Addendum to SRM Certificates
2709 San Joaquin Soil
2710 Montana Soil
2711 Montana Soil

Leachable Concentrations Using U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry and Inductively Coupled Plasma Atomic Emission Spectrometry

The certified concentrations of constituent elements in essentially all National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) are given as total concentrations. The certified concentrations are based on measurements obtained by two or more independent methods or techniques. The measurement methods require complete sample decomposition, or the sample may be analyzed nondestructively. Where complete sample decomposition is required, it can be accomplished by digestion with mixed acids, or by fusion. For mixed acid decomposition, hydrofluoric acid must be included in the acid mixture used to totally decompose siliceous materials such as soils and sediments.

For a number of environmental monitoring purposes, the concentrations of labile or extractable fractions of elements are more useful than total concentrations. Concentrations of labile or extractable fractions are generally determined using relatively mild leach conditions which are unlikely to totally decompose the sample. It should be noted that results obtained using the mild leach conditions are often erroneously depicted in reports as total concentrations. However, reported concentrations of labile or extractable fractions of elements are generally lower than total concentrations; recovery can be total if an element in a given sample is completely labile. Results are often presented as measured concentration in the leachate in comparison to the total or certified concentration. The recovery of an element as a percent of total concentration is a function of several factors such as the mode of occurrence in the sample, leach medium, leach time and temperature conditions, and pH of the sample-leach medium mixture. References 1-27 may be consulted for detailed discussions of these factors and their effect on leach results. Some of these references provide leach data for one or more reference materials.

In its monitoring programs the U.S. Environmental Protection Agency (EPA) has established a number of leach methods for the determination of labile or extractable elements. They include Methods 3015, 3050, and 3051. A number of cooperating laboratories using the variation to U.S. EPA Method 3050 for FAAS and ICP-AES measurements, have reported data for SRMs 2709, 2710, and 2711. This variation of the method uses hydrochloric acid in its final step which is different from Method 3050 for ICP-MS and HGA-AAS measurements. The data obtained are presented in Tables 1, 2, and 3 of this addendum. The names of the cooperating laboratories are listed in Table 4. Several laboratories provided replicate (3-6) analyses for each of the three soil SRMs. The number of results for a given element varied from only one to as many as nine, as indicated in the data presented in Tables 1-3. Because of the wide range of interlaboratory results for most elements, only the data range and median of the individual laboratory means are given. Ranges differ somewhat from those in Ref. 26, since this addendum is based on a larger data set than had been available previously.

For SRMs 2710 and 2711, seventeen laboratories provided data as part of contract work for the U.S. EPA. Each SRM was treated as a blind sample in one quarter of 1992. Since there was no within-laboratory replication of analysis in the design of the exercise, the 17-laboratory means of results were treated as single laboratory results from laboratories using replication, in establishing the median of the full data set. In a few cases, however, the contract laboratories mean was the only result available for a particular element (e.g., Sb in 2710). In others, the contract laboratories mean is also the median for the full leach data set (e.g., As in 2710). An asterisk identifies those cases where the contract laboratories means are given as the median value.

Please note none of the values in Tables 1-3 are certified, but are given as information on the performance of the three (3) soils when used to evaluate, or to provide quality control for Method 3050 followed by FAAS and ICP-AES measurements only. The data should not be used for any other purpose. **The certified values, provided as total concentrations, are the best estimate of the true concentrations.**

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Table 1

Leach Data from Cooperating Laboratories for Soil SRM 2709

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	2.0 - 3.1	2.6	5	35
Calcium	1.4 - 1.7	1.5	5	79
Iron	2.5 - 3.3	3.0	8	86
Magnesium	1.2 - 1.5	1.4	5	93
Phosphorus	0.05 - 0.07	0.07	3	100
Potassium	0.26 - 0.37	0.32	5	16
Silicon	--- ---	<0.01	1	<1
Sodium	0.063 - 0.11	0.068	4	6
Titanium	0.03 - 0.04	0.038	3	11
	mg/kg			
Antimony	--- ---	<10	1	···
Arsenic	--- ---	<20	2	···
Barium	392 - 400	398	2	41
Cadmium	--- ---	< 1	5	···
Chromium	60 - 115	79	5	61
Cobalt	10 - 15	12	5	90
Copper	26 - 40	32	7	92
Lead	12 - 18	13	5	69
Manganese	360 - 600	470	7	87
Molybdenum	--- ---	< 2	2	···
Nickel	65 - 90	78	7	89
Selenium	nr nr	0.014	1	<1
Strontium	100 - 112	101	3	44
Vanadium	51 - 70	62	3	55
Zinc	87 - 120	100	7	94

† % Leach Recovery = $100 \times \left[\frac{\text{Median Value}}{\text{Certified/Info. Value}} \right]$

--- at or below the detection limit

··· no % Leach Recovery calculated

nr no range reported by the laboratory

Table 2

Leach Data from Cooperating Laboratories for Soil SRM 2710

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	1.2 - 2.6	1.8	6	28
Calcium	0.38 - 0.48	0.41	7	33
Iron	2.2 - 3.2	2.7	9	80
Magnesium	0.43 - 0.60	0.57	6	67
Phosphorus	0.106 - 0.11	0.11	2	100
Potassium	0.37 - 0.50	0.45	6	21
Silicon	--- ---	<0.01	1	<1
Sodium	0.049 - 0.062	0.054	5	5
Titanium	0.092 - 0.11	0.10	3	35
	mg/kg			
Antimony	3.4 - 12	7.9*	1*	21
Arsenic	490 - 600	590	3	94
Barium	300 - 400	360	3	51
Cadmium	13 - 26	20	8	92
Chromium	15 - 23	19	6	(49)
Cobalt	6.3 - 12	8.2	7	(82)
Copper	2400 - 3400	2700	8	92
Lead	4300 - 7000	5100	8	92
Manganese	6200 - 9000	7700	8	76
Mercury	27 - 37	32*	1*	98
Molybdenum	13 - 27	20	2	(100)
Nickel	8.8 - 15	10.1	8	71
Silver	24 - 30	28	3	79
Selenium	nr - nr	0.002	1	...
Strontium	94 - 110	100	3	(42)
Thallium	0.50 - 0.76	0.63*	1*	(48)
Vanadium	37 - 50	43	4	56
Zinc	5200 - 6900	5900	9	85

† % Leach Recovery = $100 \times \left\{ \frac{\text{Median Value}}{\text{Certified/Info. Value}} \right\}$

( ) indicates that information value was used

--- at or below the detection limit

... no % Leach Recovery could be calculated

nr no range reported by laboratory

* U.S. EPA contract laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 3

Leach Data from Cooperating Laboratories for Soil SRM 2711

Element	Range			Median	N	% Leach Recovery†
	Wt %					
Aluminum	1.2	-	2.3	1.8	5	28
Calcium	2.0	-	2.5	2.1	5	73
Iron	1.7	-	2.6	2.2	7	76
Magnesium	0.72	-	0.89	0.81	5	77
Phosphorus	0.06	-	0.09	0.088	3	100
Potassium	0.26	-	0.53	0.38	5	16
Silicon	---		---	<0.01	1	<1
Sodium	0.020	-	0.029	0.026	4	2.3
Titanium	0.039	-	0.048	0.042	2	14
	mg/kg					
Antimony	---		---	<10	1	···
Arsenic	88	-	110	90	3	86
Barium	170	-	260	200	2	28
Cadmium	32	-	46	40	6	96
Chromium	15	-	25	20	4	(43)
Cobalt	7	-	12	8.2	5	(82)
Copper	91	-	110	100	6	88
Lead	930	-	1500	1100	7	95
Manganese	400	-	620	490*	7	77
Molybdenum	---		---	<2	2	···
Nickel	14	-	20	16	7	78
Silver	2.5	-	5.5	4.0	1	86
Selenium	nr		nr	0.009	1	<1
Strontium	48	-	55	50	3	20
Vanadium	34	-	50	42	3	51
Zinc	290	-	340	310	7	89

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left(\frac{\text{Median Value}}{\text{Certified/Info. Value}} \right)$$

( ) indicates information value was used

--- at or below detection limit

··· no Leach Recovery could be calculated

nr no range reported by laboratory

* U.S. EPA contract laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 4

Leach Study for Cooperating Laboratories

SRMs 2709, 2710, and 2711

S.A. Wilson, U.S. Geological Survey; Lakewood, CO.

J. Lipinski and T. Plebanski, Polish Committee for Standardization, Measures and Quality Control; Warsaw, Poland.

E. Gorecka, Polish Geological Institute; Warsaw, Poland.

M. Paul, Research Institute of Vegetable Crops; Skierniewice, Poland.

I. Matuszczyk, Forest Research Institute; Warsaw, Poland.

Z. Jonca, Institute of Environmental Protection; Warsaw, Poland.

B. Ksiazek, Geological Enterprise; Warsaw, Poland.

I. Twardowska, Polish Academy of Sciences, Institute of Environmental Engineering; Zabrze, Poland.

SRMs 2710 and 2711

L. Butler and D. Hillman, U.S. Environmental Protection Agency; Las Vegas, NV and 17 contract laboratories.

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