



# Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Steam Distillation<sup>1</sup>

This standard is issued under the fixed designation E 1385; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This practice covers the procedure for separating visible quantities of water insoluble hydrocarbons from samples of fire debris.

1.2 This practice is recommended only for samples which have a detectable odor of petroleum distillates when examined at room temperature.

1.3 This practice can yield useful extracts by the application of a solvent to the distillation trap in the event that only small quantities of hydrocarbons are obtained.

1.4 Alternate separation and concentration procedures are listed in the referenced documents.

1.5 *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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

E 752 Practice for Safety and Health Requirements Relating to Occupational Exposure to Carbon Disulfide<sup>3</sup>

E 1386 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction<sup>4</sup>

E 1387 Test Method for Ignitable Liquid Residues in Extracts from Samples of Fire Debris by Gas Chromatography<sup>4</sup>

E 1412 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration<sup>4</sup>

E 1413 Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Dynamic Headspace Concentration<sup>4</sup>

E 1459 Guide for Physical Evidence Labeling and Related Documentation<sup>4</sup>

E 1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory<sup>4</sup>

E 1618 Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry<sup>4</sup>

## 3. Summary of Practice

3.1 The sample of fire debris is introduced into a container of appropriate size, and an appropriate amount of water is added to the sample and brought to a boil. Vapors produced are condensed in a volatile oil distillation apparatus. Petroleum distillate residues float on top of a column of water and are collected as visible liquids.

## 4. Significance and Use

4.1 Steam distillation is a classical separation technique, useful for preparing extracts for analysis by gas chromatography GC/MS or GC/IR. Distillates are suitable for analysis according to Test Method E 1387 or Guide E 1618.

4.2 The visible oily liquid extract can be used as a courtroom exhibit, unlike extracts produced by other separation procedures which are solutions rather than a neat liquid.

4.3 This practice is not useful for the separation of water soluble ignitable liquids such as alcohols or ketones.

4.4 Alternate separation and concentration methods are suggested if the concentration of flammable or combustible liquid residues is not detectable by odor.

4.5 This is a destructive technique that should only be used when a representative portion of the sample can be reserved for reanalysis. Those portions of the sample subjected to this procedure may not be suitable for resampling. Consider using passive headspace concentration as described in Practice E 1412.

## 5. Apparatus

5.1 *Distillation Apparatus—A Flask*, or container of appropriate size, which allows the sample to be introduced into the flask through the mouth, and a volatile oil distillation trap fitted with a condensing column or a cold finger.

## 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

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of E30.01 on Criminalistics.

Current edition approved Dec. 10, 2000. Published March 2001. Originally published as E 1385 – 90. Last previous edition E 1385 – 95.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01

<sup>3</sup> **Discontinued.** See 1995 *Annual Book of ASTM Standards*, Vol 11.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

Analytical Reagents of the American Chemical Society where such specifications are available.<sup>5</sup> 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.

6.1.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 *Antifoam*—A silicone emulsion antifoaming agent<sup>6</sup> may be added as necessary to reduce foaming in the sample.

6.3 *Extracting Solvent*—Any high-purity solvent, such as carbon disulfide, chloroform, carbon tetrachloride, diethyl ether, or pentane.

## 7. Procedure

7.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E 1492.

7.1.1 Open and examine the fire debris sample in order to determine that it is consistent with its description.

7.1.1.1 Resolve any discrepancies between the submitting agent's description of the evidence and the analyst's observation with the submitting agent prior to the completion of the report.

7.2 Quickly check the sample for odor at room temperature. A sample which does not have a distinct ignitable liquid odor, or which has the odor of a water-soluble ignitable liquid, may be more suitably separated and concentrated by other procedures such as Practices E 1386, E 1412, and E 1413.

7.3 Place a representative portion of the sample in a distillation flask, or other appropriate container.

7.3.1 Record in the case notes a description of the portion of the sample extract.

7.4 Add the minimum amount of water necessary to prevent pyrolysis or combustion of the sample. Heat the flask on a hot

plate, or in the case of a large reaction kettle, in a heating mantle, and bring to a boil.

7.5 Adjust the temperature to achieve a condensation rate in the trap of approximately one-drop per second. Take care to ensure that the trap remains sufficiently cool to prevent re-volatilization of lighter hydrocarbons.

7.6 If the concentration of petroleum distillate in the sample is sufficiently high, a two-phase system will condense in the trap. Periodically drain the trap. Continue the distillation until the water insoluble phase ceases increasing in volume. At this point, cool the system, drain the trap, and reserve the condensate for later testing.

7.6.1 If, after boiling for 1 h, no visible oily liquid has collected in the trap, or only a small amount (too small to manipulate heat) has collected, allow the trap to cool, and rinse with a small amount of suitable solvent, such as *n*-pentane, *n*-hexane, diethyl ether or carbon disulfide, and reserve the resulting solution for testing.

7.6.1.1 *n*-Pentane, *n*-hexane, and diethyl ether produce relatively large signals when passing through a flame ionization detector, possibly obscuring the signal from lighter hydrocarbons.

## 8. Labeling

8.1 Collect the oily layer or extract in a test tube or septum seal vial, or in the case of larger samples, in an appropriately sized container.

8.1.1 Code or label the container holding the extract with the case number, sample number, date, separation method, and analyst's initials.

8.1.2 Follow the evidence documentation and handling procedures described in Guide E 1459 and Practice E 1492.

## 9. Cleaning

9.1 Clean the distillation apparatus thoroughly using a strong detergent in hot water, followed by an acetone rinse, followed by a final rinse in hot water to remove any acetone. Allow the apparatus to dry before reusing.

## 10. Keywords

10.1 fire debris samples; steam distillation

<sup>5</sup> Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the ACS, 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.

<sup>6</sup> Dow Corning Antifoam B has been found satisfactory for this purpose.

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