

**U.S. DEPARTMENT OF THE INTERIOR**

**U.S. GEOLOGICAL SURVEY**

**The Determination of Forty Four Elements in Aqueous Samples by  
Inductively Coupled Plasma – Mass Spectrometry**

by

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**Open-File Report 99-151**

This report is preliminary and has not been reviewed for conformity with U.S. Geological Survey editorial standards. Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

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## **Introduction**

This report describes the validation of an Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) technique for the determination of 44 elements in aqueous samples. This report is a supplement to Open-File Report 96-525, Analytical Methods Manual for the Mineral Resources Program, which was published in 1996. The format of this report is consistent with other published methods that have been approved by the quality assurance coordinator.

## **Principle**

In aqueous samples, 44 major, minor, and trace elements are determined using inductively coupled plasma-mass spectrometry (ICP-MS). The ICP-MS is calibrated using 4 commercially available multi-element standard solutions in conjunction with one USGS WRD standard reference sample. Samples must be filtered and acid-preserved prior to analyses, but no digestion is required for the determination of dissolved elements in aqueous samples.

## **Interferences**

ICP-MS interferences come from matrix effects, instrumental drift, and isobaric overlap of some elemental isotopes. Molecular ions formed in the plasma can also result in enhancement of measured ion intensity due to isobaric interferences (Montaser, 1998). Internal standards are added to compensate for matrix effects and instrumental drift. The isotopes measured are selected to minimize isobaric overlap from other elements and molecular species that might be present. For a few isotopes, isobaric overlap corrections are computed based on relative isotopic abundances of the elements involved, and oxide or double-charged ion intensities (Lichte and others, 1987).

## **Scope**

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability of ICP-MS determinations (USEPA SW-846 Method 6020A, 1998) are included in this work. In addition to the 23 EPA approved elements, an additional 21 elements are included in this protocol per the provisions of Method 6020A. The elements analyzed and their reporting limits are shown in Table 1.

Table 1. Lower limits of determination (LLD) for elemental analysis of water samples by inductively coupled plasma-mass spectrometry .

<u>Element</u>	<u>LLD</u>	<u>Element</u>	<u>LLD</u>
Ag, ug/L	3	Ni, ug/L	0.1
Al, ug/L	0.1	P, mg/L	0.01
As, ug/L	3	Pb, ug/L	0.05
Ba, ug/L	0.1	Rb, ug/L	0.01
Be, ug/L	0.05	S, mg/L	0.5
Ca, mg/L	0.05	Sb, ug/L	0.1
Cd, ug/L	0.02	Sc, ug/L	0.1
Ce, ug/L	0.01	Se, ug/L	5
Co, ug/L	0.02	Si, mg/L	0.2
Cr, ug/L	1	Sr, ug/L	0.5
Cs, ug/L	0.01	Ta, ug/L	0.02
Cu, ug/L	0.5	Th, ug/L	0.005
Fe, ug/L	50	Ti, ug/L	0.1
Ga, ug/L	0.02	Tl, ug/L	0.05
Ge, ug/L	0.02	U, ug/L	0.005
K, mg/L	0.03	V, ug/L	0.1
La, ug/L	0.01	W, ug/L	0.02
Li, ug/L	0.1	Y, ug/L	0.01
Mg, mg/L	0.01	Zn, ug/L	0.5
Mn, ug/L	0.01	Zr, ug/L	0.05
Mo, ug/L	0.2		
Na, mg/L	0.01		
Nb, ug/L	0.02		
Nd, ug/L	0.01		

### Apparatus

- Inductively Coupled Plasma Mass Spectrometer, Perkin-Elmer Elan 6000
- 13x100 mm disposable polypropylene test tubes with caps

### Reagents

- Deionized (DI) water
- Nitric acid (HNO<sub>3</sub>), conc ULTREX grade (70 %)
- 1% HNO<sub>3</sub>: Dilute 10 mL conc HNO<sub>3</sub> to 1000 mL with DI water

### Internal standards

- Prepare 2 L of a solution containing 500 ug/L  $^6\text{Li}$ , 20 ug/L Rh, and 10 ug/L Ir by performing serial dilutions of commercial aqueous standards using 1%  $\text{HNO}_3$ . This solution is mixed in a 1:1 ratio with the sample to be analyzed using a dual channel peristaltic pump equipped with a mixing manifold and coil.

### Calibration standards

- Standard 1 contains 0.200 mg/L of the following elements in 1%  $\text{HNO}_3$ : Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Rb, Se, Sr, Tl, U, V, Zn
- Standard 2 contains 1.00 mg/L of the following elements in 1%  $\text{HNO}_3$ : Ge, Mo, Nb, P, S, Si, Ta, Ti, W, Zr
- Standard 3 contains 0.100 mg/L of the following elements in 1%  $\text{HNO}_3$ : Ce, La, Nd, Sc, Y
- Standard 4 contains 1.00 mg/L of Fe in 1%  $\text{HNO}_3$ .
- Standard 5 is USGS WRD standard reference water sample T-143.

### Safety precautions

All laboratory personnel must wear safety glasses, a lab coat or apron, and gloves when working in the laboratory. All personnel must read the laboratory Chemical Hygiene Plan (CHP) and the Material Safety Data Sheets (MSDS) for each procedure.

### Procedure

Prior to analyzing samples, a dual detector calibration and auto-lens adjustment are performed daily according to manufacturer recommendations (Perkin-Elmer Corp., 1997). The instrument is then calibrated using the calibration solutions listed above, and the operating parameters shown in table 2. The calibration is checked using NIST SRM's 1640 and 1643d (NIST 1995, 1998).

**Table 2. Operating conditions for Perkin-Elmer Elan 6000 ICP-MS**

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Sweeps/Reading:	35
Readings/Replicate:	1
Number of Replicates:	1
Tuning File:	default.tun
Optimization File:	default.dac
QO Enabled:	Yes
Settling Time:	Normal

**Table 2. Operating conditions for Perkin-Elmer Elan 6000 ICP-MS -- Continued**

<u>Analyte</u>	<u>Mass</u>	<u>Scan Mode</u>	<u>MCA Channels</u>	<u>Dwell Time</u>	<u>Integration Time</u>
Ag	108.905	Peak Hopping	1	50.0 ms	1750 ms
Al	26.982	Peak Hopping	1	10.0 ms	350 ms
As	74.922	Peak Hopping	1	100.0 ms	3500 ms
Ba	134.906	Peak Hopping	1	30.0 ms	1050 ms
Be	9.012	Peak Hopping	1	50.0 ms	1750 ms
Ca	42.959	Peak Hopping	1	20.0 ms	700 ms
Cd	113.904	Peak Hopping	1	100.0 ms	3500 ms
Ce	139.905	Peak Hopping	1	10.0 ms	350 ms
Co	58.933	Peak Hopping	1	30.0 ms	1050 ms
Cr	51.941	Peak Hopping	1	10.0 ms	350 ms
Cs	132.905	Peak Hopping	1	10.0 ms	350 ms
Cu	64.928	Peak Hopping	1	20.0 ms	700 ms
Fe	53.940	Peak Hopping	1	10.0 ms	350 ms
Ga	70.925	Peak Hopping	1	10.0 ms	350 ms
Ge	73.922	Peak Hopping	1	10.0 ms	350 ms
K	38.964	Peak Hopping	1	10.0 ms	350 ms
La	138.906	Peak Hopping	1	10.0 ms	350 ms
Li	7.016	Peak Hopping	1	50.0 ms	1750 ms
Mg	24.986	Peak Hopping	1	10.0 ms	350 ms
Mn	54.938	Peak Hopping	1	10.0 ms	350 ms
Mo	97.906	Peak Hopping	1	20.0 ms	700 ms
Na	22.990	Peak Hopping	1	10.0 ms	350 ms
Nb	92.906	Peak Hopping	1	20.0 ms	700 ms
Nd	145.913	Peak Hopping	1	10.0 ms	350 ms
Ni	59.933	Peak Hopping	1	20.0 ms	700 ms
P	30.994	Peak Hopping	1	10.0 ms	350 ms
Pb	207.977	Peak Hopping	1	20.0 ms	700 ms
Rb	84.912	Peak Hopping	1	10.0 ms	350 ms
S	33.968	Peak Hopping	1	10.0 ms	350 ms
Sb	120.904	Peak Hopping	1	100.0 ms	3500 ms
Sc	44.956	Peak Hopping	1	10.0 ms	350 ms
Se	81.917	Peak Hopping	1	100.0 ms	3500 ms
Si	28.977	Peak Hopping	1	100.0 ms	3500 ms
Sr	87.906	Peak Hopping	1	10.0 ms	350 ms
Ta	180.948	Peak Hopping	1	10.0 ms	350 ms
Th	232.038	Peak Hopping	1	10.0 ms	350 ms
Ti	48.948	Peak Hopping	1	20.0 ms	700 ms
Tl	204.975	Peak Hopping	1	50.0 ms	1750 ms
U	238.050	Peak Hopping	1	10.0 ms	350 ms
V	50.944	Peak Hopping	1	10.0 ms	350 ms
W	183.951	Peak Hopping	1	10.0 ms	350 ms



**Table 2. Operating conditions for Perkin-Elmer Elan 6000 ICP-MS -- Continued**

<u>Analyte</u>	<u>Mass</u>	<u>Scan Mode</u>	<u>MCA Channels</u>	<u>Dwell Time</u>	<u>Integration Time</u>
Y	88.905	Peak Hopping	1	10.0 ms	350 ms
Zn	65.926	Peak Hopping	1	30.0 ms	1050 ms
Zr	89.904	Peak Hopping	1	10.0 ms	350 ms

**Signal Processing**

Detector Mode:	Dual
Measurement Units:	Counts/sec
AutoLens:	On
Spectral Peak Processing:	Average
Signal Profile Processing:	Average
Blank Subtraction:	After Internal Standard
Baseline Readings:	0
Smoothing:	Yes, Factor 5

**Calculations**

$$\text{Concentration (ppm)} = \frac{\text{Final volume (mL)} * \text{ICP-MS reading (ppm)}}{\text{Sample aliquot volume (mL)}}$$

**Correction Equations**

<u>Analyte</u>	<u>Mass</u>	<u>Corrections</u>
V	50.944	- 3.127 * (ClO53 - (0.113 * Cr52))
Fe	53.940	- 0.028226 * Cr52
Ge	73.922	- 0.116645 * Se77
As	74.922	- 3.087 * (Se77 - 0.825 * Se82)
Se	81.917	- 1.008696 * Kr83
Mo	97.906	- 0.110588 * Ru101
Cd	113.904	- 0.026826 * Sn118
Gd	159.927	- 0.093976 * Dy163
Dy	163.929	- 0.047917 * Er166
W	183.951	- 0.001242 * Os189

**Assignment of uncertainty**

The analytical results for selected reference materials are summarized in table 3.

**Table 3. Analytical performance summary for elements in aqueous samples by ICP-MS**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>
<b>Aluminum, Al (ug/L)</b>							
T-151	Surface water	2	8.5	1.0	--	--	--
T-147	Surface water	2	16.3	0.3	<b>14.0</b>	1.7	116
GWT-3	Ground water	3	32.5	0.4	<b>31.4</b>	1.2	104
T-153	Surface water	2	36.1	0.9	<b>35.0</b>	2.4	103
GWT-4	Ground water	2	39.7	0.4	<b>38.4</b>	1.1	103
<b>Antimony, Sb (ug/L)</b>							
GWT-3	Ground water	3	0.39	0.08	--	--	--
T-147	Surface water	2	10.7	0.1	<b>10.5</b>	1.3	102
GWT-4	Ground water	2	14.0	0.1	<b>13.5</b>	1.0	104
T-153	Surface water	2	27.3	0.4	<b>25.7</b>	1.3	106
T-151	Surface water	2	27.7	0.1	<b>26.8</b>	0.5	103
<b>Arsenic, As (ug/L)</b>							
T-151	Surface water	2	<3	--	--	--	--
T-153	Surface water	2	<3	--	--	--	--
GWT-3	Ground water	3	<3	--	<b>0.93</b>	--	--
T-147	Surface water	2	3.3	0.4	<b>2.4</b>	13	138
GWT-4	Ground water	2	13.2	0.1	<b>12.7</b>	0.5	104
<b>Barium, Ba (ug/L)</b>							
GWT-3	Ground water	3	27.5	0.4	<b>25.3</b>	1.3	109
T-151	Surface water	2	43.6	0.5	<b>40.7</b>	1.1	107
T-147	Surface water	2	77.4	2.2	<b>73.0</b>	2.8	106
GWT-4	Ground water	2	92.9	0.5	<b>88.5</b>	0.5	105
T-153	Surface water	2	196	2.1	<b>184</b>	1.1	106
<b>Beryllium, Be (ug/L)</b>							
T-153	Surface water	2	<0.05	--	--	--	--
GWT-4	Ground water	2	12.8	0	<b>12.1</b>	0	106
GWT-3	Ground water	3	16	0.2	<b>15.4</b>	1.3	104
T-147	Surface water	2	16.7	0.2	<b>16.0</b>	1.3	104
T-151	Surface water	2	26.5	0.1	<b>25.6</b>	0.5	103
<b>Cadmium, Cd (ug/L)</b>							
GWT-4	Ground water	2	13.3	0.2	<b>12.8</b>	1.6	104
T-147	Surface water	2	16.4	0.5	<b>15.9</b>	3.0	103
T-153	Surface water	2	16.8	0.3	<b>16.0</b>	1.7	105
T-151	Surface water	2	27.9	0.1	<b>26.8</b>	0.5	104
GWT-3	Ground water	3	47.0	0.2	<b>45.7</b>	0.4	103

**Table 3. Analytical performance summary for elements in aqueous samples by ICP-MS —Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>
<b>Calcium, Ca (mg/L)</b>							
T-153	Surface water	2	27.5	0.2	<b>27.5</b>	0.8	100
GWT-3	Ground water	3	36.8	0.4	<b>35.9</b>	1.0	102
T-151	Surface water	2	37.4	0	<b>37.9</b>	0	99
T-147	Surface water	2	40.9	1.0	<b>41.1</b>	2.6	99
GWT-4	Ground water	2	102	0.7	<b>104</b>	0.7	98
<b>Chromium, Cr (ug/L)</b>							
GWT-3	Ground water	3	1.7	0	<b>1.9</b>	0	89
T-147	Surface water	2	12.6	0.2	<b>12.8</b>	1.7	98
GWT-4	Ground water	2	13.9	0.3	<b>13.6</b>	2.0	102
T-153	Surface water	2	15.6	0.1	<b>14.9</b>	0.5	104
T-151	Surface water	2	30.7	0.3	<b>30.1</b>	0.9	102
<b>Cobalt, Co (ug/L)</b>							
GWT-3	Ground water	3	0.08	0.01	--	7.5	--
T-147	Surface water	2	0.16	0.01	--	4.6	--
GWT-4	Ground water	2	7.5	0.1	<b>7.5</b>	0.9	100
<b>Copper, Cu (ug/L)</b>							
GWT-3	Ground water	3	4.3	0.1	<b>4.2</b>	2.3	103
GWT-4	Ground water	2	10.7	0	<b>10.5</b>	0	102
T-147	Surface water	2	11.8	0.4	<b>11.4</b>	3.0	103
T-153	Surface water	2	24.7	0.2	<b>24.0</b>	0.9	103
T-151	Surface water	2	33.5	0	<b>33.0</b>	0	102
<b>Iron, Fe (ug/L)</b>							
T-147	Surface water	2	<50	--	<b>8.4</b>	--	--
T-151	Surface water	2	<50	--	<b>10.0</b>	--	--
GWT-4	Ground water	2	<50	--	<b>24.2</b>	--	--
GWT-3	Ground water	3	<50	--	<b>57.2</b>	--	--
T-153	Surface water	2	57	1.4	<b>75</b>	2.5	76
<b>Lead, Pb (ug/L)</b>							
GWT-4	Ground water	2	8.1	0.1	<b>8.17</b>	1.8	99
T-147	Surface water	2	13.8	0	<b>13.8</b>	0	100
T-151	Surface water	2	19.2	0.1	<b>19.8</b>	0.4	97
GWT-3	Ground water	3	31.4	0.5	<b>31.4</b>	1.5	100
T-153	Surface water	2	46.1	0.6	<b>46.2</b>	1.4	100
<b>Lithium, Li (ug/L)</b>							
GWT-4	Ground water	2	10.7	0.1	<b>10.4</b>	1.3	103
T-147	Surface water	2	17.8	0.2	<b>18.0</b>	1.2	99
T-151	Surface water	2	28	0.3	<b>27.6</b>	1.0	101
GWT-3	Ground water	3	34.7	0.3	<b>33.9</b>	0.9	102
T-153	Surface water	2	55.4	0.5	<b>53.4</b>	0.9	104

**Table 3. Analytical performance summary for elements in aqueous samples by ICP-MS —Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>
<b>Magnesium, Mg (mg/L)</b>							
T-147	Surface water	2	8.0	0.2	<b>8.2</b>	2.7	98
T-153	Surface water	2	8.7	0.2	<b>8.7</b>	1.9	100
GWT-3	Ground water	3	12.6	0.2	<b>12.5</b>	1.2	101
T-151	Surface water	2	16.9	0.4	<b>17.5</b>	2.1	96
GWT-4	Ground water	2	18.0	0.1	<b>18.6</b>	0.4	97
<b>Manganese, Mn ug/L)</b>							
GWT-3	Ground water	3	5.9	0.1	<b>5.6</b>	1.7	106
T-151	Surface water	2	13.9	0.1	<b>13.0</b>	0.5	107
T-147	Surface water	2	18.0	0.4	<b>17.2</b>	2.0	104
GWT-4	Ground water	2	22.8	0	<b>21.6</b>	0	106
T-153	Surface water	2	77.7	1.0	<b>74.5</b>	1.3	104
<b>Molybdenum, Mo (ug/L)</b>							
GWT-3	Ground water	3	8.3	1.5	<b>7.0</b>	18	118
T-147	Surface water	2	12.0	0.4	<b>11.8</b>	3.0	101
GWT-4	Ground water	2	13.8	0.4	<b>14.8</b>	2.6	93
T-151	Surface water	2	26.3	2.1	<b>29.6</b>	8.1	89
T-153	Surface water	2	145	0	<b>154</b>	0	94
<b>Nickel, Ni (ug/L)</b>							
T-151	Surface water	2	10.5	0.1	<b>10</b>	0.7	105
GWT-4	Ground water	2	11.7	0.14	<b>11</b>	1.2	106
T-147	Surface water	2	13.9	0.2	<b>13.6</b>	1.5	102
T-153	Surface water	2	32.7	0	<b>32.2</b>	0	102
GWT-3	Ground water	3	36.9	0.2	<b>36.6</b>	0.6	101
<b>Potassium, K (mg/L)</b>							
GWT-3	Ground water	3	0.99	0.01	<b>1.03</b>	1.0	96
T-153	Surface water	2	1.6	0	<b>1.6</b>	0	100
T-151	Surface water	2	1.9	0	<b>1.95</b>	0	97
GWT-4	Ground water	2	2.35	0.1	<b>2.4</b>	3.0	98
T-147	Surface water	2	3.45	0.1	<b>3.52</b>	2.1	98
<b>Selenium, Se (ug/L)</b>							
T-151	Surface water	2	<5	--	<b>1.8</b>	--	--
GWT-4	Ground water	2	8.55	0.4	<b>6.77</b>	4.1	126
T-153	Surface water	2	10.5	0.4	<b>9</b>	3.4	116
T-147	Surface water	2	13.2	0.4	<b>10.1</b>	2.7	130
GWT-3	Ground water	3	15.5	0.4	<b>12.4</b>	2.3	125

**Table 3. Analytical performance summary for elements in aqueous samples by ICP-MS —Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>
<b>Silicon, Si (mg/L)</b>							
T-151	Surface water	2	0.65	0.1	<b>0.66</b>	11	98
T-153	Surface water	2	2.75	0.1	<b>2.7</b>	2.6	102
GWT-4	Ground water	2	7.65	0.1	<b>7.62</b>	0.9	100
GWT-3	Ground water	3	11.2	0.1	<b>10.7</b>	0.9	105
T-147	Surface water	2	11.4	0.1	<b>11.2</b>	1.2	102
<b>Silver, Ag (ug/L)</b>							
GWT-3	Ground water	3	<3	--	--	--	--
GWT-4	Ground water	2	3.9	0.4	<b>6.2</b>	11	63
T-153	Surface water	2	4.5	0.1	<b>6.2</b>	0.8	71
T-147	Surface water	2	5.33	0.4	<b>7.6</b>	6.9	70
T-151	Surface water	2	9.4	0.4	<b>11.1</b>	4.6	84
<b>Sodium, Na (mg/L)</b>							
T-153	Surface water	2	28.4	0.1	<b>28.7</b>	0.5	99
GWT-4	Ground water	2	40.9	0.1	<b>41.4</b>	0.4	99
T-147	Surface water	2	52.3	1.4	<b>52.6</b>	2.7	99
T-151	Surface water	2	54.3	0.6	<b>55</b>	1.0	99
GWT-3	Ground water	3	142	1.1	<b>136</b>	0.8	105
<b>Strontium, Sr (ug/L)</b>							
T-153	Surface water	2	321	1.4	<b>311</b>	0.4	103
T-147	Surface water	2	321	12	<b>313</b>	3.8	102
T-151	Surface water	2	402	6.4	<b>387</b>	1.6	104
GWT-3	Ground water	3	592	5.9	<b>551</b>	1.0	108
GWT-4	Ground water	2	730	7.8	<b>711</b>	1.1	103
<b>Thallium, Tl (ug/L)</b>							
GWT-3	Ground water	3	0.6	0.1	--	22	--
T-147	Surface water	2	18.8	0.3	<b>20</b>	1.5	94
T-153	Surface water	2	19.6	0.1	<b>20.4</b>	0.7	96
T-151	Surface water	2	62.5	0.2	<b>62</b>	0.3	101
<b>Uranium, U (ug/L)</b>							
T-147	Surface water	2	3.2	0.01	<b>3.2</b>	0.2	100
GWT-4	Ground water	2	4.2	0.03	<b>4.3</b>	0.7	99
T-153	Surface water	2	7.3	0.1	<b>6.9</b>	1.2	106
T-151	Surface water	2	15.3	0.2	<b>15</b>	1.4	102
<b>Vanadium, V (ug/L)</b>							
GWT-3	Ground water	3	0.97	0.06	--	6.0	--
GWT-4	Ground water	2	14.4	0.4	<b>14.2</b>	2.5	101
T-147	Surface water	2	15.6	0.4	<b>15.2</b>	2.3	102
T-153	Surface water	2	20.1	0.4	<b>19</b>	2.1	106
T-151	Surface water	2	59.9	0.7	<b>59</b>	1.2	102

**Table 3. Analytical performance summary for elements in aqueous samples by ICP-MS —Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>
<b>Zinc, Zn (ug/L)</b>							
T-151	Surface water	2	10.7	0.07	<b>6.6</b>	0.7	162
T-147	Surface water	2	16.9	0.1	<b>14</b>	0.8	121
GWT-4	Ground water	2	51.9	0.4	<b>51</b>	0.7	102
T-153	Surface water	2	75.8	0.9	<b>72.6</b>	1.1	104
GWT-3	Ground water	3	233	3.2	<b>227</b>	1.4	103

n = number of samples

s = standard deviation

pv = proposed value taken from published reference material compilations (Farrar 1997, 1998).

% RSD = percent relative standard deviation

%R = percent recovery

-- = no data available

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