Table 1. Comparison of cup horn ultrasonic soil dispersion, in weight percent, with Calgon soil dispersion for soils from the Honcut Creek chronosequence

[All trials are averages of three replicates. From Busacca, 1982]

Sample	Treatment	Sand						Silt	<2 μm clay
		Very coarse	Coarse	Medium	Fine	Very fine	Total		
1021	Sonic	0.0	0.0	0.2	9.2	26.1	35.5	50.2	14.3
	Calgon	.1	.2	.5	12.7	23.8	37.3	46.9	15.8
1029	Sonic	.2	.5	.7	4.6	16.0	22.0	54.1	23.9
	Calgon	.1	.8	1.2	6.1	12.5	20.7	55.4	23.9
1102	Sonic	3.2	6.9	5.0	12.6	13.0	40.7	42.7	16.6
	Calgon	4.2	7.6	5.9	12.6	12.0	42.3	40.9	16.8
1106	Sonic	.6	1.6	.8	2.1	11.6	16.7	50.5	32.8
	Calgon	.6	2.3	1.0	2.9	9.9	16.7	49.3	34.0
1206	Sonic	2.8	8.3	6.3	9.4	5.4	32.2	24.1	43.7
	Calgon	3.3	9.2	6.8	8.8	5.1	33.2	18.8	48.0

Table 2. Sand and silt destruction, in weight percent, during ultrasonic treatment

[From Busacca, 1982]

Sample	Treatment	Sand						Silt	<2 μm clay
		Very coarse	Coarse	Medium	Fine	Very fine	Total		
Sand	Before	18.1	48.2	33.7			100.0	4.9	1.7
	After	15.8	43.1	30.3	3.4	0.8	93.4		
Sand	Before				46.6	53.4	100.0		
	After				38.8	51.0	89.3	8.1	2.6

		Coarse silt (20–47 μm)	Fine silt (2–20 μm)	Clay (<2 μm)
Coarse Silt	Before	100.0		
	After	94.3	4.2	1.5

were collected from samples that had been dispersed by standard Calgon. This suggests that sonication removes clay minerals that adhere to sand and silt more completely than does the Calgon treatment, although there is also some fracturing of primary grains. The sand and silt is visibly more neutral in color (less brown) after sonication.

In summary, the cup horn sonication method effectively disperses soil samples and gives results comparable to standard dispersion treatments. Dispersion is achieved without chemical dispersing agents, and there is neither Ti nor Al contamination as in probe-type sonication. Use of the cup horn, linked to the Heat Systems W-220 F sonicator, results in reduced sonication time and energy, when compared to the Biosonic system (comparison data for W-220 F not shown). This, in turn, results in less abrasion and fracturing and lower levels of primary mineral destruction than that shown for the cup horn and Biosonic.

Particle-Size Analysis

By Peter Janitzky, *University of California, Davis*

REFERENCES

Day, 1965.
Jackson, 1969.

PRINCIPLE

Samples are treated to remove organic matter and soluble salts. The samples are then dispersed in a sodium solution. Sands are removed by wet sieving, and the silts and clays are washed into sedimentation cylinders. The amount of silt or clay remaining in a predetermined volume of liquid in

the cylinders over time is used as a measure of the percentage of silt and clay in the sample. The basic tenet of the analysis is that spherical particles will settle in fluid at a rate proportional to their radius (Stokes' law).

The method described here is known as the "pipette method" because of the procedure used to obtain samples. The advantages over the well-known hydrometer method are (1) it is more accurate, and (2) it allows subsampling for clay or silt mineralogy. The major disadvantage is that it requires more time than the hydrometer method. Because particle-size analysis is commonly determined, it is discussed in detail. We were interested in determining reproducibility, as well as determining the effects of sample pretreatments on results.

EQUIPMENT

250-mL beaker
balance (analytical)
watchglass
tweezers
eyedropper
hot plate
filter candle apparatus
rubber policeman
teflon policeman
stirring rod
wash bottle
oven
desiccator
repipette (optional)
shaker bottle
mechanical shaker for sieves
nest of sieves (1.0, 0.5, 0.25, 0.177, 0.105 mm)
reciprocating shaker
size 6½ rubber stopper
sedimentation cylinders
Whittig-type wash bottle (optional)
glass funnel
300-mesh sieve (47μm)
large rubber stopper
weighing jar and lid
Kimwipes (optional)
plunger
thermometer
clock with sweep second hand
25-mL pipette

REAGENTS

distilled water
hydrogen peroxide 30 percent or sodium hypochlorite 5 percent isopropyl alcohol
hexametaphosphate dispersing solution (7.94 g Na_2CO_3 + 35.7 g $(NaPO_3)_6$ per liter of distilled H_2O

PROCEDURE

Preparation of Samples

1. Label 250-mL beakers with laboratory sample numbers. Weigh the empty beakers to 0.01-g accuracy. Check frequently to be certain that the balance is zeroed.
2. Add approximately 10 g of <2-mm soil to the appropriate beaker and cover with a watchglass.

Organic Matter Oxidation

1. Remove visible, undecomposed organic matter (roots, woody particles, and so forth) with tweezers. In B or C horizons this is rapidly accomplished because those horizons generally contain little organic matter. The A horizons, however, may contain large quantities of roots and fibers that must be removed. To extract most of the undecomposed particles 30-45 minutes may be necessary.
2. Wet each sample with distilled H_2O, add a few milliliters of 30 percent H_2O_2, and re-cover with the watchglass. Every 5-10 minutes add 3-5 mL of H_2O_2 and stir gently by slowly swirling the beaker. Use distilled H_2O to rinse the beaker sides of foam. If it is necessary to rapidly reduce foaming because the sample is about to overflow, add a drop or two of isopropyl alcohol directly to the foam.
3. After most foaming subsides, heat to about 70 °C and continue H_2O_2 additions for about one hour. Samples low in organic matter may be heated soon after the first addition of H_2O_2. With samples high in organic matter, however, it is preferable to allow the reaction to occur overnight, without heat, before placing them on a hotplate. This is important because organic rich samples overflow when they are heated too soon.
4. When the organic material that binds soil mineral particles together has been removed, the treatment should be stopped because it affects the mineral fraction as well. Several criteria are used to decide when to stop the oxidation:
   (a) When a light-brown foam no longer appears around the surface of the soil solution after the addition of H_2O_2 and, instead, rapid "self-oxidation" of the peroxide occurs (a vigorous reaction that usually exhausts itself within 5 minutes of H_2O_2 addition);
   (b) The appearance of bleached fragments of roots floating on the surface; and
   (c) Time.

   Do not try to completely oxidize the undecomposed organic particles. Benefits realized are not worth the time required nor the possibility of damage to minerals. Those large particles are not involved in binding minerals.

Filter Candling

1. Clean the watchglass and sides of each beaker with a rubber spatula (or finger), rinsing the residue into the beaker.

2. Place the beakers in a filtering rack, add a filter candle to each (do this before adding any water to prevent overflow), add some distilled water, stir to suspend the silt and clay, and then fill to the top with distilled water.
3. Turn on the vacuum after emptying the vacuum bottle.
4. When the last beaker is drained (less than 25 mL remaining), close off the vacuum with stopcocks, stop the suction, open the rinse water reservoir stopcock, and turn off the vacuum. Re-open the stopcocks to the beakers so that rinse water can flow through them. Rinse the filter candles with a wash bottle; use a finger to remove any residue from the candles, then rinse the finger!
5. Resuspend the silt and clay; refill with distilled water, repeating parts 3 and 4 until five beaker volumes (1,000-1,250 mL) have been filtered.
6. After the final filtering, carefully clean the sides and bottom of each filter candle, washing all mineral particles back into the beakers. Cover the beakers with watch-glasses and place in an oven for 24 hours at 105 °C.

Measuring the Oven-dry Weight

1. After oven drying, place the beakers in a desiccator to cool.
2. When cool (about 2 hours) weigh each one to 0.01-g accuracy, minimizing their exposure to ambient air before weighing. This is the weight used in all calculations, it must be accurate.

Dispersal

1. Add 10 mL of hexametaphosphate solution and 10-20 mL of distilled water to each sample and to a blank. (Note: We dispense hexametaphosphate from a repipette container. To assure solution uniformity, gently agitate the solution with a slow motion of the container. Calibrate the pipette and dispense one aliquot into a waste container to eliminate air bubbles in the neck of the dispenser.)
2. Label wide-mouthed shaker bottles, including one for the hexametaphosphate blank.
3. Carefully clean the beakers with a teflon policeman, being certain to remove the baked-on residue from the sides. Transfer the suspended soil to the appropriate shaker bottle and fill each bottle two-thirds full with distilled water. Seal the bottles with size 6½ rubber stoppers.
4. Save the tared beakers for the sand analysis.
5. Shake the solutions overnight (14-16 hours) on a reciprocating shaker, checking to be certain that they do not rattle—they will break. Avoid leaving the bottles on the shaker more than 16 hours; abrasion of mineral particles can cause small segments to break off, giving a measured particle-size distribution different from the natural distribution.
6. That same evening, fill the required number of labeled sedimentation cylinders with distilled water so that the water will equilibrate to room temperature.

Sand Separation

1. Construct a modified Whittig wash bottle (optional) (fig. 7) and fill it with distilled water from the first of the sedimentation cylinders. Save the remainder of the room-temperature water from the cylinder; it will be used to refill the wash bottle.
2. Place a glass funnel and a 300-mesh sieve on top of the first empty, labeled sedimentation cylinder.
3. Carefully remove the stopper from the first bottle and rinse the soil solution clinging to it back into the bottle.
4. Pour the soil suspension from the bottle through the sieve and into the cylinder. Be careful not to upset the sieve—it is precariously balanced.
5. Rinse the sides of the shaker bottle with distilled water, using the water jet to resuspend the silt and clay particles. Allow the sand to settle for 10-20 seconds, then pour the suspension through the sieve. Repeat the process five or six times. Careful decanting of silt and clay before the sand reduces sieve clogging and increases sieving speed.
6. Transfer the soil remaining in the shaker bottle to the appropriate beaker saved from the filter candling, using several rinses because fine particles resist transfer. Hold the shaker bottle in the light to be certain that all the soil has been transferred. (Note: An effective method for rinsing is to first aim a jet of water down the sides of the shaker bottle so that all soil particles move to the bottom. Then tilt the bottle to one side and wash the particles to the lower sides of the bottom. Pour the soil solution into the beaker and finally, while the opening of the bottle is still tilted downward toward the beaker, rinse the lower

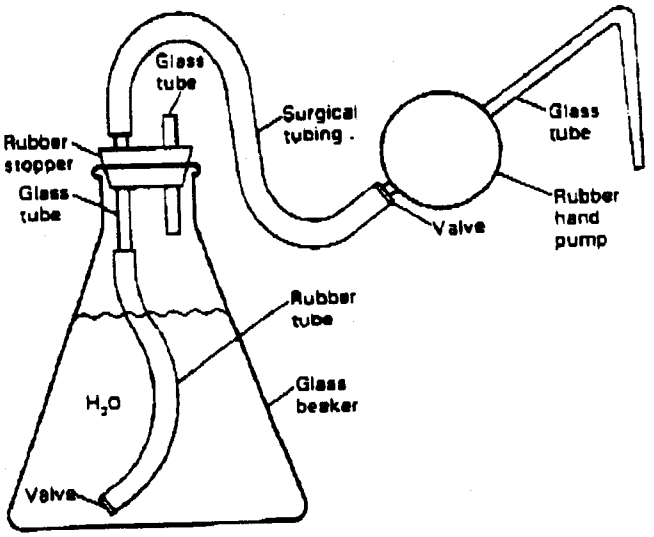

Figure 7. Construction of a Whittig-type wash bottle.

sides of the bottle where the remaining soil particles have collected, allowing the backwash to fall into the beaker.)

7. After each rinse, gently suspend the soil in the beaker and decant the suspension as was done in step 5 above. When no silt or clay remains in the beaker, any rinses of the shaker bottle that are still necessary should go directly through the sieve rather than into the beaker.
8. Use a wash bottle to gently rinse the inside of the sieve (especially the screen) so that the silt and clay particles that may have been trapped pass into the sedimentation cylinder. Next, rinse the outside of the screen so that silt and clay particles that have clung to it will fall into the cylinder. Rinse the funnel and fill the cylinder exactly to the 1-L mark. Cover with a large rubber stopper.
9. Use a jet of water to wash the sand on the inside of the sieve back into the beaker. This is done by aiming the wash bottle at the outside of the sieve and through the screen so that the wash will fall into the beaker. Rotate the sieve so that all sides are cleaned. Hold the sieve in the light to check for sand particles that still cling to the inside; rinse again if necessary.
10. Cover the beaker with a watchglass and dry at 105 °C for 24 hours.
11. Repeat for each sample including the hexametaphosphate blank.

Taring of Weighing Jars

1. Tare the weighing jars, recording jar number, sample number, and tare weight. The jars must be spotlessly clean and equilibrated to the temperature of the weighing room (2 hours to be safe). The lid, sides, and bottom of the jars should be wiped clean just prior to weighing; at the 0.1-mg accuracy required, fingerprints can cause significant errors.

Mixing of Samples in Sedimentation Cylinders

1. Use the plunger to suspend the silt and clay in the sedimentation cylinders at intervals of 2½ minutes or more (2½ minutes is the approximate time needed to take a pipette sample). Mix the blank as well. Record the exact time each sample is mixed. (Note: The sample is most safely mixed by rapid upward strokes and slow downward strokes of the plunger. Frequent short strokes at the cylinder bottom plus occasional long strokes are best. Slow the velocity of the plunger whenever it approaches the solution's surface.)
2. When removing the plunger from the cylinder, gently tap it against the lip of the cylinder so that little soil solution is removed on its stem and head. Rinse the plunger with distilled water over the sink and move to the next cylinder.

Pipette Sampling

1. If the cylinders are not in a constant temperature environment, record the temperature of the blank solution every 2-4 hours. Draw a plot of time (horizontal axis) vs. temperature. The average temperature for the settling period is at the point on the vertical axis crossed by a horizontal line that bisects the area defined by the temperature-time curve (see fig. 8). If available, the sedimentation should be done in a constant-temperature room.
2. Use the average temperature to determine the proper sedimentation time (table 3).
3. Prepare a 25-mL pipette with a rubber hand pump attached. The pipette must be cleaned with dichromate and rinsed with distilled H_2O. Become familiar with the use of the pipette before sampling; errors at this stage are simple to make and costly.
4. At the appropriate time, carefully sample at the 10-cm depth using the 25-mL pipette. Try to sample over a 10-12-s period. Empty the pipette into a labeled weighing jar. Rinse the pipette into the jar with distilled water. Continue this procedure on the remaining samples.
5. Evaporate the samples to dryness in a forced-draft oven at 105 °C (usually 24 hours). Place the jars, with lids open, in a desiccator to cool to room temperature. Before removing the jars from the desiccator, seal the jars with the lids. Measure and record the weights after wiping away fingerprints. Minimize the time the sample is exposed to ambient air by keeping all but the one sample being weighed in the covered desiccator. Reweigh the first sample to see if it adsorbed moisture; a weight change greater than 0.0005 g means that the samples must be redried and reweighed.
6. If <1-μm clay content is to be determined, do not remix the solutions. Instead, allow them to continue settling, taking temperature readings every 4 hours. Determine the average temperature from a graph as before; 28 hours from mixing is a typical sampling time. The sampling procedure is identical to that for <2-μm particles. Solution should be extracted at 10 cm below the original surface

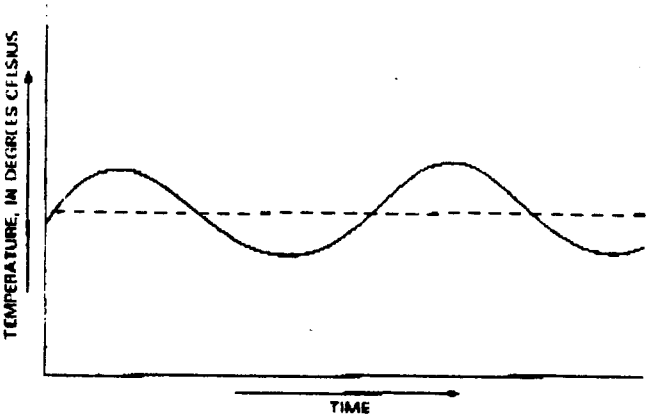

Figure 8. Example of temperature-time diagram used in particle-size analysis. Solid line indicates actual temperature; dashed line indicates average temperature.

Table 3. Settling times[1] for 2-, 5-, and 20-m particles to pass through 10 cm of water

[See Jackson (1969) for details on other temperatures and size fractions]

Temperature (°C)	2 μm (hour:min)	5 μm (hour:min)	20 μm (min:seconds)
20	8:00	1:17	4:48
21	7:49	1:15	4:41
22	7:38	1:13	4:35
23	7:27	1:11	4:38
24	7:17	1:10	4:22
25	7:70	1:08	4:16
26	6:57	1:07	4:10
27	6:48	1:05	4:04
28	6:39	1:04	4:00
29	6:31	1:03	3:55
30	6:22	1:01	3:49

[1]Assumes a particle density of 2.60.

because settling has been from that point. Drying and weighing are the same as in step 5.

7. Save the suspension until after the clay fraction calculations are completed in order to repeat analyses if necessary. A serious error has been made if the <1-μm fraction is larger than the <2-μm fraction.
8. If clay mineralogy is to be determined, resuspend the samples and take a sample which will supply a minimum of 50 mg of <2-μm material.

Sand Sieving

1. After cooling in a desiccator, weigh the sand fraction of each sample in the appropriate tared beaker.
2. Use a spatula to loosen them and transfer the sands to the top sieve in the sieve nest.
3. Shake the sands for 3 minutes on the mechanical shaker. Weigh and record the weights, save any fractions that are desired.

CALCULATIONS

1. Clay:

[weight jar + oven-dry sample] − [weight jar] − [average weight hexametaphosphate] = weight of particle fraction.

$$\frac{\text{weight of clay particle fraction}}{\text{weight of mineral fraction}} \times \frac{\text{1,000-mL solution}}{\text{25-mL sample}} \times 100$$

= percent clay-particle fraction in sample.

2. Sand:

$$\frac{\text{weight sand}}{\text{total weight of mineral fraction}} \times 100 = \text{percent sand in sample.}$$

3. Silt:

silt percent = 100 − (clay percent + sand percent)

ADDITIONAL REMARKS

We tested the efficacy of hand subsampling compared to using a sample splitter (table 4) and citrate-bicarbonate-dithionite (CBD) pretreatment (table 5). We found no significant differences in particle-size distribution between samples split or hand subsampled, and subsampling by hand was faster than sample splitting. There were some differences in particle-size distribution between pretreated and non pretreated samples. For example, percent sand was higher for sample 102, which received no CBD pretreatment, compared to pretreated samples. The same was not true for sample 132 (table 5; fig. 9). Because of the non-uniform effects caused by CBD pretreatment and because it is an additional and apparently unnecessary step in the procedure, we elected not to include it in our standard procedure.

Additional triplicate analyses further illustrate the level of precision which can be obtained by careful laboratory practice (table 6).

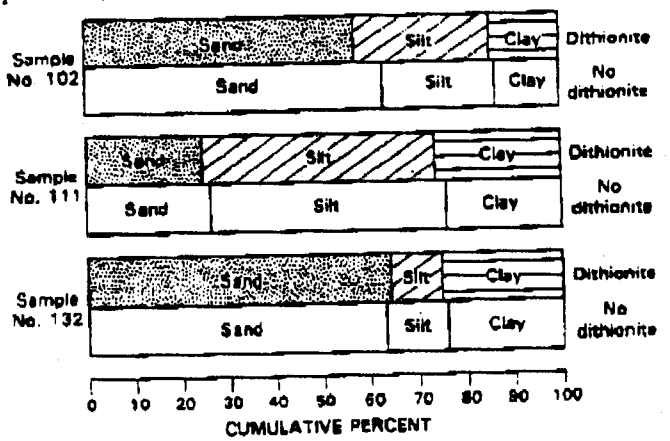

Figure 9. Effects of dithionite pretreatment on particle-size distribution. All samples were pretreated with Na hexametaphosphate; dithionite-treated samples were also extracted by citrate-bicarbonate-dithionite (CBD) reagent (Jackson, 1969). Replicates are indicated by a, b, c.

Table 4. Repeat determinations, in weight percent, of particle size for sample splitter vs. hand sampling

[111-1, 111-2, 111-3 sampled by spoon from carton of prepared soil after stirring contents of carton. 111-4, 111-5, 111-6 sampled using a sample splitter to obtain samples of desired size. Sample 111 is a BCt horizon (94-122 cm) from an R28 profile, Dry Creek chronosequence. $\bar{X}$, mean value for replicates; SD, standard deviation; CV, coefficient of variability (SD divided by $\bar{X}$)]

Sample	Oven-dry weight (g)	Oven-dry sand (g)	Sand	Silt	Clay		Sand				
					<2 μm	<1 μm	Very coarse	Coarse	Medium	Fine	Very fine
111-1	14.585	3.940	27.0	49.9	23.1	19.2	0.8	1.3	1.6	12.0	11.3
111-2	15.165	4.170	27.5	49.8	22.7	19.1	.6	1.5	1.7	12.1	11.6
111-3	14.745	4.060	27.5	49.5	23.0	19.1	.7	1.4	1.7	11.7	12.1
		$\bar{X}$	27.3	49.7	22.9	19.1	.7	1.4	1.6	11.9	11.6
		SD	.29	.21	.21	.06	.1	.1	.06	.04	.4
		CV	.01	.004	.009	.003	.1	.07	.03	.02	.03
111-4	14.355	3.900	27.2	49.6	23.2	19.2	.8	1.5	1.8	11.7	11.4
111-5	14.280	3.875	27.1	49.8	23.1	19.1	.7	1.5	1.8	11.9	11.3
111-6	14.500	3.905	26.9	50.1	23.0	19.1	.9	1.3	1.8	11.9	11.9
		$\bar{X}$	27.1	49.8	23.1	19.1	.8	1.4	1.8	11.8	11.5
		SD	.15	.25	.09	.06	.1	.1	.0	.11	.3
		CV	.005	.005	.004	.003	.1	.08	.0	.009	.03

Table 5. Effects of citrate-bicarbonate-dithionite pretreatment on particle-size distribution, in weight percent

[For each of the three samples: -1, -2, -3 received dithionite pretreatment; -4, -5, -6 did not. Sample 102 is a Bt2 (33-70 cm) from the M42 profile, 111 is a BCt (94-122 cm) from R28, and 132 is a Bt1 (33-80 cm) from the T11 profile, all from the Dry Creek chronosequence]

Sample	Oven-dry weight (g)	Oven-dry sand (g)	Sand	Silt	Clay		Sand				
					<2 μm	<1 μm	Very coarse	Coarse	Medium	Fine	Very fine
102-1	9.520	5.51	57.8	28.8	13.4	10.2	7.2	12.1	12.7	18.0	7.8
102-2	10.050	5.84	58.1	28.2	13.7	10.3	7.8	12.1	12.3	17.8	8.1
102-3	9.900	5.81	58.7	27.9	13.4	10.3	7.8	12.4	12.6	17.8	8.1
102-4	10.215	6.51	63.7	24.4	11.9	9.3	9.9	15.0	13.9	17.8	7.1
102-5	10.970	6.98	63.6	23.9	12.5	9.5	10.5	15.6	12.6	17.8	7.1
102-6	10.260	6.50	63.4	24.4	12.2	9.0	11.0	15.9	12.3	17.8	7.2
111-1	9.685	2.53	26.1	48.7	25.2	20.7	0.6	0.9	1.2	11.6	11.8
111-2	9.755	2.53	25.9	49.0	25.1	21.7	.6	1.1	1.5	11.5	11.2
111-3	10.210	2.63	25.7	48.9	25.4	21.4	.5	1.1	1.4	11.6	11.1
111-4	14.585	3.94	27.0	49.9	23.1	19.2	.8	1.3	1.6	12.0	11.3
111-5	15.165	4.17	27.5	50.2	22.7	19.1	.6	1.5	1.7	12.1	11.6
111-6	14.745	4.06	27.5	49.5	23.0	19.1	.7	1.4	1.7	11.7	12.1
132-1	11.025	7.11	64.5	10.4	25.1	24.3	8.2	22.0	13.2	14.7	6.4
132-2	10.710	7.00	65.4	10.6	24.0	22.6	9.6	22.4	13.0	14.7	5.7
132-3	10.780	7.04	65.2	10.7	24.1	22.5	9.7	21.7	13.0	14.9	5.9
132-4	10.450	6.88	65.9	10.5	23.6	23.1	9.5	22.7	13.4	14.6	5.7
132-5	10.690	7.00	65.5	10.7	23.8	22.3	9.8	22.6	13.0	14.2	5.8
132-6	10.835	6.56	60.5	18.6	20.9	20.6	10.9	21.4	11.4	11.9	5.0

Preparation of Soil Samples for X-Ray Diffraction Analysis

By Peter Janitzky,
University of California, Davis

REFERENCE

L. D. Whittig, written commun., 1983.

PRINCIPLE

A 10-20-mg clay sample is smeared upon the surface of

Table 6. Reproducibility, in weight percent, of particle-size analysis by the pipette method

[Replicates are indicted by a, b c. Samples are from Merced River chronosequence (Harden,1985). $\bar{X}$, mean value of replicates; SD, standard deviation; CV, coefficient of variability in percent=SD divided by $\bar{X}$. Percent standard error estimated by average CV for samples from any chronosequence that had replicate determinations]

Sample description		Sand	Silt	Clay <2 μm	Clay <1 μm	Sand: Very coarse	Sand: Coarse	Sand: Medium	Sand: Fine and very fine
Post-Modesto No.14 VI C3 (n)	a	96.9	2.4	0.7	0.4	3.9	57.2	25.6	10.2
	b	96.5	2.6	.9	.9	4.5	60.1	23.4	8.5
	c	96.1	3.3	.6	.4	5.0	58.6	23.9	8.6
	$\bar{X}$	96.5	2.7	.7	.6	4.5	58.6	24.3	9.1
	SD	.40	.47	.15	.29	.55	1.45	1.15	.95
	CV	.41	17.8	20.8	50.9	12.3	2.4	4.7	10.5
Post-Modesto No. 17 II C3 (ox)	a	64.0	29.4	6.6	4.9	0.0	.1	1.7	62.2
	b	63.2	29.7	7.1	4.5	.0	.1	1.1	62.0
	c	65.4	27.7	6.9	5.0	.1	.2	1.8	63.3
	$\bar{X}$	64.2	28.9	6.9	4.8	.03	.1	1.5	62.5
	SD	1.11	1.08	.25	.26	0.06	.06	.38	.70
	CV	1.7	3.7	3.7	5.5	173.2	43.3	24.7	1.12
Post-Modesto No.14-2 A12	a	56.0	35.0	9.0	5.8	.1	3.2	5.8	46.8
	b	55.3	37.2	7.5	5.5	.1	2.5	6.0	46.6
	c	48.7	42.9	8.4	6.3	.1	3.3	4.4	40.9
	$\bar{X}$	53.3	38.4	8.3	5.8	.1	3.0	5.4	44.8
	SD	4.02	4.07	.75	.40	.0	.43	.87	3.35
	CV	7.5	10.6	9.1	6.9	.0	14.5	16.1	7.5
Post-Modesto No. 14 A12	a	37.3	51.6	11.1	7.6	.1	1.4	2.6	38.2
	b	42.3	45.5	11.2	7.9	.1	1.0	2.3	39.9
	c	36.6	52.0	11.4	8.0	.1	1.4	1.9	33.2
	$\bar{X}$	40.7	49.7	11.2	7.8	.1	1.3	2.3	37.1
	SD	3.68	3.64	.15	.21	.0	.23	.35	3.48
	CV	16.1	7.3	1.3	2.6	.0	18.2	15.5	9.4
Percent standard error for samples	$\bar{X}$	6	10	11	17	46	12	6	7
	n	4	4	35	4	4	35	35	4

a porous ceramic plate. By means of a suction device, the clay material is successively saturated with the cations required in the various steps of X-ray analysis. The plate is reusable after cleaning.

EQUIPMENT

diamond saw and grinder for preparation of the ceramic plate

beaker, medium size

suction apparatus. This consists of a metal plate (45-cm long, 10-cm wide, 1-cm thick) supported by four short legs (5-cm long). The plate has lengthwise a row of 12 holes (15-mm diameter) at distances of 35 mm. A copper tube (2-cm diameter) with respective holes, soldered airtight to the bottom side of the plate, is connected to a suction flask and serves as a drain for the holes. Glued to the top side of the plate is a rubber gasket in which rectangles (25 by 18 mm) are cut for each hole. Rubber stoppers (No. 0) close all holes not occupied by the ceramic plates during the saturation procedure.

centrifuge

centrifuge tubes, 100 mL (2)

muffle furnace

spatula

REAGENTS AND MATERIALS

sodium chloride, saturated solution.

magnesium acetate, 1 *N* solution. Disolve 122 g of $Mg(CH_3COO)_2 \cdot 4H_2O$ in H_2O and dilute to 1,000 mL. Transfer some amount into a wash bottle for use. (Note: all solutions are made with distilled water.)

hydrochloric acid, pH 3.5. Set approx. 500 mL H_2O in a beaker on the magnetic stirrer, insert electrode of the pH meter, and add a few drops of 1 *N* HCl until a pH of 3.5 is reached. Store in wash bottle.

Field and Laboratory Procedures Used in a Soil Chronosequence Study

Michael J. Singer and Peter Janitzky, *Editors*

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To Steve Reneau	From E. McDonald	
Co./Dept. EES-1 LANL	Co. DRI	
Phone #	Phone #	
Fax # 505 665 3285	Fax # 702 673 7302	

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James M. Ray

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Date

James M. Ray

Authorizing Party (Please Print)