



Designation: C696 – 11

Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets¹

This standard is issued under the fixed designation C696; 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 These test methods cover procedures for the chemical, mass spectrometric, and spectrochemical analysis of nuclear-grade uranium dioxide powders and pellets to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

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Uranium by Ferrous Sulfate Reduction in Phosphoric Acid and Dichromate Titration Method	2
C1267 Test Method for Uranium By Iron (II) Reduction In Phosphoric Acid Followed By Chromium (VI) Titration In The Presence of Vanadium	3
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2. Referenced Documents

2.1 ASTM Standards:³

- C753** Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder
- C761** Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride
- C776** Specification for Sintered Uranium Dioxide Pellets
- C1267** Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium
- C1287** Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry
- C1347** Practice for Preparation and Dissolution of Uranium Materials for Analysis
- C1408** Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method
- C1413** Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions by Thermal Ionization Mass Spectrometry
- C1453** Test Method for the Determination of Uranium by

¹ These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Sept. 1, 2011. Published October 2011. Originally approved in 1972. Last previous edition approved in 2005 as C696 – 99(2005). DOI: 10.1520/C0696-11.

² Discontinued January 1999. See C696–80.

³ 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.

⁴ Discontinued September 2011.

⁵ Discontinued as of May 30, 1980.

- Ignition and the Oxygen to Uranium (O/U) Atomic Ratio of Nuclear Grade Uranium Dioxide Powders and Pellets
C1457 Test Method for Determination of Total Hydrogen Content of Uranium Oxide Powders and Pellets by Carrier Gas Extraction
C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide
D1193 Specification for Reagent Water
E115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis (Withdrawn 2002)⁶
E130 Practice for Designation of Shapes and Sizes of Graphite Electrodes (Withdrawn 2013)⁶
E402 Test Method for Spectrographic Analysis of Uranium Oxide (U₃O₈) by Gallium Oxide-Carrier Technique (Withdrawn 2007)⁶

3. Significance and Use

3.1 Uranium dioxide is used as a nuclear-reactor fuel. In order to be suitable for this purpose, the material must meet certain criteria for uranium content, stoichiometry, isotopic composition, and impurity content. These test methods are designed to show whether or not a given material meets the specifications for these items as described in Specifications **C753** and **C776**.

3.1.1 An assay is performed to determine whether the material has the minimum uranium content specified on a dry weight basis.

3.1.2 The stoichiometry of the oxide is useful for predicting its sintering behavior in the pellet production process.

3.1.3 Determination of the isotopic content of the uranium in the uranium dioxide powder is made to establish whether the effective fissile content is in compliance with the purchaser's specifications.

3.1.4 Impurity content is determined to ensure that the maximum concentration limit of certain impurity elements is not exceeded. Determination of impurities is also required for calculation of the equivalent boron content (EBC).

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used 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.⁷ 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

⁶ The last approved version of this historical standard is referenced on www.astm.org.

⁷ *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.

5. Safety Precautions

5.1 Proper precautions should be taken to prevent inhalation, or ingestion of uranium dioxide powders or dust during grinding or handling operations.

6. Sampling

6.1 Criteria for sampling this material are given in Specification **C753** and Specification **C776**.

6.2 Samples can be dissolved using the appropriate dissolution techniques described in Practice **C1347**, but final determination of applicability must be made by the user.

URANIUM BY FERROUS SULFATE REDUCTION IN PHOSPHORIC ACID AND DICHROMATE TITRATION METHOD

This test method was withdrawn in January 1999 and replaced by Test method **C1267.**

URANIUM AND OXYGEN TO URANIUM ATOMIC RATIO BY THE IGNITION (GRAVIMETRIC) IMPURITY CORRECTION METHOD

This test method was withdrawn in September 2011 and replaced by Test Method **C1453.**

CARBON (TOTAL) BY DIRECT COMBUSTION-THERMAL CONDUCTIVITY METHOD

This test method was withdrawn in January 1999 and replaced by Test Method **C1408.**

TOTAL CHLORINE AND FLUORINE BY PYROHYDROLYSIS ION-SELECTIVE ELECTRODE METHOD

This test method was withdrawn in September 2011 and replaced by Test Method **C1502.**

MOISTURE BY THE COULOMETRIC ELECTROLYTIC MOISTURE ANALYZER METHOD

7. Scope

7.1 This test method covers the determination of moisture in uranium dioxide samples. Detection limits are as low as 10 µg.

8. Summary of Test Method

8.1 The sample is heated in an oven (up to 400°C) to drive off any water. The moisture is carried from the oven into the

electrolytic cell by a flowing stream of *dry* nitrogen. Two parallel platinum wires wound in a helix are attached to the inner surface of the tube, the wall of which is evenly coated with phosphorus pentoxide (P_2O_5) (a strong desiccant that becomes electrically conductive when wet). A potential applied to the wires produces a measurable electrolysis current when moisture wets the desiccant. Electrolysis of the water continuously regenerates the cell enabling it to accept additional water.

8.2 Precautions must be taken to prevent interference from the following sources. Hydrogen fluoride will cause permanent damage to the cell and sample system and should not be run under any conditions. Corrosive acidic gases, such as chlorine and hydrogen chloride, will corrode the instrument. Entrained liquids and solids can cause cell failure and should be prevented from entering the gas stream. Ammonia and other basic materials react with the acidic cell coating and renders the cell unresponsive. Hydrogen, and to a lesser extent, oxygen or air, may cause a high reading due to recombination, in the cell, or in the case of hydrogen, due to reaction with oxide coating of the sample boat to produce water. Alcohols and glycols, particularly the more volatile ones, respond like water and therefore must not be present.

9. Apparatus

9.1 *Moisture Analyzer*, for solids, with quartz glass oven capable of being heated from ambient temperatures to 1000°C. The assembly includes electrolytic cell, flow meter, range 30 to 140 cm³/min air, and a dryer assembly.⁸

9.2 *Balance*,⁹ for weighing samples in the range from 1 to 100 mg.

9.3 *Nitrogen Gas Cylinder*, with a pressure regulator, a flow meter and a drying tower.

10. Reagents

10.1 *Barium Chloride Dihydrate* ($BaCl_2 \cdot 2 H_2O$).

11. Operation

11.1 Turn the main power switch ON.

11.2 Adjust nitrogen gas pressure to 41.4 kPa (6 psi) and the flow rate to 50 mL/min measured at the exit of the apparatus.

11.3 Weigh the sample into a small, dry, aluminum boat (**Note 1**) and insert it into the instrument oven as follows:

NOTE 1—For samples that have been reduced in a hydrogen atmosphere and thus contain excess hydrogen, the use of a platinum boat in place of the aluminum tube and nickel boat will minimize any interference due to the hydrogen.

⁸ A CEC Solids Moisture Analyzer, of Type 26-321A-MA is available from DuPont Instruments Inc., S. Shamrock Ave., Monrovia, CA 91016. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁹ A Cahn Electrobalance, or equivalent, available from Cahn Division, Ventrum Instrument Corp., Paramount, CA has been found satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

11.3.1 Open the top of the analyzer and remove the TFE-fluorocarbon plug. Do not touch with gloves.

11.3.2 With forceps pull the nickel boat one third of the way out of the tube and place the aluminum boat and the sample inside the nickel boat, then reposition the nickel boat near the center of the heating coils.

11.3.3 Replace the TFE-fluorocarbon plug and close the lid of the analyzer.

11.4 Reset the counter to 0 μ g.

11.5 Set the timer at 1 h.

11.6 Set the temperature at 400°C. This will activate the analyzer and start the heating cycle.

11.7 When the preset temperature has been reached and the counter ceases counting, record the reading, *S*.

12. Standardization

12.1 Determine the blank by processing dry, empty, aluminum boats according to steps 11.3 – 11.7 until constant values are obtained.

12.2 Weigh and analyze replicate 5-mg samples of $BaCl_2 \cdot 2 H_2O$ until consistent results are obtained. Sodium tungstate dihydrate ($Na_2WO_4 \cdot 2 H_2O$) may also be used for calibration.

13. Calculation

13.1 Calculate the moisture recovery, *Z*, for the standard as follows:

$$Z = (A - B)147.2Y \quad (1)$$

where:

A = micrograms of moisture on counter when standard is tested,

B = micrograms of moisture on counter from blank, and
Y = milligrams of $BaCl_2 \cdot 2 H_2O$. Each milligram of $BaCl_2 \cdot H_2O$ contains 147.2 μ g of water.

13.2 Calculate the percent moisture in the sample as follows:

$$\text{Moisture, \%} = [(S - B)/1000 WZ] \times 100 = (S - B)/10 WZ \quad (2)$$

where:

S = micrograms of moisture on counter when sample is tested,

B = micrograms of moisture on counter from blank,

W = milligrams of sample, and

Z = recovery of moisture from standard.

14. Precision

14.1 The relative standard deviation for moisture in a concentration range of 100 μ g/g is approximately 2 % but increases to 10 % at the 20 μ g/g level.

NITROGEN BY THE KJELDAHL METHOD

15. Scope

15.1 This test method covers the determination of nitride nitrogen in uranium dioxide in the range from 10 to 250 μ g.

16. Summary of Test Method

16.1 The sample is decomposed with acid, the resulting solution is made strongly alkaline with sodium hydroxide solution, and the nitrogen is separated as ammonia by steam distillation. The distillate is collected in boric acid solution and the ammonia present is titrated with 0.01 *N* standard acid using a mixed indicator.

NOTE 2—Although a simple acid digestion is usually adequate for dissolution of uranium samples, some uranium nitrides do not yield to such treatment. The use of potassium dichromate in phosphoric acid (1)¹⁰ has proved to be successful with nitrides that are difficult to decompose. Therefore, this medium has been recommended although, in most cases, a mixture of phosphoric and sulfuric acids would be adequate.

17. Interferences

17.1 There should be no interferences in nuclear-grade uranium dioxide.

18. Apparatus

18.1 *Nitrogen Distillation Apparatus*, micro.¹¹

18.2 *Heater*, 750-W electric, full-control.

18.3 *Burner*, bunsen-type.

18.4 *Buret*, micro, class A, 5- or 10-mL capacity, graduated in 0.02-mL divisions.

19. Reagents

19.1 *Ammonia-Free Water*—Prepare by distillation or from an ion-exchange column.

19.2 *Boric Acid-Indicator Solution*—Dissolve 20 g of boric acid (H_3BO_3) in 800 mL of hot ammonia-free water, cool the solution, add 4 mL of mixed indicator solution (52.3), and dilute to 1 litre.

19.3 *Mixed Indicator Solution*—Mix 100 mL of a 1 % alcoholic solution of bromocresol green and 20 mL of a 1 % alcoholic solution of methyl red.

19.4 *Phosphoric Acid (H_3PO_4 , 85 %)*—Heat acid to 190°C to remove excess water.

NOTE 3—Some lots of H_3PO_4 give high blanks and cannot be used.

19.5 *Potassium Dichromate Solution (65 g/litre)*—Dissolve 65 g of potassium dichromate ($K_2Cr_2O_7$) in ammonia-free water and dilute to 1 litre. If necessary to reduce the blanks prepare the dichromate by recrystallization of K_2CrO_4 from alkaline solution (1).

19.6 *Sodium Hydroxide Solution*—Dissolve 500 g of sodium hydroxide (NaOH) in 1 litre of ammonia-free water.

19.7 *Sulfuric Acid, Standard*—(H_2SO_4 , 0.01 *N*)—Standardize against a standard sodium hydroxide solution that has been standardized against potassium hydrogen phthalate.

¹⁰ The boldface numbers in parentheses refer to the list of references at the end of these methods.

¹¹ Kemmerer-Hallett Type, Fisher Scientific Co., has been found satisfactory. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

NOTE 4—Hydrochloric acid (HCl, 0.01 *N*) may be used instead of H_2SO_4 .

20. Procedure

20.1 Blank Determinations:

20.1.1 Fill the boiler of the distillation apparatus with ammonia-free water and distill for at least 30 min with a digestion flask in place in order to purge the apparatus of any traces of ammonia present.

20.1.2 Place 10 mL of H_3PO_4 and 15 mL of potassium dichromate solution (65 g/litre) in a digestion flask and attach to the apparatus. Add 50 mL of NaOH solution and start passing the steam from the boiler through the digestion flask.

20.1.3 Place a 125-mL Erlenmeyer flask containing 5 mL of the boric acid-indicator solution over the tip of the condenser and collect 25 mL of distillate. Lower the flask so that the tip of the condenser is above the level of the distillate and continue the distillation for an additional 30 s to rinse down the inside of the tube.

20.1.4 Titrate the distillate with the 0.01 *N* H_2SO_4 from a microburet until the solution turns to a pink color.

20.1.5 Repeat the blank determination, steps 20.1.2 – 20.1.4, until the blanks are constant. If the blank exceeds 0.03 to 0.04 mL, look for a source of contamination.

20.2 Analysis of the Sample:

20.2.1 Transfer up to 2 g of a weighed, powdered sample (Note 5) to the digestion flask.

NOTE 5—Samples in pellet form must be crushed in a diamond mortar to – 100 mesh powder and sampled by riffing or quartering to obtain a representative sample.

20.2.2 Add 10 mL of H_3PO_4 and heat the flask gently with a small burner until a clear green solution is obtained. Inspect the solution carefully to ensure that no undissolved uranium nitrides remain.

20.2.3 Cool the flask, then add 15 mL of $K_2Cr_2O_7$ solution (65 g/litre) slowly with mixing. Warm at low heat for 3 to 4 min.

20.2.4 Attach the digestion flask to the distillation apparatus and add 50 mL of NaOH solution.

20.2.5 Place the receiving flask containing 5 mL of the boric acid-indicator solution over the condenser tip and distill and titrate following the procedure used to determine the blank.

21. Calculation

21.1 Calculate the nitrogen content as follows:

$$N, \mu\text{g/g on } UO_2 \text{ basis} = (A - B) 14.01 N \times 10^3 / W \quad (3)$$

where:

- A = millilitres of standard acid to titrate sample,
- B = millilitres of standard acid to titrate blank,
- N = normality of standard acid solution, and
- W = grams of UO_2 sample.

22. Precision

22.1 This test method will determine nitrogen to within 7 μg of the amount present.

**ISOTOPIC URANIUM COMPOSITION BY
MULTIPLE-FILAMENT SURFACE-IONIZATION
MASS SPECTROMETRIC METHOD**
(This test method was withdrawn in 1980 and replaced
by Test Method **C1413**.)

**SPECTROCHEMICAL DETERMINATION OF TRACE
ELEMENTS IN HIGH-PURITY URANIUM DIOXIDE**

23. Scope

23.1 This test method covers the spectrographic analysis of nuclear-grade UO₂ for the 26 elements in the ranges indicated in **Table 1**.

23.2 For simultaneous determination of trace elements by plasma emission spectroscopy refer to Test Method **C761**.

24. Summary of Test Method

24.1 The sample of UO₂ is converted to U₃O₈ and mixed with a spectrochemically pure carrier consisting of 16.4 mol % strontium fluoride in silver chloride. A given quantity of this mixture is placed in a special cupped electrode and excited in a d-c arc. The spectrum is recorded on photographic plates and the selected lines are either visually compared with standard plates or photometrically measured and compared with synthetically prepared standards exposed on the same plate.

25. Significance

25.1 Carrier distillation methods for the analysis of uranium over the past years have used a variety of carriers. Test Method

E402, approved by ASTM Committee E-2 on Emission Spectroscopy, called for gallium oxide as the carrier. This method involves the use of a mixture of silver chloride and strontium fluoride (**2, 3**). The fluoride gives an increased sensitivity for aluminum, zirconium, titanium, and niobium.

25.2 For the analysis of refractory elements in uranium, a separation is required for maximum sensitivity. However, recent work (**4, 5**) has improved the sensitivity of some elements using a mixed carrier technique.

26. Apparatus

26.1 *Spectrograph*—A spectrograph with sufficient resolving power and linear dispersion to separate the analytical lines from other lines in the spectrum of the sample in the spectral region 4200 to 7000 Å is required. Instruments with a reciprocal linear dispersion of approximately 5 Å/mm, first order or less, are satisfactory. A direct-reading spectrograph of comparable quality may be substituted for the equipment listed, in which case the directions given by the manufacturer should be followed rather than those given in the succeeding steps of this procedure.

26.2 *Excitation Source*—Use a high-voltage spark source capable of providing a 14-A d-c arc (short circuit).

26.3 *Excitation Stand*—Conventional type with adjustable water-cooled electrode holders.

26.4 *Developing Equipment*—Use developing, fixing, washing, and drying equipment conforming to the requirements of Practice **E115 (6)**.

26.5 *Microphotometer*, having a precision of at least ± 1 % for transmittances.

26.6 *Mixer*, for dry materials.¹²

26.7 *Platinum Crucible*, 10-mL capacity.

26.8 *Venting Tool*—See **Fig. 1** for diagram.

26.9 *Calculating Boards*, or other special equipment are optional, their use depending to a large extent on how frequently analyses are made and how much speed is required.

26.10 *Muffle Furnace*, capable of heating up to 900°C.

26.11 *Electrode Forceps*, with each V-tip bent to form a semicircular grasp around the electrodes.

26.12 *Balances*, torsion-type, one with a capacity up to 1 g and capable of weighing to ±0.1 mg, and one with a capacity of 500 g.

27. Reagents and Materials

27.1 *Agate Mortars*.

**TABLE 1 Recommended Analytical Spectral Lines and
Concentration Range of Trace Elements**

Element	Analytical Line, °A ^A	Concentration range, µg/g of U
Ag ^B	3280.68	0.1 to 50
Al	2367.06	10 to 200
As	2349.84	5 to 50
B	2497.73	0.10 to 5
Ba	4554.04	10 to 300
Be	2348.61	0.1 to 3
Bi	3067.72	5 to 50
Ca	4226.73	1 to 50
Cd	2288.02	0.15 to 5
Co	3453.51	5 to 50
Cr	2843.25	10 to 100
Cu	3247.54	1 to 10
Fe	2462.64	10 to 300
In	3256.09	5 to 50
Mg	2779.83	10 to 100
Mn	2605.69	5 to 50
Mo	3132.59	0.5 to 10
Na	3302.32	80 to 400
Ni	3050.82	5 to 100
P	2553.28	50 to 500
Pb	2833.07	5 to 50
Sb	2598.05	1 to 50
Si	2435.16	10 to 200
Sn	3175.02	5 to 50
Ti	3361.26	1 to 100
V	3183.41	1 to 100
Zn	3345.02	20 to 300
Zr	3438.23	25 to 300

^A All of the above lines are photographed in the second order, except barium and calcium which are first order lines.

^B A gallium oxide carrier must be used for silver. See Test Method **E402**.

¹² The Fisher-Kendall mixer was found to be satisfactory for large quantities and the Wig-L-Bug (Spex Industries) for small quantities. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

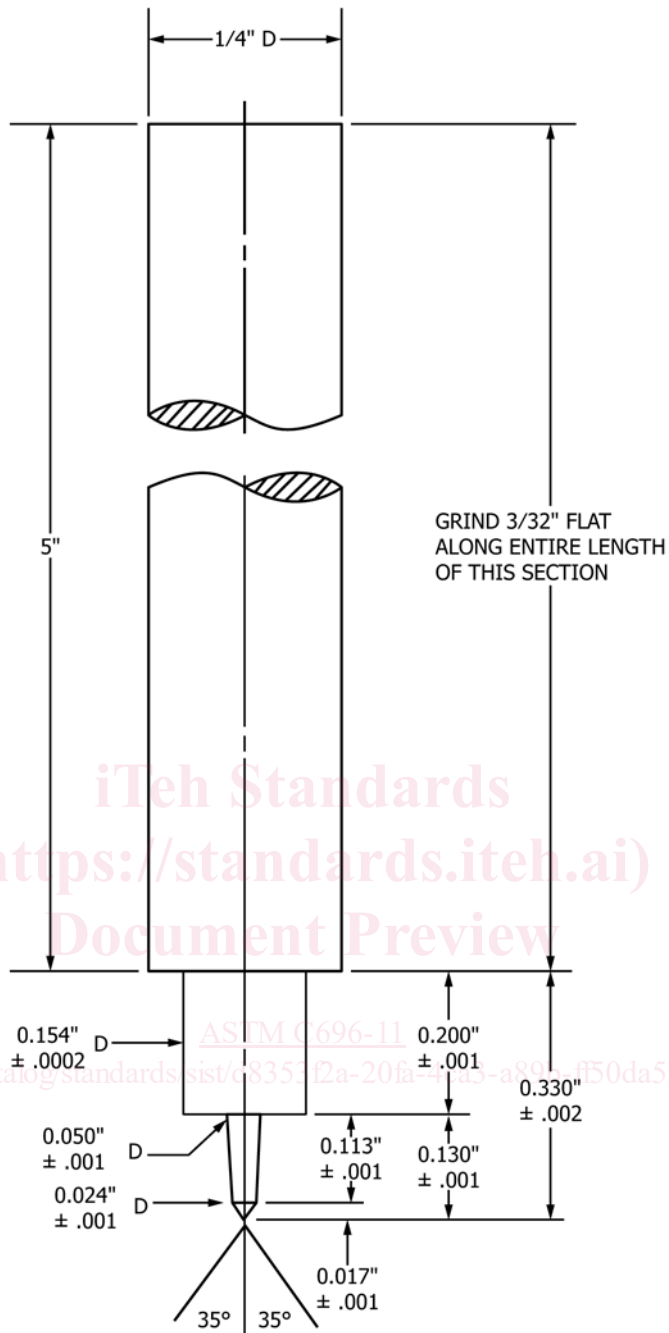


FIG. 1 Venting Tool

27.2 *Electrodes*—The anode, pedestal, and counter electrodes should be respectively of the S-1, S-2, and C-1 types as given in Practice E130.¹³

¹³ Upper electrode, Ultra Carbon 1992, lower electrode, Ultra Carbon 1998, electrode pedestal, Ultra Carbon 1993.

27.3 *Glassine Paper*.

27.4 *Tissue*—A suitable wiping tissue is necessary.

27.5 *Mixing Vial*, plastic, having a 12.7-mm (½-in.) diameter and a 25.4-mm (1-in.) length with cap, and a 9.6-mm (¾-in.) diameter plastic ball.