



Designation: C1111 – 10

## Standard Test Method for Determining Elements in Waste Streams by Inductively Coupled Plasma-Atomic Emission Spectroscopy<sup>1</sup>

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 ( $\epsilon$ ) 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.

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:<sup>2</sup>

- 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
- 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:<sup>3</sup>

- ISO 1042 Laboratory Glassware—One-mark Volumetric Flasks
- ISO 3585 Borosilicate Glass 3.3—Properties
- ISO 8655 Piston-Operated Volumetric Instruments (6 parts)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

**TABLE 1 Analytical Wavelengths and Applicable Concentration Ranges<sup>A</sup>**

Element	Lower Limit, <sup>B</sup> 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

<sup>A</sup> 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.

<sup>B</sup> These limits obtained using a Jarrell-Ash ICAP-9000 ICP Spectrometer.

### 2.3 US EPA Standard:

**Method 6010, Inductively Coupled Plasma Method, SW-846, Test Methods for Evaluating Solid Waste<sup>4</sup>**

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology **C859**, Terminology **E135**, and Practice **C1109**.

## 4. Summary of Test Method

4.1 The general principles of emission spectrometric analysis are given in Ref. **(1)**.<sup>5</sup> 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.

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

## 6. Interferences

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

6.1.1 *Interelement Interferences*—Interelement 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.

<sup>4</sup> 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>.

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

**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									
Iron	271.44		0.0039			-0.0001				-0.0002	
Iron	271.44					-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		0.0017	0.0002	
Strontium	421.55										
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

6.2 *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 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 requirements of ISO 3585. Glassware should be cleaned before use by soaking in nitric acid and then rinsing thoroughly with water.

7.3 *Filters*, inert membrane, having pore size of 2.5  $\mu\text{m}$ .

7.4 *Piston-operated Volumetric Pipettors and Dispensers*, complying with the requirements of ISO 8655, for pipetting and dispensing of solutions, acids, and so forth.

7.5 *Bottles*, tetrafluoroethylene or polyethylene, for storage of calibration and check solutions.

7.6 *Disposable Gloves*, impermeable, for protection from corrosive substances. Polyvinyl chloride (PVC) gloves are suitable.

7.7 *Inductively Coupled Plasma – Atomic Emission Spectrometer*, computer controlled, with a spectral bandpass of 0.05 nm or less.

NOTE 1—A bandpass of 0.05 nm or less is required to provide the necessary spectral resolution.

NOTE 2—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.

NOTE 3—An autosampler having a flowing rinse is recommended.

## 8. Reagents

8.1 *Purity of Reagents*—Chemicals used in the preparation of the standards must be of ultrahigh purity grade. Chemicals used in the preparation of the samples shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,<sup>6</sup> where such specifications are available.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Specification D1193, Type I, or water exceeding these specifications.

<sup>6</sup> *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.