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Results of the Second 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 Second 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.

Fifteen laboratories participated in this round robin analysis program.* Each laboratory was sent three samples of overburden material that was ground to pass a 2 mm 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.

Each laboratory was asked to respond to a questionnaire. A summary of the workload information from this questionnaire is presented as figure A1 in the appendix. Responses indicate that the mining industry supplies most of the annual workload to five of the responding laboratories. For only two laboratories 80-100% of the workload is research oriented, while for the majority of the laboratories it represents less than 20% of their activity. Similarly, only one laboratory reports 80-100% of its workload from agriculture; the majority of laboratories report that less than 20% of their workload is from agriculture. Plant samples represent a small fraction of the effort of a majority of laboratories, while water and soil or overburden samples show no distinct workload trend.

The remainder of this report is a presentation of the data obtained from the second round robin. Any statistical tests applied to the data must be interpreted with caution because of the small number of samples (n of 15 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 1-3 for the three samples. 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 1-3. The laboratories performing the analyses are coded to conceal the identity of the individual laboratories. Judgements on laboratory quality, based on comparisons of reported values, are inappropriate because many of the

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

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 4-6. Only pH ranged by a factor of two or less for all three sample types. At the other extreme, the range in values reported for exchangeable sodium percentage (ESP) differed by 100 times or more, and boron, available N, molybdenum, selenium, and acid potential ranged by a factor of 10 times or more for all three 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, or instrumental analysis 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.

Histograms showing the frequency distribution for the values reported by all laboratories for each parameter in each of the three samples 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 1-3.

LABORATORY METHODS

The techniques reported by each participating laboratory to determine each parameter are summarized in tables 7-21. 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 13, the sample aliquot used for CEC ranged from 2-5gm. The sample preparation ranged from using the sample as received to resieving or further grinding to as fine as a 60-mesh size. The amount and kind of extracting solution, and the sample-to-solution ratio was inconsistent among laboratories.

It would be informative to graphically evaluate the effects of sample aliquot, sample preparation, sample-to-solution ratio, extraction procedure, and reaction time on the reported values for each parameter. Because the number of laboratories providing detailed information on their methods for each parameter was inadequate, only a few evaluations are provided. Reaction time appears to have little or no effect on the four parameters shown in figure 1. The largest differences appear to be among laboratories using the same reaction time. The same statement can be made for different extractants used on two additional parameters (fig. 2). These two figures illustrate that, even though the same reaction times or extractants are used on splits of the same sample, widely different results can be obtained due to differences in the laboratory techniques, equipment, personnel, or some other unidentified factors.

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. 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.

5. 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.

Table 1. Reported data for 'SAMPLE-3' sample from the Second 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 4; description of acronyms are given in appendix table A2.]

Laboratory Code	pH	Conduc-tivity	Soluble Ca	Soluble Mg	Soluble Na	SAR	Saturation Percentage	Sand	Silt	Clay	Carbon-Ash	Carbon-Organic
A	7.60	1.95	2.94	10.30	8.70	3.38	44.30	69.3	16.9	13.8	--a	.260
B	8.10	1.30	2.40b	7.00b	7.80 b	3.62	--	--	--	--	--	--
C	7.76	.79	1.10	4.00	3.70	2.30	--	68.0	14.0	18.0	--	--
D	7.90	1.73	2.50	8.40	6.00	2.57	30.66	60.0	17.5	22.0	6.44	--
E	8.10	2.01	2.77	9.44	8.22	3.33	44.10	66.0	22.0	12.0	3.77	--
F	8.00	1.30	6.50	2.60	7.20	2.90	53.10	--	--	--	8.71	--
G	7.80	1.57	2.57	7.73	6.75	2.97	46.90	61.8	23.2	16.4	2.90	--
H	7.90	1.70	2.30	7.70	6.90	3.10	47.00	66.0	20.0	14.0	6.00	--
I	8.10	1.47	3.28	7.79	7.04	2.99	44.00	63.0	21.0	16.0	4.50	--
J	8.13	1.49	2.19	7.54	6.61	3.00	51.00	58.0	22.0	20.0	--	.230
K	8.22	1.65	2.69	10.78	7.22	2.78	40.20	58.0	20.0	22.0	--	--
L	7.86	1.52	2.90	9.20	4.20	1.70	44.00	60.0	19.1	20.9	--	.998
M	7.72	2.10	29.00	65.00	2.00	.31	45.95	68.0	16.0	16.0	--	.260
N	7.80	1.35	2.60	7.20	6.50	2.90	53.10	60.0	18.0	22.0	--	--
O	8.32	.84b	1.90b	6.30b	6.40 b	3.16	60.30	67.8	13.5	18.7	--	1.290

Laboratory Code	CEC	Exch Na	ESP	B	Avail. N	Cu	Mo	Se	Acid Potential	Neut. Potential	Acid-Base Potential
A	18.90	1.61	6.50	<.20	--	1.80	1.800	<.010	.100	9.430	91.2
B	--	--	--	--	--	--	--	--	.070	8.770 b	--
C	15.00	.78	2.70	<.03	--	10.50 c	.400 c	.080 c	.040	9.259 b	--
D	18.50	1.26	.07	2.65	--	2.70	<1.000	.003	.080	--	--
E	17.60	.72	2.05	<.10	1.40	1.40	<.050	.010	.0	8.700 b	87.0
F	19.20	--	--	.30	3.60	3.40	<.200	.150	.040	8.620 b	84.9
G	12.90	1.31	7.70	.49	2.65	.69	.050	.015	.040 b	9.820 b	97.0
H	12.00	.80	6.70	.40	2.00	1.80	.070	<.025	.030	8.800	87.0
I	13.10	.56	4.30	.60	1.20	1.89	.034	<.005	.020	8.400	84.0
J	14.11	1.00	4.70	.40	<.10	1.33	.020	<.012	.024 b	10.100	93.5b
K	13.98	.62	4.43	.48	.08	2.09	.610	<.010	--	--	--
L	--	--	--	.70	3.50	1.50	--	--	--	25.000	--
M	--	--	--	.21	1.43	--	--	--	--	--	--
N	22.20	--	--	.40	4.40	--	--	--	<.010	13.100 b	131.0
O	13.20	.89	6.73	--	--	--	--	--	.0	10.400 b	104.2

a, Not determined.
b, Reported value converted to common units.
c, Total amount, not extractable amount, reported.

Table 2. Reported data for 'SAMPLE-4' sample from the Second 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 4; description of acronyms are given in appendix table A2.]

Laboratory Code	pH	Conductivity	Soluble Ca	Soluble Mg	Soluble Na	SAR	Saturation Percentage	Sand	Silt	Clay	Carbon-Ash	Carbon-Organic
A	5.20	5.48	30.20	70.40	2.57	.36	45.60	50.7	27.3	22.0	-- a	2.55
B	4.80	3.20	15.50	45.80	2.40	.43	--	--	--	--	--	--
C	5.61	3.30	24.00 b	39.00 b	1.10 b	.20	--	40.0	28.0	32.0	--	--
D	5.90	4.65	20.00	42.50	1.20	.21	31.64	55.0	27.5	17.5	14.70	--
E	5.50	5.12	23.90	51.70	2.05	.33	48.20	46.0	32.0	22.0	16.30	--
F	5.40	3.50	1.60	24.40	43.60	.30	66.30	--	--	--	17.89	--
G	5.10	4.61	25.10	45.30	2.31	.39	50.20	56.4	30.0	13.6	13.80	--
H	5.40	5.10	25.00	49.00	1.70	.28	49.00	44.0	33.0	23.0	17.00	--
I	5.70	3.90	22.60	61.10	1.82	.28	49.00	35.0	31.0	34.0	16.20	--
J	5.79	4.00	11.20	36.85	1.41	.29	76.00	39.0	28.0	33.0	--	5.64
K	6.78	4.35	21.76	53.30	1.85	.30	48.10	30.0	46.0	24.0	--	--
L	5.59	4.10	30.10	56.10	2.40	.40	44.00	44.0	23.8	32.2	--	10.06
M	5.45	5.90	14.00	19.00	77.00	18.90	44.22	55.0	21.0	24.0	--	6.18
N	5.10	4.15	20.20	44.80	1.60	.30	55.90	34.0	28.0	38.0	--	--
O	5.34	2.40 b	18.40 b	37.90 b	1.80 b	.34	60.00	25.3	24.4	49.3	--	8.95

Laboratory Code	CEC	Exch Na	ESP	B	Avail. N	Cu	Mo	Se	Acid Potential	Neut. Potential	Acid-Base Potential
A	31.40	1.76	5.20	<.20	--	5.76	1.100	<.010	.690	1.760	-3.96
B	--	--	--	--	--	--	--	--	.780	1.470 b	--
C	14.40	.17 b	1.00	.03	--	36.50 c	.700c	1.350 c	.900	1.318 b	--
D	31.50	.40	.01	2.95	--	4.50	.800	<.003	.630	--	--
E	29.00	.10	<.10	<.10	5.40	5.30	<.050	.030	.320 b	.800 b	-2.00
F	36.60	--	--	.70	10.20	6.80	<.200	.110	.660	3.380 b	13.20
G	18.20	.63	2.82	.57	6.95	1.69	.090	.040	.660 b	.978 b	-10.80
H	21.00	.19	.90	.70	11.00	4.70	.210	.083	.740	1.400	-9.00
I	24.30	.15	.60	1.00	9.60	6.08	.034	<.005	.590	1.200	-6.00
J	25.39	<.54	<1.71	.73	4.58	4.22	.076	<.012	.780 b	4.050 b	16.13
K	18.53	.20	1.08	.84	.41	5.08	.870	<.010	--	--	--
L	--	--	--	1.30	5.00	5.40	--	--	--	2.300	--
M	--	--	--	.52	8.32	--	--	--	--	--	--
N	18.80	--	--	1.50	10.00	--	--	--	.690	.788 b	-13.80
O	19.80	.28	1.40	--	--	--	--	--	.016 b	.774 b	7.24

a, Not determined.
b, Reported value converted to common units.
c, Total amount, not extractable amount, reported.

Table 3. Reported data for 'SAMPLE-5' sample from the Second 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 4; description of acronyms are given in appendix table A2.]

Laboratory Code	pH	Conductivity	Soluble Ca	Soluble Mg	Soluble Na	SAR	Saturation Percentage	Sand	Silt	Clay	Carbon-Ash	Organic Carbon
A	7.70	6.30	13.10	18.50	55.50	14.00	57.70	33.6	45.3	21.1	-- a	.84
B	8.00	3.50	4.60	11.00	30.00	10.80	--	--	--	--	--	--
C	7.48	3.10	6.00 b	8.70 b	26.00 b	9.60	--	38.0	34.0	28.0	--	--
D	7.70	5.81	12.00	16.00	37.00	9.89	34.84	27.5	42.5	30.0	7.91	--
E	8.00	6.45	12.00	17.00	49.60	13.00	62.60	30.0	52.0	18.0	6.26	--
F	7.90	3.80	34.40	8.30	11.00	11.00	82.30	--	--	--	10.03	--
G	7.60	5.23	11.20	14.00	42.90	12.10	63.50	27.3	50.9	21.8	5.10	--
H	7.80	5.20	7.70	10.00	29.00	9.70	74.00	31.0	48.0	21.0	9.00	--
I	7.80	4.91	12.10	19.30	41.60	15.10	58.00	29.0	48.0	23.0	6.40	--
J	7.96	4.23	3.77	10.74	30.10	11.18	92.00	24.0	48.0	28.0	--	1.18
K	8.16	5.03	10.18	16.86	44.15	12.01	56.40	28.0	42.0	30.0	--	--
L	7.92	4.43	11.10	15.40	44.00	12.10	62.00	25.3	43.3	31.4	--	2.49
M	7.73	6.70	3.00	10.00	8.00	3.30	52.99	35.0	41.0	24.0	--	1.21
N	7.70	3.90	8.90	10.80	30.20	9.60	55.90	24.0	44.0	32.0	--	--
O	8.20	2.42 b	6.40 b	9.00 b	33.90 b	12.30	81.00	44.9	23.4	31.7	--	1.60

Laboratory Code	CEC	Exch Na	ESP	B	Avail. N	Cu	Mo	Se	Acid Potential	Neut. Potential	Acid-Base Potential
A	22.60	5.76	11.00	<.20	--	3.56	1.20	<.010	.2000	9.310	86.8
B	--	--	--	--	--	--	--	--	.1500	8.640 b	--
C	12.90	4.40	12.00	<.03	--	29.00 c	.80c	<.003c	.1500	9.475 b	--
D	17.70	4.35	.25	1.65	--	4.00	.40	.003	.1500	--	--
E	17.60	3.80	3.98	<.10	7.50	3.80	<.05	.040	.0064 b	8.300 b	81.0
F	18.70	--	--	.30	11.50	6.30	<.20	.070	.1500	8.420 b	79.5
G	11.10	4.21	13.40	.54	12.20	1.47	.07	.020	.1700 b	9.260 b	87.3
H	12.00	4.30	36.00	.40	14.00	3.70	.16	.041	.1800	8.500	79.0
I	12.50	1.74	13.90	.60	10.80	4.77	.02	<.005	.1200	7.600	72.0
J	13.45	4.52	13.01	.48	4.76	3.62	<.02	<.012	.2400 b	2.800 b	20.5
K	13.17	2.49	18.91	.28	.54	4.01	.71	<.010	--	--	--
L	--	--	--	.36	6.30	4.30	--	--	--	27.300	--
M	--	--	--	.18	10.41	--	--	--	--	--	--
N	19.20	--	--	1.30	11.00	--	--	--	.1100	12.400 b	120.0
O	11.10	4.70	4.23	--	--	--	--	--	.0013 b	9.360 b	93.6

a, Not determined.
b, Reported value converted to common units.
c, Total amount, not extractable amount, reported

TABLE 4. Summary statistics for 'SAMPLE-3' from the Second Western Task Force Round Robin Soil and Overburden Analysis Program

Parameter	Units	Range Min.	Range Max.	Median	Mean	Std. Dev.	n
pH	standard	7.6	8.32	7.9	8.0	0.20	15
Conductivity	mmhos/cm	0.79	2.1	1.5	1.5	0.38	15
Soluble - Ca	meq/L	1.1	29.0	2.6	4.5	6.87	15
- Mg	meq/L	2.6	65.0	7.7	11.4	14.98	15
- Na	meq/L	2.0	8.7	6.8	6.3	1.78	15
Sodium Absorption Ratio (SAR)	none	0.31	3.62	3.0	2.7	0.81	15
Saturation Percentage	%	30.66	60.3	46.0	46.5	7.14	13
Particle Size - sand	%	58.0	69.3	63.0	63.5	4.14	13
- silt	%	13.5	23.2	19.1	18.7	3.06	13
- clay	%	12.0	22.0	18.0	17.8	3.42	13
Carbon - Ash	%	2.9	8.71	4.5	5.4	2.10	6
- Organic Carbon	%	0.23	1.29	0.26	0.61	0.50	5
Cation Exchange Capacity	meq/100g	12.0	22.2	14.1	15.9	3.25	12
Exchangeable Na	meq/100g	0.56	1.61	0.80	0.96	0.34	10
Exchangeable Sodium Percentage (ESP)	%	0.07	7.7	4.4	4.6	2.42	10
Boron	mg/kg (ppm)	<0.03	2.65	0.4	.7	0.71	13
Available N	mg/kg (ppm)	0.08	4.4	1.4	2.3	1.39	10
Copper[1]	mg/kg (ppm)	0.69	3.4	1.8	1.9	0.75	10
Molybdenum[1]	mg/kg (ppm)	0.02	1.8	0.07	0.43	0.71	9
Selenium[1]	mg/kg (ppm)	0.003	0.15	0.01	0.04	0.071	9
Acid Potential (AP)	% Total S	0.0	0.10	0.03	0.04	0.032	12
Neutralization Potential (NP)	% $CaCO_3$	8.4	25.0	9.3	10.9	4.63	12
Acid Base Potential (ABP)	Tons $CaCO_3$/1000 Tons	84.0	131.0	91.2	95.5	14.79	9

[1]Total analyses from laboratory "C" are not included in the summary.

TABLE 5. Summary statistics for 'SAMPLE-4' from the Second Western Task Force Round Robin Soil and Overburden Analysis Program

Parameter	Units	Range Min.	Range Max.	Median	Mean	Std. Dev.	n
pH	standard	4.8	6.78	5.5	5.5	0.45	15
Conductivity	mmhos/cm	2.4	5.9	4.2	4.3	0.93	15
Soluble - Ca	meq/L	1.6	30.2	21.8	20.2	7.43	15
- Mg	meq/L	19.0	70.4	45.3	45.1	13.14	15
- Na	meq/L	1.1	77.0	1.9	9.6	21.51	15
Sodium Absorption Ratio (SAR)	none	0.20	18.9	0.3	1.6	4.80	15
Saturation Percentage	%	31.64	76.0	49.0	51.4	11.13	13
Particle Size - sand	%	26.3	56.4	44.0	42.7	9.81	13
- silt	%	21.0	46.0	28.0	29.2	6.05	13
- clay	%	13.6	49.3	24.0	28.0	9.55	13
Carbon - Ash	%	13.8	17.89	16.2	16.0	1.50	6
- Organic Carbon	%	2.55	10.06	6.2	6.7	2.96	5
Cation Exchange Capacity	meq/100g	14.4	36.6	21.0	24.1	6.78	12
Exchangeable Na	meq/100g	0.10	1.76	0.20	0.43	0.52	10
Exchangeable Sodium Percentage (ESP)	%	0.01	5.2	1.0	1.6	1.65	10
Boron	mg/kg (ppm)	0.03	2.95	0.70	0.99	0.76	13
Available N	mg/kg (ppm)	0.41	11.0	7.0	7.1	3.32	10
Copper[1]	mg/kg (ppm)	1.69	6.8	5.1	5.0	1.38	10
Molybdenum[1]	mg/kg (ppm)	0.034	1.1	0.20	0.45	0.45	9
Selenium[1]	mg/kg (ppm)	<0.003	0.11	0.01	0.07	0.037	9
Acid Potential (AP)	% Total S	0.016	0.90	0.66	0.62	0.24	12
Neutralization Potential (NP)	% $CaCO_3$	0.774	4.05	1.3	1.7	1.06	12
Acid Base Potential (ABP)	Tons $CaCO_3$/1000 Tons	-13.8	16.13	-4.0	-1.0	10.73	9

[1]Total analyses from laboratory "C" are not included in the summary.

TABLE 6. Summary statistics for 'SAMPLE-5' from the Second Western Task Force Round Robin Soil and Overburden Analysis Program

Parameter	Units	Range Min.	Range Max.	Median	Mean	Std. Dev.	n
pH	standard	7.48	8.2	7.80	7.8	0.20	15
Conductivity	mmhos/cm	2.42	6.7	4.9	4.7	1.27	15
Soluble - Ca	meq/L	3.0	34.4	10.2	10.4	7.14	15
- Mg	meq/L	8.3	19.3	11.0	13.0	3.83	15
- Na	meq/L	8.0	55.5	33.9	34.2	13.08	15
Sodium Absorption Ratio (SAR)	none	3.3	15.1	11.2	11.0	2.69	15
Saturation Percentage	%	34.84	92.0	62.0	64.1	14.96	13
Particle Size - sand	%	24.0	44.9	29.0	30.6	6.01	13
- silt	%	23.4	52.0	44.0	43.3	7.61	13
- clay	%	18.0	32.0	28.0	26.2	4.84	13
Carbon - Ash	%	5.1	10.03	6.4	7.5	1.86	6
- Organic Carbon	%	0.84	2.49	1.2	1.5	0.63	5
Cation Exchange Capacity	meq/100g	11.1	22.6	13.2	15.2	3.80	12
Exchangeable Na	meq/100g	1.74	5.76	4.3	4.0	1.14	10
Exchangeable Sodium Percentage (ESP)	%	0.25	36.0	12.0	12.7	9.94	10
Boron	mg/kg (ppm)	<0.03	1.65	0.36	0.61	0.48	13
Available N	mg/kg (ppm)	0.54	14.0	10.4	8.9	4.08	10
Copper[1]	mg/kg (ppm)	1.47	6.3	3.8	4.0	1.19	10
Molybdenum[1]	mg/kg (ppm)	<0.02	1.2	0.16	0.43	0.46	9
Selenium[1]	mg/kg (ppm)	<0.003	0.07	0.01	0.03	0.025	9
Acid Potential (AP)	% Total S	0.0013	0.20	0.15	0.14	0.07	12
Neutralization Potential (NP)	% $CaCO_3$	2.73	12.4	8.6	10.1	5.83	12
Acid Base Potential (ABP)	Tons $CaCO_3$/1000 Tons	20.5	120.0	81.0	80.0	26.20	9

[1]Total analyses from laboratory "C" are not included in the summary.

Table 7 . Summary of the techniques used by participating laboratories to determine pH.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	250gm±	-10 mesh	Paste with water	Overnight
B	---[2]	---	Saturated paste	---
C	---	---	1:1 extract	---
D	250gm	2mm	Water	16-18 hrs.
E	---	2mm	Saturated paste	Overnight
F	---	As received	Water saturated paste	Wyoming DEQ
G	300gm	As received	Deionized water	24 hrs.
H	---	As Received	Water saturated paste	24 hrs.
I	---	As received	Saturated paste	14 hrs.
J	---	2mm	Water to saturation	4 hrs.
K	202gm	As received	81.2ml water	20 hrs.
L	90gm	2mm	Paste	16 hrs.
M	250gm±	2mm	Saturated soil	4 hrs.
O	300gm	2.0mm	181ml deionized water	4 hrs.

[1]Laboratories not reporting datails are excluded.

[2]No details reported.

Table 8. Summary of the techniques used by participating laboratories to determine conductivity.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	250gm	-10 mesh	Water paste	overnight
B	---[2]	---	Saturated paste	---
D	250gm	2mm	Water	16-18 hrs.
E	---	2mm	Paste extract	overnight
F	---	As received	Water saturated paste	Wyoming DEQ
G	300gm	-10 mesh	Deionized water	24 hrs.
H	---	As received	Water paste extract	24 hrs.
I	---	As received	Saturated paste	14 hrs.
J	---	2mm	Water to saturation	4 hrs.
K	202gm	As received	81.2ml water	20 hrs.
L	90gm	2mm	20ml water	16 hrs.
M	250gm±	2mm	Saturated soil paste	---
O	300gm	2.0mm	181ml deionized water	4 hrs.

[1]Laboratories not reporting datails are excluded.

[2]No details reported.

Table 9. Summary of the techniques used by participating laboratories to determine soluble calcium, magnesium, and sodium.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	250gm	---[2]	Water paste	Overnight
B	---	---	Saturated paste	---
C	---	---	1:1 extract	---
D	250gm	2mm	Water	16-18 hr.; AA[3]
E	---	---	Paste extract	Overnight; AA
F	---	As received	Water saturated paste	Wyoming DEQ
G	300gm	-10 mesh	---	24 hrs.; AA
H	---	As received	Paste extract	24 hrs.; AA-flame
I	---	As received	Saturated paste	14 hrs.; AA-flame
J	---	2mm	Water to saturation	4hrs.; AA-flame
K	202gm	As received	81.2ml water	24 hrs.; AA-flame
L	90gm	---	15ml water	16 hrs.; AA
M	---	---	Saturated soil paste	AA
O	300gm	2.0mm	181ml deionized water	AA

[1]Laboratories not reporting details are excluded.

[2]No details reported.

[3]Atomic absorption.

Table 10. Summary of the techniques used by participating laboratories to determine saturation percentage.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	250gm±	---[2]	Water paste	Overnight
D	9gm	2mm	Water	24 hrs.
E	---	2mm	Saturated paste	Overnight
F	---	As received	Water saturated paste	Wyoming DEQ
G	35gm	-10 mesh	Deionized water	24 hrs.
H	---	As received	Paste with water	24 hrs.
I	---	As received	Saturated paste	14 hrs.
J	---	2mm	Water to saturation	4 hrs.
K	202gm	As received	81.2ml water	20 hrs.
L	90gm	2mm	---	16 hrs.
M	20gm±	2mm	---	4 hrs.
O	300gm	2.0mm	181ml deionized water	4 hrs.

[1]Laboratories not reporting details are excluded.

[2]No details reported.

Table 11. Summary of the techniques used by participating laboratories to determine particle size (texture).

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	55gm	---[2]	Hexametaphosphate	Overnight
D	40gm	2mm	---	2 hr.; hydrometer
E	50gm	2mm	100ml 6% Na-hexametaphosphate	40 sec./8 hrs.; hydrometer
G	55gm	-10 mesh	100ml calgon	30 sec./8 hrs.; hydrometer
H	40gm	As received	USDA method, set 12 hrs.	30 sec./8 hrs.; hydrometer
I	40gm	As received	Set 14 hrs.	Hydrometer
J	50gm	2mm	50ml 0.08M $(NaPO_3)_6$	40 sec./ 4 hrs.; hydrometer
K	50gm	As received	10ml 1N $Na_6(PO_4)_6$	40 sec./8 hrs.; hydrometer
M	50gm	2mm	---	40 sec./2 hrs.
O	100gm	2.0mm	---	16 hrs.

[1] Laboratories not reporting details are excluded.

[2] No details given.

Table 12. Summary of the techniques used by participating laboratories to determine organic carbon.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	1gm	---[2]	---	4 hrs.
D	1gm	2mm	---	4 hrs.; muffle furnace
E	1gm	2mm	---	2 hrs. 250°C; muffle furnace
F	---	---	---	Method D3174
G	2gm	-60 mesh	---	8 hrs. 550°C
H	1gm	As received	---	12 hrs. 750°C; muffle furnace
I	10gm	As received	---	7 hrs.; high temperature furnace
J	0.25gm	2mm	2.5ml 0.2M $K_2Cr_2O_7$ / 5ml conc. H_2SO_4	10 sec./12 hrs.;Spec 20
L	0.5gm	0.25mm	20ml $H_2SO_4/K_2Cr_2O_7$/DI-water	2hrs./set 20 min.; Spec 20
M	0.2gm±	0.5mm	5gm treated with 12ml 4N HCl to remove carbonates	LECO CHN600
O	10gm	2mm	NTIS, 1978 method	---

[1]Laboratories not reporting details are excluded.

[2]No details given.

Table 13 . Summary of the techniques used by participating laboratories to determine cation exchange capacity.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	5gm	---[2]	100ml 1N $NaOAc/NH_4OAc$	Sample dependent
D	4gm	2mm	NH_4OAc	---
E	5gm	2mm	50ml 1M $NaOAc/NH_4OAc$	AA[3]-flame
F	5gm	-60 mesh	$NaOAc/NH_4OAc$ 3-33ml washings	---
G	5gm	-10 mesh	100ml $MgNO_3$	Flame photometer
H	---	As received	NaOAc saturation/NH_4OAc repl.	10 min/wash; AA-flame
I	2gm	As received	50ml 1M NaOAc	30 min; AA-flame
J	2gm	2mm	1M NaOAc/95% EtOH/1M NH_4OAc 3-25ml washings	Shake 15 min., centrifuge 15 min.; AA-flame
K	5gm	As received	ASA Monog. 9, method 8.3	Na by AA, Cl by titration
O	4gm	2mm	Not Applicable	AA

[1]Laboratories not reporting details are excluded.

[2]No details reported.

[3]Atomic absorption.

Table 14. Summary of the techniques used by participating laboratories to determine exchangeable

A	5gm	---[2]	100ml 1N NH_4OAc	Sample dependent
C	---	---	EDTA	---
D	1gm	2mm	NH_4OAc	AA[3]
E	5gm	2mm	50ml 1M NH_4OAc	30 min.; AA-flame
G	5gm	-10 mesh	50ml 1N NH_4OAc	30 min.; AA
H	---	As received	100ml NH_4OAc	10 min./wash; AA-flame
I	2gm	As received	50ml 1M NH_4OAc	30min.; AA-flame
J	2gm	2mm	1M NH_4OAc, 3-10ml washings	Shake 15 min, set 15 min.; AA-flame
K	5gm	As received	100ml 1N NH_4OAc, 4-25ml washings	Exch. Na = Ext. Na - Sol. Na
O	4gm	2.0mm	Not applicable	AA

[1]Laboratories not reporting details are excluded.

[2]No details given.

[3]Atomic absorption.

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

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	5gm	---[2]	50 ml water	Boil 30 min.
C	---	---	Hot water	---
D	20gm	2mm	---	Azomethine method
E	25gm	2mm	50ml 1% $CaCl_2$	Boil 5 min.; ICAP[3]
F	20gm	---	40ml water	Wyoming DEQ
G	25gm	-10 mesh	50ml 0.1% $CaCl_2$	Autoanalyzer
H	10gm	As received	20ml hot water	30 min.; spectrometer
I	10gm	As received	Water	30 min.; Autoanalyzer, Azo H
J	10gm	2mm	50ml water	Boil 1 min.; Spec 20
K	20gm	As received	40ml 0.01M $CaCl_2$	Boil 5 min.; Curcumin method
L	90gm	2mm	Water	Set 16 hrs.; Spec 20
M	---	---	Saturated soil paste	ASA Monog. 9, method 25-3; Spectonic 21

[1]Laboratories not reporting details a excluded.

[2]No details given.

[3]Induction coupled plasma.

Table 16. Summary of the techniques used by participating laboratories to determine available nitrogen.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
E	5gm	2mm	50ml 1% $CaCl_2$	30 min.; autoanalyzer, Cd reduction
F	10gm	60 mesh	40ml water	Salicylic acid
G	25gm	-10 mesh	50ml 1% $CaCl_2$	Cd reduction
H	10gm	As received	50ml 1N NaCl	1 hr.; autoanalyzer
I	10gm	As received	Water	---[2]
J	5gm	2mm	25ml water	15 min.; specific ion electrode
K	10gm	As received	100ml 2M KCl	Colorimetric, Cd reduction
L	0.5gm	2mm	Disodium chromotropic acid/ antimony sulfate/ D-water	Shake 15 min., set 1 hr.; Spec 20
M	5gm	2mm	50ml 1M KCl	30 min.; autoanalyzer

[1]Laboratories not reporting details are excluded.

[2]No details given.

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

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	25gm	---[2]	50ml 0.1N DTPA	Overnight
C	---	---	Total analysis	---
D	5gm	2mm	---	---
E	25gm	2mm	50ml DTPA	2 hrs.; AA[3]-flame
F	20gm	60 mesh	40ml AB-DTPA	Wyoming DEQ; AA
G	25gm	-10 mesh	50ml DTPA	2 hrs.; AA
H	20gm	As received	40ml DTPA	2 hrs.; AA-flame
I	40gm	As received	80ml AB-DTPA	15 min.; AA-flame
J	5gm	2mm	20ml 0.005M DTPA	2 hrs.; AA-furnace
K	15gm	As received	30ml 0.005M DTPA	Shake 2 hrs. at 240 cpm; AA-furnace
L	10gm	---	AB-DTPA	AA

[1]Laboratories not reporting details are excluded.

[2]No details given.

[3]Atomic absorption.

Table 18. Summary of the techniques used by participating laboratories to determine molybdenum.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	5gm	---[2]	50ml 1.0N oxalate	Overnight
C	---	---	Total analysis	---
D	---	2mm	"See procedure"	---
E	10gm	2mm	50ml 1M $(NH_4)_2CO_3$	6hrs; AA[3]-furnace
F	20gm	60 mesh	40ml AB-DTPA	Wyoming DEQ; AA
G	5gm	-10 mesh	10ml $(NH_4)_2CO_3$	8 hrs.; AA-furnace (rod)
H	15gm	As received	30ml $(NH_4)_2C_2O_4$	AA-furnace
I	40gm	---	80ml AB-DTPA	15 min.; HGA-AA
J	10gm	2mm	20ml 1M $(NH_4)_2CO_3$	6 hrs.;AA-furnace
K	10gm	As received	100ml ammonium oxalate/ oxalic acid	Shake overnight at 120cps; AA-furnace

[1]Laboratories not reporting details are excluded.

[2]No details given.

[3]Atomic absorption.

Table 19. Summary of the techniques used by participating laboratories to determine selenium.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	5gm	---[2]	50ml water	overnight
C	---	---	Total analysis	---
D	---	2mm	"See procedure"	---
E	25gm	2mm	50ml 1% $CaCl_2$	Boil 5 min.; AA[3] furnace
F	20gm	---	40ml AB-DTPA	Wyoming DEQ; AA
G	25gm	-10 mesh	50ml $CaCl_2$	40 min.; AA/H_2O_2/HCl
H	10gm	As received	50ml water	30 min.; AA-furnace
I	10gm	As received	50ml water	30 min.; HGA-AA
J	10gm	2mm	50ml water	Boil 1 min.; AA-hydride
K	5gm	As received	50ml water	4 hrs. at 100°C; AA-furnace EPA method 270.2

[1]Laboratories not reporting details are excluded.

[2]No details given.

[3]Atomic absorption.

Table 20. Summary of the techniques used by participating laboratories to determine acid potential.

Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	0.2gm	---[2]	---	---
B	---	---	Total S, pyritic S	---
C	---	---	Total S, organic S, sulfate S, pyritic S	---
D	1gm	2mm	ASTM D1757 method B	---
E	---	0.25mm	Leco S analyzer	---
F	0.2gm	---	Leco	---
G	0.2gm	-60 mesh	S-analyzer	---
H	---	---	Fisher S analyzer	---
I	2.0gm	---	Total S by Eschuka method	---
J	0.25gm	2mm	Leco S analyzer	---
O	2gm	2mm	Wyoming DEQ	Not applicable

[1]Laboratories not reporting details are excluded.

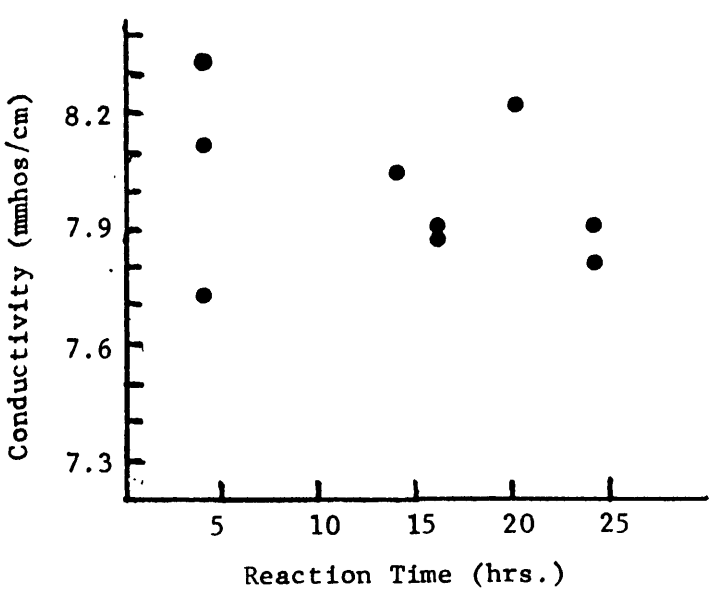
[2]No details given.

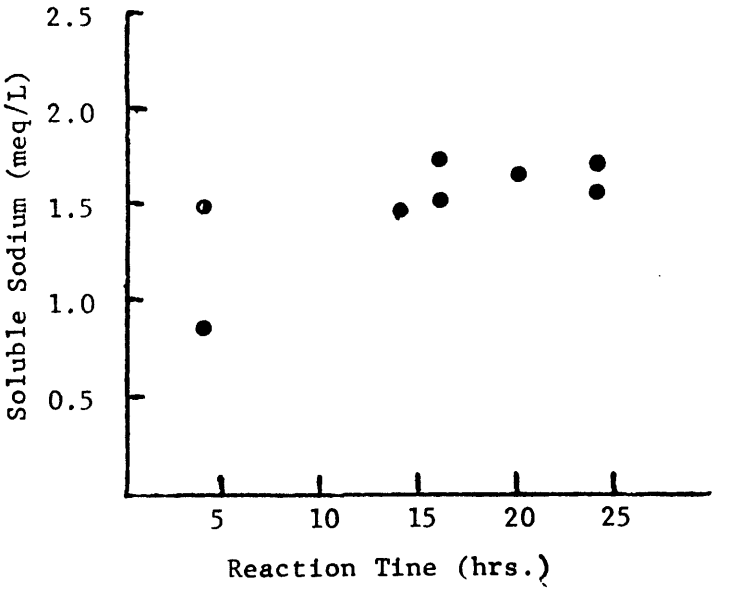
Table 21. Summary of the techniques used by participating laboratories to determine neutralization potential

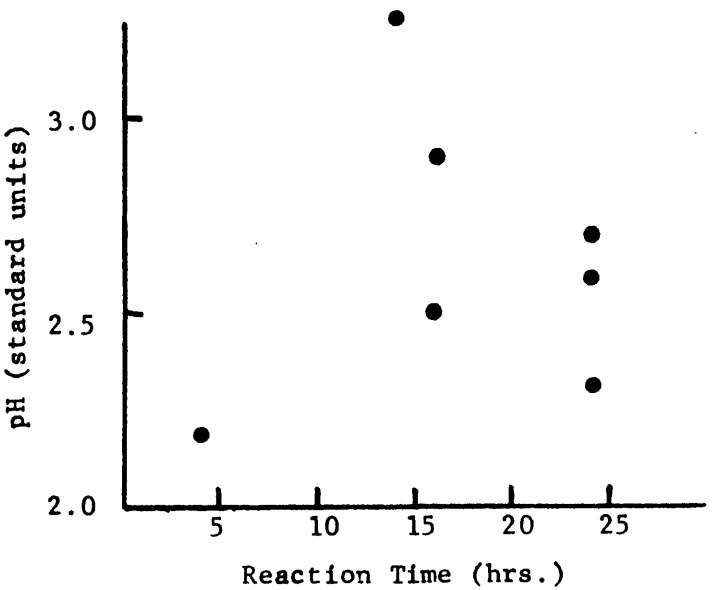
Laboratory code[1]	Sample aliquot	Sample preparation	-----Extraction procedure------	-------Reaction time-----
A	5.0gm	---[2]	50ml 0.25M HCl	Heat to boiling 30 min.
E	5gm	0.25mm	25ml 0.5M HCl/0.5M NaOH	---
F	2gm	---	20ml HCl	---
G	2gm	-60 mesh	40ml 0.11M HCl/ titration	1 hr.
H	5gm	As received	20ml 0.5M HCl/titration	30 min.
I	---	---	20ml 0.2M HCl	---
J	2gm	2mm	Various amounts of 0.2M H_2SO_4	Boil 3 to 5 min.
L	---	2mm	3N HCl	Set 2 hrs.
O	2gm	2mm	Not applicable	---

[1]Laboratories not reporting details are excluded.

[2]No details given.







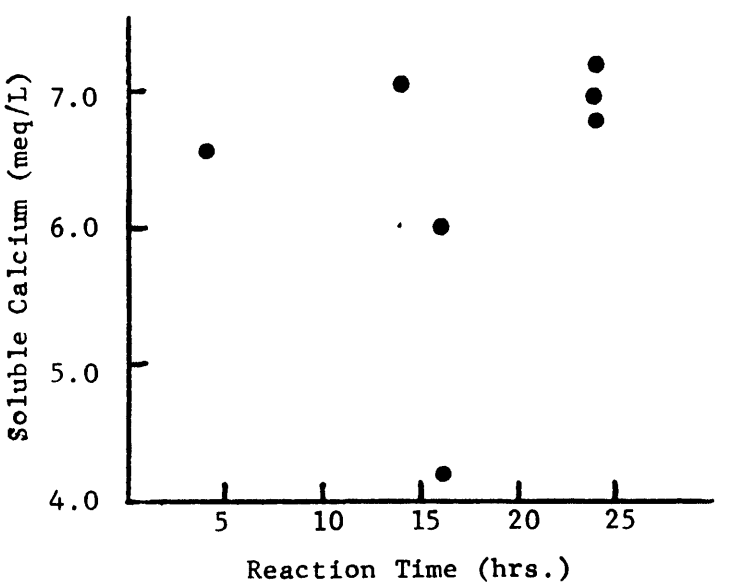


Figure 1. The effect of reaction time on four parameters.

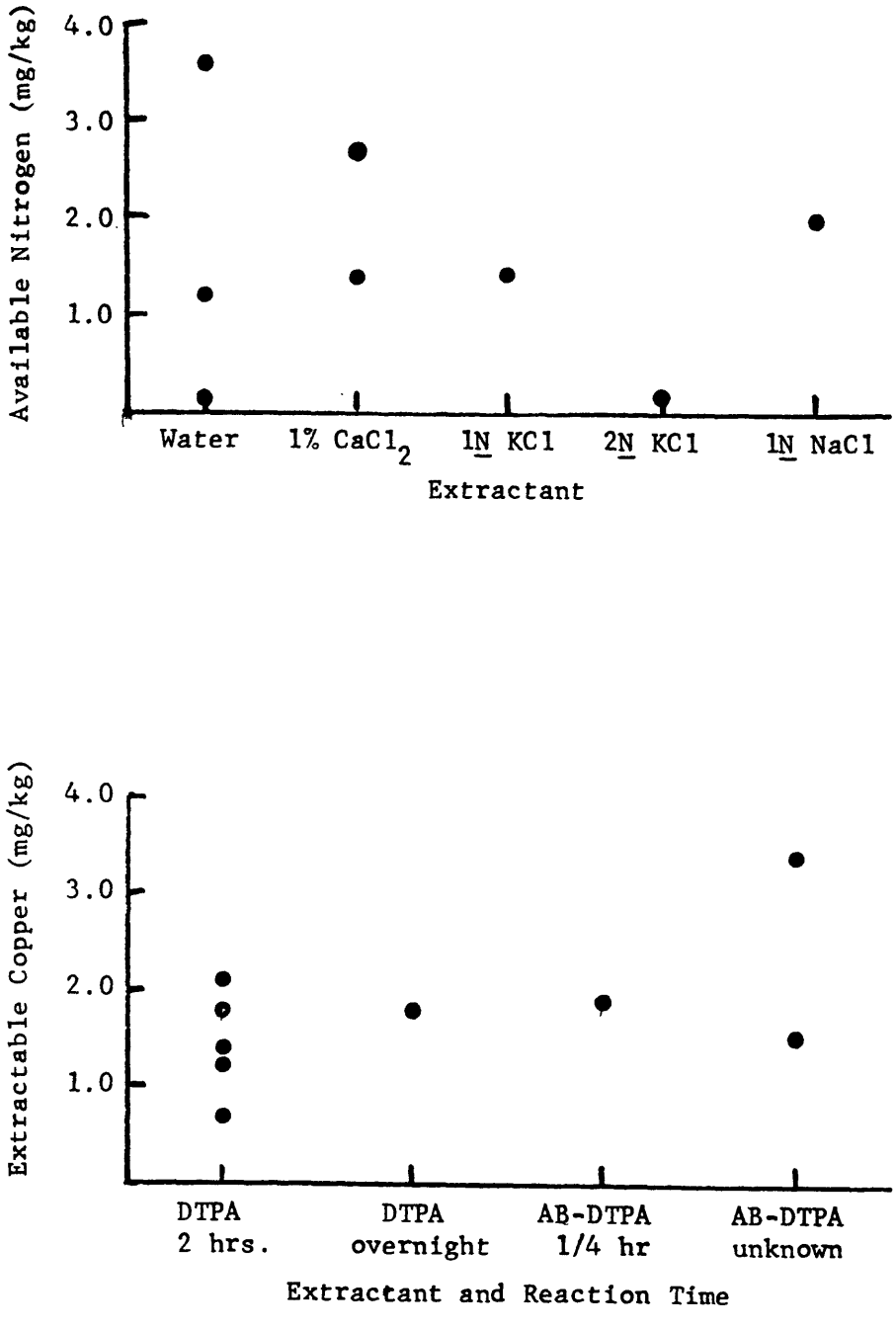


Figure 2. The effect of different extractants on two parameters.

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

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ACCU Labs Research, Inc.
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Attn: Ken Pohlman
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ACZ Inc./Bookcliffs
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Steamboat Springs, CO 80477

Casa Del Sol, Inc.
Attn: Dr. Joe Bowden
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Commercial Testing and Engineering Co.
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CORE Laboratories

CORE Laboratories
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Energy Laboratories
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Minnesota Valley Testing Laboratory
Attn: Jeff Reiser
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Native Plants, Inc.
Applied Ecology - Soils Lab.
Attn: Von Isaman
417 Wakara Way
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Northern Engineering & Testing
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Railroad Commission of Texas
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Reclamation Research Unit
Attn: Dennis Neuman
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Montana State University
Bozeman, MT 59717

Table A2. Recommended procedures for the Second 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

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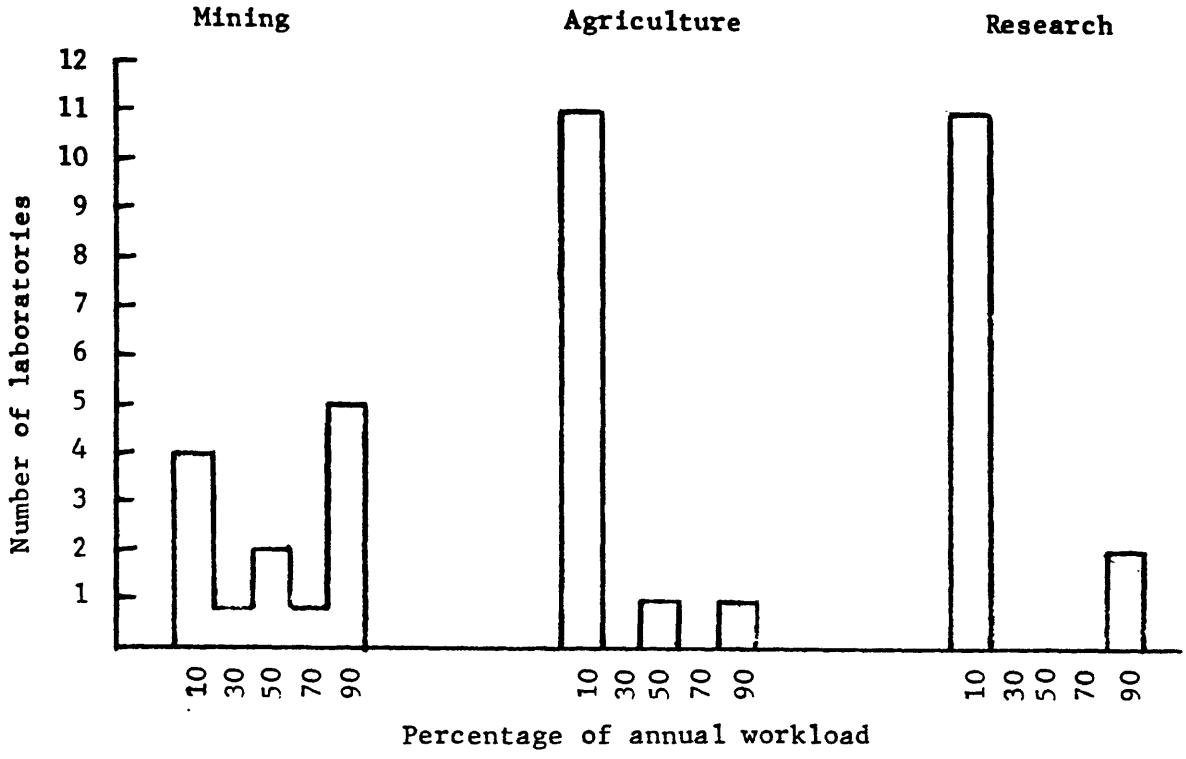

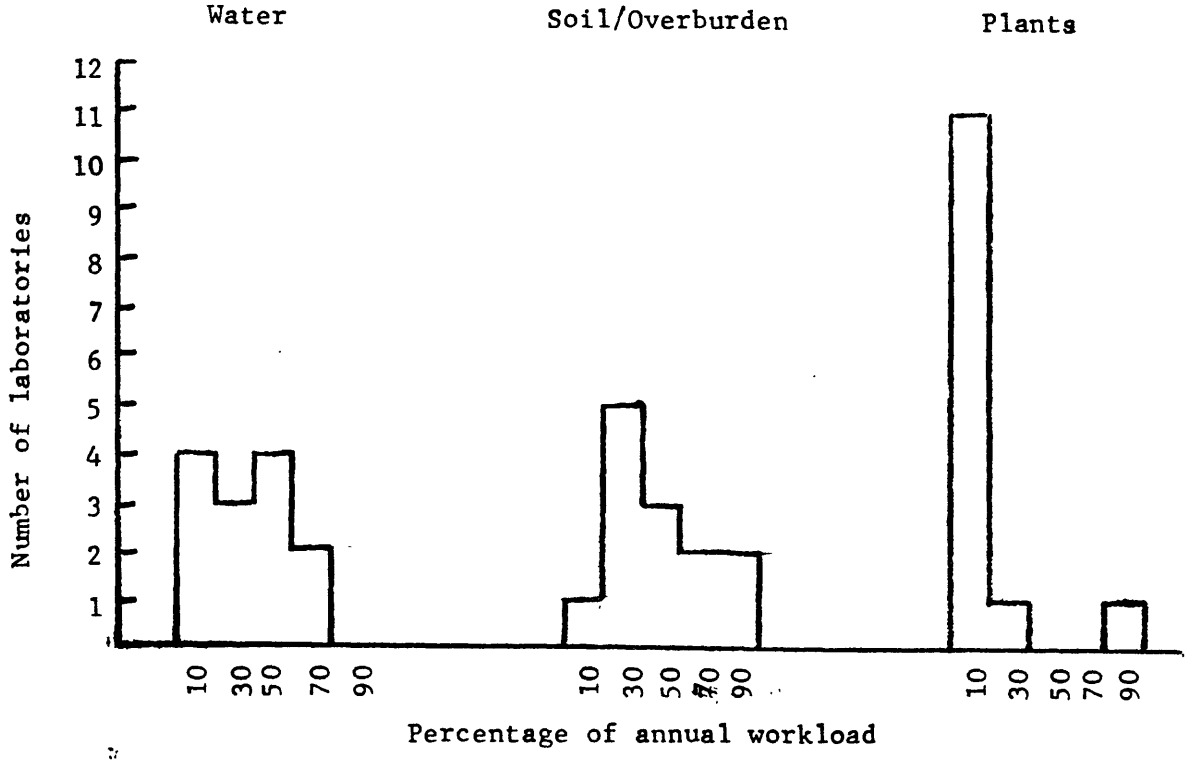

Figure A1. Results of the workload survey from the second round robin analysis program