



Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography¹

This standard is issued under the fixed designation E 1387; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the identification of residues of ignitable liquids in extracts from fire debris samples. Extraction procedures are described in the referenced documents.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 6.3.

2. Referenced Documents

2.1 ASTM Standards:

- E 260 Practice for Packed Column Gas Chromatography²
- E 752 Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide³
- E 1385 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation²
- E 1386 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction²
- E 1389 Practice for Cleanup of Fire Debris Sample Extracts by Acid Stripping²
- E 1412 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration²
- E 1413 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration²
- E 1618 Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry²

¹ This test method is under the jurisdiction of ASTM Committee E-30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

Current edition approved Oct. 10, 1995. Published December 1995. Originally published as E 1387 – 90. Last previous edition E 1387 – 90.

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 11.03.

3. Summary of Test Method

3.1 The sample extract or preparation is introduced into the gas chromatographic column containing a liquid phase suitable for the separation of common ignitable liquid components. The resulting chromatogram is interpreted by techniques of *pattern recognition* and *pattern comparison* described in this test method. Ignitable liquids may fall into one of five major classifications, or into a “miscellaneous” category described herein.

4. Significance and Use

4.1 The identification of an ignitable liquid residue in a sample of fire debris can support a field investigator’s opinion regarding the incendiary nature of a fire.

4.1.1 The identification of an ignitable liquid residue in a fire scene does not necessarily lead to the conclusion that a fire was incendiary in nature. Further investigation may reveal a legitimate reason for the presence of ignitable liquids.

4.2 Due to the volatility of ignitable liquids and to variations in sampling technique, the absence of detectable quantities of ignitable liquid residues does not necessarily lead to the conclusion that ignitable liquids were not present at the fire scene.

4.3 When the gas chromatographic pattern is not sufficiently complex, as described in 10.3, additional analytical techniques are required.

5. Apparatus

5.1 *Gas Chromatograph*—A chromatograph equipped with a flame ionization or mass spectral detector may be used. Other detectors may be used if it can be shown that they have sensitivity and selectivity equal to the above detectors.

5.1.1 *Sensitivity*—The system shall be capable of detecting 0.05 volume % in carbon disulfide (or any appropriate solvent) of any common ignitable mixtures.

5.1.2 *Sample Inlet System*—A sample inlet system that allows a reproducible volume of liquid to be injected.

5.1.3 *Column*—A non-polar capillary column is recommended, but any column may be used provided that, under the conditions of use, the test mixture can be resolved into its component peaks.

5.1.3.1 The test mixture shall consist of equal parts by weight of the even-numbered normal alkanes ranging from

n-hexane through n-eicosane, plus the following aromatic components: toluene, *p*-xylene, *o*-ethyltoluene, *m*-ethyltoluene, and 1,2,4-trimethylbenzene.

5.1.3.2 If total resolution of the test mixture cannot be obtained on a single column or program, changing columns or programs is permitted as long as using both columns or programs results in the complete resolution of the test mixture.

5.2 *Column Oven*—A column oven capable of reproducible temperature and temperature program settings in the range of 50 to 300°C should be used.

5.3 *Strip Chart Recorder*—A recording potentiometer with a full scale deflection of 10 mV or less should be used. The full scale response of the recorder should not exceed 1 s. An integrator, or computerized data station and printer which meets or exceeds these requirements, is acceptable.

5.4 *Syringes*:

5.4.1 *For liquid samples*—a microsyringe, capable of reproducibly introducing sample sizes in the range of 0.1 to 10.0 mL.

5.4.2 *For gas samples*—a gas-tight syringe capable of reproducibly introducing sample sizes in the range of 0.5 to 5 mL.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.⁴ Other grades may 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 *Solvents*—The solvent in which the extract is dissolved will depend upon the extraction technique employed. Frequently check solvents for purity by running appropriate blanks, both neat and evaporated, to at least twice the extent used in the analysis.

6.2.1 If Carbon disulfide is used, read and follow the safety precautions described in Practice E 752.

6.3 *Carrier Gas*—**Caution:** hydrogen, helium, and nitrogen are compressed under high pressure, and hydrogen is an extremely flammable gas.

6.4 *Combustion Gases*—Air and hydrogen (if a flame ionization detector is used).

6.5 Activated Charcoal.

7. Sample Handling

7.1 Methods of obtaining the extracts or preparations for analysis are described in Practices E 1385, E 1386, E 1389, E 1412, and E 1413.

7.2 Due to the volatility of the solvents and the analytes, take care to ensure that samples do not evaporate or otherwise

change composition. Extracts in carbon disulfide may be covered with water prior to removing the extracts from the sample preparation hood. Alternatively, septum seal vials may be used for storing any solvents or extracts.

7.2.1 If water is used as a sealant, exercise care to avoid the introduction of water onto DMCS treated columns.

7.2.2 Avoid the use of water as a sealant if the presence of water soluble compounds is suspected.

8. Calibration

8.1 Calibrate the chromatographic instrument frequently using standards of known concentrations of known ignitable liquids as well as blanks. Optimize gas flows periodically. Refer to Practice E 260 for detailed instructions on operation of the gas chromatograph.

8.1.1 Run appropriate blanks and controls periodically.

8.1.1.1 Clean syringes thoroughly between injections.

8.1.2 Run known standards as necessary.

8.1.2.1 Standard chromatograms must be run under the same chromatographic conditions as those used to produce the sample chromatogram.

8.1.2.2 Every case file that includes a positive identification of an ignitable liquid or residue must include the standard chromatogram used to confirm the identification.

8.2 *Chromatogram Evaluation*—A good chromatogram for comparison work is one in which the peaks of interest are 50 to 100 % of full scale. Rerun samples, or replot chromatogram, using different parameters (attenuation or sample size) to achieve a good chromatogram.

8.2.1 In addition to the chromatogram described above, it is sometimes necessary to produce other, off-scale plots, in order to bring some features into view for comparison. Such off-scale plots may be required when there are one or more components present at a significantly higher concentration than the other components in the residue.

9. Petroleum Distillate Classification System

9.1 Six major classes of complex liquid products are recognized as usually identifiable by GC patterning alone when recovered from fire debris.

9.1.1 This test method is only intended to allow isolated residues to be characterized as to one of the types of products listed in Table 1. Other characterizations of samples may be possible, but are not within the scope of this test method.

10. Procedure

10.1 Obtain a chromatogram of the fire debris sample extract.

10.2 Obtain a chromatogram of a matching, or nearly matching, standard, and compare the pattern of peaks visually.

10.2.1 The essential requirement for making a classification using this procedure is the matching of the sample chromatogram with a known standard chromatogram obtained under similar conditions, noting sufficient significant points of correlation or similarities. Make all comparisons using only good chromatograms, as described in 8.2.

10.2.2 Pattern matching requires that the entire pattern used for comparison be displayed at the same sensitivity.

10.2.2.1 To provide sufficient detail for some comparisons,

⁴ *Reagent Chemicals, American Chemical Society Specifications*, 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Ignitable Liquid Classification System

Class Number (Class Name)	"Peak Spread" Based on N-Alkane Carbon Numbers (Un evaporated Liquid)	Examples
1 Light Petroleum Distillates (LPD)	C ₄ -C ₁₁	Petroleum ethers, Pocket lighter fuels, Some rubber cement solvents, Skelly solvents, V M & P Naphtha, Some camping fuels
2 Gasoline	C ₄ -C ₁₂	All brands and grades of automotive gasoline, including gasohol.
3 Medium Petroleum Distillates (MPD)	C ₈ -C ₁₂	Mineral spirits, Some paint thinners, Some charcoal starters, "Dry-cleaning" solvents, Some torch fuels, Some solvents for insecticides and polishes, some lamp oils
4 Kerosene	C ₉ -C ₁₇	Number 1 Fuel Oil, Jet-A (aviation) fuel, Insect sprays, Some charcoal starters, Some torch fuels, Some paint thinners, Some solvents for insecticides and polishes, some lamp oils
5 Heavy Petroleum Distillates (HPD)	C ₉ -C ₂₃	Number 2 fuel oil, Diesel fuel
0 Miscellaneous	Variable	Single compounds, Turpentines, Specialty mixtures that cannot be further classified into one of the categories below
0.1 Oxygenated solvents	Variable	Alcohols, Esters, Ketones
0.2 Isoparaffins	Variable	Isoparaffin products, Some charcoal starters, Some copier fluids, Some aviation gasolines, Some lamp oils, Some solvents for insecticides and polishes, Some camping fuels
0.3 Normal alkanes	Variable	Specialty products formulated from normal alkanes, Some lamp oils, Some solvents for insecticides and polishes
0.4 Aromatic solvents	Variable	Light, medium and heavy "aromatic naphtha" used as solvents for paints and plastics
0.5 Naphthenic/paraffinic solvents	Variable	Specialty solvent/fuel products made from Class 3 or Class 4 distillates-treated to remove normal alkanes and aromatics, with higher cycloalkane content than isoparaffin products

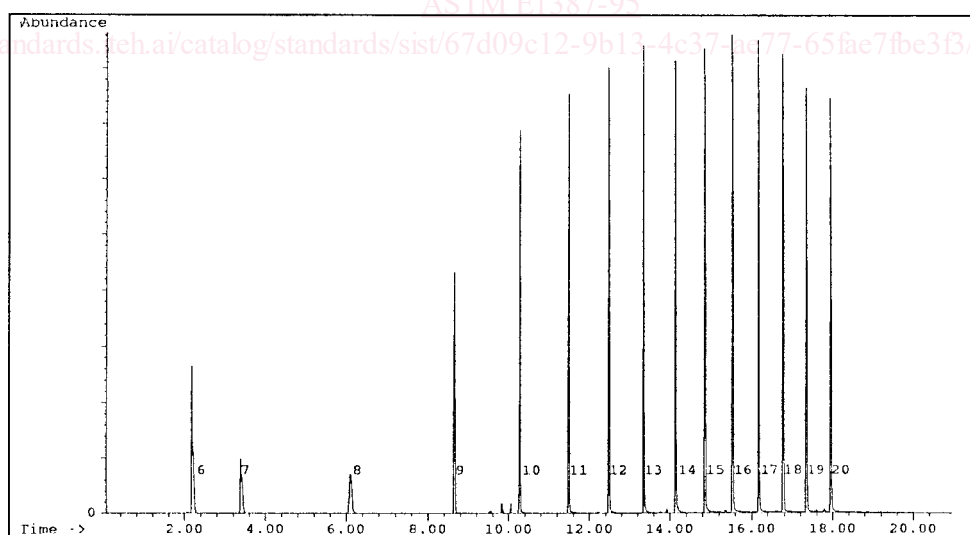
different amplitudes or presentations of the data may be necessary.

10.2.2.2 The carbon number range is determined by comparing the chromatogram to a standard containing known normal alkanes, as shown in Fig. 1.

10.2.3 Store the standard chromatogram(s) in the case file, along with the sample chromatogram(s).

10.3 Use the following criteria to determine whether sufficient similarities exist between the sample and the standard to classify the residue:

10.3.1 . *Class 1*—At least four major peaks matching a known Class 1 standard are in the C₄ to C₁₁ range. No major peak beyond C₁₁ is associated with ignitable liquids. Examples of Class 1 liquids are shown in Fig. 2 and Fig. 3.



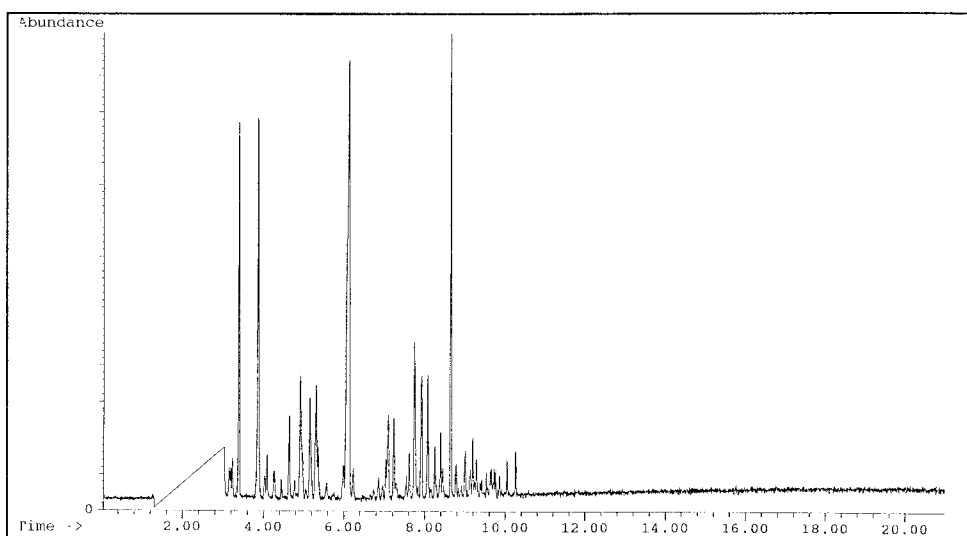
Chromatographic Conditions

Instrument: HP 5890 Gas Chromatograph
 Detector: HP 5971 Mass Selective Detector
 Column: 25 m, HP-1, 0.2 mm, ID, 0.5 µm film thickness
 All injections are 1 µL of a 1 % solution in diethyl ether.

Initial Temperature: 60°C, 6 min
 Ramp: 20°C/min
 Final Temperature: 280°C, 4 min

NOTE 1—These are typical chromatograms, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundances of individual components, depending on sample history, separation procedure, and chromatographic conditions.

FIG. 1 C₆-C₂₀ Normal Alkanes

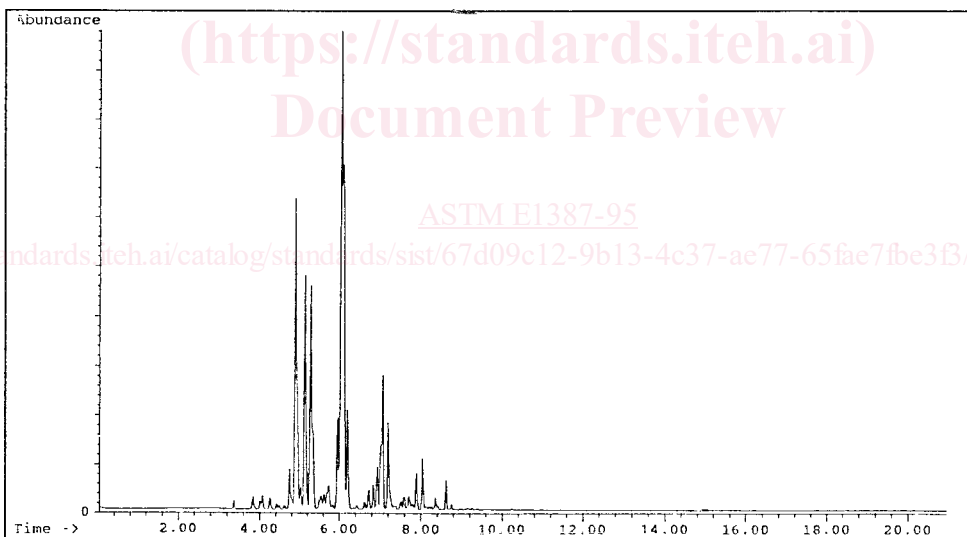


Chromatographic Conditions

Instrument: HP 5890 Gas Chromatograph	Initial Temperature: 60°C, 6 min
Detector: HP 5971 Mass Selective Detector	Ramp: 20°C/min
Column: 25 m, HP-1, 0.2 mm, ID, 0.5 µm film thickness	Final Temperature: 280°C, 4 min
All injections are 1 µL of a 1 % solution in diethyl ether.	

NOTE 1—These are typical chromatograms, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundances of individual components, depending on sample history, separation procedure, and chromatographic conditions.

FIG. 2 Example of a Class 1 Pattern; Brand A Cigarette Lighter Fluid



Chromatographic Conditions

Instrument: HP 5890 Gas Chromatograph	Initial Temperature: 60°C, 6 min
Detector: HP 5971 Mass Selective Detector	Ramp: 20°C/min
Column: 25 m, HP-1, 0.2 mm, ID, 0.5 µm film thickness	Final Temperature: 280°C, 4 min
All injections are 1 µL of a 1 % solution in diethyl ether.	

NOTE 1—These are typical chromatograms, provided for illustration only. Each laboratory is likely to obtain slightly different retention times and relative abundances of individual components, depending on sample history, separation procedure, and chromatographic conditions.

FIG. 3 Example of a Class 1 Pattern; Brand B Cigarette Lighter Fluid, Evaporated 90 %

10.3.2 Class 2—The C₃ alkylbenzene four-peak group, which represents *m*- and *p*-ethyltoluene, 1,3,5-trimethylbenzene, *o*-ethyltoluene, and 1,2,4-trimethylbenzene (pseudocumene), must be present; this group occupies the range between C₉ and C₁₀ and is still present in gasolines that have lost as much as 90 % of their initial (“fresh”) weight by

evaporation or combustion. Other peak groupings characteristic of gasoline must also be present. Examples of Class 2 liquids are shown in and Fig. 4. A time-expanded view of the C₃ alkylbenzene four-peak group is shown in Fig. 5.

10.3.2.1 The mere presence of these alkylbenzenes does not justify an identification of gasoline. These compounds must be