



Designation: ~~C1111-04~~ Designation: C1111 - 10

Standard Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy¹

This standard is issued under the fixed designation C1111; 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 trace, minor, and major elements in waste streams by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) following an acid digestion of the sample. Waste streams from manufacturing processes of nuclear and non-nuclear materials can be analyzed. This test method is applicable to the determination of total metals. Results from this test method can be used to characterize waste received by treatment facilities and to formulate appropriate treatment recipes. The results are also usable in process control within waste treatment facilities.

1.2 This test method is applicable only to waste streams that contain radioactivity levels that do not require special personnel or environmental protection.

1.3 A list of the elements determined in waste streams and the corresponding lower reporting limit is found in Table 1.

1.4 This test method has been used successfully for treatment of a large variety of waste solutions and industrial process liquids. The composition of such samples is highly variable, both between waste stream types and within a single waste stream. As a result of this variability, a single acid digestion scheme may not be expected to succeed with all sample matrices. Certain elements may be recovered on a semi-quantitative basis, while most results will be highly quantitative.

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1.5 This test method should be used by analysts experienced in the use of ICP-AES, the interpretation of spectral and non-spectral interferences, and procedures for their correction.

1.6 No detailed operating instructions are provided because of differences among various makes and models of suitable ICP-AES instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

1.7 This test method contains notes that are explanatory and are not part of the mandatory requirements of the method.

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

1.9 This standard does not purport to address all of the safety problems, 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

C859 [Terminology Relating to Nuclear Materials](#)

C1109 [Practice for Analysis of Aqueous Leachates from Nuclear Waste Materials Using Inductively Coupled Plasma-Atomic Emission Spectroscopy](#)

C1234 [Practice for Preparation of Oils and Oily Waste Samples by High-Pressure, High-Temperature Digestion for Trace Element Determinations](#) ~~D1129 Terminology Relating to Water~~

D1193 [Specification for Reagent Water](#)

E135 [Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

E177 [Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)

2.2 *ISO and European Standards:*³

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. Current edition approved Jan. Oct. 1, 2004-2010. Published February-2004-October 2010. Originally approved in 1988. Last previous edition approved in 1998-2004 as E111-98-C1111-04. DOI: 10.1520/C1111-104.

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 Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

TABLE 1 Analytical Wavelengths and Applicable Concentration Ranges^A

Element	Lower Limit, ^B mg/L	Upper Limit, mg/L	Wavelength, nm
Aluminum	0.02	5000	308.22, 237.01
Barium	0.001	100	493.41
Beryllium	0.0003	100	313.04
Boron	0.004	200	249.68
Cadmium	0.003	200	226.50
Calcium	0.004	1000	317.93, 393.37
Chromium	0.01	5000	267.72, 298.92
Cobalt	0.005	150	228.62
Copper	0.004	150	324.75
Iron	0.004	5000	271.44, 259.94
Lead	0.05	200	220.35
Lithium	0.004	150	670.78
Magnesium	0.0005	5000	293.65, 279.55
Manganese	0.001	150	257.61
Nickel	0.01	5000	231.60, 341.48
Phosphorus	0.2	250	178.29
Potassium	0.6	1000	766.49
Silver	0.006	150	328.07
Sodium	0.02	200	330.29, 588.99
Strontium	0.0004	100	421.55
Thorium	0.2	250	283.73
Titanium	0.003	150	334.94
Uranium	0.03	1000	409.01
Vanadium	0.005	250	292.40
Zinc	0.001	250	213.86
Zirconium	0.005	250	339.20

^A The estimated upper and lower concentration limits are to be used only as a general guide. These values are instrument and sample dependent, and as the sample matrix varies, these concentrations may be expected to vary also.

^B These limits obtained using a Jarrell-Ash ICAP-9000 ICP Spectrometer.

ISO 1042 Laboratory Glassware—One-mark Volumetric Flasks

ISO 3585 Borosilicate Glass 3.3—Properties

ISO 8655 Piston-Operated Volumetric Instruments (6 parts)

2.3 US EPA Standard:

Method 6010, Inductively Coupled Plasma Method, SW-846, Test Methods for Evaluating Solid Waste⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129C859, Terminology E135, and Test Method, and Practice C1109.

4. Summary of Test Method

4.1 Elements are determined, either sequentially or simultaneously, by inductively coupled plasma-atomic emission spectroscopy (Method 6010, SW-846). If the sample is a clear acidified solution, the elements are determined with no further pretreatment. If the sample contains undissolved solids, the elements are determined using an aliquot of the thoroughly mixed sample after a nitric acid digestion:

4.1 The general principles of emission spectrometric analysis are given in Ref. (1).⁵ In this test method, elements are determined, either sequentially or simultaneously, by ICP-AES (Method 6010, SW-846).

4.2 If the sample is a clear acidified solution, the elements are determined with no further pretreatment. If the sample contains undissolved solids, the elements are determined using an aliquot of the thoroughly mixed sample after a nitric acid digestion.

5. Significance and Use

5.1 This test method is useful for the determination of concentrations of metals in many waste streams from various nuclear and non-nuclear manufacturing processes. The test method is useful for characterizing liquid wastes and liquid wastes containing undissolved solids prior to treatment, storage, or stabilization. It has the capability for the simultaneous determination of up to 26 elements.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

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.

⁴ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

⁵ The boldface numbers in parentheses refer to a list of references at the end of this standard.

5.2 The applicable concentration ranges of the elements analyzed by this procedure are listed in Table 1.

6. Interferences

6. Interferences in ICP-AES are primarily spectral and can be compensated for in the following ways:

6.1 *Spectral interferences* in ICP-AES, and ways to compensate for them, include the following:

6.1.1 *Inter-element Interferences* —Inter-element interferences are characterized by spectral overlap of one element line over another. This interference can be compensated for by correction of the raw data, which requires measurement of the interfering element at the wavelength of interest. Table 2 lists some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for indicating potential spectral interferences. Various analytical systems may exhibit somewhat different levels of interferences. Therefore, the interference effects must be evaluated for each individual system.

6.1.2 *Molecular Band Interference* —Molecular band interference arising from overlap of molecular band spectra at the wavelength of interest can be eliminated by careful selection of wavelength.

6.1.3 *High Background*—High background effects from scattered light, etc., can be compensated for by background correction adjacent to the analyte line.

6.2 *Physical Interferences*—Physical interferences are effects associated with nebulization and transport processes in samples with either high solids or acid concentrations. These effects are reduced by a tenfold dilution of the sample and the use of a peristaltic pump in conjunction with a high-solids nebulizer. *Non-Spectral Interferences* —These include physical or chemical effects, such as high solids content or high acid concentration, that affect nebulization or the transport of the sample to the plasma and its vaporization, atomization, or excitation in the plasma. Effects due to high solids content or high acid concentration can be reduced by a tenfold dilution of the sample and the use of a peristaltic pump in conjunction with a high-solids nebulizer.

7. Apparatus

7.1 *Spectrometer*—An inductively coupled plasma emission spectrometer with a spectral bandpass of 0.05 nm or less is required. The spectrometer may be of the simultaneous multielement or sequential scanning type. The spectrometer may be of the air path, inert gas path, or vacuum type, with spectral lines selected appropriately for use with the specific instrument. Either an analog or digital readout system may be used.

7.1 Ordinary laboratory apparatus are not listed, but are assumed to be present.

7.2 *Glassware*, volumetric flasks complying with the requirements of ISO 1042, made of borosilicate glass complying with the

TABLE 2 Analyte Concentration Equivalents Arising from Interferents at the 1000 mg/L Level

Analyte	Wave-lengths, nm	Interferent, mg/L									
		Aluminum	Chromium	Copper	Iron	Nickel	Antimony	Silicon	Tin	Uranium	Vanadium
Aluminum	308.22							0.0020		0.0044	0.0199
Aluminum	237.21		-0.0022		-0.0084					0.0350	
Barium	493.41										
Beryllium	313.04										0.0013
Boron	249.68				0.0015						
Cadmium	226.50				0.0002	-0.0004					
Calcium	317.93									-0.0018	
Calcium	393.37							0.0002			
Chromium	267.72									0.0025	0.0018
Chromium	298.92									0.0560	
Cobalt	228.62		0.0001							0.0001	
Copper	324.75										
Iron	259.94	0.0001				-0.0001				-0.0002	
Iron	271.44		0.0039			-0.0015				0.0220	
Lead	220.35	-0.0012	-0.0028		0.0002	0.0006				0.0016	
Lithium	670.78							0.0003			
Magnesium	279.55										
Magnesium	293.65		-0.0270		-0.1390					0.0350	
Manganese	257.61									0.0002	
Nickel	231.60				-0.0002			0.0003		0.0003	
Nickel	341.48								0.0001	0.0027	
Phosphorus	178.29	0.0002			-0.0079	0.0120	0.0004			0.0044	
Potassium	766.49		0.0010							-0.0005	0.0014
Silver	328.07									0.0003	
Sodium	330.29	0.0035	-0.0220		-0.0145					-0.1580	
Sodium	588.99							0.0006		0.0002	
Strontium	421.55								0.0017		
Thorium	283.73		0.0007		0.0005	0.0049				0.0500	
Titanium	334.94		0.0003								
Vanadium	292.40		-0.0029								-0.0014
Zinc	213.85			0.0034	0.0001	0.0038					
Zirconium	339.20				-0.0003	-0.0002				-0.0005	