation for the types of compounds that are significant in the interpretation and classification of ignitable liquids using Test Method E(INTRP).

12.1.3 Limited availability of ignitable liquid reference materials or limited searchable databases can hinder the ability to perform meaningful comparisons. However, the

maintenance of appropriate reference ignitable liquids (see Section 6.5) and mass spectral libraries (see Section 5.4) can minimize this limitation.

13. Keywords

13.1 forensic science, fire debris; gas chromatography; ignitable liquid; mass spectrometry

ANNEX

(Mandatory Information)

A1. CARRYOVER STUDIES

A1.1. Carryover studies are required when the GC-MS method does not specify the utilization of analysis of blanks between samples.

A1.2. Carryover studies are performed as part of the GC-MS method validation or verification.

A1.3. Carryover studies are designed to evaluate the potential for carryover when using a specific GC-MS method over the range of samples anticipated to be encountered during casework.

A1.4. Minimum sample analysis requirements for a carryover study:

A1.4.1 Analyze solvent blanks between samples throughout the entire GC-MS method validation or verification process.

A1.4.2 Analyze a variety of samples consisting of reference ignitable liquids and matrix materials, the types and approximate concentrations of which are reflective of those that are reasonably anticipated to be found in casework scenarios. Include vapors, liquids, and extracted samples as applicable for the specific GC-MS method to be utilized.

A1.4.2.1 If the GC-MS method will be utilized for casework samples prepared using solvent extraction as in Practice E1386, include extracts of samples consisting of burned matrix materials, vegetable oils, and lubricating oils.

A1.4.2.2 Matrix components routinely encountered as potential interferents (e.g. styrene) can be analyzed as single compound samples using a reference material that is fit for purpose for qualitative work, in accordance with Practice E3255.

A1.4.3 Analyze highly concentrated samples of reference ignitable liquids or single compounds across the entire volatility range and composition of interest applicable to the GC-MS method utilized. These samples are deliberately designed to exceed the reasonable concentrations found in typical casework scenarios, and they are specifically intended to assess the conditions under which carryover will occur.

A1.5. Evaluate the total ion chromatogram (TIC) and extracted ion profiles (EIPs) of all solvent blanks for the presence of carryover in the form of peaks which have a signal-to-noise ratio greater than approximately three times the average local baseline, and that are also observed as a component of the previous sample.

NOTE: Address peaks not attributable to carryover (e.g. peaks arising from suspected contamination or siloxane peaks arising from septum bleed) separately within the GC-MS method validation or verification.

A1.6. Use the information from the evaluation of solvent blanks in Section A1.5 to assess the risk of carryover during the analysis of routine casework scenarios.

A1.7 Based on the assessment in Section A1.6, implement guidelines for the evaluation of all casework data for the presence of carryover, as well as mitigation procedures in the event that carryover is observed in a sample.

A1.7.1 When data from a sample does not meet the requirements listed in Sections 8.1.2 through 8.1.4, reanalyze the sample in accordance with Section 10.5, as well as the subsequent sample(s) in order to mitigate the potential risk of carryover.

A1.8. Maintain all carryover study data and associated documentation as part of the GC-MS method validation or verification, in accordance with Practice E3255 and WK72631.

APPENDIX 1

(Nonmandatory Information)

X1. EXAMPLES OF GC-MS PARAMETERS

X1.1 Examples of GC-MS methods suitable for the analysis of suspected ignitable liquids are provided in the table below.

X1.1.1 Method 1 is suitable for the general screening of ignitable liquids, including light oxygenates. This method can be useful for achieving relatively rapid throughput and narrow peaks due to its temperature and pressure programs.

X1.1.2 Method 2 is suitable for the general screening of ignitable liquids, but does not scan for light oxygenates due to the higher start temperature and a delay in initiating the detector.

X1.1.3 Method 3 is suitable for the general screening of ignitable liquids, including light oxygenates. This method uses a longer column which can enhance resolution; however, peak shapes are wider and total run time is increased.

X1.1.4 Method 4 is suitable for targeted screening only for light oxygenated compounds.

X1.2 Verify instrumental methods prior to use, as these parameters are only intended to serve as a starting point for method development.

	Method 1	Method 2	Method 3	Method 4
Column				
Type	HP-1MS	HP-5MS	RX-1MS	HP-1MS
Length	30 m	30 m	60 m	30 m
Inner Diameter	250 µm	250 µm	250 µm	250 µm
Film Thickness	0.25 µm	0.25 µm	0.25 µm	0.25 µm
Injector				
Inlet Temperature	290 C	250 C	250 C	290 C
Injection Volume	2 µL	1 µL	1 µL	Manual
Split Ratio	20:1	50:1	5:1	20:1
GC				
Mobile Phase	Helium	Helium	Helium	Helium
Flow Rate	1.8 mL/min then 20 mL/min per min to 1.2 mL/min for 9.97 min, then 20 mL/min per min to 1.8 mL/min for 0 min.	1.25 mL/min	1 mL/min hold for 5 min, then 0.4 mL/min per min to 1.6 mL/min	0.6 mL/min then 20 mL/min per min to 1.2 mL/min for 9.97 min, then 20 mL/min per min to 1.8 mL/min for 0 min.
Temperature Program	40 C hold for 1.5 min; 20 C/min to 140 C, hold 0 min; 30C/min to 300 C, hold 5.17 min	50 C hold for 3.0min; 10 C/min to 280 C, hold for 4 min	40 C hold for 0 min; 6 C/min to 80 C, hold 0 min; 15 C/min to 250 C, hold for 0 min	35 C hold 4 min; 10 C/min to 100 C, hold for 0 min
MS				
Source Temp	230 C	230 C	230 C	230 C
Interface Temp	300 C	280 C	280 C	290 C
Mass Scan Range	14-200 m/z for 2 min; 14-400 m/z for 12 min; 14-600 m/z to end of run	33-400 m/z	10-74 m/z for 5.05 min; 29-450 m/z to end of run	10-150 m/z
Mass Threshold	150	150	100	50
A/D Samples	4	4	4	8
Total Run Time	17.003 min	30 min	26 min	10.5 min

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