Designation: C1517 – 16

Standard Test Method for
Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy[1]

This standard is issued under the fixed designation C1517; 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 describes the steps necessary for the preparation and determination of impurity metals in uranium metal and uranium compounds by DC arc emission spectroscopy.

1.2 The method is valid for those materials that can be dissolved in acid or converted to an oxide in a muffle furnace, or both (see Practice C1347).

1.3 This method uses the carrier distillation technique to selectively carry the impurities into the arc, leaving the uranium oxide in the electrode. If it is necessary to determine the carrier metal (usually a silver or strontium, or gallium compound) as an impurity, another technique must be chosen for that element.

1.4 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 *This standard may involve hazardous materials, operations and equipment. 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
C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride
C776 Specification for Sintered Uranium Dioxide Pellets
C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals
C859 Terminology Relating to Nuclear Materials
C967 Specification for Uranium Ore Concentrate
C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis
E130 Practice for Designation of Shapes and Sizes of Graphite Electrodes (Withdrawn 2013)[3]
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminologies C859 and E135.

4. Summary of Test Method

4.1 Uranium metal, solutions and compounds are converted to uranium oxide (U_3O_8) in a muffle furnace. A weighed amount of the oxide is mixed with an appropriate spectrographic carrier and loaded into a graphite electrode. The electrode is excited in a DC arc and the light is dispersed by a spectrograph or spectrometer. The resulting spectrum is measured electronically using a CCD, CID, or CMOS camera sensitive to the proper regions. The line intensities are compared directly to calibration curves derived from the arced standards.

5. Significance and Use

5.1 This test method is applicable to uranium metal, uranium oxides and compounds soluble in nitric or sulfuric acid, and uranium solutions which can be converted to uranium oxide (U_3O_8) in a muffle furnace. It may be used to determine the impurities in uranium compounds as listed in Specifications C753, C776, C788, and C967.

6. Apparatus

6.1 *Spectrograph or Spectrometer*—A spectrograph with sufficient resolving power and linear dispersion to separate the

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[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

analytical lines from other lines in the spectrum of the sample in the spectral region of 230 to 855 nm is required. Some spectrographs may be able to access wavelengths lower than 230 nm that may allow the determination of other analytes. Instruments with a reciprocal linear dispersion in the first order of 0.5 nm/mm or less are satisfactory. The spectrometer should include a CCD, CID, or CMOS camera for electronic measurement of the resulting spectrum.

6.2 *Excitation Source*—Use an arc power source capable of providing a dc arc of up to 14-A dc, depending on the carrier used and electrode design.

6.3 *Excitation Stand*—Conventional type with adjustable water-cooled electrode holders (may be fitted with automatic sample changers if desired).

6.4 *Mixer,* for dry materials.

6.5 *Platinum Crucible.*

6.6 *Venting Tool,* (see Fig. 8, Test Methods C761 – 01).

6.7 *Muffle Furnace,* 1000°C capability.

7. Reagents and Materials

7.1 *Purity of Materials*—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 of Analytical Reagents of the American Chemical Society[4] where such specifications are available. 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 *Electrodes*—The anode and counter electrodes should be of the S-2, S 16 and C-1 types as given in Practice E130 (or equivalent).

NOTE 1—Exact shapes and dimensions of the electrodes are not as critical as given in Practice E130; however, dimensions of the electrodes used should be consistent and it is essential that the same dimension electrodes be used for standards and samples.

7.3 *Nitric Acid (HNO_3)*—concentrated (70 %), electronic grade, or equivalent.

7.4 *Sulfuric Acid (H_2SO_4)*—concentrated, electronic grade, or equivalent.

7.5 *Spectrographic Carrier*—The following spectrographic carriers have been used successfully for DC Arc analysis:

7.5.1 *Silver Chloride-Lithium Fluoride,* 11:1 w/w ratio.

7.5.2 *Silver Chloride-Silver Fluoride,* 4:1 w/w ratio.

7.5.3 *Gallium Oxide,* 99.99 % or better.

7.5.4 *Silver Chloride-Strontium Fluoride,* 16.4 mol % SrF_2 in AgCl.

7.5.5 *Gallium Oxide-Lithium Fluoride,* 11:1 w/w ratio.

7.6 *Mixing Vial,* plastic, 12.7 mm (1/2 in.) by 25.4 mm (1 in.) with cap and 10 mm (3/8 in.) plastic mixing bead.

TABLE 1 Carrier—Sample Combinations

Carrier Material	Carrier Wt, (mg)	Oxide Wt, (mg)	Electrode Charge, (mg)	Mixing Time, (s)
AgCl/AgF (4:1)	50	250	50	180
AgCl/LiF (11:1)	30	270	100	60
AgCl/SrF_2 (~6:1)	50	450	100	30
Ga_2O_3/LiF (11:1)	20	380	100	25
Ga_2O_3[A]	7	343	100	60

[A] For the determination of Ag and Li only.

NOTE 1—The above listed combinations of carrier, uranium oxide and electrode charge have been successfully used for the determination of impurities in uranium oxide. Other combinations may be available or suitable. However, the user must demonstrate comparable precision and bias.

7.7 *Standard Uranium Oxide (U_3O_8) Diluent*—Use NBL CRM 129[3] (or its replacement or equivalent) of known impurity level as a diluent.

8. Precautions

8.1 Consult manufacturer's Safety Data Sheets (SDS) for chemical incompatibilities, specific hazards, or spill cleanup for any hazardous materials used in this method.

8.2 All mixing and weighing operations involving uranium oxides should be carried out in properly functioning hoods or gloveboxes.

9. Standardization and Calibration

9.1 *Standards:*

9.1.1 Standards may be synthesized by adding the impurity elements to purified U_3O_8 (NBL CRM 129-A[5], or equivalent) and homogenizing. Impurities in powder form, preferably as oxides, may be blended in U_3O_8; impurities in solution may be added to U_3O_8 and the mixture dried, blended and reignited, or the impurities and uranium may be combined in solution and reconverted to U_3O_8. The individual elements should grade in such a ratio as to facilitate visual comparisons covering the desired analytical range for each.

9.1.2 The compounds used to make U_3O_8 impurity standards should be of the highest purity available.

9.1.3 Alternatively, commercially available uranium impurity standards, such as NBL CRM 123[5] and 124[5] series standards, may be used. (Other standards may be available; the user should determine quality or applicability, or both, prior to use.) These may be supplemented by synthetic standards to extend calibration ranges, if necessary.

9.1.4 For each standard used, prepare in the same ratio of uranium oxide to carrier as for samples (see Table 1 for further details).

9.1.5 Charge the electrode and arc at the same conditions as determined to be optimum for the instrument in use.

9.2 *Calibration Curves:*

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[4] *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] Available from the US Department of Energy, New Brunswick Laboratory, Building 350, 9800 South Cass Avenue, Argonne, IL 60439, ATTN: Reference Material Sales. http://science.energy.gov/nbl/.

9.2.1 In using a direct reading spectrometer, calibrate according to manufacturer's instruction.

10. Procedure

10.1 *Sample Preparation*

10.1.1 *Liquid Samples:*

10.1.1.1 Transfer into a platinum crucible a sufficient amount of liquid to yield not more than 2 g uranium oxide.

10.1.1.2 Place the crucible on a hot plate and evaporate to dryness. Do not allow to boil.

TABLE 2 Typical Wavelengths and Limits of Detection

Element	Wavelength (nm)	LOD using AgCl/AgF (ppm)	LOD using AgCl/LiF (ppm)	LOD using Ga_2O_3/LiF (ppm)
Al	308.21	1	5	1
	236.70			
As	234.98	10	. . .	
Au	267.59	1	. . .	
B	249.77	0.1	0.4	0.5
Ba	455.40	2	1	
	493.41			
Be	234.86	0.1	1	1
	249.45			
Bi	306.77	1	. . .	
Ca	396.84	10	25	
	422.64			
Cd	228.80	0.1	0.3	0.5
Co	240.72	1	5	
	340.51			
Cr	284.33	2	10	3
	425.44			
	427.48			
Cs	852.11	10	. . .	
Cu	324.75	1	2	1
	327.40			
Fe	248.33	10	20	2
	252.28			
	302.06			
Ga	294.36	1	. . .	
Ge	265.12	1	. . .	
K	769.89	10	. . .	
Li	670.78	0.2		
Mg	279.55	2	10	1
	280.27			
Mn	279.48	1	10	1
	279.83			
Mo	313.21	10		
Na	588.99	1	10	
	589.59			
Nb	316.34	10	. . .	
Ni	300.25	1	5	2
	341.48			
P	255.33	100	25	
	213.62			
Pb	283.31	4	3	1
Pd	340.46	1	. . .	
Rb	780.02	20	. . .	
Sb	259.81	2	10	
Si	250.69	10	10	1
	251.61			
	288.16			
Sn	283.99	10	2	1
	317.50			
Sr	407.77	20	. . .	
Ta	265.33		35	
Ti	334.90	4	5	
	337.28			
V	292.40	1	5	
	318.34			
W	294.70	100	. . .	
Zn	330.25	10	10	30
	213.86			

NOTE 2—The addition of H_2SO_4 may be necessary for solutions of uranyl chloride to effect complete conversion to oxide.

10.1.1.3 Continue the preparation at 10.1.2.2.

10.1.2 *Miscellaneous Uranium Oxides, Solids or Compounds:*

10.1.2.1 Transfer not more than 2 g of the uranium compound to a platinum crucible.

10.1.2.2 Place in a cool muffle and set the muffle to 900 ± 50°C.

NOTE 3—Some impurities (for example, Mo, W, Cr) may be lost at high temperatures. Do NOT place samples directly in hot muffle. Lower temperatures may be used if it is determined that complete oxide conversion is achieved.

10.1.2.3 Leave the crucibles and samples in the muffle until conversion to U_3O_8 is complete.

NOTE 4—Times will vary depending on amount and type of sample; the user must determine optimum time experimentally for their sample types.

10.2 *Addition of Carrier:*

10.2.1 Weigh an appropriate amount of uranium oxide into a mixing vial and add the amount of carrier chosen (see Table 1).

10.2.2 Mix on a mixer for at least 1 min.

NOTE 5—Exact time will depend on efficiency of mixing action of the mixer chosen, the amount of sample and carrier used. The user must determine this experimentally for their sample type (see Table 1).

10.3 Charge each electrode with the amount of uranium oxide and carrier determined to be optimum. For each sample charge duplicate electrodes.

10.4 Grip each electrode with forceps and pack the charge by tapping on a solid surface.

10.5 Further compress and vent the charge with the venting tool shortly before arcing the samples. Wipe the venting tool with tissue between different samples.

NOTE 6—Venting is dependent on the carrier used and may not be required. A combination tamping/venting tool is permissible.

10.6 Arc the electrodes at the conditions determined optimum for the instrument in use.

10.7 In using a direct reading spectrograph, the sample emission spectra will be acquired as each electrode is arced.

10.8 Determine each impurity by comparing the lines for each element using the calibration curves established in 9.2.

11. Precision and Bias

11.1 Within the different stages of the nuclear fuel cycle many challenges lead to the inability to perform interlaboratory studies for precision and bias. These challenges may include variability of matrices of material tested, lack of suitable reference or calibration materials, limited laboratories performing testing, shipment of materials to be tested, and regulatory constraints. Because of these challenges each laboratory utilizing these test methods should develop their own precision and bias as part of their quality assurance program.

11.2 There are not certified materials for the determination of all elements that may be analyzed by this method. Nor would all the elements be expected in these materials.

TABLE 3 Typical Data Using AgCl/AgF Carrier for NBL CRM 124-2 (Data from Lab A)

Element	# Points	Value (ppm)	Average (ppm)	Absolute Difference (Avg-Value)	% Relative Difference	Std. Dev	% Rel Std. Dev
Al	30	105	103	-2	2	15	15
Cr	30	52	61	9	17	12	20
Cu	30	25	27	2	8	6	22
Fe	30	110	116	6	5	27	23
Mo	30	50	53	3	6	11	21
Ni	30	102	113	11	11	18	16
Si	30	102	83	-19	19	13	16

TABLE 4 Typical Data Using AgCl/LiF Carrier Synthetic Standards (U_3O_8 Spiked with Traceable Impurities) (Data from Lab B)

Element	# Points	Value (ppm)	Average (ppm)	Absolute Difference (Avg-Value)	% Relative Difference	Std. Dev	% Rel Std. Dev
Al	143	140	138	-2	1	12	9
Ca	139	60	60	0	0	10	16
Cr	142	60	58	-2	3	10	17
Fe	135	220	239	19	9	39	16
Ni	130	120	119	-1	1	14	12
Si	110	100	91	-9	9	9	10

However, there are standards available from the New Brunswick Laboratory certified for many of the elements of interest. Data using these standards and other available standards are listed in Tables 3 and 4. Each user must determine the proper quality control and assurance measures for their application and the carrier/sample combination used. Relative standard deviations are calculated relative to average value. For different elements the relative standard deviation was found to be between 9 % and 23 %. These sigma are an indication of precision. The relative difference from the certified values vary from 0 % to 20 %. These numbers are an indication of the potential bias. This data was collected over a 12-month period in two different laboratories by several different technicians and is typical of data for these techniques. For other elements not in these standards, the precision is normally −50 % to +100 % of the determined value. The user can expect precision and bias values for some elements (those with weak emission lines or highly refractory elements) to be greater than the values listed in Tables 3 and 4. Those values must be determined by the user.

11.3 The data acquired in generation of results in Tables 3 and 4 was generated using DC arc systems using photographic plates or film whose use was described in earlier versions of this method. The use of CCD, CID, or CMOS cameras currently mentioned in 6.1 may provide differing performance which the user should characterize for their specific application. Such instruments may allow for time resolved acquisition of characteristic wavelength intensity and the use of wavelengths below 230 nm that may allow for additional analytes.

12. Keywords

12.1 carrier distillation; dc-arc; emission spectroscopy; impurities in uranium

APPENDIX

(Nonmandatory Information)

X1. ALTERNATIVE CARRIER APPROACHES

X1.1 There are alternative carrier mixes beyond that mentioned in Table 1 and non-carrier approaches that a practitioner of the DC arc methodology may wish to be familiar with (**1-3**).[6]

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

REFERENCES

(1) Rehana, I., Zafar Iqbal, M., and Mohamad, D., "Studies on Vaporization Behaviour of Trace Level Elements in U_3O_8-Matrix by Carrier Distillation DC-Arc Technique," *Acta Chemica Hungarica*, Vol 127, (5), 1990, pp. 645–653.

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(3) Sastry, M. D., Bhide, M. K., Svaitri, K., Babu, Y., and Joshi, B. D., Spectrographic Determination of Traces of B, Be, Cd, Si, and Co in U_3O_8 by the d.c. Arc Cathode-Region Method," *Fresenius Z. Anal. Chem*, Vol 308, 1981, pp. 456–460.

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