Designation: C1502 – 16

Standard Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide[1]

This standard is issued under the fixed designation C1502; 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 chlorine and fluorine in nuclear-grade uranium dioxide (UO_2) powder and pellets, nuclear grade gadolinium oxide (Gd_2O_3) powder and gadolinium oxide-uranium oxide (Gd_2O_3-UO_2) powder and pellets.

1.2 With a 2 gram UO_2 sample size the detection limit of the method is 4 µg/g for chlorine and 2 µg/g for fluorine. The maximum concentration determined with a 2 gram sample is 500 µg/g for both chlorine and fluorine. The sample size used in this test method can vary from 1 to 10 grams resulting in a corresponding change in the detection limits and range.

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

1.4 *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]

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets

C859 Terminology Relating to Nuclear Materials

C888 Specification for Nuclear-Grade Gadolinium Oxide (Gd_2O_3) Powder

C922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets

D1193 Specification for Reagent Water

3. Terminology

3.1 *Definitions*—Except as otherwise defined herein, definitions of terms are given in Terminology C859.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accelerator*—a chemical compound or a flux that will decrease the reaction time or prohydrolysis time.

4. Summary of Test Method

4.1 The halogens are separated from the test materials by pyrohydrolysis in a quartz reaction tube with a stream of wet oxygen or air at a temperature of 900 to 1000°C (**1-4**). Chloride and fluoride are volatilized simultaneously as acids, absorbed in an absorption solution as chloride and fluoride and measured with ion selective electrodes (**4-6**).

5. Significance and Use

5.1 The method is designed to show whether or not the tested materials meet the specifications as given in either Specification C753, C776, C888 or C922.

6. Interferences

6.1 The absorption solution controls the pH of the measured solution to avoid hydroxide ion interference or the formation of hydrogen complexes with fluoride.

6.2 Bromide, iodide, cyanide and sulfide, if present in the condensate, interfere in the measurement of chloride with ion-selective electrodes, but have very little effect upon the measurement of fluoride with ion-selective electrodes.

6.3 As the ionic activity of the chloride and fluoride ions is temperature dependent, the standard solutions and sample solutions should be measured at the same temperature.

7. Apparatus

7.1 *Pyrohydrolysis Equipment,* the assembly of suitable equipment is shown in Fig. 1.

7.2 *Gas Flow Regulator and Flowmeter.*

7.3 *Hot Plate,* used to warm the water saturating the sparge gas to 50 to 80°C.

7.4 *Combustion Tube Furnace,* having a bore of about 32 mm with a length of about 300 mm and the capability of

[1] This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] 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.

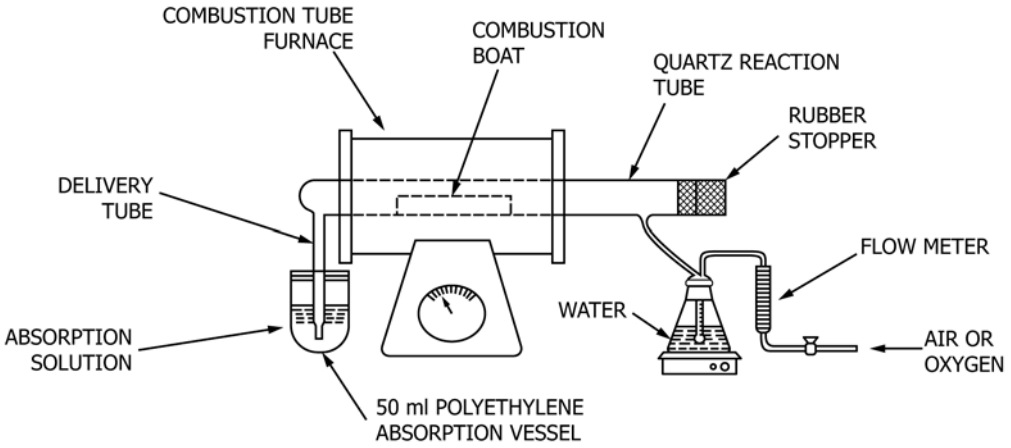


FIG. 1 Pyrohydrolysis Equipment

maintaining a temperature of 950 ± 25°C. Combustion tube furnaces with different dimensions may be satisfactory. Temperatures between 900 and 1000°C have been found to be satisfactory.

7.5 *Quartz Reaction Tube* (Fig. 2)—The exit end should not extend more than 50 mm beyond the furnace with a ground joint connecting to the delivery tube. The delivery tube extends into a polyethylene or Pyrex absorption vessel with a tip capable of giving a stream of very fine bubbles. A second absorption vessel connected in series, may be necessary to ensure complete collection of the fluorine and chlorine from the sample.

7.6 *Combustion Boat,* a ceramic, platinum or quartz boat with a 10 mL capacity (approx. 90 to 100 mm long, 13 mm wide, and 10 mm high). Boats with different dimensions may be satisfactory.

7.7 *Absorption Vessel,* a 50-ml polyethylene graduate or tube is satisfactory.

7.8 *Ion-Selective Electrodes,* fluoride-selective activity electrode, chloride-selective activity electrode. Combination electrodes may be suitable.

7.9 *Double-Junction Reference Electrode,* such as a silver-silver chloride with appropriate filling solutions.

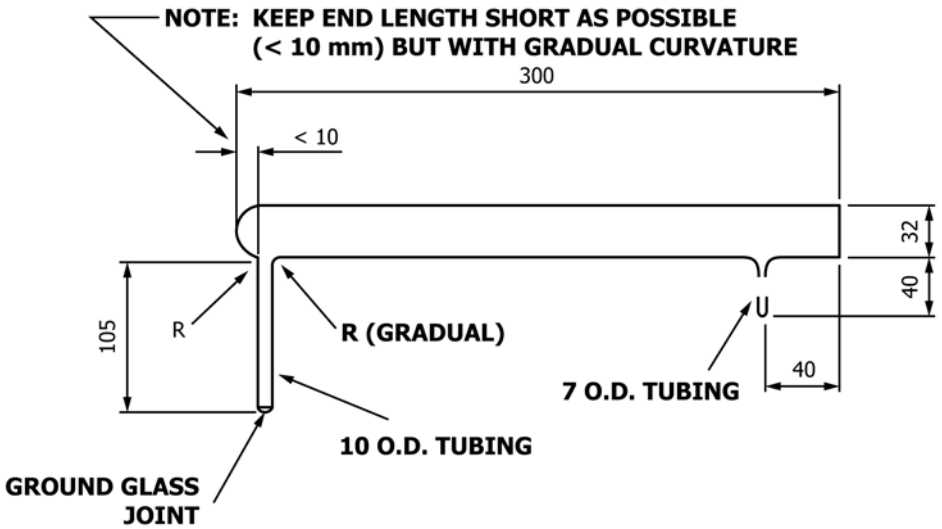
7.10 *pH/mV Meter*—The meter should have minimum resolution of 1 mV.

7.11 *Magnetic Stirrer.*

7.12 *Beakers,* 50 mL polyethylene.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that



NOTE 1—All dimensions in millimetres.

FIG. 2 Quartz Reaction Tube