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An Interlaboratory Study of Inductively Coupled Plasma Atomic Emission Spectroscopy Method 6010 and Digestion Method 3050

PROJECT SUMMARY
AN INTERLABORATORY STUDY OF INDUCTIVELY COUPLED PLASMA
ATOMIC EMISSION SPECTROSCOPY METHOD 6010
AND DIGESTION METHOD 3050

by

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NOTICE

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ABSTRACT

The design, execution, and results of an interlaboratory study of Method 6010, "Inductively Coupled Plasma Atomic Emission Spectroscopy," are described. This study examined the application of the method to the analysis of solid-waste materials for 23 elements. Part of the interlaboratory study included a study of Method 3050, "Acid Digestion of Sediments, Sludges and Soils," which is integral to Method 6010 when considering the analysis of certain solid wastes. The overall study was designed so that the variability of the two methods was separable. Method performance data, including precision and accuracy, are presented and discussed. A comparison of the inductively coupled plasma atomic emission and atomic absorption spectroscopic techniques is presented, as well as a comparison of results from two different types of inductively coupled plasma spectrometers. The limitations of the methods are described, and suggestions are given to improve the general application of Method 6010.

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INTRODUCTION

An interlaboratory study of solid wastes using the EPA analytical Method 6010 entitled "Inductively Coupled Plasma Atomic Emission Spectroscopy" (ICP-AES), which is included in the EPA methods publication SW-846, was performed with nine participating laboratories. This interlaboratory study concentrated on the application of Method 6010 for the determination of 23 elements in seven solid materials including dried sludges, sediments, and fly ash. The 23 target elements are: Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, K, Pb, Mg, Mn, Mo, Ni, Se, Ag, Na, Tl, V, and Zn. This study followed a single-laboratory evaluation that investigated the application of Method 6010 to a variety of aqueous and solid-waste samples. The different waste matrices studied in the single-laboratory evaluation required the utilization of several different digestion procedures. In contrast, this interlaboratory study examined Method 6010 for the analysis of solid wastes that were digested using a single digestion procedure.

Since the digestion of solid samples is necessary to apply Method 6010 for the analysis of wastes, a thorough study of

Method 6010 must also include digestion as a variable. Consequently, a parallel study of Method 3050 (Acid Digestion of Sediments, Sludges, and Soils) was included as an integral part of the interlaboratory study. The present study was designed to determine the performance of Method 6010 both independent of and together with the Method 3050 digestion procedure.

Seven solid materials, representative of solid wastes, were selected as the method evaluation materials. Three of the materials (river sediment, coal fly ash, and estuarine sediment) are Standard Reference Materials from the National Bureau of Standards, and one material (the mine tailing) is an EPA reference material. The other three solids (a contaminated soil and two industrial sludges) were obtained from the EPA. A detailed homogeneity study was performed by the coordinating laboratory before the solids were distributed to the participating laboratories. The results indicated that the solid samples were homogeneous.

Sixteen grams of these homogeneous solids were distributed to the laboratories to be digested by Method 3050, both unspiked and spiked. The spiking solutions provided to the laboratories contained 19 of the 23 target elements. They were designed to be added to the solids prior to digestion to bring the concentrations of the 19 elements in the laboratories' digests to minimum levels of about 100 times the corresponding "Estimated Instrumental Detection Limits" given in Method 6010. It was not necessary to spike Al, Ca, Fe, and Mg into the solids because of the high endogenous concentrations of these metals in the 7 solid samples. Having each laboratory spike portions of the solid samples with the spiking solutions prior to digestion assured that each laboratory used equally spiked aliquots of the solids. This procedure eliminated the need to create uniformly spiked solids for distribution. The resulting digests were analyzed by Method 6010.

In order to remove sample-preparation variability from measurement variability, bulk digests of the 7 solid samples were prepared by the coordinating laboratory for distribution to the participating laboratories. These bulk digests were spiked with the same spiking solutions that were used to spike the solid samples. Thus, the spiked bulk digests of the seven solid samples were very similar in composition to the spiked solids digests that were prepared by the laboratories. Therefore, data from the Method 6010 analyses of these spiked bulk digests could be compared to data from the spiked solids in order to estimate the variances due to the digestion and analysis procedures. In order to test the effects of high levels of V and Mo on the determination of the other analytes by Method 6010, the spiked bulk digest from the fly ash solid was also spiked to contain 0.1 percent of these interfering elements.

In addition to the solid samples and the spiked bulk digests, two QC solutions containing the target elements were provided to the participating laboratories for analysis with and without digestion. Because these solutions were carefully prepared and verified by the coordinating laboratory, the results could be used to estimate the accuracy of the Methods. Other solutions were provided to the participating laboratories to insure high ICP-AES data quality. These were initial calibration verification solutions and an interference check solution.

The results of this collaborative study yielded quantitative information on the precision and accuracy of Method 6010, independently and together with Method 3050. Data obtained on sequential and simultaneous ICP-AES instruments as well as by atomic absorption spectroscopy (AAS) were compared statistically, and the results are reported. The method of standard additions (MSA) is a conditional requirement of Method 6010, so its effect on data quality was investigated.

RESULTS AND DISCUSSION

This multilaboratory evaluation of Method 6010 demonstrates that the method, as described, is capable of achieving excellent accuracy and precision for the determination of the 23 elements in quality control (QC) solutions. These QC solutions contained the 23 elements at concentrations of approximately 100 times the instrumental detection limits, and the solutions were interference-free in that no interfering elements were present at high concentrations. Accuracy for the multilaboratory analyses of the QC solutions (when the mean values are expressed as a percentage of the target values) varies from 95 percent to 104 percent for the solutions analyzed without digestion and varies from 93 percent to 103 percent (silver excluded) for the solutions digested before being analyzed. Digestion of the QC solution containing silver resulted in a mean silver value that is only 53 percent of the target value whereas the mean silver value is 100 percent of the target value for the direct analyses of this QC solution. The percent RSD's for the elements range from 3.1 percent to 9.1 percent for the QC solutions that were analyzed by Method 6010 without digestion and from 2.6 percent to 13 percent (when silver is excluded) for the QC solutions that were analyzed after digestion by Method 3050. The median percent RSD's for the 2 sets of QC solutions are 6.5 and 6.7 percent, respectively. This precision is considered excellent for these solutions. Silver with a percent RSD of 52 is the lone outlier in the QC solution set that was digested before analysis.

The interlaboratory precision for Method 6010, with digestion eliminated as a variable, was determined for the 23 elements in the spiked bulk digests of six representative solid complex matrices, including river and estuarine sediments and industrial sludges (Table 1). The analyte concentrations in these spiked bulk digests were about 100 times the instrumental detection limits. The median percent RSD's for the 6 sediments across 23 elements range from 6.8 percent to 11 percent. Thus, the precision for the measurement of the target elements in these complex solutions is very good.

The seventh spiked bulk digest, from coal fly ash, was spiked with very high levels of molybdenum and vanadium (0.1 percent). The median percent RSD's for the determination of

TABLE 1. PERCENT RSD's FOR THE DETERMINATION OF THE 23 TARGET ELEMENTS IN THE SPIKED BULK DIGESTS

ELEMENTS	HAZARDOUS WASTE 1	RIVER SEDIMENT	FLY ASH	ESTUARINE SEDIMENT	INDUSTRIAL SLUDGE	ELECTRO-PLATING SLUDGE	MINE TAILING
Al	11	19	16	1.9	11	13	7.6
Sb	5.6	52	73	8.7	3.2	24	4.4
As	13	11	83	22	25	8.6	5.3
Be	5.8	5.8	57	4.8	6.4	9.9	8.5
Cd	11	6.6	5.7	7.6	3.1	9.8	12
Ca	8.8	9.4	5.6	5.3	8.5	7.0	7.9
Cr	6.2	5.5	36	7.6	5.8	7.8	39
Co	11	14	21	6.8	6.7	11	15
Cu	4.4	4.3	9.7	6.0	11	7.8	12
Fe	6.6	8.3	8.8	6.0	6.9	8.4	8.4
Pb	15	7.2	22	4.7	3.9	5.6	8.0
Mg	8.8	8.1	15	9.4	8.0	20	10
Mn	10	13	14	11	11	9.6	5.5
Mo	20	33	19	28	16	36	21
Ni	9.4	8.9	8.1	5.4	5.1	9.2	12
Se	7.5	13	16	6.2	13	13	19
Ag	44	23	17	46	47	19	27
Tl	19	13	22	29	30	20	29
V	12	58	7.5	7.3	5.5	11	18
Zn	9.1	6.7	7.6	15	10	2.5	16
Ba	11	10	8.7	6.4	8.0	20	11
Na	17	38	49	4.7	5.8	9.8	7.9
K	8.8	7.4	4.2	4.8	13	5.8	7.9
MEDIAN PERCENT RSD	10	10	16	6.8	8.0	11	11

the 23 elements in this spiked digest range from 4.2 percent to 83 percent with a median of 16 percent (Table 1). The 12 percent median RSD for fly ash digests without added Mo and V (Table 2) suggests that these two elements decreased the measurement precision of many of the target elements.

When Method 6010 and Method 3050 are applied in combination for the determination of the 23 elements in spiked solids, the apparent measurement precision decreases (Table 2) when compared to the corresponding spiked bulk digest. The median percent RSD's for the 7 solids across the 23 elements range from 11-17 percent. The spiked solid samples were spiked prior to digestion to assure that the concentrations of the analytes in the resulting digests were approximately 100 times greater than the instrumental detection limits. The accuracy of the ICP Method 6010 can be estimated for these complex matrices by comparing the average concentrations of the elements in the spiked bulk digests (as determined by Method 6010) to the corresponding concentrations which were determined by AAS by one of the participating laboratories. A null hypothesis approach that is based on the mean and on the corresponding standard deviation was used to determine if the ICP-AES and AAS values are significantly different at the 95 percent confidence level. The results indicate that only two

TABLE 2. PERCENT RSD's FOR THE DETERMINATION OF THE 23 TARGET ELEMENTS IN THE SPIKED SOLIDS

ELEMENTS	HAZARDOUS WASTE 1	RIVER SEDIMENT	FLY ASH	ESTUARINE SEDIMENT	INDUSTRIAL SLUDGE	ELECTRO-PLATING SLUDGE	MINE TAILING
Al	17	24	20	22	14	18	26
Sb	27	56	25	62	28	40	58
As	13	26	16	22	20	20	22
Be	16	13	7.6	11	18	7.0	16
Cd	13	8.4	9.3	14	19	18	20
Ca	7.3	9.0	12	10	12	14	12
Cr	7.9	22	9.7	7.1	18	12	26
Co	18	22	12	9.2	18	13	18
Cu	12	14	10	9.7	19	9.4	12
Fe	14	19	44	16	18	14	18
Pb	15	6.4	9.6	11	20	19	5.8
Mg	5.9	8.4	17	9.0	16	10	10
Mn	14	9.0	11	10	16	18	9.4
Mo	19	31	24	18	18	43	20
Ni	13	20	9.7	10	20	15	17
Se	13	9.4	9.8	10	15	18	12
Ag	19	7.6	50	34	30	27	50
Tl	19	28	34	28	18	43	44
V	18	19	12	10	18	39	24
Zn	14	12	11	13	20	8.2	20
Ba	8.4	9.8	7.2	14	16	30	7.2
Na	14	40	32	9.4	20	15	12
K	19	17	18	18	22	5.7	16
MEDIAN PERCENT RSD	14	17	12	11	18	18	18

out of 184 elemental measurements by the two methods are significantly different. The ICP-AES mean value was statistically higher than the AAS value for Ca in the digests of the Estuarine Sediment and the Mine Tailing Waste. In some cases where the ICP/AAS ratios are very different (less than 0.75 or greater than 1.25), the standard deviations in the ICP measurements are very high, and, therefore, the differences in the means are not significant. Overall, the agreement between ICP and AAS is excellent.

The median percent RSD's for the same 7 solids, unspiked, range from 17-27 percent (Table 3). This poorer precision when compared to the spiked solids results because over 50 percent of the reported concentration values are less than 100 times the average of the instrumental detection limits. In other words, as the concentrations approach the instrumental detection limits the precision decreases as indicated by the higher percent RSD values. Four elements among those with the highest median percent RSD's are antimony, selenium, silver and arsenic. For those elements that were present in the digests of the unspiked solids at concentrations 100 times greater than the IDL's (due to their occurrence in high concentrations in the unspiked solids), the precision is comparable to the precision for the spiked solid samples.

TABLE 3. PERCENT RSD'S FOR THE DETERMINATION OF THE 23 TARGET ELEMENTS IN THE UNSPIKED SOLIDS

ELEMENTS	HAZARDOUS WASTE 1	RIVER SEDIMENT	FLY ASH	ESTUARINE SEDIMENT	INDUSTRIAL SLUDGE	ELECTRO-PLATING SLUDGE	MINE TAILING
Al	19	32	19	23	15	23	17
Sb	38	78	--	--	47	68	57
As	53	48	32	18	83	44	28
Be	31	27	27	35	42	70	41
Cd	37	17	57	52	17	22	59
Ca	9.0	13	10	11	10	17	8.6
Cr	11	19	28	22	12	12	90
Co	24	60	23	12	21	46	30
Cu	10	9.4	16	17	17	12	20
Fe	13	24	52	10	14	12	18
Pb	8.0	12	33	37	16	17	17
Mg	6.0	11	20	10	18	14	9.2
Mn	8.6	17	24	10	18	21	11
Mo	30	42	20	58	56	49	26
Ni	14	25	34	21	16	20	40
Se	42	61	--	30	43	74	77
Ag	41	43	47	1.4	38	54	60
Tl	31	30	--	--	38	45	120
V	21	72	15	17	28	35	47
Zn	14	12	20	8.6	12	9.2	20
Ba	7.4	11	4.3	14	24	38	8.8
Na	66	52	34	9.1	16	17	13
K	23	34	20	17	32	9.6	24
MEDIAN PERCENT RSD	21	27	23	17	18	22	26

The Method 6010 variance and the Method 3050 variance can be calculated from the data base resulting from the analyses of the spiked bulk digests and the spiked solid samples (Table 4). A statistical analysis of the data shows that in general, the digestion procedure and the ICP-AES analytical procedure contribute about equally to the overall measurement uncertainty or precision (variance) for the determinations of the 23 target elements in digests of these 7 homogeneous solids.

The method of standard additions was required for less than 10 percent of the total analyses. Results by ICP-AES using the method of standard additions were compared with non-MSA data for the spiked bulk digest samples. The comparison of this limited data set (Table 5) indicates that on the average there is no consistent improvement in the data quality when the method of standard additions is used with Method 6010 for the analysis of the solid matrices that were used in this study.

A comparison between data obtained on simultaneous and sequential inductively coupled plasma spectrometers indicated that the concentration values were statistically indistinguishable.

TABLE 4. ESTIMATED PERCENTAGE CONTRIBUTIONS OF METHOD 6010 ICP VARIANCE AND METHOD 3050 DIGESTION VARIANCE TO TOTAL VARIANCE

Elements	6010 ICP	3050 Digestion
Al	41	59
Cd	26	74
Ca	50	50
Co	39	61
Cu	38	62
Fe	11	89
Pb	66	34
Mg	100	0
Mn	68	32
Mo	100	0
Ni	27	73
Se	89	11
Tl	63	37
Zn	55	45
Ba	37	63
K	22	76
Be	25	75
V	24	76
Sb	3	97
As	35	65
Cr	26	74
Na	25	75
Ag	100	0
Median:	46	55

TABLE 5. COMPARISON OF MSA AND NON-MSA RESULTS[a]

SPIKED BULK DIGESTS

SAMPLE NAME	ELEMENT	NON-MSA N	NON-MSA MEAN CONC.[b]	NON-MSA SD	MSA N	MSA MEAN CONC.[b]	MSA SD	%RATIO	SIG. DIF.[c]
HAZARDOUS WASTE	Cd	5	894	117	3	940	84	95	NO
HAZARDOUS WASTE	Tl	5	4410	788	3	4510	1130	98	NO
HAZARDOUS WASTE	Zn	5	4310	426	3	4560	250	95	NO
RIVER SEDIMENT	Tl	7	3160	2210	3	5050	675	63	NO
FLY ASH	Cd	5	754	422	3	897	219	84	NO
FLY ASH	Cr	5	1480	885	3	2390	1090	62	NO
FLY ASH	Pb	4	4100	634	4	6770	3300	61	NO
FLY ASH	Mn	4	1910	233	3	1750	304	109	NO
FLY ASH	Ni	3	1530	154	4	1350	500	113	NO
FLY ASH	Tl	4	5530	3730	3	1950	2470	284	NO
ESTUARINE SEDIMENT	Tl	5	3870	1290	3	3340	2850	116	NO
INDUSTRIAL SLUDGE	Tl	5	4470	872	3	4620	2230	97	NO
ELECTROPLATING SLUDGE	Tl	3	4600	740	4	5350	1120	86	NO
MINE TAILING	Cd	5	850	69	3	985	112	86	NO

[a] Only those elements that required the application of the MSA by three or more laboratories are included as statistically significant.

[b] Concentration for liquids in ug/L; concentration for solids in mg/kg.

[c] Result of a null hypothesis approach used to indicate whether MSA and non-MSA results are significantly different.

N - Number of cases.

% Ratio - non-MSA to MSA mean concentrations.

(continued)

TABLE 5. (continued)

UNSPIKED SOLIDS

SAMPLE NAME	ELEMENT	NON-MSA N	NON-MSA MEAN CONC.[b]	NON-MSA SD	MSA N	MSA MEAN CONC.[b]	MSA SD	%RATIO	SIG. DIF.[c]
HAZARDOUS WASTE	Be	4	0.8	0.1	3	0.7	0.2	93	NO
HAZARDOUS WASTE	Cr	6	95	8.4	3	111	10	86	YES
HAZARDOUS WASTE	Co	6	8.0	2.4	3	9.1	1.5	88	NO
HAZARDOUS WASTE (DUP.)	Ni	5	17	1.3	4	13	8.9	128	NO
RIVER SEDIMENT	Sb	6	325	266	3	169	246	192	NO
RIVER SEDIMENT	Cd	6	11	2.5	3	11	3.5	103	NO
RIVER SEDIMENT	Co	5	21	16	4	21	19	99	NO
RIVER SEDIMENT	Ni	6	44	20	3	27	7.0	161	NO
RIVER SEDIMENT (DUP.)	Cd	6	10	1.6	3	10	0.7	107	NO
RIVER SEDIMENT (DUP.)	Ni	6	39	13	3	38	19	105	NO
FLY ASH	Be	6	3.0	0.8	3	2.6	1.2	114	NO
MINE TAILING	Cd	4	2.3	1.6	3	1.9	1.1	122	NO
MINE TAILING	Zn	6	372	44	3	340	119	109	NO
MINE TAILING (DUP.)	Cd	4	2.4	1.6	3	1.5	0.8	158	NO
MINE TAILING (DUP.)	Co	6	7.3	2.5	3	8.8	3.1	83	NO
MINE TAILING (DUP.)	Ni	5	21	5.6	4	21	11	100	NO
MINE TAILING (DUP.)	Zn	6	365	43	3	345	122	106	NO
ELECTROPLATING SLUDGE	Cd	6	113	24	3	96	41	118	NO
ELECTROPLATING SLUDGE	Mn	6	226	31	3	254	126	89	NO
ELECTROPLATING SLUDGE (DUP.)	As	6	33	20	3	41	20	80	NO
ELECTROPLATING SLUDGE (DUP.)	Mo	5	14	11	3	21	7.3	68	NO
INDUSTRIAL SLUDGE	As	4	11	6.6	3	26	11	41	YES

(Continued)

TABLE 5. (concluded)

SPIKED SOLIDS									
		NON-MSA			MSA				
SAMPLE NAME	ELEMENT	N	MEAN CONC.[b]	SD	N	MEAN CONC.[b]	SD	%RATIO	SIG. DIF.[c]
HAZARDOUS WASTE	Co	6	45	8.2	3	30	2.2	149	YES
HAZARDOUS WASTE	Pb	6	340	104	3	238	14	143	NO
HAZARDOUS WASTE	Mo	6	39	20	3	29	2.8	134	NO
HAZARDOUS WASTE	Ni	6	57	10	3	37	2.9	152	YES
HAZARDOUS WASTE (DUP.)	Co	6	48	4.8	3	56	11	85	NO
HAZARDOUS WASTE (DUP.)	Pb	6	390	29	3	338	112	115	NO
HAZARDOUS WASTE (DUP.)	Ni	6	61	3.5	3	58	14	106	NO
ESTUARINE SEDIMENT	Cd	6	46	4.7	3	53	2.2	87	NO
ESTUARINE SEDIMENT	Mo	6	37	19	3	47	2.5	79	NO
ESTUARINE SEDIMENT	Ni	6	65	6.7	3	73	1.3	89	NO
ESTUARINE SEDIMENT	Tl	6	180	65	3	239	24	75	NO
ESTUARINE SEDIMENT (DUP.)	Ni	6	63	6.9	3	74	3.3	86	YES
MINE TAILING	Ni	6	64	7.9	3	60	15	108	NO
MINE TAILING (DUP.)	Ni	6	63	6.9	3	64	19	99	NO
ELECTROPLATING SLUDGE (DUP.)	Tl	6	160	46	3	304	104	53	YES

RECOMMENDATIONS

The experimental design used in this multilaboratory study has resulted in several excellent sets of multidimensional analytical data that deserve consideration beyond the intended scope of this report. Further analysis and interpretation of this data base is suggested.

The presence of high concentrations (0.1 percent) of added vanadium and molybdenum in the fly ash spiked bulk digest could account for the apparent decrease in the precision of Method 6010 for the determination of many of the 23 target elements in this matrix compared to the 6 other solid digests. The interfering effects in this matrix should be studied further.

The poor precision, accuracy, and spike recoveries for silver demonstrated in this study, should be noted in both Method 3050 and Method 6010. The possibility of precipitation in the nitric/hydrochloric acid digestion matrix as well as phototransformation should be discussed in Method 3050.

The poor spike recovery of antimony, observed in this study, should be noted in Method 3050. In particular, the possibility of the formation of oxide and oxo-chloride precipitates of antimony in the nitric/hydrochloric acid digestion matrix should be discussed.

The application of the method of standard additions (MSA), a conditional requirement of Method 6010, affects the economics, the turnaround time of analysis, the practicality of the Method, as well as the data quality. Although this report indicates that, on the average, MSA data were not consistently different from non-MSA data, the requirement for the application of the MSA should be investigated further.

When soil-containing matrices are being analyzed by Method 6010, the authors are of the opinion that the method of standard additions should not be required for those elements that are endogenous to soils in high concentrations. The high-concentration endogenous elements in soils include Al, Ca, Fe, Mg, K, and Na.

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The complete report, entitled "An Interlaboratory Study of Inductively Coupled Plasma Atomic Emission Spectroscopy Method 6010 and Digestion Method 3050," will be available only from:

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