

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

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

Prepared by
Ignitable Liquids, Explosives & Gunshot Residue Subcommittee
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1 **Standard Practice for**
2 **Gas Chromatography Electron Ionization Mass Spectrometry Analysis of**
3 **Ignitable Liquids**
4

5 **1. Scope**

6 1.1 This practice covers the instrumental analysis of ignitable liquids, as well as extracts
7 from fire debris samples, by gas chromatography-electron ionization mass spectrometry (GC-
8 MS).

9 1.2 This practice describes performance criteria for use during initial GC-MS method
10 development and optimization, data evaluation and acceptance criteria, quality assurance and
11 quality control considerations, and limitations.

12 1.3 *This standard is intended for use by competent forensic science practitioners with the*
13 *requisite formal education, discipline-specific training (see Practice E2917), and demonstrated*
14 *proficiency to perform forensic casework.*

15 1.4 *The values stated in SI units are to be regarded as the standard. No other units of*
16 *measurement are included in this standard.*

17 1.5 *This standard does not purport to address all of the possible safety concerns, if any,*
18 *associated with its use. It is the responsibility of the user of this standard to establish*
19 *appropriate safety, health, and environmental practices and determine the applicability of*
20 *regulatory requirements prior to use.*

21
22 **2. Referenced Documents**

23 2.1 *ASTM Standards*¹:

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

26 **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.

- 27 **E1412** Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by
28 Passive Headspace Concentration with Activated Charcoal
- 29 **E1413** Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by
30 Dynamic Headspace Concentration onto an Adsorbent Tube
- 31 **E(CLASS)** Classification for Ignitable Liquids Encountered in Forensic Fire Debris Analysis
- 32 **E(INTRP)** Test Method for Interpretation of Gas Chromatography-Electron Ionization Mass
33 Spectrometry Data for the Identification of Ignitable Liquid Classes in Forensic Fire
34 Debris Analysis
- 35 **E1732** Terminology Relating to Forensic Science
- 36 **E2154** Practice for Separation and Concentration of Ignitable Liquid Residues from Fire
37 Debris Samples by Passive Headspace Concentration with Solid Phase Microextraction
38 (SPME)
- 39 **E2451** Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from
40 Fire Debris Samples
- 41 **WK72631 (E2549 Revision)** Practice for Validation and Verification of Analytical Methods
42 for Forensic Science Service Providers Performing Forensic Chemistry Analysis
- 43 **E2881** Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire
44 Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry
- 45 **E2917** Practice for Forensic Science Practitioner Training, Continuing Education, and
46 Professional Development Programs
- 47 **E3189** Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by
48 Static Headspace Concentration onto an Adsorbent Tube
- 49 **E3197** Terminology Relating to Examination of Fire Debris
- 50 **E3245** Guide for Systematic Approach to the Extraction, Analysis, and Classification of
51 Ignitable Liquids and Ignitable Liquid Residues in Fire Debris Samples
- 52 **E3255** Practice for Quality Assurance of Forensic Science Service Providers Performing
53 Forensic Chemical Analysis

54

55 **3. Terminology**

56 3.1 *Definitions:*

57 3.1.1 For definitions of terms that can assist in interpreting this Practice, refer to
58 Terminology **E1732**, Terminology **E3197**, and Classification **E(CLASS)**.

59 3.2 *Definitions of Terms Specific to This Standard:*

60 3.2.1 **analytical batch**, *n*—a set of samples that are analyzed together on the same
61 instrument; also known as a sample sequence

62 3.2.2 **carryover**, *n*—material left over from a previous sample that results in signal that is
63 observed in the data of a subsequent sample

64 3.2.3 **GC-MS method**, *n*—the complete set of instrument parameters utilized in the
65 analysis of a test sample.

66 3.3 *Abbreviations*:

67 3.3.1 **C#** – total number of carbon atoms associated with the substitutions on a base
68 molecule; for example, C3 alkyl benzenes include n-propyl benzene, 3-ethyl toluene, 1,2,4-
69 trimethyl benzene, etc.

70

71 4. Significance and Use

72 4.1 This Practice is useful for generating GC-MS data from ignitable liquids and extracts
73 from samples suspected to contain ignitable liquid residues. Refer to Guide [E3245](#) for
74 perspective on the use of GC-MS within the framework of ignitable liquids analysis. Refer to
75 Practices [E1386](#), [E1388](#), [E1412](#), [E1413](#), [E2154](#), and [E3189](#) for sample preparation procedures.
76 Refer to Test Method [E\(INTRP\)](#) for interpretation of data generated using this Practice.

77 4.2 This Practice is intended to be used in conjunction with quality assurance procedures
78 covered in Practice [E3255](#).

79

80 5. Apparatus

81 5.1 Gas Chromatograph (GC)—A chromatograph capable of using capillary columns and
82 being interfaced to a mass spectrometer.

83 5.1.1 Sample Inlet System—A sample inlet system that can be operated in either split or
84 splitless mode with capillary columns.

85 5.1.2 GC Oven—A column oven capable of reproducible temperature program operation
86 in the range from at least 35 to 300 °C.

87 5.1.3 Column—A capillary, bonded phase, methylsiloxane or phenylmethylsiloxane
88 column or equivalent.

89 5.1.3.1 Alternate column stationary phases can be used for targeted analyses. As an
90 example, screening for oxygenated compounds could be improved by use of a more polar
91 column than those listed above.

92 5.2 Mass Spectrometer (MS)—A mass selective detector capable of acquiring mass spectra
93 from m/z 10 to m/z 400 with unit resolution or better.

94 5.3 Data Station—A computerized data station capable of performing the following functions
95 in conjunction with the GC-MS, either through its operating system or by user programming:

96 5.3.1 Recording instrument parameters used during sample runs.

97 5.3.2 Storing time sequenced mass spectral data from sample runs.

98 5.3.3 Retrieving and displaying sample data files.

99 5.3.4 Generating extracted ion profiles.

100 5.3.5 Retrieving a specified mass spectrum from a data file and comparing it against one
101 or more mass spectral libraries.

102 5.3.6 Preparing data presentations in electronic or hard copy format.

103 5.4 Mass Spectral Library—One or more searchable databases containing mass spectra of
104 individual chemical compounds.

105 5.5 Sample introduction devices.

106 5.5.1 For liquid samples, a syringe capable of introducing a sample size appropriate for
107 the volume of the inlet upon expansion of the injected solvent. Typical liquid sample sizes
108 are in the range from 0.1 to 10 microliters.

109 5.5.2 For vapor samples, a vapor-tight syringe capable of introducing a sample size
110 appropriate for the volume of the inlet. Typical vapor sample sizes are in the range from 0.5
111 to 5 milliliters.

112 5.5.3 For some adsorbent tubes (see Practices [E1413](#) and [E3189](#)) and for SPME fibers
113 (see Practice [E2154](#)), a system capable of desorbing the trapped volatile compounds and
114 introducing them into the GC-MS.

115

116 **6. Chemicals, Reagents, and Reference Materials**

117 6.1 *Purity of Reagents*—Use reagent grade chemicals or better. Unless otherwise indicated,
118 it is intended that all reagents conform to the specifications of the Committee on Analytical
119 Reagents of the American Chemical Society where such specifications are available.² Other
120 grades can be used, provided it is first ascertained that the reagent is of sufficiently high purity to
121 permit its use without lessening the accuracy of the determination.

122 6.2 *Solvent/Diluent*—Use carbon disulfide, diethyl ether, pentane, or other solvent that does
123 not interfere with the analysis. It is generally desirable to use a solvent whose volatility greatly
124 exceeds that of most expected solutes to facilitate sample concentration by evaporation, if
125 necessary. The polarity of the solvent should also be considered.

126 6.3 *Carrier Gas*—Use helium or hydrogen of purity 99.995% or higher.

127 6.4 *Test Mixture*—Use a test mixture that is suited to the GC-MS method being utilized.
128 More than one test mixture can be used.

129 6.4.1 The composition of a general-purpose ignitable liquids test mixture at minimum
130 consists of the following components diluted in solvent:

131 6.4.1.1 A mixture of alkanes (at least five normal alkanes ranging from n-hexane to n-
132 eicosane) and aromatic compounds that cover the approximate volatility and polarity
133 range of the ignitable liquid classes for which the GC-MS method was developed, and

134 6.4.1.2 At least one pair of closely-eluting compounds, such as isomers or closely-
135 eluting oxygenates, to facilitate the evaluation of the adequacy of the resolution of the
136 GC-MS method.

137 6.4.2 The composition of a targeted test mixture consists of compounds that cover the
138 volatility range and composition of interest.

139 6.4.3 Oxygenated compounds can be added to the general-purpose ignitable liquids test
140 mixture or prepared as a separate targeted test mixture.

141 6.4.4 Examples of suitable test mixtures are listed in Table 1. Refer to Test Method
142 E2881 for information about test mixtures for use with vegetable-oil based (VOB) products.

143

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.

144 **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 Acetone 2-Propanol (Isopropanol) 2-Butanone Even-numbered n-alkanes (C ₆ -C ₂₀) Toluene p-xylene 1-methyl-2-ethylbenzene 1-methyl-3-ethylbenzene 1,2,4-trimethylbenzene	N-alkanes (C ₈ -C ₂₀) Methylcyclohexane 1-methyl-2-ethylbenzene 1-methyl-3-ethylbenzene 1,3,5-trimethylbenzene 1-methyl-4-ethylbenzene	Ethanol Acetone 2-Propanol (Isopropanol) 2-Butanone (Methyl ethyl ketone)

145

146 6.5 Reference Ignitable Liquids—Maintain reference ignitable liquids for the various
 147 ignitable liquid classes defined in Table 1 of Practice E(CLASS).

148 6.5.1 Reference ignitable liquids can be obtained from commercial or retail sources.
 149 Certified ignitable liquid standards are not required.

150 6.5.2 Assess reference ignitable liquids in accordance with Appendix 2 of Practice E3255
 151 prior to use.

152 **7. Instrument Maintenance**

153 7.1 Establish and follow an instrument maintenance routine that takes into consideration the
 154 recommendations of the instrument manufacturer and the overall use of the equipment.

155 7.1.1 Continually evaluate data features to determine if maintenance is necessary to
 156 optimize performance. Data features that can indicate a need for maintenance include
 157 changes in retention time, peak abundance, peak shape, or the presence of unexplainable or
 158 extraneous peaks.

159 7.2 Clean syringes thoroughly with a suitable solvent (see Section 6.2) between injections to
 160 minimize the potential for carryover.

161 7.2.1 Analyzing a solvent blank between samples is recommended but is not required if
 162 studies performed in accordance with Annex A1 demonstrate that the cleaning procedure
 163 prevents carryover.(1)³

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

164

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

166 8.1 Any GC-MS method can be utilized for ignitable liquids analysis, provided it meets the
167 following performance criteria:

168 8.1.1 *All components of interest are eluted.*

169 8.1.1.1 For a general-purpose ignitable liquids GC-MS method, demonstrate that, at a
170 minimum, light, medium, and heavy range components are present in the total ion
171 chromatogram (TIC).

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

175 8.1.1.2 For a targeted GC-MS method, demonstrate that the compounds of interest are
176 present in the TIC.

177 8.1.2 *Peak shapes are approximately Gaussian with narrow widths.*

178 8.1.2.1 Gaussian (i.e. symmetrical “bell-shaped”) peaks are ideal; however, some
179 asymmetry and tailing is normal with temperature gradient programs.(2, 3)

180 8.1.2.2 Some factors that can affect peak width are column specifications, column
181 efficiency, temperature program(s), GC flow rate, and sample concentration.(4, 5)

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

186 8.1.3 *Peak resolution is sufficient for the recognition of patterns associated with the*
187 *components of interest, or identification of targeted components.* Due to the number of
188 similar compounds present in ignitable liquids, baseline resolution of all compounds is not
189 necessary.

190 8.1.3.1 For a general-purpose ignitable liquids GC-MS method, at a minimum,
191 demonstrate that peak resolution in the C3 and C4 alkyl benzene groups in gasoline, and

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

194 8.1.3.2 Adequate peak resolution can usually be achieved through the selection of
195 appropriate column specifications and gas chromatographic method parameters.

196 8.1.4 *Retention times of components of interest are sufficiently reproducible.*

197 8.1.4.1 For a general-purpose ignitable liquids GC-MS method, at a minimum,
198 demonstrate that peaks in the light range (e.g. toluene), medium range (e.g. 1,2,4-
199 trimethylbenzene), and heavy range (e.g. n-hexadecane) have sufficiently reproducible
200 retention times (e.g. no more than the greater value of either $\pm 1\%$ or ± 0.1 minute (6)
201 deviation from the mean value from 10 repetitions performed during the method
202 development process).

203 8.1.4.2 For a targeted GC-MS method, at a minimum, demonstrate that the peak for
204 one compound of interest has sufficiently reproducible retention times (e.g. no more than
205 the greater value of either $\pm 1\%$ or ± 0.1 minute (6) deviation from the mean value from 10
206 repetitions performed during the method development process).

207 8.1.5 *Mass spectra are collected over one or more mass-to-charge (m/z) ranges which*
208 *encompass the relevant ions used for identification of the components of interest.*

209 8.1.5.1 For a general-purpose ignitable liquids GC-MS method, start scans at a m/z
210 value no greater than 33 for the lower limit, and scan to an upper limit at or greater than a
211 m/z value of 400.

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

215 8.1.6 *Relevant ions in the mass spectra of the components of interest are of sufficient*
216 *intensity for identification of the components, either by computer library search or by*
217 *comparison with reference mass spectra, in accordance with Test Method **E(INTRP)**.*

218 8.1.6.1 Contributions from extraneous ions are minimized in the mass spectra. Some
219 sources of extraneous ions are column bleed, carrier gas impurities, detector noise,
220 cleanliness of the mass selective detector, and poor vacuum performance.

221 8.1.6.2 Parameters that can affect the intensity of ions in the mass spectra include the
222 mass threshold, analog-to-digital (A/D) samples, tune method (e.g. auto tune, standard
223 spectra tune, etc.), and tune parameters.

224 8.2 Optimize GC-MS methods for sensitivity, to reduce the potential for carryover, and for
225 run time efficiency while also meeting the performance criteria in 8.1.

226 8.2.1 Sensitivity can be optimized by adjusting injection, inlet, and detector parameters.

227 8.2.2 Reducing the potential for carryover can be optimized by adjusting injector
228 parameters or syringe wash parameters, or both, and by running blanks.

229 8.2.3 Run time efficiency (i.e. minimization of total run time that satisfies performance
230 criteria) can be optimized by adjusting temperature, flow, and column parameters.

231 8.3 Examples of GC-MS method parameters for the analysis of samples suspected to contain
232 ignitable liquids are provided in Appendix 1.

233

234 **9. Sample Preparation, Analysis, and Preservation**

235 9.1 Prepare samples in accordance with Section 9 of Guide [E3245](#) in conjunction with one or
236 more of Practices [E1386](#), [E1388](#), [E1412](#), [E1413](#), [E2154](#), or [E3189](#), as appropriate.

237 9.2 Analyze the sample by GC-MS.

238 9.2.1 Use an appropriate GC-MS method that has been developed and optimized for use
239 in ignitable liquids analysis (see Section 8) and validated in accordance with Practice [E2549](#).

240 9.2.2 Use an appropriate sample introduction device (see Section 5.5) to deliver the
241 sample to the GC-MS.

242 9.3 When analysis is complete, preserve and store samples according to Practice [E2451](#).

243

244 **10. Data Evaluation and Acceptance Criteria**

245 10.1 For all sample types, verify that the sample was properly introduced to the GC-MS, that
246 the instrument performed correctly, and that data were recorded.

247 10.1.1 Confirm that a TIC and mass spectra were recorded.

248 10.1.2 Evaluate chromatographic features of the TIC.

249 10.1.2.1 Confirm that peaks are present in the TIC as appropriate for the sample and
250 GC-MS method utilized.

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

253 10.1.2.2 Confirm that most peak shapes are approximately Gaussian with narrow
254 widths (see also Section 8.1.2).

255 10.1.2.3 Confirm that the baseline is stable (e.g. with respect to drift, noise, or
256 spikes).

257 10.2 For blanks, also confirm that there are no peaks present in the TIC that correspond to
258 compounds of interest that would interfere with the ability to interpret the data for the presence
259 of ignitable liquids, in accordance with Test Method E(INTRP).

260 10.3 For test mixtures, also confirm that retention times for selected peaks are within the
261 greater value of either $\pm 1\%$ or ± 0.1 minute (6) of the retention times established during
262 development and optimization from previously established values for the GC-MS method used.

263 10.3.1 A sudden shift of retention times or decrease in abundances could indicate that
264 instrument maintenance is necessary.

265 10.3.2 If maintenance is conducted that would reasonably be expected to change
266 retention times (e.g. column trimming or column change) or abundances (e.g. ion source
267 cleaning), run the test mixture a minimum of three times to determine the new retention times
268 or abundances to be used for comparison.

269 10.4 Reject data from samples that do not satisfy the evaluation criteria as follows:

270 10.4.1 For questioned samples, use the criteria in Section 10.1.

271 10.4.2 For blanks, use the criteria in Sections 10.1 and 10.2.

272 10.4.3 For test mixtures, use the criteria in Sections 10.1 and 10.3.

273 10.5 Samples that produce rejected data can be reanalyzed. Additional sample treatment,
274 such as dilution of overconcentrated samples, can be utilized as needed prior to reanalysis.

275 10.6 Maintain records of rejected data, any additional sample treatment utilized, and any
276 reanalysis performed as a portion of the casework documentation.

277

278 **11. Quality Assurance and Quality Control**

279 11.1 Validate or verify GC-MS instruments and GC-MS methods according to Practice
280 **WK72631** and Practice **E3255** in order to demonstrate their suitability for use in ignitable liquids
281 analysis prior to use on unknown samples.

282 11.1.1 Maintain records of all validations and verifications in accordance with Practice
283 **WK72631** and Practice **E3255**.

284 11.2 Tune the mass spectrometer in accordance with manufacturer's recommendations in
285 order to ensure correct calibration of mass to charge ratios and to optimize mass spectrometer
286 system performance.

287 11.2.1 Tune using perfluorotributylamine (PFTBA), or another generally accepted tuning
288 compound in accordance with manufacturer's recommendations.

289 11.2.2 Evaluate tune results against acceptance criteria established in accordance with
290 manufacturer's recommendations.

291 11.2.3 Maintain tuning records as a portion of either the quality control documentation or
292 the casework documentation.

293 11.3 Analyze at least one process blank per analytical batch in order to demonstrate that
294 materials such as solvents, adsorption media, glassware, and other sample processing hardware
295 are free from contaminants that can cause interferences.

296 11.3.1 Use the type of blank appropriate for the phase (i.e. vapor or liquid) of the
297 questioned sample to be analyzed and, if applicable, to the method used to isolate the
298 questioned sample from the original fire debris material (see Practices **E1388**, **E1412**, **E3189**,
299 **E1413**, **E2154**, and **E1386**).

300 11.3.2 Evaluate the data from blanks according to Sections 10.1 and 10.2.

301 11.3.3 Maintain data from blanks as a portion of the casework documentation.

302 11.4 Analyze at least one test mixture at the beginning of each analytical batch in order to
303 demonstrate proper instrument performance with respect to the components of interest.

304 11.4.1 Use a test mixture that is appropriate for the GC-MS method being performed (see
305 Section 6.4).

306 11.4.2 Evaluate the data from test mixtures to confirm proper instrument performance
307 according to Sections 10.1 and 10.3.

308 11.4.3 Maintain data from test mixtures as a portion of the casework documentation or
309 the quality control documentation, or both.

310 11.5 Maintain data from reference ignitable liquids that are to be used for the interpretation
311 of questioned samples and that have been analyzed using the same GC-MS methods as the
312 questioned samples.

313

314 **12. Limitations**

315 12.1 In general, analysis of ignitable liquids by GC-MS is subject to limitations, including
316 the following:

317 12.1.1 Co-eluting compounds can result in no identification or misidentification.
318 However, with appropriate GC-MS method development (see Section 8) and the use of
319 closely eluting compounds in the test mixture (see Section 6.4) the possibility of co-eluting
320 compounds in ignitable liquids analysis can be minimized. Refer to Test Method **E(INTRP)**
321 for interpretation of data suspected to contain co-eluting compounds arising from substrate
322 interferences.

323 12.1.2 Compounds outside of the approximate volatility and polarity range for which a
324 specific GC-MS method was developed and optimized might not be observed in the data, or
325 might not give rise to data that are suitable for comparisons. However, the use of one or
326 more GC-MS methods developed and optimized to cover the light through heavy range
327 components of ignitable liquids (see Section 8) can be used to minimize this limitation for
328 the types of compounds that are significant in the interpretation and classification of ignitable
329 liquids using Test Method **E(INTRP)**.

330 12.1.3 Limited availability of ignitable liquid reference materials or limited searchable
331 databases can hinder the ability to perform meaningful comparisons. However, the

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

334 **13. Keywords**

335 13.1 forensic science, fire debris; gas chromatography; ignitable liquid; mass spectrometry

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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.

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

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

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

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385 **APPENDIX 1**

386 **(Nonmandatory Information)**

387 **X1. EXAMPLES OF GC-MS PARAMETERS**

388

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

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

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

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

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

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

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	Method 1	Method 2	Method 3	Method 4
<u>Column</u>				
Type	HP-1MS	HP-5MS	RX-1MS	HP-1MS
Length	30 m	30 m	60 m	30 m
Inner Diameter	250 μ m	250 μ m	250 μ m	250 μ m
Film Thickness	0.25 μ m	0.25 μ m	0.25 μ m	0.25 μ m
<u>Injector</u>				
Inlet Temperature	290 C	250 C	250 C	290 C
Injection Volume	2 μ L	1 μ L	1 μ L	Manual
Split Ratio	20:1	50:1	5:1	20:1
<u>GC</u>				
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
<u>MS</u>				
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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