Pipette Method To Supplement Hydrometer Test for Particle-Size Determination in Soils

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IN investigations being carried on with loess soils, a comparatively simple test was desired to supplement the standard hydrometer test for determining particle-size distribution in the clay range below 0.005 mm. and for extending measurements below 0.001 mm. A pipette method with gravity settling was developed for this purpose. The method and illustrative results obtained in the combined pipette-hydrometer test are presented in this paper. Discussions of the basic principles of settlement analysis of soils and the significance of particle-size distribution in the clay ranges of the soils tested are also included.

● THE behavior characteristics of soils are governed to a large extent by the clay minerals present. Since particle-size distribution below 0.005 mm. is somewhat diagnostic of the kinds of clay minerals found in soils, measurements in this size range are important in the study of the clay fraction of soils.

To measure particle sizes in the silt and clay ranges down to 0.001 mm., the hydrometer test is used in civil-engineering soil laboratories because of its simplicity and rapidity. For such measurements in agricultural soil laboratories, the more complex and time-consuming pipette method (14) has been adopted internationally. Bouyoucos (2, 3, 4, 5, 6,) has reported close agreement between the results of the hydrometer and the pipette tests.

Particle-size determinations in the clay range finer than 0.001 mm. have been somewhat neglected due to the difficulty of measurement. The standard hydrometer test and the usual pipette methods employing gravity settling are unsatisfactory because of the long settling time necessary. A centrifuge is generally used to speed up settling, and such a centrifuge-pipette method is described by Steele and Bradfield (16). A centrifuge-hydrometer method was used by Norton and Speil (13) and Hauth and Davidson (9). In these methods the effects of swirling and currents in the centrifuge tube are questioned by some authorities. A special centrifuge with a sector-shaped tube was developed by Kamack (10) to achieve radial settling without

interference from the straight walls of the ordinary centrifuge tube. Marshall (11) developed a two-layer method involving two liquids in a centrifuge tube which, while shown to be accurate (12), is rather time consuming.

In the investigations being carried on with loess soils in the Soil Research Laboratory of the Iowa Engineering Experiment Station, a comparatively simple test was desired to supplement the standard hydrometer test for determining the particle-size distribution in the clay range below 0.005 mm. and for extending measurements below 0.001 mm. A pipette method with gravity settling was developed for this purpose. The method and some illustrative results obtained with loess soils are described in this paper.

BASIC PRINCIPLES OF SETTLEMENT ANALYSIS

The settling velocity of spheres of equal size, falling through a medium, may be calculated from Stokes' law; and from the velocity the distance and time of settling may be determined. In a soil suspension, the time is measured from when the suspension was last stirred, and the depth or distance of settling is measured from the surface of the suspension. The calculated diameter of particles falling through this distance is then the maximum diameter of particles remaining at that depth. Since smaller particles are still in suspension at that depth, their concentration is a measure of the percent of particles smaller than the

specified diameter. The hydrometer and pipette are used to measure this concentration of particles.

Hydrometer

Particle concentrations are determined with the hydrometer by measuring the specific gravity of the suspension. As seen in Figure 1, the hydrometer does not measure the specific gravity at any particular depth but through a range of depths in the suspension. Concentrations of larger and smaller particles lower and higher in the suspension will thus affect the reading, and the particle concentrations measured will be over a range
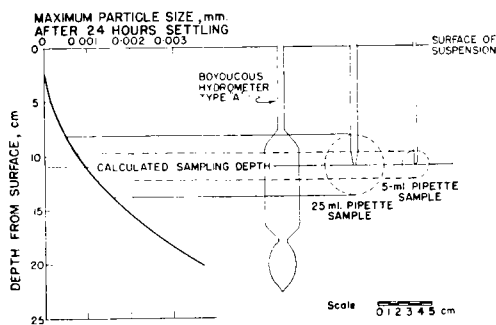

Figure 1. Graph shows particle size at left in depth ranges covered by hypothetical hydrometer and pipette measurements after 24 hr. of settling.

of particle sizes instead of being just one size. However, since particle-size accumulation curves in natural soils are usually smooth, this is not too serious an error. Hydrometers of the type illustrated (Fig. 1) have been calibrated on the basis of pipette tests (18), and there is fairly close agreement in results obtained with both.

Pipette

In the pipette test instead of measuring the specific gravity of the suspension, a sample of known volume is withdrawn at the specified depth; then it is dried and weighed. Although much criticism has been directed at the hydrometer test because it measures particle-size ranges, not particle sizes, it is usually overlooked that the pipette does this also (Fig. 1). A sphere of material is withdrawn from the suspension, but the smaller the pipette the smaller the range of depths sampled.

Measurement of Colloidal Sizes

A number of authors have pointed out that gravity settling of a suspension is not practical for particle-size determinations below 0.001 or possibly 0.0005 mm. due to the effect of Brownian movement and convection currents arising from slight changes in temperature. However, gravity sedimentation was used for determinations down to 0.0005 mm. by Steele and Bradfield (16), and Svedburg and Rinde (17) extended the lower limit to 0.0001 mm. in a study of colloidal suspensions of gold.

Colloidal particles in a suspension will reach equilibrium when the force of gravity on the particles is counterbalanced by the kinetic forces resulting from greater particle concentrations lower in the suspension. After equilibrium is reached there will be far fewer particles in suspension near the surface than deeper in the suspension. Therefore a shallow sampling depth will tend to minimize the effects of colloidal interference in the settling. In addition to the colloidal effect, other errors are present in unknown amounts. These errors include the assumption of a spherical particle shape in the application of Stokes' Law, and the uncertain effective specific gravity of the clay particles, which may vary with the amount of adsorbed water and the kind of clay mineral.

SUPPLEMENTAL PIPETTE METHOD

Apparatus

With the pipette apparatus described in this paper, the lower limit of the clay size range was extended to 0.0002 mm. A 5-ml. automatic pipette[1] fastened in a Shaw pipette rack[2] was used in this study. The rack was swivelled to a ring stand in such a way that sedimentation cylinders could be set in a circle around the stand (Fig. 2). This arrangement permits the settling of up to 12 suspensions at one time. The soils to be tested were dispersed according to the ASTM standard method (1) (ASTM Designation: D422-39) as modified by Davidson and Chu (8), using sodium metaphosphate as the dispersing agent. Standard 1000-ml. glass graduates are

[1] Available from A. S. Aloe Company, St. Louis, Missouri.
[2] Available from American Instrument Company, Silver Spring, Maryland.

used, and the air-jet dispersion apparatus of Chu and Davidson (7) is well adapted to the method.
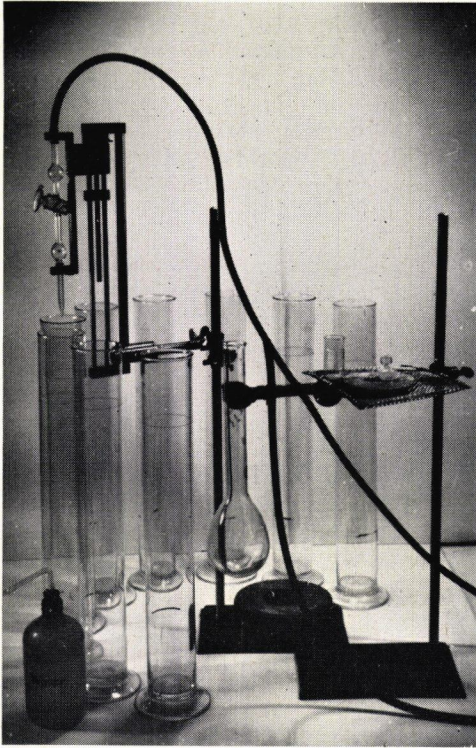

Figure 2. Circular arrangement of pipette apparatus for running nine tests simultaneously.

Settling Time and Sampling Depth

The time of settling for spherical particles of any diameter may be calculated from Stokes' law, expressed as

$$T = \frac{30\eta L}{980 \, d^2(G - G_1)},$$

where T is the time in minutes, η the viscosity of the settling medium in poises, L the depth in centimeters, d the particle diameter in millimeters, and G and G_1 the specific gravities of the particles and the settling medium respectively.

The specific gravity of the soil particles, G, should represent material in the size range being studied. Since only the specific gravity of the minus-10-sieve portion of the soil is

usually determined in engineering laboratories, this value is used in the ASTM standard hydrometer test. It may be used in the supplemental pipette test, and the error will probably be in keeping with the limitations of Stokes' law. In working with the clay fraction,
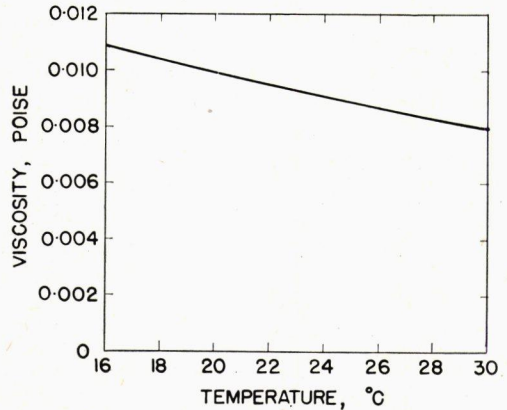

Figure 3. Relation of viscosity of water to temperature.
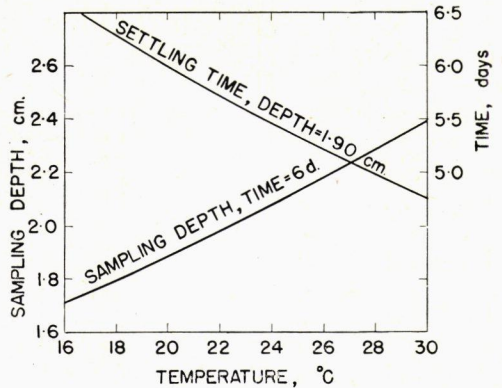

Figure 4. Settling time with a constant depth or sampling depth with a constant time as influenced by variations in temperature; d equals 0.0002 mm., G equals 2.70.

the error will vary with the types of clay minerals present.

Since η, the viscosity of water, varies with temperature (Fig. 3), adequate temperature control of the sedimentation cylinders is necessary. The control is either by means of a water bath or by keeping the cylinders in a room with a fairly constant temperature. Variations in temperature during the settling period may be compensated for by adjust-

ments of the settling time or of the sampling depth. This is illustrated in Figure 4 for the 0.0002 mm. clay determination. If the variation is not great, the average temperature over the settling period may be used in calculations. Ordinarily samples are taken at predetermined depths, and the time is varied to compensate for changes in temperature. With adequate temperature control this is not necessary. Following is an example of time and depth calculations from Stokes' law: If

$$T = \frac{30\eta L}{980 \, d^2(G - G_1)}$$

the average temperature throughout settling is 67 F. (19.4 C.), $\eta = 0.0102$ poise. If $d = 0.002$ mm., $L = 5$ cm., $G = 2.60$,

$$T = \frac{30(0.0102)(5)}{980(0.002)^2(2.60 - 1.00)}$$

$$= 244 \text{ min.} = 4 \text{ hr. } 4 \text{ min.}$$

Sampling

Sampling with the pipette may be done at shallow depths to lessen the settling time. However, since a sphere of material is withdrawn from the suspension, the sampling depths must be at least equal to the radius of the sphere. A small-volume pipette will thus allow shallower sampling depths. Since weighing errors will increase with decreasing sample size, a 5-ml. pipette is probably the minimum practical size. Table 1 shows a typical sampling schedule for pipette analysis with a 5-ml. pipette.

Use of the Automatic Pipette

Before using the pipette it is calibrated with mercury to determine its exact capacity. The pipette is filled to the level of the stopcock. Turning the stopcock 180 deg. allows the material to flow out.

In the pipette method, as previously discussed, samples are taken at predetermined depths. In order to obtain a uniform soil-water mixture at the start of the settling period the suspension is shaken by hand in 1000-ml. glass graduates according to the ASTM standard method (1).

The Shaw pipette rack (Fig. 5) provides an accurate method of lowering the pipette a set distance into the suspension. When the pipette is at the desired sampling depth,

suction is applied by means of an aspirator bottle adjusted so that the filling time is about 8 sec. Suction can also be applied by

TABLE 1
A SAMPLING SCHEDULE FOR PIPETTE ANALYSIS

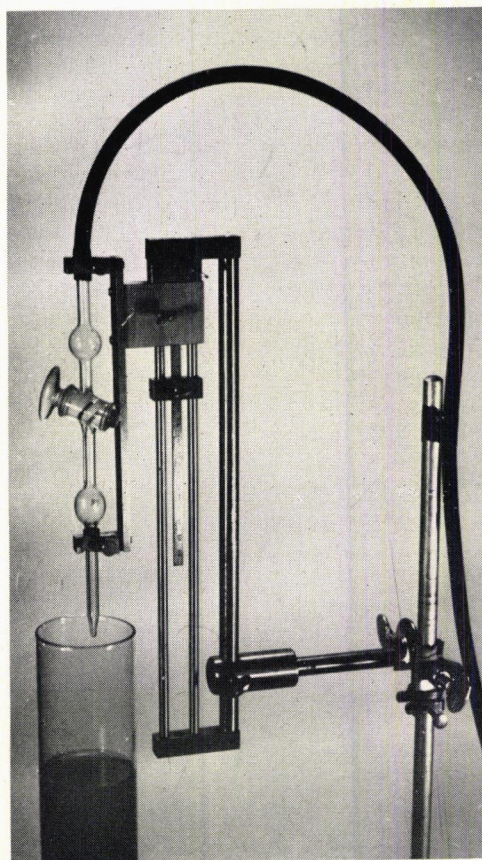| Equivalent spherical particle diameter	Average temperature	Sampling depth	Settling time
mm.	C.	cm.	
0.002	20	5.0	3 hr. 46 min.
0.001	20	3.0	9 hr. 5 min.
0.0005	20	2.0	24 hr. 10 min.
0.0002	20	1.9	6 days

Figure 5. The 5-ml. automatic pipette in position in a Shaw pipette rack.

means of the mouth. Because successively shallower sampling depths are used, reshaking the suspension between samplings was

found to be unnecessary. Considerable time is saved by this procedure. Reshaking between sampling was also omitted in the methods of Steele and Bradfield (*16*) and Rittenhouse (*15*).

After the pipette is filled and withdrawn from the soil suspension, it is emptied into a 12- or 15-ml. weighing bottle. Particles clinging to the inside of the pipette are washed into the bottle with distilled water. The pipette is rinsed with acetone to speed drying and is connected to an aspirator or vacuum line to dry it. Weighing bottles must be washed, oven-dried and weighed again before reusing.

The filled weighing bottle, after being dried in a constant-temperature (105 to 110 C.) oven, is weighed on an analytical balance. The oven-dry weight of the sample must be corrected for the amount of dispersing agent present. Since sodium metaphosphate remains hydrated at oven dryness, the correction may be obtained by oven drying a pipetteful of sodium-metaphosphate solution of the same concentration used in the soil suspension. The corrected weight of the sample can then be obtained and used to calculate the percentage by weight of particles finer than the maximum particle size measured. The calculation procedure is illustrated by the following example:

Total wt. of air-dry soil used in test	50.0 gm.
Hygroscopic moisture content	5.00%
Wt. of soil in an oven-dry condition	47.6 gm.

The pipette sample was taken to measure material finer than 0.002 mm:

| Wt. of oven-dried pipette sample and bottle	19.1644 gm.
Oven-dry wt. of bottle	19.0722 gm.
Wt. of oven-dry pipette sample	0.0922 gm.
Calibrated volume of the pipette	5.025 ml.
Oven-dry wt. of dispersing agent in 5.025 ml. of solution	0.0159 gm.
Wt. of oven-dry soil in pipette sample	0.0922 − 0.0159 = 0.0763 gm.
Oven-dry wt. of soil particles finer than 0.002 mm. in the whole suspension	$(0.0763)\dfrac{1000}{5.025} = 15.19$ gm.
Percent by weight of the soil particles finer than 0.002 mm.	$(100)\dfrac{15.19}{47.6} = 31.8\%$
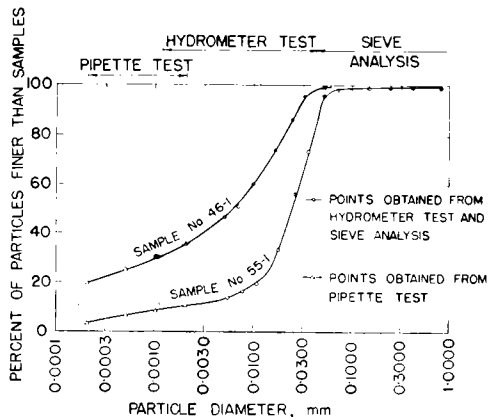
Figure 6. Particle-size accumulation curves for soils tested which had the lowest (No. 55-1) and highest (No. 46-1) clay contents.

TABLE 2
CLAY CONTENTS BY HYDROMETER AND PIPETTE TESTS[a]

Sample No.	Clay Contents from Hydrometer Test, % oven-dry wt.[b]		Clay Contents from Pipette Test, % oven-dry wt.			
	0.002 mm.	0.001 mm.	0.002 mm.	0.001 mm.	0.0005 mm.	0.0002 mm.
55-1	12.0	11.4	10.5	8.6	6.5	3.8
20-2	16.0	14.5	15.4	12.7	9.3	4.0
61-2	19.6	18.0	18.8	15.7	11.2	6.9
26-1	22.4	20.0	21.5	18.1	14.2	8.3
26-2	23.1	19.8	23.4	19.8	15.1	7.8
29-1	25.0	22.2	24.0	20.7	16.6	11.4
36-1	28.9	25.0	28.4	24.6	20.5	14.3
43½-1	33.0	29.8	31.4	27.6	23.2	17.3
46-1	36.2	32.0	35.3	31.2	25.4	19.8
49-3	31.4	28.4	28.2	25.6	20.5	12.8
49-4	24.6	22.4	22.8	19.7	15.8	10.5

[a] All clay contents are in terms of percent finer than particle diameter indicated.
[b] All hydrometer test values are interpolated from particle-size accumulation curves.
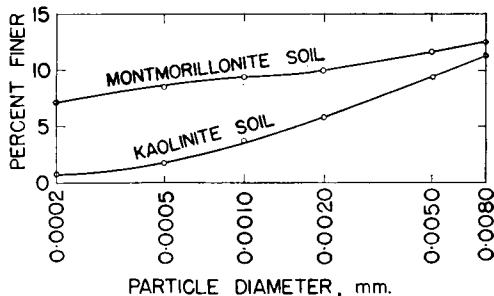

Figure 7. Particle-size accumulation curves for the clay range of synthetic montmorillonite and kaolinite soils.

ILLUSTRATIVE RESULTS OF COMBINED
PIPETTE-HYDROMETER ANALYSIS

Particle-size accumulation curves for two loess soils with high and low clay contents are presented in Figure 6. The smooth transition between hydrometer and pipette test data is shown. Table 2 presents a comparison of hydrometer and pipette test values for a

the results checked within 1 percent. In normal testing procedure duplicate tests would not be necessary.)

As previously mentioned, the shapes of particle-size accumulation curves in the clay range should be an indication of the types of clay minerals present. The particle-size curves in Figure 7 illustrate the effect the kind of clay
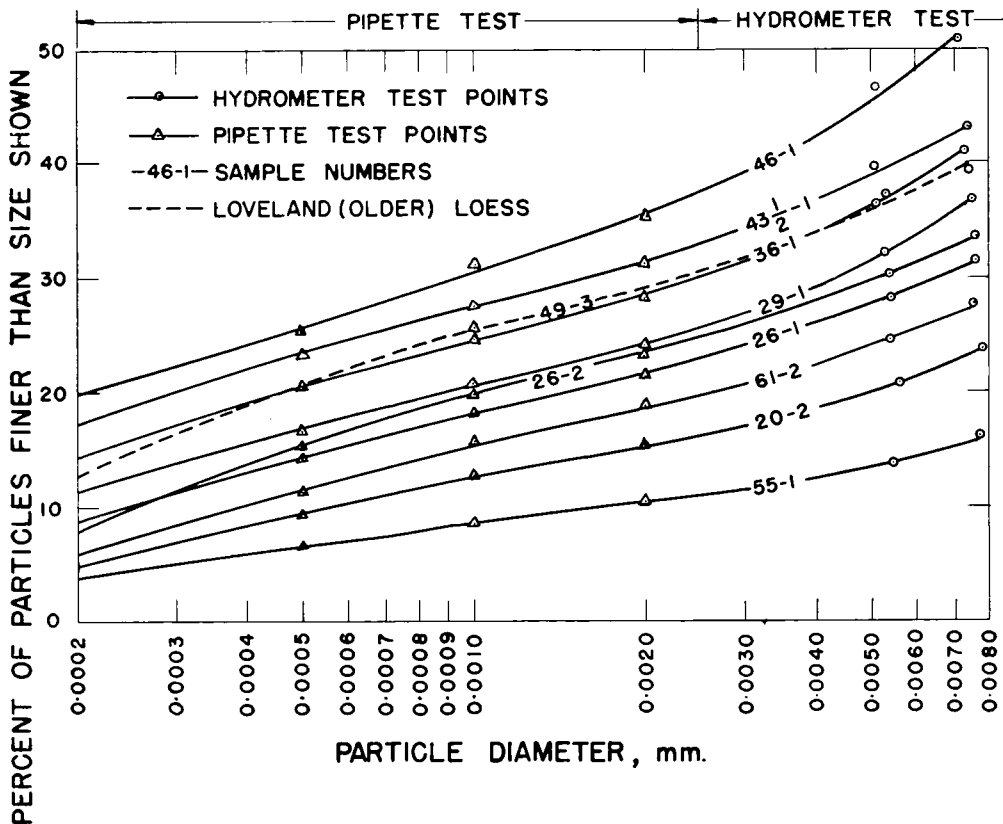

Figure 8. Particle-size accumulation curves for the clay range of some of the loess tested.

number of loess soils. The data in the range where the test methods overlap show close agreement. Corresponding values taken from the hydrometer tests and from the pipette tests for the 0.002 mm. clay vary up to 3.2 per cent, the average difference in values being 1.2 percent. In the 0.001-mm. clay size, the average difference is 1.7 percent. For both the 0.001-mm. and 0.002-mm. sizes the hydrometer test values tend to be higher. (All pipette tests were run in duplicate, and

mineral has on the particle-size distribution of two synthetic soils so made as to have approximately equal amounts of 0.005-mm. clay. The clay minerals in the two soils are kaolinite in one and montmorillonite in the other, and the nonclay portions of both are cleaned quartz. Much more work is needed with relatively pure clay minerals and known mixtures of clay minerals before the significance of the shape of the particle-size curve in the clay range of soils can be fully interpreted.

This approach to clay-mineral identification should prove to be a useful supplement to other identification methods.

Particle-size accumulation curves for the clay range of some of the loess soils tested by the combined pipette-hydrometer method are presented in Figure 8. With two exceptions (Samples 49-3 and 26-2) the shapes of the curves are markedly similar, though they represent soils having 0.005-mm. clay contents varying from 13 to 45 percent. From differential thermal analyses (*9*), the clay minerals in these soils are the same, being probably illite or a mixture of illite and montmorillonite. The shapes of the particle-size curves in Figure 8 appear to confirm this.

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TABLE 3

PROPERTIES OF SOILS TESTED WHICH MAY AFFECT MECHANICAL ANALYSIS

Sample No.	Material	Age	Location (All in SW Iowa) (County)	Sampling Depth	P.I.	Cation Exchange Capacity	Organic Matter	CaCO₃ Content	Engineering Classification (AASHO)
				ft.	%	me./100 gm.	%	%	
55-1	Loess	Upper Wisconsin (?)	Harrison	2-3	2.3	11.2	0.24	11.0	A-4 (8)
20-2	Loess	Wisconsin	Harrison	39-40	6.2	13.4	0.17	10.2	A-4 (8)
61-2	Loess	Wisconsin	Ida	17-18	10.8	14.2	0.15	11.5	A-6 (8)
26-1	Loess	Wisconsin	Shelby	4-5	12.5	18.2	0.18	1.4	A-6 (9)
26-2	Loess	Wisconsin	Shelby	10-11	17.8	17.9	0.17	3.6	A-6 (9)
29-1	Loess	Wisconsin	Audubon	5-6	18.0	19.5	0.25	2.9	A-6 (11)
36-1	Loess	Wisconsin	Montgomery	5½-6½	20.8	21.0	0.21	1.8	A-7-6 (13)
43½-1	Loess	Wisconsin	Fremont	4½-5½	33.4	24.4	0.37	0.5	A-7-6 (18)
46-1	Loess	Wisconsin	Page	5-6	32.7	22.6	0.30	1.5	A-7-6 (19)
49-3	Weathered Loess Soil	Loveland	Pottawattamie	55-56	24.6	22.7	0.19	1.2	A-7-6 (15)
49-4	Loess	Loveland	Pottawattamie	65-66	15.3	16.6	0.11	7.0	A-6 (10)

Sample 26-2 differs in that it is gray-colored and unoxidized whereas the other samples are buff-colored and oxidized. Sample 49-3 is an older, highly weathered soil. Other data which may affect size analysis of these soils are presented in Table 3.

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Laboratory Compaction Tests of Coarse-Graded Paving and Embankment Materials

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THIS report presents results of a laboratory study of the applicability of the Proctor type of compaction test to the compaction of coarse-graded materials. Three representative granular materials (gravel, limestone, and slag) were tested through a range of carefully controlled artificial gradations in which the ratio of coarse to fine fractions was varied.

These tests indicate that a modified Proctor procedure can be used successfully to compact coarse-graded materials in either a 4- or 6-in. diameter mold. The maximum density of mixtures of coarse and fine materials increased with increasing percentages of coarse material, up to a point of optimum gradation. Beyond this point the further addition of coarse material resulted in lower densities. The optimum gradation for the materials used in this study occurred when the mixtures contained about 40 to 60 percent of material retained on the No. 4 sieve. When the mixture contained more than 80 percent of coarse material, results obtained from the Proctor compaction test were erratic.

Degradation of coarse aggregate during compaction increased with increasing percentages of coarse material. Breakage in the 4-in. mold was negligible when the plus-4 material was less than 30 percent.

The increase in maximum density which is gained by adding coarse materials to fine-graded mixtures can be predicted by a correction formula applied to the density of the finer, or minus-4, portion of the material. Correction formulas were not applicable to mixtures coarser than the optimum gradation.

● IN the standard Proctor soil compaction test (ASTM Designation D698-42T) all of the particles retained on the No. 4 sieve are removed before the sample is compacted. Proctor test results, therefore, cannot be translated directly into specification require-