Designation: D2284 – 11

Standard Test Method for Acidity of Sulfur Hexafluoride[1]

This standard is issued under the fixed designation D2284; 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 determination of the acidic fluorides of sulfur hexafluoride (SF_6).

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

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 Acidic constituents of the sample are absorbed in slightly alkaline water containing an indicator and the excess base titrated with a standard acid solution. The resultant acidity is expressed as equivalents of hydrofluoric acid (HF).

4. Significance and Use

4.1 Acidic fluorides are undesirable in SF_6 used as an electrical insulating gas in that they may contribute to corrosion or constitute dielectric hazard.

4.2 This test method is valid for both new and used SF_6. In used SF_6, it will only measure those active species which are hydrolyzable.

5. Apparatus

5.1 *Gas Washing Bottles,* 500-mL capacity, two required.

5.2 *Microburet,* graduated to 0.01 mL.

5.3 *Wet Test Meter.*

NOTE 1—The meter is used to measure the volume of dry gas in liters and hence the weight of SF_6 samples. The density of sulfur hexafluoride at 0°C and 1 atm. of pressure (STP) is 6.52 g/L. The density of sulfur hexafluoride at 25°C and 1 atm. of pressure (normal temperature and pressure) is 5.97 g/L. Alternatively, the sample weight may be determined by weighing the sample cylinder before and after sampling.

6. Reagents

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. 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. [3] 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Specification D1193.

6.3 *Phenolphthalein Indicator Solution* (10 *g/L*)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

6.4 *Sodium Hydroxide Solution (0.01 N)*—Dissolve 0.4 ± 0.01 g of sodium hydroxide (NaOH) in water and dilute to 1 L. Prepare a fresh solution weekly. Standardize by titrating against a weighed amount of potassium acid phthalate.

6.5 *Sulfuric Acid (0.01 N)*—Add 0.25 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to water and dilute to 1 L. Standardize against standard 0.01 *N* NaOH solution.

7. Sampling

7.1 Take the sample as a liquid from the cylinder to be sampled. This may be done by inverting the cylinder so that the outlet valve is at the bottom.

8. Preparation of Apparatus

8.1 Place the cylinder to be sampled as suggested in Section 7 and connect a needle valve to the cylinder outlet. Connect the

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

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

[3] *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 *Annual 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.

inlet of one of the gas sampling bottles to the cylinder needle valve and the outlet to the wet test meter. Flexible rubber or plastic tubing can be used. Set the meter to zero and note the reading.

9. Procedure

9.1 Boil 600 mL of deionized water in a 1-L beaker for about 5 min and cool quickly to room temperature. Add 10 drops of phenolphthalein indicator solution and enough 0.01 *N* NaOH solution to color the solution faintly pink. Pour half of the solution into each of the two gas washing bottles and add 2.00 mL of 0.01 *N* NaOH solution to each. Replace the caps on both bottles and set one aside to serve as a blank.

NOTE 2—Every precaution should be taken to ensure that both solutions are treated in an identical manner to ensure that the quantities of atmospheric contaminants such as CO_2 or other acidic vapors absorbed into the test and blank solutions are equal.

9.2 After placing the gas sampling tube in position (see Sections 7 and 8) carefully open the sample cylinder needle valve so that the sample gas passes through the solution at a rate of about 1 L/min. After 6 to 8 L of sample have passed through the tube, close the needle valve, remove the bubbler, and record the gas meter reading. Note that the sensitivity of the technique may be increased by using a larger volume of gas.

NOTE 3—Terminate sampling immediately if the solution fades to a pale pink.

9.3 Titrate the solution in each of the gas washing bottles with standard 0.01 *N* H_2SO_4 until they are faintly pink and match in color exactly.

10. Calculation

10.1 Calculate the acidity as HF as follows:

$$\text{Acidity as HF, mL/kg (ppm)} = \frac{[(B - A)N \times 0.020]}{DW} \times 10^6$$

where:

A = H_2SO_4 required by the sample solution, mL
B = H_2SO_4 required by the blank solution, mL
D = density of SF_6 gas, g/L,
N = normality of the H_2SO_4, and
W = sample used, L.

NOTE 4—When using the sample weight rather than the volume, bubble a minimum of 50 g of sample and use the weight of the sample as the denominator of the above equation.

11. Precision and Bias

11.1 *Precision*—The repeatability standard deviation on the difference between two test results at an average value of 1.12 mL of titrant has been determined to be ±0.018 for a single operator in a single laboratory. The 95 % repeatability limit for duplicate determinations should agree within 7.1 % of the average of the two results. It is not feasible to specify the reproducibility of the procedure at this time because no other laboratories have been found to participate in an ILS study. The data used to determine the precision is provided in Table 1.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of acidity in sulfur hexafluoride, no statement on bias can be made.

11.3 *Detection Limit*—The calculated lower limit is 0.04 ppm acidity as HF based on the burette which has a 0.01 mL increment.

12. Keywords

12.1 acid; electrical; fluoride; gas; hexafluoride; insulating; sulfur

TABLE 1 Data Used to Develop the Repeatability Statement

Sample A	mL of titrant
Test 1	1.14
Test 2	1.10
Test 3	1.14
Test 4	1.12
Test 5	1.10
Average	1.12
Standard Deviation	0.0179
Number of Determinations	5
Number of DE (n – 1)	4
Minimum Value	1.10
Maximum Value	1.14
95 % Repeatability, ASTM	±0.08
ASTM Repeatability of Average	7.1 %