UNITED STATES DEPARTMENT OF THE INTERIOR

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Results of the Third Western Task Force Round Robin Soil and Overburden Analysis Program

By

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INTRODUCTION

Regulatory guidelines for surface mining require the assessment of various physical and chemical properties of soil and overburden materials before mining, and minesoil and regraded spoil after mining. The list of properties, or parameters, that are required to evaluate suitability of materials is not consistent among regulatory agencies from all western states, or the federal Office of Surface Mining. In addition, the analytical requirements within each state may differ for soil before mining, redistributed soil after mining, and overburden material. The parameters requested in this Third Western Task Force Round Robin Soil and Overburden Analysis Program may, or may not, be required by each state for each type of material. Many of the requested parameters are, however, required by each state for all types of materials listed above.

Sixteen laboratories participated in this round robin analysis program.* Each laboratory was sent two samples of overburden material that was ground to pass a 60-mesh sieve and then homogenized. The participating laboratories are listed in the appendix (table A1). The parameters that were requested along with the procedures are listed in appendix (table A2).

The remainder of this report is a presentation of the data obtained from the third round robin. Any statistical tests applied to the data must be interpreted with caution because of the small number of samples (n of 16 or less), and because of the differences in methods used for each parameter by the participating laboratories.

REPORTED DATA

The values reported by each laboratory for each parameter are listed in tables 1-2 for the two sample sets. A value reported by a laboratory in different units than the suggested units for that parameter (table A2), was converted to the appropriate units. Converted values are identified on tables 1-2. The laboratories performing the analyses are coded to conceal the identity of the individual laboratories. Judgments on laboratory quality, based on comparisons of reported values, are inappropriate because many of the analyses were made utilizing different analytical techniques and no correct value can be assigned to an individual parameter.

Summaries of the reported values are given in tables 3-4. Only pH and carbon ranged by a factor of two or less for both sample sets. At the other extreme, the range in values reported for available nitrogen and acid potential differed by 100 times or more, and cation exchange capacity (cec), molybdenum, and neutralization potential ranged by a factor of 10 times or more for both sample types. The remainder of the parameters ranged between 2 times and 100 times, depending on the parameter and the sample type. The intermediate and large differences for many parameters suggest that inconsistencies in sample homogeneity, sample preparation, analytical methods, instrumental analysis, or some combination of these can produce results with deviations large enough to make some data unreliable for estimating a simple average value. The sources of the deviations could not be identified from the information provided by the participating laboratories.

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*A round robin analysis program is an informal, interlaboratory comparison of analytical precision based on analysis of uncertified sample splits.

Histograms showing the frequency distribution for the values reported by all laboratories for each parameter in each of the two sample sets are not presented as they were in the first round robin (Severson and Fisher, 1985). Histograms can be constructed, if desired, from the data in tables 1-2.

LABORATORY METHODS

The techniques reported by each participating laboratory to determine each parameter are summarized in tables 5-19. Several laboratories reported only information such as sample aliquot or sample preparation; therefore, we do not know if the recommended method was used, or if a different procedure was used but not reported. From the information provided, however, it becomes obvious that the techniques used for any single parameter are not consistent among laboratories. For example, in table 11, the sample aliquot used for CEC ranged from 2-5 gm. The sample preparation ranged from using the sample as received to resieving to 60-mesh size. The amount of extracting solution and the sample-to-solution ratio was inconsistent among laboratories.

RECOMMENDATIONS

1. Laboratories should carefully check the results they are reporting. Some values reported in this round robin appear to be in error because of a mistake in the placement of the decimal point. Other common errors are in calculation, transcription, and conversion of data from one unit to another.

2. Sample aliquot, sample preparation, soil-to-solution ratio, reaction time, and other special techniques used by the laboratory should be reported so that changes in these variables could be related to the reported values to determine whether or not they affect the reported values in a predictable way.

3. The range in reported values for most parameters was wide. This may be due to the different techniques used by the laboratories for a single parameter, and to the potential errors listed in item 2 above. Lack of homogeneity of the sample split sent to each laboratory might also contribute to error. We recommend that these data not be used as a best estimate of a single "correct value" for each parameter in each of the three samples because of the different techniques used by the participating laboratories.

4. Round-robin programs should probably be conducted on a state-by-state basis, where all state regulatory agencies recommend the same methods for analysis of soil and overburden parameters. To provide most useful data, the same methods must be used by all participating laboratories so that the results can be easily summarized and provide a basis for comparison of individual laboratory results to the group average.

5. The round-robin results point out that when determining an exchangeable, soluble, or available fraction of the total, the same method must be used by all participating laboratories in order to obtain comparable results. If different methods are used by participating laboratories, then the techniques used must be given in sufficient detail so that the effects of the variation in technique on reported values can be assessed.

6. If the purpose of an analysis is to comply with regulatory guidelines for soil and overburden, then the method used for analysis should be the one recommended by the regulatory agency unless there is a demonstrated correlation between the recommended method and the alternative method. It should be the responsibility of the regulatory agency to recommend methods that will provide data that are useful, accurate, and reliable in predicting the suitability or unsuitability of soil or overburden. It should be the responsibility of the laboratory using an alternative method to demonstrate the relationship between the recommended and alternative methods.

REFERENCES CITED

Severson, R. C., and Fisher, Scott E., Jr., 1985, Results of the first western task force round robin soil and overburden analysis program: U.S. Geological Survey Open-File Report 85-220, 54 p.

Severson, R. C., and Fisher, Scott E., Jr., 1986, Results of the second western task force round robin soil and overburden analysis program: U.S. Geological Survey Open-File Report 86-49, 30 p.

Table 1. Reported data for samples 1-21 from the Third Western Task Force Round Robin Soil and Overburden Analysis Program.

[N, not detected; <, detected but below the limit of determination shown; >, determined to be greater than the value shown. Units are given in table 3; descriptions of acronyms are given in appendix table A2.]

Laboratory	pH	Conductivity	Soluble Ca	Soluble Mg	Soluble Na	SAR	Saturation Percentage	Sand	Silt	Clay	Carbon-Ash	Carbon-Organic
A	6.15	5.32	16.50	19.70	68.00	16.00	60.00	38.0	38.0	24.0	--[1]	--
B	6.35	4.76[2]	32.40[2]	19.30[2]	37.70[2]	7.44	57.70	50.0	32.5	17.5	43.7	--
C	6.50	1,800.00	1,690.00	618.00	1,200.00	7.98	68.40	21.2	17.4	21.4	40.5	--
D	6.90	6.40	19.60	16.70	35.30	8.30	66.00	38.0	32.0	30.0	43.0	--
E	6.20	4.20	13.90	11.90	28.70	8.00	78.30	40.0	28.0	32.0	41.1	--
F	6.30	6.40	33.70	19.20	40.00	7.80	60.90	20.8	38.4	40.8	43.1	26.10
G	6.10	5.10	26.00	17.00	35.00	7.50	62.70	36.0	42.0	23.0	42.0	--
H	5.40	6.70	32.21	21.31	43.31	8.37	58.86	45.5	25.0	29.6	--	26.60
I	6.18	4.92	29.29	15.30	37.32	7.90	40.00	35.0	37.0	28.0	--	23.45
J	6.30	4.82	24.70	12.10	25.80	6.01	62.00	42.7	31.1	26.2	40.2	--
K	6.10	5.26	17.50	19.90	33.90	7.84	62.00	--	--	--	43.0	--
L	5.70	1.60	16.90	8.82	16.90	4.72	53.60	29.0	33.0	38.0	45.5	--
M	6.10	5.60	28.50	14.20	31.90	6.90	70.30	35.0	34.0	31.0	40.0	--
N	6.16	4.82	24.80	15.60	34.80	7.74	69.80	33.0	34.0	33.0	39.8	--
O	5.96	4.20	26.80	13.80	21.50	4.80	54.80	26.5	42.2	31.3	--	19.20
P	5.90	5.32	28.00	16.70	37.20	7.87	57.70	40.0	34.0	26.0	42.5	--

Table 1. Reported data for samples 1-21 from the Third Western Task Force Round Robin Soil and Overburden Analysis Program.--Continued

Laboratory	CEC	Exch. Na	ESP	B	Avail. N	Cu	Mo	Se	Acid Potential	Neut. Potential	Acid-Base Potential
A	--	--	--	2.40	--	--	--	--	--	--	--
B	--	--	--	1.84	8.000	--	--	1.60	.46[2]	2.20[2]	7.70
C	67.40	3.00	4.50	3.00	.480	62.50	1.650	.05	.30	2.03[2]	10.00
D	32.40	--	--	3.70	10.100	6.50	--	--	--	--	--
E	12.60	1.00	7.90	1.90	26.000	7.00	.970	.38	.86	2.90	2.10
F	44.40	4.70	5.00	2.90	8.000	7.40	.800	.34	.98	<.10	-30.60
G	68.70	5.10	7.40	3.20	9.000	8.90	.640	<.05	1.00	2.50	-6.20
H	2.90	--	--	--	119.300	--	--	--	--	--	2.63
I	41.47	2.99	9.41	2.27	14.990	4.78	.570	.36	1.31[2]	1.78[2]	-23.10
J	52.20	4.44	5.44	1.80	6.550	2.56	.390	.15	0.98[2]	1.81[2]	-12.50
K	35.30	2.37	6.70	4.15	9.600	9.93	.420	.22	.93	1.30	-16.00
L	34.50	2.94	8.52	4.10	.041	--	--	--	.94	1.70	-12.30
M	33.60	2.32	6.90	2.70	67.000	11.10	1.380	.37	.98	1.68	13.82
N	32.01	3.32	10.37	2.95	9.550	10.98	.368	.37	.61	.88	-10.30
O	32.70	3.00	9.20	4.50	7.000	5.50	.650	.16	1.19	2.34	-13.80
P	36.70	4.89	7.48	2.10	9.600	7.20	.090	.23	.96	2.20[2]	12.00

[1] Not determined

[2] Reported values were converted to common units.

Table 2. Reported data for samples 1W-21W from the Third Western Task Force Round Robin Soil and Overburden Analysis Program.

[N, not detected; <, detected but below the limit of determination shown; >, determined to be greater than the value shown. Units are given in table 3; descriptions of acronyms are given in appendix table A2.]

Laboratory	pH	Conductivity	Soluble Ca	Soluble Mg	Soluble Na	SAR	Saturation Percentage	Sand	Silt	Clay	Carbon-Ash	Carbon-Organic
A	6.57	2.48	11.60	9.80	26.70	8.20	38.30	22.00	46.0	32.00	--[1]	--
B	7.60	2.78	11.25	8.67	11.30	3.60	52.80	29.25	49.0	21.75	5.40	--
C	--	--	--	--	--	--	--	--	--	--	--	--
D	7.00	3.20	8.80	8.50	13.40	4.50	54.10	19.00	46.0	35.00	--	.63
E	6.80	1.80	5.80	5.40	13.00	5.50	64.50	20.00	40.0	40.00	4.40	--
F	6.80	3.30	13.00	10.10	15.30	4.50	51.00	21.80	46.2	32.00	4.80	.37
G	6.70	3.00	11.00	4.60	13.00	4.70	55.00	18.00	48.0	35.00	5.50	--
H	6.50	3.20	11.39	9.62	15.44	4.76	51.18	45.40	25.0	29.60	--	.49
I	6.82	2.12	9.33	6.99	12.35	4.32	36.74	16.00	44.0	40.00	--	1.68
J	6.80	2.04	8.53	5.88	9.49	3.53	79.50	16.90	41.3	41.80	3.68	--
K	6.70	2.25	11.00	7.97	12.40	4.03	53.00	23.00	44.0	33.00	5.00	--
L	6.70	.96	5.11	3.95	6.16	2.89	58.20	6.00	47.0	47.00	<1.00	--
M	6.70	2.30	8.00	5.80	10.60	4.00	66.80	22.00	41.0	37.00	4.20	--
N	6.80	2.07	7.00	6.10	11.60	4.53	67.30	18.00	45.0	37.00	3.80	--
O	6.54	1.57	7.40	5.00	7.00	3.00	47.60	20.00	47.0	33.00	--	.60
P	6.50	2.97	11.90	9.24	13.10	4.03	51.40	18.00	52.0	30.00	4.60	--

Table 2. Reported data for samples 1W-21W from the Third Western Task Force Round Robin Soil and Overburden Analysis Program.--Continued

Laboratory	CEC	Exch. Na	ESP	B	Avail. N	Cu	Mo	Se	Acid Potential	Neut. Potential	Acid-Base Potential
A	--	--	--	1.500	--	--	--	--	--	--	--
B	--	--	--	.280	4.000	--	--	1.00	.046[2]	.712[2]	5.67
C	--	--	--	--	--	--	--	--	--	--	--
D	9.10	--	--	.520	4.700	4.30	--	--	--	--	--
E	7.10	.10	1.40	.800	16.000	4.80	.440	.49	.18	.800	2.40
F	16.20	1.35	3.50	.490	4.000	4.50	.420	.63	.12	<.100	-3.80
G	61.80	1.20	1.90	.300	4.000	6.50	.300	<.05	.12	.310	-.65
H	3.30	--	--	--	42.400	--	--	--	--	--	.51
I	19.98	.94	4.86	.457	7.690	2.77	.170	.64	1.40[2]	.280[2]	-41.00
J	24.80	1.59	3.37	.700	4.460	1.09	.160	.19	.16[2]	.610[2]	1.11
K	11.70	.34	2.90	.550	3.100	5.97	.180	.55	.08	.100	-2.00
L	13.30	.50	3.76	.230	.017	--	--	--	.02	.250	-.31
M	10.50	.45	4.30	.200	26.000	6.00	.340	.44	.06	.250	.65
N	9.73	.77	7.91	.560	7.350	7.26	.128	.64	.11	.085	-2.59
O	10.50	.65	6.00	.690	5.100	3.70	.140	.21	.14	.590	1.50
P	14.40	1.14	3.24	.200	4.800	4.48	<.050	.30	.07	.200[2]	.0

[1] Not determined

[2] Reported values were converted to common units.

Table 3. Summary statistics for samples 1-21 from the Third Western Task Force Round Robin Soil and overburden analysis program (Only unqualifided values are included).

Parameter	Units	Range		Mean	Standard Deviation	Number of Samples
		Minimum	Maximum			
pH	standard	5.4	6.9	6.1	0.331	16
Conductivity[1]	mmhos/cm	1.6	6.7	5.0	1.21	15
Soluble - Ca[1]	meq/L	13.9	33.7	24.7	6.39	15
- Mg[1]	meq/L	8.8	21.3	16.1	3.51	15
- Na[1]	meq/L	16.9	68.0	35.2	11.5	15
Sodium Adsorption Ratio (SAR)	none	4.7	16.0	7.8	2.46	16
Saturation Percentage	%	40.0	78.3	61.4	5.58	16
Particle Size - Sand	%	20.8	50.0	35.4	8.35	15
- Silt	%	17.4	42.2	33.2	6.41	15
- Clay	%	17.5	40.8	28.8	6.12	15
Carbon - Ash	%	39.8	45.5	42.0	1.75	12
- Organic Carbon	%	19.2	26.6	23.8	3.39	4
Cation Exchange Capacity	meq/100g	2.9	68.7	37.3	17.7	12
Exchangeable Sodium	meq/100g	1.0	5.1	3.3	1.23	12
Exchangeable Sodium Percentage (ESP)	%	4.5	10.4	7.4	1.82	12
Boron	mg/kg (ppm)	1.8	4.5	2.9	0.885	15
Available Nitrogen	mg/kg (ppm)	0.041	119	20.4	31.7	15
Copper	mg/kg (ppm)	2.6	62.5	12.0	16.1	12
Molybdenum	mg/kg (ppm)	0.09	1.7	0.72	0.461	11
Selenium	mg/kg (ppm)	0.05	1.6	0.38	0.418	11
Acid Potential (AP)	% Total S	0.30	1.31	0.88	0.277	13
Neutralization Potential (NP)	% $CaCO_3$	0.88	2.9	1.94	0.543	12
Acid Base Potential (ABP)	Tons $CaCO_3$/1000 Tons	-30.6	13.8	-5.5	13.7	14

[1] Values for laboratory "C" are not included in the summary statisitcs.

Table 4. Summary statistics for samples 1W-21W from the Third Western Task Force Round Robin Soil and overburden analysis program (Only unqualifided values are included).

Parameter	Units	Range		Mean	Standard Deviation	Number of Samples
		Minimum	Maximum			
pH	standard	6.5	7.6	6.8	0.268	15
Conductivity	mmhos/cm	0.96	3.3	2.4	0.676	15
Soluble - Ca	meq/L	5.1	13.0	9.4	2.40	15
- Mg	meq/L	4.0	10.1	7.2	2.08	15
- Na	meq/L	6.2	26.7	12.7	4.66	15
Sodium Adsorption Ratio (SAR)	none	2.9	8.2	4.4	1.25	15
Saturation Percentage	%	36.7	79.5	55.2	11.1	15
Particle Size - Sand	%	6.0	45.4	21.0	8.32	15
- Silt	%	25.0	52.0	41.1	6.15	15
- Clay	%	21.8	47.0	34.9	6.00	15
Carbon - Ash	%	3.7	5.5	4.6	0.646	9
- Organic Carbon	%	0.37	1.7	0.75	0.528	5
Cation Exchange Capacity	meq/100g	3.3	61.8	16.3	14.7	13
Exchangeable Sodium	meq/100g	0.10	1.6	0.82	0.464	11
Exchangeable Sodium Percentage (ESP)	%	1.4	7.9	3.9	1.84	11
Boron	mg/kg (ppm)	0.2	1.5	0.53	0.397	14
Available Nitrogen	mg/kg (ppm)	0.017	42.4	9.5	11.5	14
Copper	mg/kg (ppm)	1.1	7.3	4.7	1.76	11
Molybdenum	mg/kg (ppm)	0.13	0.44	0.25	0.123	9
Selenium	mg/kg (ppm)	0.19	1.0	0.51	0.243	10
Acid Potential (AP)	% Total S	0.02	1.4	0.21	0.378	12
Neutralization Potential (NP)	% $CaCO_3$	0.085	0.80	0.38	0.251	11
Acid Base Potential (ABP)	Tons $CaCO_3$/1000 Tons	-41.0	5.7	-3.0	11.7	13

Table 5. Summary of techniques used by participating laboratories to determine pH.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	166g	---[2]	Deionized water (99ml)	24hr; USDA Handbook 60, p.102
B	300g	2mm	Deionized water (173ml)	16hr
C	---	---	Saturated paste	3hr
D	~300g	---	Saturated paste	2hr; USDA Handbook 60
F	---	---	Saturated paste	Overnight
G	25g	---	Deionized water (16.5ml)	24hr
H	150g	---	Saturated soil paste (89ml)	24hr
I	250g	-8 mesh	Deionized water	16hr; USDA Handbook 60, p.102
J	200g	---	---	24hr; USDA Handbook 60
K	---	-60 mesh	Saturated paste	24hr
M	35g	As received	Deionized water paste	24hr; USDA Handbook 60 Method 21a
N	Cup-full	As received	Saturated paste	24hr; USDA Handbook 60, Method 21a
O	50g	---	50ml	24hr
P	400g	---	Paste extract	12hr (Overnight)

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 6. Summary of techniques used by participating laboratories to determine conductivity.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	166g	---[2]	Deionized water (99ml)	24hr; USDA Handbook 60, p. 89)
B	300g	2mm	Deionized water (173ml)	16hr
C	---	---	Saturated paste	3hr
D	~300g	---	Saturated paste	2hr; USDA Handbook 60
F	---	---	Saturated paste	Overnight
G	25g	---	Deionized water (16.5ml)	24hr
H	150g	---	Saturated soil paste (89ml)	24hr
I	250g	-8 mesh	Deionized water	16hr; USDA Handbook 60, p.89)
J	200g	---	---	24hr; USDA Handbook 60
K	---	-60 mesh	Saturated paste	24hr
M	35g	As received	Deionized water paste	24hr; USDA Handbook 60 Method 21a
N	Cup-full	As received	Saturated paste	24hr; USDA Handbook 60, Method 3a & 46)
O	50g	---	50ml	24hr
P	400g	---	Paste extract	12hr (Overnight)

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 7. Summary of techniques used by participating laboratories to determine soluble calcium, magnesium, and sodium.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	166g	---[2]	Deionized water (99ml)	24hr; USDA Handbook 60, p. 84)
B	300g	2mm	Deionized water (173ml)	16hr
C	5g		25ml	15min
D	~300g	---	Saturated paste	2hr; USDA Handbook 60
F	---	---	Saturated paste	Overnight
G	25g	---	Deionized water (16.5ml)	24hr
H	150g	---	Saturated soil paste (89ml)	24hr
I	250g	-60 mesh	Deionized water	16hr; USDA Handbook 60, p.84)
J	200g	---	---	24hr
K	---	-60 mesh	Saturated paste	24hr
M	35g	As received	Deionized water paste	24hr; USDA Handbook 60 Method 3a
N	Cup-full	As received	Saturated paste	24hr; USDA Handbook 60, Method 3a
O	50g	---	50ml	24hr
P	400g	---	Paste extract	12hr (Overnight)

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 8. Summary of techniques used by participating laboratories to determine saturation percentage.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	166g	---[2]	Deionized water (99ml)	24hr; USDA Handbook 60, p.107
B	300g	2mm	Deionized water (173ml)	16hr
C	12g		Saturated paste	---
D	~300g	---	Saturated paste	2hr
F	---	---	Saturated paste	Overnight
G	25g	---	Deionized water (16.5ml)	---
H	150g	---	Saturated soil paste (89ml)	24hr; USDA Handbook 60, Method 27a
I	250g	-8 mesh	Deionized water	4hr; USDA Handbook 60, p.107
J	30g	---	---	24hr; USDA Handbook 60
K	---	-60 mesh	Saturated paste	24hr
M	---	As received	Deionized water	24hr; USDA Handbook 60 Method 27b
N	Cup-full	As received	Saturated paste	24hr; USDA Handbook 60, Method 27a
O	50g	---	---	---
P	30-40g	---	Paste extract	12hr drying

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 9. Summary of techniques used by participating laboratories to determine particle size (texture).

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	50g	---[2]	Deionized water/tap water	24hr; ASA Monograph p. 562
B	40g	2mm	---	Not applicable
C	50g	---	1000ml	---
D	25g	---	5ml Calgon, 5ml $NaSiO_3$	Modified Bouyoucos hydrometer
F	15g	---	Peroxide pretreatment, $Na_3(PO_4)$	---
G	40g	---	---	Hydrometer
H	50g	---	---	J. Agronomy 54:469, 1962
I	50g	-8 mesh	Water, 125ml $NaPO_3$	ASA Monog. Method 43-5, p.562-566
J	55g	---	100ml Calgon	ASA Monog. Method 43-5
K	40g	-60 mesh	Not applicable	Hydrometer
M	---	As received	Calgon	8hr; Sieve and hydrometer
N	50g	As received	100ml $NaPO_3$	40sec. and 4hr readings
O	50g	---	---	Pipette
P	50g	---	100ml Hexametaphosphate	40sec. and 8hr hydrometer reading

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 10. Summary of techniques used by participating laboratories to determine organic carbon.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
B	5g	2mm	---[2]	Not applicable
C	7g	---	---	Combustion
D	---	---	---	Furnace, dry ash
F	---	---	$K_2Cr_2O_7 + H_2SO_4$	Wet oxidation
F	---	---	550°C	Ash overnight
G	1g	---	---	Loss on ignition
H	0.5g	---	---	ASA Monog. Method 29.352
I	10g	-60 mesh	4hr @ 105°C; 7hr @ 400°C	Low temperature combustion
J	2g	---	7-8hr @ 550°C	---
K	5g	-60 mesh	7hr	Furnace
M	1g	As received	7hr @ 550°C	ASTM D3174-82
N	1g	As received	4hr @ 550°C	ASTM D3174-82
O	2g	---	Walkley-Black	---
P	1g	---	2hr @ 550°C	Loss on ignition

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 11. Summary of techniques used by participating laboratories to determine cation exchange capacity.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
C	4g	---[2]	---	USDA Handbook 60, p.100
D	4g	---	1.0N Na acetate, multiple extract	USDA Handbook 60, p.101
F	2g	---	NaOAc/proponal/NaOAc leach	ICP[3]
G	5g	---	100ml 0.5N $MgNO_3$	6hr, AA[4]
H	4g	---	---	USDA Handbook 60, Method 19
I	5g	-60 mesh	100ml 1N NaOAc/1N NH_4OAc	3-5min; USDA Handbk. 60, Meth. 19
J	5g	---	100ml 1N NH_4OAc	USDA Handbook 60, p.101
K	2g	-60 mesh	50ml 1N Na acetate/ 1N NH_4 acetate	0.75hr, ICP
L	---	---	---	USDA Handbook 60
M	---	As received	1N NaOAc	0.5hr, ASA Monog. 8-3
N	2g	As received	3-25ml leaches; 1N NaOAc/ 95% EtOH/ 1N NH_4OAc	USDA Handbook 60, Method 19; AA
O	2g	---	pH 8.2 NaOAc	---
P	5g	---	50ml 1N NaOAc/NH_4OAc	30min.; Flame AA

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Induction coupled plasma.
[4]Atomic absorption.

Table 12. Summary of techniques used by participating laboratories to determine available sodium.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
C	4g	---[2]	---	USDA Handbook 60, p.101
F	2.5g	---	25ml 1N NH_4OAc	ICP[3]
G	5g	---	100ml 1N NH_4OAc	6hr, AA[4]
I	4g	-60 mesh	3-33ml leaches; 1N NH_4OAc	5min.; USDA Handbk. 60, Meth. 18
J	5g	---	100ml 1N NH_4OAc	USDA Handbook 60, Method 18
K	2g	-60 mesh	50ml 1N NH_4 acetate	0.75hr, ICP
M	1g	As received	20ml 1N NH_4OAc	---
N	2g	As received	3-10ml leaches; pH 7.0 1N NH_4OAc	ASA Monog. Method 13-4.3; AA
O	2g	---	pH 8.0 NH_4OAc	---
P	5g	---	50ml 1N NH_4OAc	30min.; Flame AA

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Induction coupled plasma.
[4]Atomic absorption.

Table 13. Summary of techniques used by participating laboratories to determine boron.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
A	166g	---[2]	99ml deionized water	ASA Monog. Pt. 2, p.442
B	20g	2mm	---	Pye Unicam UV/VIS 6-550
C	20g	---	40ml (?)	2hr; ICP[3]
D	20g	---	40ml $BaCl_2$; boil 5 min	Comm.SoilSci.PlantAnal. 2:363(1971)
F	10g	---	Saturation extract/Hot water	Overnight/5min; ICP
G	2g	---	Deionized water	0.75hr;Technicon Autoanalyzer
I	20g	-60 mesh	40ml water/1ml 1N $BaCl_2$	5min. reflux; ICP
J	25g	---	50ml 10% $CaCl_2$	30min.; ASA Monog. 25-9.1
K	20g	-60 mesh	100ml hot water	1hr; ICP
M	20g	As received	40ml hot water	0.083hr; ASA Monog.; Curcumin
N	20g	As received	40ml 0.01M $CaCl_2$; reflux 5 min	ASA Monog. Method 25-9.1/25-5
O	20g	---	40ml 0.01N $CaCl_2$	5 min.; Colorimetric
P	25g	---	50ml 0.5% $CaCl_2$	30min.; ICP

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Induction coupled plasma.

Table 14. Summary of techniques used by participating laboratories to determine available nitrogen.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
B	10g	2mm	---[2]	Pye Unicam UV/VIS 6-550
C	10g	---	50ml 0.2N (?)	15min
D	5g	---	25ml $Ca(OH)_2$	0.25hr; APHA 14th Ed. 1975 p.429 Colorimetric, chromotropic acid
F	5g	---	25ml 2M KCl	1hr; Flow injection analysis
G	10g	---	50ml 2N NaCl	2hr;Technicon Autoanalyzer
H	10g	---	100ml 2N KCl	1hr;Technicon Autoanalyzer II
I	10g	-60 mesh	100ml 2M KCl	1hr; ASA Monog. Method 33-3.2
J	25g	---	50ml 10% $CaCl_2$	30min.; ASA Monog. 33-3.2
K	20g	-60 mesh	100ml hot water	1hr; Auto-Cd reduction
M	10g	As received	50ml 2N NaCl	1hr
N	5g	As received	25ml deionized water	Ion-specific electrode for NO_3-N
O	10g	---	40ml 2N KCl	1hr; Colorimetric, Cd reduction
P	5g	---	50ml 0.5% $CaCl_2$	30min.; Cd reduction

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Induction coupled plasma.

Table 15. Summary of techniques used by participating laboratories to determine copper.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
C	20g	---[2]	40ml 0.5N (?)	2hr; AA[3]
D	20g	---	40ml DTPA	2hr
F	10g	---	20ml DTPA/AB-DTPA	2hr/15min; ICP[4]
G	20g	---	40ml 0.005M AB-DTPA	0.25hr; AA
I	20g	-60 mesh	40ml AB-DTPA	1hr; ASA Monog. Method 3-5.2.3; AA
J	25g	---	50ml DTPA	2hr; ASA Monog. 19-3.3
K	25g	-60 mesh	50ml AB-DTPA	0.25hr; ICP
M	20g	As received	40ml AB-DTPA	0.5hr
N	5g	As received	20ml 0.005M DTPA	2hr; ASA Monog. Method 19-3.3; AA
O	10g	---	20ml DTPA	2hr; Flame AA
P	25g	---	50ml DTPA	2hr; ICP

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Atomic absorption.
[4]Induction coupled plasma.

Table 16. Summary of techniques used by participating laboratories to determine molybdenum.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
C	1g	---[2]	50ml (?)	Digestion; ICP[3]
F	5g	---	50ml Acid ammonium oxalate	Overnight; ICP
F	10g	---	20ml AB-DTPA	15min; ICP
G	15g	---	30ml Ammonium oxalate	12hr; AA[4]
I	20g	-60 mesh	40ml AB-DTPA	1hr; ASA Monog. Method 3-5.2.3; AA
J	5g	---	10ml NH_4CO_3	8hr; Rod-AA
K	10g	-60 mesh	100ml Acid ammonium oxalate	10hr; ICP
M	20g	As received	40ml 1M Na_2CO_3	6hr; AA
N	10g	As received	20ml 1M $(NH_4)_2CO_3$	6hr; CSU Scientific Series Paper 2155
O	5g	---	25ml Ammonium oxalate/oxalic acid	12hr; Graphite furnace AA
P	10g	---	50ml 1M AB-DTPA	30 min; ICP

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Induction coupled plasma.
[4]Atomic absorption.

Table 17. Summary of techniques used by participating laboratories to determine selenium.

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
B	0.25g	2mm	---[2]	Varian AA[3]-5
C	1g	---	50ml (?)	Digestion; AA
F	10g	---	50ml Hot water	30min boil; Hydride ICP[4]
F	10g	---	20ml AB-DTPA	15min; Hydride ICP
G	10g	---	50ml (?)	Hydride AA
I	20g	-60 mesh	40ml AB-DTPA, 30% H_2O_2, HCl	1hr; ASA Monog. Method 3-5.5.4; AA
J	25g	---	50ml 10% $CaCl_2$	30min; Hydride AA
K	20g	-60 mesh	100ml Hot water	1hr; Hydride
M	10g	As received	50ml Hot water	30min; H_2O_2 reduction, Hydride
N	10g	As received	50ml Hot deionized water	1min boil
O	10g	---	50ml Hot water	Graphite furnace AA
P	25g	---	50ml 0.5% $CaCl_2$	30 min; Furnace AA

[1]Laboratories not reporting details are excluded.
[2]No details reported.
[3]Atomic absorption.
[4]Induction coupled plasma.

Table 18. Summary of techniques used by participating laboratories to determine acid potential (total sulfur).

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
B	---[2]	2mm	---	---
C	---	---	X-ray and SO_4	---
F	0.25g	---	50ml $HNO_3/HClO_4$ digest	Ion chromatography for SO_4
G	~0.2g	---	---	Sulfur analyzer
I	0.5g	-60 mesh	---	LECO sulfur analyxer
J	0.1g	---	---	Sulfur analyzer
K	2g	-60 mesh	Not applicable	Eschka method
M	0.25g	As received	---	LECO SC 32
N	1g	As received	---	LECO sulfur analyzer
O	0.1-0.2g	---	---	LECO
P	0.3-0.5g	---	Hot water wash	LECO

[1]Laboratories not reporting details are excluded.
[2]No details reported.

Table 19. Summary of techniques used by participating laboratories to determine neutralization potential ($CaCO_3$).

Laboratory code[1]	Sample aliquot	Sample preparation	------Extraction procedure------	------Reaction time/ method------
B	2g	2mm	Not applicable	---[2]
C	---	---	---	Calculation
G	2g	---	20ml 0.5N HCl	Titration
H	15g	---	50ml ~0.5N HCl; Boil 5min.	USDA Handbook 60 Method 23(c)
I	2g	-60 mesh	20ml 0.5N HCl; Boil 5min.	USDA Handbook 60 Method 23(c)
J	2g	---	20ml 0.1330 N HCl	1hr; Smith, 1974
K	2g	-60 mesh	20ml 0.21 N HCl	24hr; Acid neutralization
L	---	---	---	EPA 600/2-78-054
M	2g	As received	25ml 0.1N HCl	USDA Handbook 60 Method 23(c)
N	2g	As received	Excess 0.4N H_2SO_4; Boil 3-5min.	Smith, 1974 Mine spoil potential for soil and water quality.
O	2g	---	---	USDA Handbook 60
P	5g	---	50ml 0.5N HCl	Titration with NaOH

[1]Laboratories not reporting details are excluded.
[2]No details reported.

TABLE A1. Laboratories, in alphabetical order, participating in the third round robin analysis program

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ACCU Labs Research, Inc.
Attn: William R. Gilgren
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(303) 423-2766

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Energy Laboratories
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USDA-ARS
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Intermountain Laboratories, Inc.
Attn: Roger Pasch
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Sheridan, WY 82801

Native Plants, Inc.
Applied Ecology - Soils Lab.
Attn: Von Isaman
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Peabody Coal Company
Central Laboratory
Attn: R. L. Wilburn
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Freeburg, IL 62243

Utah State University
Soil Testing Laboratory
Attn: Karl Topper
Agricultural Experiment Station
Logan, UT 84322

Table A2. Recommended procedures for the Third Western Task Force Round Robin Soil and Overburden Analysis Program

	Parameter	Reported As	Procedure
1.	pH	standard units	USDA Handbook 60, Method (21a), pg. 102.
2.	Conductivity	mmhos/cm @ 25°C	USDA Handbook 60, Method (3a), pg. 84 and Method (4b), pg. 89.
3.	Soluble calcium (Ca), magnesium (Mg), sodium (Na)	meq/L	USDA Handbook 60, Method (3a), pg. 84. Analysis by AA or ICP.
4.	Sodium absorption ration (SAR)		Calculated from: USDA Handbook 60, pg. 26
5.	Saturation %	%	USDA Handbook 60, Method (27a) or (27b), pg. 107
6.	Particle size analysis	% sand, silt, clay	ASA Mono. No. 9, Pt 1, Method (43-5), pg. 562-566.
7.	Texture	USDA textural class	
8.	Organic Carbon	%	ASTM, Method (D3174-82), pg. 396-397.
9.	Cation Exchange Capacity (CEC)	meq/100g	ASA Mono. No. 9, Pt 2 (2nd Ed), Method (8-3), pg. 152-154.
10.	Exchangeable sodium (ES)	meq/100g	ASA Mono. No. 9, Pt 2, (2nd Ed), Method (13-4.3), pg. 238-240.
11.	Exchangeable sodium percentage (ESP)	%	Calculated: $\frac{ES}{CEC} \times 100$
12.	Boron (B)	ppm	ASA Mono. No. 9, Pt 2, (2nd Ed), Method (25-9.1), pg. 443 and Method (25-5), pg. 435-436.
13.	Available nitrogen (N)	ppm	ASA Mono. No. 9, Pt 2 (2nd Ed), Method (33-3.2), pg. 649 and Method (33-8.2), pg. 679-682.
14.	Copper (Cu)	ppm	ASA Mono. No. 9, Pt 2 (2nd Ed), Method (19-3.3), pg. 331-333. Analysis by AA or ICP.

Table A2.--continued

Parameter	Reported As	Procedure
15. Molybdenum (Mo)	ppm	ASA Mono. No. 9, Pt 2 (1st Ed), Method (74-2), pg. 1062-1063. Analysis by Furnace AA or ICP.
16. Selenium (Se)	ppm	ASA Mono. No. 9, Pt 2 (1st Ed), Method (80-3.2), pg. 1122 and hydride generation for AA or ICP by ASA Mono. No. 9, Pt 2 (2nd Ed), Method (3-5.5.3), pg. 60.
17. Acid Potential (AP)	% Total Sulfur	LECO Sulfur Analyzer
18. Neutralization Potential (NP)	% $CaCO_3$	USDA Handbook 60, Method (23c), pg. 105.
19. Acid Base Potential (ABP)	Tons Ca CO_3/1000 tons material	Calculated:[1] ABP = NP-AP

[1]The following calculations are necessary for conversion of % total sulfur and % $CaCO_3$ to common units of tons $CaCO_3$/1000 tons material:

% S x (31.24) = tons $CaCO_3$ required/1000 tons material

% $CaCO_3$ x (10) = tons $CaCO_3$ present/1000 tons material.

References

ASA Monograph No. 9, Part 1, (First Edition), C. A. Black (Ed), Methods of Soil Analysis - Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling; American Society of Agronomy, Inc., Madison, Wisconsin, 1965.

ASA Monograph No. 9, Part 2 (First Edition), C. A. Black (Ed), Methods of Soil Analysis - Chemical and Microbiological Properties. American Society of Agronomy, Inc., Madison, Wisconsin, 1965.

ASA Monograph No. 9, Part 2 (Second Edition), A. L. Page (Ed), Methods of Soil Analysis, Chemical and Microbiological Properties. American Society of Agronomy, Inc., Madison, Wisconsin, 1982.

American Society of Testing and Materials, Part 26, Gaseous Fuels, Coal and Coke - Atmospheric Analysis. 1982. Philadelphia, PA.

USDA Handbook 60, U.S. Salinity Laboratory Staff. 1954. Diagnosis and Improvement of Saline and Alkali Soils; Washington, D. C.