



Designation: E 1230 – 96

Standard Test Method for the Approximate Determination of Low Molecular Weight Hydrocarbons in Anhydrous Hydrogen Fluoride¹

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1. Scope

1.1 This test method is applicable for the approximate determination of C1–C6 hydrocarbons in anhydrous hydrogen fluoride (HF) in the range of 0.0001 to 0.1 %.

1.2 The values as stated in SI units are to be regarded as the standard, except in cases where measures for fittings are given and where standard gases are certified in terms of part per million. The values given in parentheses are given 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.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:

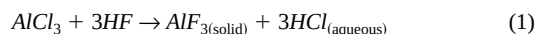
D 1193 Specification for Reagent Water²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

E 260 Practice for Packed Column Gas Chromatography⁴

3. Summary of Test Method

3.1 This test method is designed to measure contaminant hydrocarbons in liquid anhydrous hydrogen fluoride (HF). An anhydrous HF sample is collected in a 10 mL sample cylinder. The cylinder is then attached to a larger cylinder (500 mL) that has been fitted with a septum at one end and contains a solution of aluminum chloride (AlCl₃). The HF sample is allowed to flow into the 500-mL cylinder and mix with the AlCl₃ producing the following reaction.



The hydrocarbons are expelled into the vapor space, and a vapor sample is obtained from the cylinder via the septum by

using a gas tight syringe. The vapor sample is analyzed by gas chromatography using a flame ionization detector (FID). The resulting chromatogram permits qualitative and semi-quantitative determination of the hydrocarbons. A discussion of gas chromatography is presented in Practice E 260.

4. Significance and Use

4.1 Anhydrous HF may be contaminated with low levels of hydrocarbons. This is due in part to the high affinity of hydrocarbons to HF. This test method provides a means for the approximate determination of hydrocarbons in commercial anhydrous HF.

5. Interferences

5.1 The impurities in the aluminum chloride hexahydrate solution, from which the HF precipitates solid AlF₃, can be a source of interference. Whenever interfering compounds are traced to the neutralizing solution, a new source of aluminum chloride solution should be obtained. This procedure is quantitative as long as the solution interference contributes less than 10 % to the component concentration in the sample.

5.2 Compounds that have little or no response in the flame ionization detector and hence do not interfere are hydrogen sulfide gas (H₂S), sulfur dioxide gas (SO₂), carbon dioxide gas (CO₂), carbon monoxide gas (CO), silicon tetrachloride (SiCl₄), trichlorosilane (SiHCl₃), and silicon tetrafluoride (SiF₄).

6. Apparatus

NOTE 1—All apparatus or equipment used in direct contact with HF must be constructed from stainless steel or materials resistant to HF.

6.1 *Sample Cylinder*, consisting of a 10-mL stainless steel cylinder with 1/8-in. (3.1-mm) female NPT end fittings⁵ to which are attached stainless steel shut-off valves.⁶ The outlet of each valve is a 1/4-in. (6.2-mm) stainless steel Swagelok fitting. (See Fig. 1.)

6.2 *Reaction Cylinder* (Fig. 2), consisting of a 500-mL

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on Product Standards.

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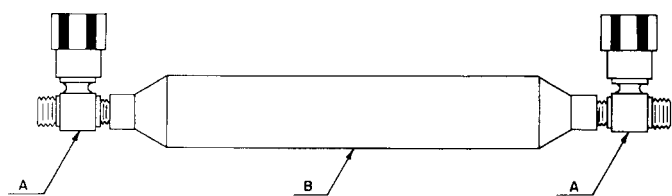
² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ *Annual Book of ASTM Standards*, Vol 14.01.

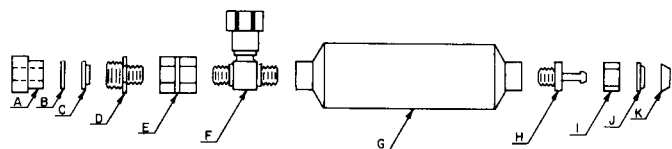
⁵ A sampling cylinder available from Hoke Inc., 1 Tenakill Park, Cresskill, NJ 07626, has been found satisfactory for this purpose. Request Catalogue No. 2HDY10.

⁶ A shut-off valve available from the Whitey Co., 318 Bishop Rd., Highland Heights, OH 44143, has been found satisfactory for this purpose. Request Catalogue No. SS-ODKM2-S4.



CODE	DESCRIPTION
A	1/4" Swagelok X 1/8" NPT Needle Valve
B	10mL Cylinder

FIG. 1 HF Sample Cylinder



CODE	DESCRIPTION
A	Cap
B	12.5mm Septum
C	Back Ferrule
D	1/4" NPT Male Adapter
E	Female Hex Coupling
F	1/4" MNPT Needle Valve
G	500mL TFE Lined Cylinder
H	1/4" NPT Male Tube Connector
I	1/4" Swagelok Nut
J	Back Ferrule
K	Front Ferrule

FIG. 2 Reaction Cylinder

stainless steel (polytetrafluoroethylene) PTFE lined cylinder⁷ with 1/4-in. (6.2-mm) female NPT end fittings at both ends.

6.2.1 The septum assembly attaches to the 500-mL reaction cylinder (Fig. 2) in the following manner: A knurled cap (A) encloses a 12.5-mm septum (B),⁸ backed by a ferrule (C) and a 1/4-in. (6.2-mm) NPT male adapter (D).⁹ The septum assembly is joined to the 500-mL cylinder by a stainless steel hex coupling (E)¹⁰ and a stainless steel shutoff valve¹¹ with 1/4-in. (6.2-mm) NPT end fittings (F).

6.2.2 The 500-mL cylinder attaches to the HF sample cylinder (Fig. 1) in the following manner: The 1/4-in. (6.2-mm)

⁷ A cylinder available from the Whitey Co., 318 Bishop Rd., Highland Heights, OH 44143, has been found satisfactory for this purpose. Request Catalogue No. 304-L-HDF4-500T.

⁸ A septum available from Supelco, Inc., Supelco Park, Bellefonte, PA 16823, has been found satisfactory for this purpose. Request Catalogue No. 2-0413.

⁹ The Ultra-Torr male adapter, available from the Cajon Co., 9760 Shepard Road, Macedonia, OH 44056, has been found satisfactory for this purpose. Request Catalogue No. SS-6-UT-1-4.

¹⁰ A hex-coupling, available from the Cajon Co., 9760 Shepard Road, Macedonia, OH 44056, has been found satisfactory for this purpose. Request Catalogue No. SS-4-HCG.

¹¹ A shut-off valve, available from the Whitey Co., 318 Bishop Road, Highland Hts., OH 44143, has been found satisfactory for this purpose. Request Catalogue No. SS-14DKM4.

NPT male tube adapter (H)¹² is fitted with a 1/4-in. (6.2-mm) Swagelok nut (I)¹³ with back (J) and front ferrules (K).

6.3 *Gas Chromatograph*, having accessories and capabilities as follows:

6.3.1 *Sample Injection Port*, lined with stainless steel and maintained at 125°C.

6.3.2 *Column Oven*, capable of isothermal operation at 55°C.

6.3.3 *Detector*, flame ionization type, capable of operation at 150°C.

6.3.4 *Column Packed with Chromosil 310*,¹⁴ 8-ft by 1/8-in. (2.4-m by 3.1-mm) outside diameter polytetrafluoroethylene (PTFE).

NOTE 2—The column described in 6.3.4 has been found suitable for this analysis. If other columns are used, the precision and bias of this test method should be ascertained.

6.3.5 *Attenuator Switch*, to permit accurate, variable attenuation of the detector signal.

6.4 *Recorder*, range to match chromatograph output signal, 1-s full scale deflection with a chart speed of approximately 3 cm/min or other convenient speed that will produce a satisfactory chromatogram. Alternatively, an integrating recorder can be used to facilitate measurement of the peak areas.

6.5 *Timer*, 60-min, adjustable to 1-min intervals.

6.6 *Cylinder Rotator*, adjustable to approximately 6 rpm.

6.7 *Vacuum Pump*, capable of reducing pressure to less than 10 torr.

6.8 *Syringe*, gas tight, 1-mL capacity.

6.9 *Syringe*, gas tight, 10-mL capacity.

6.10 *Syringe*, gas tight, 50-mL capacity.

7. Reagents

7.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.¹⁵ 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D 1193.

7.3 *Aluminum Chloride Hexahydrate Solution* (120 g/L)—Dissolve 120 g of aluminum chloride hexahydrate (AlCl₃·6H₂O) in water and dilute to 1 L.

¹² A fractional tube adapter to male pipe, available from the Cajon Co., 9760 Shepard Road, Macedonia, OH 44056, has been found satisfactory for this purpose. Request Catalogue No. SS-4-TA-1-4.

¹³ The fractional tube size nut, available from Swagelok Co., 31400 Aurora Road, Solon, OH 44139, has been found satisfactory for this purpose. Request Catalogue No. SS-402-1.

¹⁴ A Chromosil 310 column, available from Supelco, Inc., Supelco Park, Bellefonte, PA 16823, has been found satisfactory for this purpose. Request Catalogue No. 1-1501.

¹⁵ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.