

**Designation:** D 4190 - 03

# Standard Test Method for Elements in Water by Direct-Current Plasma Atomic Emission Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation D 4190; 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.

This standard has been approved for use by agencies of the Department of Defense.

# 1. Scope\*

- 1.1 This test method covers the determination of dissolved and total recoverable elements in water, which includes drinking water, lake water, river water, sea water, snow, and Type II reagent water by direct current plasma atomic emission spectroscopy (DCP).
- 1.2 The information on precision and bias may not apply to other waters.
- 1.3 This test method is applicable to the 15 elements listed in Annex A1 (Table A1.1) and covers the ranges in Table 1.
- 1.4 This test method is not applicable to brines unless the sample matrix can be matched or the sample can be diluted by a factor of 200 up to 500 and still maintain the analyte concentration above the detection limit.
- 1.5 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:
- D 1066 Practice for Sampling Steam<sup>2</sup>
- D 1129 Terminology Relating to Water<sup>3</sup>
- D 1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits<sup>3</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup>
- D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents<sup>2</sup>
- D 5810 Guide for Spiking into Aqueous Solutions<sup>2</sup>

**TABLE 1 Solutions for Analysis** 

17.522 1 Columbia for Amaryolo			
Element P	Solutions for Analysis, µg/L		
Liement F	1	2	3
Al	50	100	190
Be	50	500	1000
В	50	500	1000
Cd	1000	50	500
Cr	500	1000	50
Co	50	500	1000
Cu	1000	50	500
Fe	500	1000	50
Pb	500	200	1000
Mn	800	50	300
Hg	500	1000	200
Ni	50	300	800
Sr	600	50	300
V	1000	50	400
Zn	500	1000	50

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis<sup>4</sup>

E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis<sup>3</sup>

## 3. Terminology

- 3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.
  - 3.2 Definitions of Terms Specific to This Standard:
  - 3.2.1 *LCS*, *n*—laboratory control standard.
- 3.2.2 *total recoverable element*, *n*—a term relating to forms of each element that are determinable by the digestion method that is included in this test method.

## 4. Summary of Test Method

- 4.1 Elements are determined, either sequentially or simultaneously, by d-c plasma atomic emission spectroscopy.
- 4.2 Matrix enhancement or suppression of the emission signal can be minimized by the addition of 2000 mg/L of lithium ion to all standards, samples, and blanks.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Aug. 10, 2003. Published September 2003. Originally approved in 1982. Last previous edition approved in 1999 as D 4190 – 99.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol. 3.05.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 11.02.

- 4.3 Dissolved elements are determined by atomizing a filtered and acidified sample directly with no pretreatment.
- 4.4 If the sample is clear, total recoverable elements are determined in the same manner as dissolved elements except that sample is unfiltered and acidified.
- 4.5 If there are large particles (non-colloidal) the total recoverable elements are determined on a portion of the sample after a hydrochloric-nitric acid digestion (12.2-12.5). The same digestion procedure is used to determine all total recoverable elements in this test method.

Note 1—The volatility of mercury<sup>5,6</sup> compounds, especially the chlorides, makes it necessary to use considerable care in digesting samples containing these elements. The samples must not be boiled unless provision is made to prevent loss by volatilization.

## 5. Significance and Use

5.1 This test method is useful for the determination of element concentrations in many natural waters. It has the capability for the simultaneous determination of up to 15 separate elements. High analysis sensitivity can be achieved for some elements, such as boron and vanadium.

#### 6. Interferences

- 6.1 For commonly occurring matrix elements the following spectral interferences have been observed:
- 6.1.1 Calcium, magnesium, and boron interfere with lead at 405.78 nm.
  - 6.1.2 Calcium interferes with chromium at 425.43 nm.
  - 6.1.3 Magnesium interferes with cadmium at 214.44 nm.
  - 6.1.4 Iron interferes with cobalt at 345.35 and 240.73 nm.
  - 6.1.5 Cobalt interferes with nickel at 341.48 nm.
- Note 2—The exact magnitude of these interferences has not been determined since it depends on the concentration of the calibration standards used and the sample matrix.
- 6.2 Some additional possible interferences are listed in Annex A2 (Table A2.1) so that the analyst may be aware of and test for them.

# 7. Apparatus

7.1 See the manufacturer's instruction manual for installation and operation of DCP spectrometers, refer to Guide E 1097 for information on DCP spectrometers.

## 8. Reagents

8.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society<sup>7</sup> where such specifications are available. Other grades may be

<sup>5</sup> Standard Methods of Chemical Analysis, Editor, N. H. Furman, Vol 1, Sixth Edition, pp. 107 and 657.

<sup>6</sup> Smith, G. F., *The Wet Chemical Oxidation of Organic Compositions*, The G. Frederick Smith Chemical Co., 1965.

<sup>7</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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

- 8.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type I of Specification D 1193. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without lessening the bias and precision of the determination. Type II water was specified at the time of round robin testing of this test method.
- 8.3 *Stock Solutions*—Preparation of stock solutions for each element is listed in Annex A3 (Table A3.1) or use commercially available, ICP Grade, stock standards.
- 8.4 High Purity Hydrochloric Acid, (HCl), (sp gr 1.19), concentrated hydrochloric acid.
- 8.5 Hydrochloric Acid, (1 + 1)—Add one volume of HCl (sp gr 1.19) to one volume of water.
  - 8.6 Lithium Carbonate, ultrapure.
- 8.7 Lithium Solution (40 000 mg/L)—Dissolve 213 g of ultrapure lithium carbonate in a minimum amount of HCl (sp gr 1.19) and dilute to 1 L with water.
- 8.8 Concentrated Nitric Acid, (HNO<sub>3</sub>), (sp gr 1.42)—Highpurity acid can be prepared by distillation of concentrated nitric acid from a sub-boiling quartz still or it can be commercially purchased.
- 8.9 Dilute Nitric Acid, (1+1)—Add one volume of HNO<sub>3</sub> (sp. gr. 1.42) to one volume of water.
- 8.10 Dilute Nitric Acid, (1 + 499)—Add one volume of HNO<sub>3</sub>(sp gr 1.42) to 499 volumes of water.

Note 3—If a high reagent blank is obtained on either  $\mathrm{HNO_3}$  or  $\mathrm{HCl}$ , distill the acid or use high purity acid. When  $\mathrm{HCl}$  is distilled, an azeotropic mixture is obtained (approximately 6 N  $\mathrm{HCl}$ ); therefore, whenever concentrated  $\mathrm{HCl}$  is specified in the preparation of a reagent or in the procedure, use double the amount if distilled acid is used.

# 9. Precautions

- 9.1 Emission intensities are affected by changing viscosity so it is important to control the viscosity of blanks, standards, and samples within reasonable limits. Reagent water standards should not be used to analyze oil field brines. Alternatively, matrix matching or the method of additions can be used.
- 9.2 Organic solvents, such as alcohol, acetone, and methyl ethyl ketone have been observed to enhance emission intensity. This enhancement effect must be compensated for when organic solvents are known to be present. Alternatively, matrix matching or the method of additions can be used.

# 10. Sampling

- 10.1 Collect the samples in accordance with the applicable standards, Practice D 1066, Specification D 1192, or Practices D 3370.
- 10.2 Preserve the samples by immediately adding high purity nitric acid to adjust the pH to two at the time of collection. Normally 2 mL of  $\rm HNO_3$  is required per liter of sample. If only dissolved elements are to be determined, (Note 4) filter the sample through a 0.45  $\mu$ m membrane filter before acidification. The holding time for the sample may be calculated in accordance with Practice D 4841.

Note 4—Depending on the manufacturer, these filters have been found to be contaminated to various degrees with heavy metals. Care should be exercised in selecting a source of these filters. A good practice is to wash the filters with nitric acid and reagent water before filtering a sample.

#### 11. Calibration and Standardization

- 11.1 Prepare 100 mL of a blank and at least four standard solutions to bracket the expected concentration range of the samples to be analyzed by diluting 5.0 mL of lithium solution (see 8.7) and an appropriate volume of stock solution with  $\rm HNO_3(1+499)$ . Prepare the blank and standards each time the test is to be run.
- 11.2 Atomize the blank and standards and record the emission intensity or concentration. Atomize  $HNO_3$  (1 + 499) between each standard.
- 11.3 Using the instrument software verify that the instrument calibration is within user acceptable QC limits.

#### 12. Procedure

- 12.1 To determine dissolved elements, add 5.0 mL of lithium solution (see 8.7) to a 100.0 mL volumetric flask and bring to volume with the well-mixed acidified sample. Proceed with 12.6.
- 12.2 When determining total recoverable elements in solutions containing suspended matter or large particles (that is, noncolloidal), add 5.0 mL of HNO<sub>3</sub> (sp. gr. 1.42) and 5.0 mL of lithium solution to a 100.0-mL sample.
- Note 5—When digestion is necessary, subject the standards, sample, and blank to the same procedure.
  - 12.3 Add 5.0 mL of HCl (sp. gr. 1.19) to each sample.
- 12.4 Heat the samples in a covered beaker on a steam bath or hot plate until the volume has been reduced to 15 or 20 mL. Take care to see that the samples do not boil. Loss of sample could result from bumping or spattering.
- Note 6—For samples with high levels of dissolved solids, the amount of reduction in volume is left to the discretion of the analyst.
- 12.5 Cool and filter the samples, if necessary, through a fine ashless filter paper into 100.0 mL volumetric flasks. Wash the filter paper three times with water and adjust to volume.
- 12.6 Atomize each solution and record its emission intensity or concentration. Atomize  $HNO_3$  (1 + 499) between samples.

#### 13. Calculation

- 13.1 Calculate the concentration of elements in each sample, in mg/L, using the calibrations established in 11.3. Modern DCP instruments will provide the results in the calibrated concentration units.
- 13.2 Multiply the results for dissolved elements by the dilution factor of 1.05 to correct for the required addition of lithium solution (12.1).

#### 14. Precision and Bias 8

14.1 To facilitate handling and distribution for round robin testing, three concentrated solutions were prepared. These were acidified solutions of 15 elements.

- 14.2 The concentrated solutions, when diluted according to directions, yielded solutions for analysis with the composition as shown in Table 1. A total of eight laboratories and thirteen operators participated in this study.
- 14.2.1 Type II water was specified at the time of round robin testing of this test method.
- 14.3 *Precision*—The precision of this test method for the elements tested within their respective ranges of concentration given in Table 1 may be expressed as given in Table 2.
  - 14.4 Bias—See Table 3.
- 14.5 This section on precision and bias conforms to Practice D 2777 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of D 2777 98, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

# 15. Quality Control (QC)

- 15.1 In order to be certain that analytical values obtained using these test methods are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when analyzing each element.
  - 15.2 Calibration and Calibration Verification
- 15.2.1 Analyze at least three working standards containing concentrations of each element that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument. The calibration correlation coefficient shall be equal to or greater than 0.990. In addition to the initial calibration blank, a calibration blank shall be analyzed at the

**TABLE 2 Precision** 

Element	Reagent Water	Water of Choice
Al	$S_T = 0.093X - 0.301$	$S_T = 0.108X + 0.424$
	$S_O = 0.051X + 0.497$	$S_O = 0.044X + 3.18$
Be	$S_T = 0.066X + 0.354$	$S_T = 0.059X + 2.15$
	$S_O = 0.025X - 0.250$	$S_O = 0.042X 1.43$
В	$S_T = 0.045X + 9.34$	$S_T = 0.045X + 2.87$
	$S_O = 0.022X + 3.70$	$S_O = 0.021X + 5.12$
Cd	$S_T = 0.044X + 6.08$	$S_T = 0.066X + 2.99$
	$S_O = 0.025X + 4.96$	$S_O = 0.037X + 7.99$
Cr	$S_T = 0.060X + 2.13$	$S_T = 0.038X + 4.56$
	$S_O = 0.032X + 1.20$	$S_O = 0.027X + 3.86$
Co	$S_T = 0.062X + 4.59$	$S_T = 0.085X + 9.55$
	$S_O = 0.032X + 4.11$	$S_O = 0.040X + 3.99$
Cu	$S_T = 0.038X + 5.58$	$S_T = 0.049X + 2.75$
	$S_O = 0.031X + 0.956$	$S_O = 0.039X + 0.644$
Fe	$S_T = 0.051X + 14.3$	$S_T = 0.053X + 15.7$
	$S_O = 0.013X + 10.7$	$S_O = 0.034X + 12.2$
Pb	$S_T = 0.038X + 9.69$	$S_T = 0.037X + 18.3$
	$S_O = 0.027X + 5.36$	$S_O = 0.016X + 20.7$
Mn	$S_T = 0.058X + 2.35$	$S_T = 0.034X + 1.98$
	$S_O = 0.023X + 3.30$	$S_O = 0.018X + 3.79$
Hg	$S_T = 0.008X + 22.3$	$S_T = 0.009X + 28.0$
	$S_O = 0.003X + 14.7$	$S_O = 0.009X + 23.7$
Ni	$S_T = 0.078X + 5.47$	$S_T = 0.088X + 3.38$
	$S_O = 0.029X + 7.17$	$S_O = 0.039X5.54$
Sr	$S_T = 0.073X + 1.47$	$S_T = 0.024X + 3.56$
	$S_O = 0.034X + 1.72$	$S_O = 0.021X + 1.27$
V	$S_T = 0.053X + 1.74$	$S_T = 0.050X + 3.97$
	$S_O = 0.038X + 0.794$	$S_O = 0.048X - 0.156$
Zn	$S_T = 0.025X + 8.38$	$S_T = 0.022X + 10.9$
	$S_O = 0.011X + 6.67$	$S_O = 0.014X + 9.47$

where:

<sup>&</sup>lt;sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D19–1079.

 $S_r$  = overall prevision,  $\mu$ g/L,

 $S_O$  = single-operator precision,  $\mu$ g/L, and

X = concentration of element determined,  $\mu$ g/L.

**TABLE 3 Bias** 

Element	Amount Added, (µg/L)	Reagent Water, Type II, (% Bias)	Statistical Significance (95 % Confidence Level)	Water of Choice (% Bias)	Statistical Significance (95 % Confidence Level)
Al	50 100 190	+ 0.4 -5.3 -5.0	no yes yes	-9.2 -6.4 -6.7	yes yes yes
Be	50 500 1000	-0.2 -6.2 -7.2	no yes yes	-1.4 -5.8 -5.7	no yes yes
В	50 500 1000	-14.6 -6.6 -7.2	yes yes yes	-7.0 -5.3 -7.0	yes yes yes
Cd	50 500 1000	-0.6 -3.6 -3.5	no no no	0.0 -3.1 -3.3	no no yes
Cr	50 500 1000	-1.2 -4.2 -5.8	no yes yes	-4.5 -3.0 -3.8	yes yes yes
Co	50 500 1000	+ 2.6 -7.4 -9.9	no yes yes	+ 5.8 -8.1 -8.0	no yes yes
Cu	50 500 1000	+ 9.6 -0.6 -0.9	yes no no	+ 1.4 -1.4 -1.2	no no no
Fe	50 500 1000	+ 11.4 -4.0 -6.6	yes yes yes	+ 10.4 -5.8 -6.1	no yes yes
Pb	200 500 1000	-0.9 + 1.6 -0.9	no no no	-1.0 + 1.5 -1.0	no no no
Mn	50 300 800	+ 1.8 -5.3 -3.9	no yes yes	+ 1.6 -2.2 -2.5	no yes yes
Hg	200 500 1000	-9.8 -3.8 -2.7	yes yes no	+ 12.5 -1.6 -3.1	yes no yes
Ni	50 300 800	+ 1.0 -4.6 -6.4	no yes yes	+ 5.6 -3.5 -5.9	yes yes yes
Sr	50 300 600	+ 7.2 -5.4 -2.7	yes yes yes	+ 0.2 -4.1 -2.9	no yes yes
V	50 400 1000	-2.5 -6.4 -7.7	no yes yes	-8.8 -6.8 -6.8	yes yes yes
Zn	50 500	+ 2.6 -1.4	no no	+ 2.2 -0.1	no no

end of the batch run to ensure contamination was not a problem during the batch analysis.

15.2.2 Verify instrument calibration after standardization by analyzing a standard at the concentration of one of the calibration standards. The concentration of a mid-range standard should fall within  $\pm$  15% of the known concentration.

15.2.3 If calibration cannot be verified, recalibrate the instrument.

15.3 Initial Demonstration of Laboratory Capability

15.3.1 If a laboratory has not performed the test before, or if there has been a major change in the measurement system, for example, new analyst, new instrument, and so forth, a precision and bias study must be performed to demonstrate laboratory capability.

15.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a mid-range concentration of each element. The matrix and chemistry of the solution should be equivalent to the solution used in the collaborative study. Each replicate must be taken through the complete analytical test method including any sample preservation and pretreatment steps. The replicates may be interspersed with samples.

15.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 2. This study should be repeated until the recoveries are within the limits given in Table 2. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

15.4 Laboratory Control Sample (LCS)

15.4.1 To ensure that the test method is in control, analyze a LCS containing a known concentration of each element with each batch or ten samples. If large numbers of samples are analyzed in the batch, analyze the LCS after every ten samples. The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for a mid-range LCS shall fall within  $\pm$  15 % of the known concentration.

15.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all

the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

15.5 Method Blank

15.5.1 Analyze a reagent water test blank with each batch. The concentration of each element found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of each element is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

15.6 Matrix Spike (MS)

15.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each batch by spiking an aliquot of the sample with a known concentration of each element and taking it through the analytical method.

15.6.2 The spike concentration plus the background concentration of each element must not exceed the high calibration standard. The spike must produce a concentration in the spiked sample that is 2 to 5 times the analyte concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

15.6.3 Calculate the percent recovery of the spike (P) using the following formula:

 $P = 100 [A (V_s + V) - BV_s] / CV$  where:

A = analyte concentration (mg/L) in spiked sample;

B = analyte concentration (mg/L) in unspiked sample;

C = concentration (mg/L) of analyte in spiking solution;

 $V_s$  = volume (mL) of sample used; and

V = volume (mL) added with spike.

15.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte concentration, listed in Guide D 5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results

must be qualified with an indication that they do not fall within the performance criteria of the test method.

Note 7—Acceptable spike recoveries are dependent on the concentration of the component of interest. See Guide D 5810 for additional information.

## 15.7 Duplicate

- 15.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch. If the concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.
- 15.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an F test. Refer to 6.4.4 of Practice D 5847 for information on applying the F test.

15.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

15.8 Independent Reference Material (IRM)

15.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The concentration of the IRM should be in the concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

# 16. Keywords

16.1 direct-current plasma; DCP; elements; water

# **ANNEXES**

(Mandatory Information)

#### A1. ANALYTICAL WAVELENGTHS

A1.1 Table A1.1 shows analytical wavelengths (see 1.3).

**TABLE A1.1 Analytical Wavelengths** 

TABLE ATT Analytical Wavelengths			
Element	Wavelength, nm <sup>A</sup>	Range, μg/L	
Al	396.15	The range for the elements determined in	
Be	234.86	this study is given in Table 1. <sup>B</sup>	
В	249.68		
Cd	214.44		
Cr	425.43		
Co	345.35		
Cu	324.75		
Fe	371.99		
Pb	405.78		
Mn	279.48		
Hg	253.65		
Ni	305.08		
Sr	407.77		
V	437.92		
<i>7</i> n	213.86		

A Wavelengths other than those specified may be used if determined that they have adequate sensitivity and are linear over the working range. Alternative wavelengths should be checked for significant interferences.

<sup>&</sup>lt;sup>B</sup> It is possible to go lower by concentration of the sample or higher by dilution of the sample.



#### A2. POSSIBLE INTERFERENCES

A2.1 Table A2.1 additional possible interferences (see 6.2).

**TABLE A2.1 Some Possible Spectral Interferences** 

Element	Wavelengths, nm	Relative Intensity	Possible Interferences <sup>A</sup>
Al	236.76		Cu, Rb, Yb, Pd, Hf, V, Os, W, Er, Ir, Re
	396.15 <sup>B</sup>		Mo, U, Yb, Zr
Be	234.86		Re, Nb, Te, Ni, Os, Lu, Ta, Zr, Mo, W, Pt, Co, Nd
В	208.96	137	Zr, Pd, Nd
	249.68	456	Pd, Re, Sn, Hg, Ru
	249.77 <sup>B</sup>	1000	V, Ca, Ru, Fe, Sn, Hg, Mn, Ni, Fe
Zn	202.55	1000	In, Cu, Ir, U
	206.19	634	Zr, Ir, Ga, Pd, Sb, Ta, V
	213.86 <sup>B</sup>	131	Cu, As, Nb, Ir, Ni, Fe, Os
	334.50	186	U, Mo

<sup>&</sup>lt;sup>A</sup> L & R—When using the echelle grating spectrograph, some wavelengths may appear in two adjacent orders of reflection. "L" denotes the left side of the grating, "R" denotes the right side of the grating. It should be noted that the large majority of elements listed here as possible interferences have not been observed to cause problems in the solutions so far analyzed.

#### A3. STOCK SOLUTIONS

A3.1 Table A3.1 lists stock solutions (see 8.3).

TABLE A3.1 Preparation of Metal Stock Solutions<sup>A,B</sup>

Element (Compound)	Weight, g	Solvent
Al	1.000	HCI (1 + 1)
Ве	1.0000	HCI (sp gr 1.19)
H <sub>3</sub> BO <sub>3</sub>	5.720	Water <sup>B</sup>
CdO	1.142	HNO <sub>3</sub> (sp gr 1.42)
$K_2Cr_2O_7$	3.762	Water <sup>B</sup>
Co	1.000	HCI (sp gr 1.19)
CuO	1.252	HCI (sp gr 1.19)
Fe	1.000	HCI (sp gr 1.19)
Pb(NO <sub>3</sub> ) <sub>2</sub>	1.599	Water <sup>B</sup>
MnSO <sub>4</sub> ·H <sub>2</sub> O	3.076	Water <sup>B</sup>
HgCl <sub>2</sub>	1.354	Water <sup>B</sup>
Ni	1.000	$HNO_3(1 + 1)$
SrCO <sub>3</sub>	1.6849	HCI (sp gr 1.19)
$V_2O_5$	1.785	HCI (sp gr 1.19)
ZnO	1.245	HCI (sp gr 1.19)

A Metal stock solutions, 1.00 mL = 1.00 mg of metal. Dissolve the listed weights of each compound or metal in 20 mL of the specified solvent and dilute to 1 L. The metals and oxides require heat to increase rate of dissolution.

## **SUMMARY OF CHANGES**

Committee D19 has identified the location of selected changes to this standard since the last issue (D 4190 – 99) that may impact the use of this standard.

(1) Section 14.5 was added.

(2) Section 15 was revised.

<sup>&</sup>lt;sup>B</sup> Wavelengths used by the manufacturer and reported by users of DCP.

<sup>&</sup>lt;sup>8</sup> Where water is used as the solvent, acidify with 20 mL of HNO<sub>3</sub> (sp. gr. 1.42) and dilute to volume. See Section 8 for concentration of acids. Commercially available standards may also be used.

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