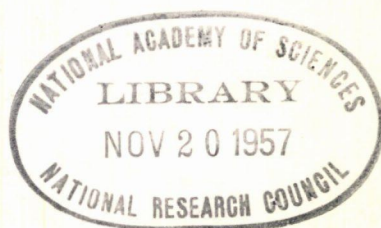


HIGHWAY RESEARCH BOARD
Bulletin 95

Laboratory Analysis of Soils
Grain Size and Liquid Limit



National Academy of Sciences—
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Grain Size and Liquid Limit

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Dispersing Agents for Particle-Size Analysis of Soils

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In the particle-size analysis of soils, mechanical agitation and the physico-chemical action of a dispersing agent are employed to disperse a soil sample in water and prepare a suspension for subsequent determination of particle size-groups by sedimentation techniques. The dispersing agent fulfills two functions vital to the accuracy of the analysis: (1) assisting the mechanical treatment in the initial separation of naturally cemented or aggregated prime particles and (2) providing conditions for normal sedimentation in the suspension during the test period, by preventing flocculation (or other interfering effects) of the dispersed particles.

Many different types of chemical dispersing agents have been employed with varying results. At present, sodium metasilicate is widely used in soil testing for engineering purposes, and sodium hexametaphosphate, sodium hydroxide, and sodium carbonate are among those used for agricultural soils. However, the reported results of numerous studies do not afford a clear evaluation of effectiveness, because of the lack of consistency in methods and techniques and, frequently, the narrow scope of experimental soils and dispersants. The investigation reported here was undertaken to provide this needed evaluation.

Many different dispersing agents were tested with a broad selection of soils under closely controlled conditions of analysis. Results showed sodium metasilicate to be relatively ineffectual in the dispersion of several soils and not suitable for general use. Several others, such as the carbonate, hydroxide, and oxalate of sodium, produced a high degree of dispersion with a few soils but failed to stabilize the suspensions of others. Sodium polyphosphos and sodium tripolyphosphate were highly and about equally effective for all of the experimental soils representing many of the great soil groups of the United States. Two others, sodium hexametaphosphate and sodium tetraphosphate, were only slightly less so. None of these four phosphates was effective with a laterite material, a red, ferruginous soil of the tropics. Conversely, this soil was highly dispersed by trisodium phosphate and tetrasodium pyrophosphate, which were generally ineffective with members of the other soil groups.

● **PARTICLE-size analysis** is one of the oldest and most-valuable test methods for soil materials. Its purpose is to determine the quantitative distribution of prime particles in several selected size-groups or separates, such as sands, silt, and clay. The resulting particle-size distribution data have long been used to characterize the physical makeup, or texture, of soils and have been especially important in soil classification for highway and other purposes.

Although details of analytical procedures vary considerably among different investigators, a required feature of all methods for particle-size analysis

is the dispersion of the soil sample. A satisfactory dispersion requires: (1) complete separation of the prime particles of the soil or the reduction of agglomerates to sizes not larger than those of the smallest size-group to be determined and (2) maintenance of the resulting soil suspension in a condition satisfactory for subsequent particle-size determinations. In methods now in general use, dispersion is accomplished by mechanical agitation of a soil-water mixture, in conjunction with an added chemical dispersing agent. Silt and clay-size groups are determined from the resulting soil-water suspension by sedimentation techniques involving

either hydrometer readings or pipetting and drying of aliquots. Sand groups are determined by standard sieving techniques.

The accuracy of the analysis is strongly affected by the degree to which the mechanical agitation and the dispersing agent satisfy the above two requirements. Mechanical dispersion is accomplished by stirring or shaking the soil-water mixture; a high-speed motor-driven stirrer has been widely used for this purpose in soil laboratories for many years. A more-recently developed air-jet device (12) has been shown to produce an even-more-complete dispersion and also to minimize the attrition of prime particles which may occur with even a brief treatment with the motor-driven stirrer. The use of either device is now optional in ASSHO Method T 88-49 (10).

The present investigation was primarily concerned with the second requirement, or chemical phase, which has been far less well resolved. This concerns the physico-chemical activity of the dispersing agent in the soil-water mixture, which assists in the initial mechanical dispersion but is more-particularly required for the maintenance of the resulting suspension. The problems involved in this part of the dispersion process result from inorganic and organic substances of clay and colloidal sizes. These adhere to and bind each other, as well as some of the larger particles, into aggregate structures which resist separation. In most highway-soil materials, the clay and colloidal particles are largely inorganic in nature and consist chiefly of minute crystallites of one or more members of the clay mineral group. The crystal structures contain an excess of negative charges which attract positively-charged ions, such as H^+ , Ca^{++} , and Na^+ . These, in turn, influence the tendency of the clay particles either to be dispersed or to remain in aggregates or floccules. The hydrogen and calcium forms of clays resist dispersion, whereas the particles of sodium clays are separated with relative ease.

Further, a dispersed suspension of soil particles in water may manifest colloidal properties, such as gelation (syneresis) or coagulation and flocculation. Coagulation and flocculation hasten particle sedimentation, and gelation retards it. In the first case, hydrometer readings made in the soil suspension for particle-

size measurements yield low results for the finer particles, and the analysis makes the soil appear coarser than it actually is; in the second, the reverse is true.

Most dispersing agents provide sodium ions to replace the flocculating cations from the soil clay and anions, such as hydroxyl, oxalate, carbonate, phosphate, or silicate, to inactivate the replaced cations. Or if instead of an electrolyte, a lyocratic (protective) colloid such as gum acacia is used, dispersion may be aided and the suspension maintained by the action of the colloid in forming protective layers around the clay particles. As often happens in practice, the dispersing agent may fail to provide these effects, with the result that particle sedimentation is interfered with and measurements of the silt and clay fractions are invalidated.

In the long history of particle-size analysis, many different chemical dispersants have been used with varying success and acceptance. Among the more common are: ammonium hydroxide and the carbonate, oxalate, and hydroxide of sodium. Sodium hexametaphosphate is now widely used with agricultural soils (6 and 11), and AASSHO Method T 88-49 (10) specifies sodium metasilicate. The extensive literature reveals the extreme variability under which analyses have been made, including: experimental procedures and apparatus; types and concentrations of electrolytes; types and amounts of soils; and pretreatments of samples for removal of organic matter, soluble salts, or exchangeable bases. Rarely are comparative data presented for more than one or two dispersing agents, and the soils studied are usually severely limited in number and variety. Thus, for the most part, the literature reveals an inadequate testing and evaluation of dispersing agents with regard to degree of dispersion obtained and to suitability for use with a wide variety of soils. It can only be concluded that, in general, the several dispersing agents used in the past vary considerably in both respects. Some are of severely limited value and none has been found suitable for use with all soils.

This report presents the principal findings of a study designed to evaluate the effectiveness of a large group of dispersing agents with a wide variety of soils under closely controlled conditions of anal-

ysis. For purposes of comparison, data obtained by the use of AASHTO Method T 88-49 are also presented. The criterion adopted for the degree of dispersion obtained is the percentage value for the size-group of particles having a diameter of one micron (0.001 mm.) and less. This is determined from the 24-hour hydrometer reading and is termed "the dispersion value." This size group was selected because it is the smallest now determined in particle-size analyses for highway purposes. Further, its upper limit is well below the 2-micron value commonly accepted for the clay fraction.

MATERIALS AND METHODS

Dispersing Agents

The 19 dispersing agents selected for experimental study are listed in Table 1, divided among seven subgroups according to chemical composition. These include most of the materials which have been widely used for soil dispersion. A few of the phosphates are new to soil-testing; others, such as Lomar PW and Marasperse CB, have not been employed with soils but are finding application as industrial dispersing agents.

With a few exceptions, the chemical-reagent grade of each material was used. Sodium tripolyphosphate and sodium polyphosphates were of technical grade, and gum acacia and gum ghatti were of pharmaceutical quality. Although the grade of Marasperse CB is not known, its manufacturer has stated that it is a highly purified product. All are readily available at a cost not too high to prohibit their use in routine laboratory work.

Soils

The soils chosen for analysis are listed and described in Table 2. In addition to the soil names, when known, their liquid and plastic limits and classifications, as derived from the limit values and the percentages passing the No. 40 and No. 200 sieves, are given as further descriptive information. All of the samples are from the B or C horizons, since this is the part of the soil profile usually used in highway construction.

The soils selected represent a fairly wide range of genetical conditions and in-

clude members of many of the great soil groups of the world. Only one of the samples, Nipe No. 6, is from outside the United States. This is a true laterite, a soil group common to the tropics, but not found in this country. Two clay mineral samples, a bentonite and a kaolin, were included for testing, since they are relatively pure examples of two of the three chief clay mineral groups common to soils. An illite sample was not available in sufficient quantity for the testing of this clay mineral group as such, but illite occurs as a major clay mineral constituent in some of the soils included in the list.

Apparatus

Samples were dispersed (at an air pressure of 20 psi. from a laboratory compressed-air line) with a Model A, air-jet dispersion cup which has been described in a previous report (12) and approved for use in AASHTO Method T 88-49 (10).

Densities of the soil suspensions were measured with an early model of the streamlined Bouyoucos hydrometer, calibrated for use at 67 F. Density readings were corrected for deviations from this temperature and for the presence of the dispersing agent.

Analytical Procedure and Methods

Essentially, the experimental work consisted of the following tests: (1) Soils 1 to 6, dispersed in the air-jet dispersion cup with each of 19 dispersants which were added in 5 or 10-ml. increments of stock solution to replicate portions of each soil. (2) Soils 7 to 14 and the clay minerals 15 and 16, dispersed in the air-jet dispersion cup with a single concentration of each of the phosphates. (3) Soils 1 to 14, dispersed with the motor-stirrer and sodium metasilicate solution.

For those tests made by the use of the air-jet dispersion cup, a 50-gram portion of air-dry soil passing the 2-mm. sieve was soaked overnight in a beaker containing about 75 ml. of distilled water. The mixture was then transferred to the dispersion cup with the use of an additional 75 ml. of water from a wash bottle; the predetermined volume of dispersant stock solution was added; and with the air-pressure adjusted to 20 psi., the mixture was dispersed for 5 minutes with soils having a plasticity

TABLE 1
EXPERIMENTAL DISPERSING AGENTS

No.	Name	Formula	Stock solution concentration	Type of reaction
1	Sodium metasilicate (sodium silicate)	$\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$	0.4N (56.8) ^f	Supplies dispersive cation and alkaline reaction Hydrolyzes to silicic acid which acts as protective colloid (3) and precipitates Ca and Mg
2	Sodium hexametaphosphate (Calgon, buffered with Na_2CO_3)	NaPO_3 or $(\text{NaPO}_3)_6$	0.4N (40.8)	Forms soluble undissociated complexes with many cations and prevents flocculation effects Also supplies a dispersive cation
3	Trisodium phosphate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	0.4N (50.7)	Do.
4	Tetrasodium pyrophosphate	$\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$	0.4N (44.6)	Do.
5	Sodium tripolyphosphate ^e	$\text{Na}_5\text{P}_3\text{O}_{10}$	0.4N (29.4)	Do.
6	Sodium tetrphosphate ^a	$\text{Na}_6\text{P}_4\text{O}_{13}$	0.4N (31.3)	Do.
7	Sodium polyphos ^e	$\text{Na}_{12}\text{P}_{10}\text{O}_{31}$	0.4N (36.1)	Do.
8	Victamide	^b	0.4N (38.6)	Do.
9	Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	0.4N (26.8)	Oxalate ion precipitates Ca and Mg. Supplies dispersive cation
10	Lithium oxalate	$\text{Li}_2\text{C}_2\text{O}_4$	0.4N (20.4)	Do.
11	Ammonium oxalate	$(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$	0.4N (28.4)	Do.
12	Sodium hydroxide	NaOH	0.4N (16.0)	Supplies dispersive cation and alkaline reaction
13	Lithium hydroxide	LiOH	0.4N (9.6)	Do.
14	Sodium carbonate	Na_2CO_3	0.4N (21.2)	Do.
15	Lithium carbonate	Li_2CO_3	0.1N (3.7)	Do.
16	Gum acacia	Complex colloidal polysaccharide	0.13% (1.3)	Prevents coalescence of particles by providing protective hydration layer.
17	Gum ghatti	Do	0.13% (1.3)	Do.
18	Lomar PW	Complex sulfonate ^c	0.4N (95.3)	Produces repelling negative charges on suspended particles
19	Marasperse CB	Complex sulfonate ^d	0.33% (3.3)	Do.

^a Sodium tetrphosphate is sold under the name of "Quadrafos" by Rumford Chemical Works, Rumford, N. J.

^b Victamide is an ammonium amido-polyphosphate furnished by the Victor Chemical Works, Chicago, Ill. Its exact formula has not been released but $(\text{NH}_4)_2\text{P}_2\text{O}_5 \cdot \text{NH} \cdot \text{NHP}_2\text{O}_5(\text{NH}_4)_2$ was used to calculate the weight needed for a 0.4N stock solution.

^c Lomar PW is the sodium salt of a condensed mono-naphthalene sulfonic acid, furnished by Jaques & Co., Passaic, N. J. The formula $(\text{C}_{10}\text{H}_7\text{SO}_3\text{NaCH}_2)_2$, $\text{C}_{10}\text{H}_7\text{SO}_3\text{Na}$ was used to calculate the weight needed for a 0.4N stock solution.

^d Marasperse CB is an indefinite mixture of partially desulfonated sodium lignosulfonates furnished by the Marathon Corp., Rothschild, Wisconsin

^e Sodium tripolyphosphate and sodium polyphosphate are products of the fusion of various combinations of phosphates, purchased from Blockson Chemical Co., Joliet, Ill

^f Figures in parentheses indicate grams of dispersing agent per liter of stock solution.

index of 5 and less, 10 minutes with those between 6 and 20, and 15 minutes with those greater than 20. These dispersion times were established previously (12) from tests of a large number of soils. The dispersed mixture was then transferred to a 1,000-ml. volumetric cylinder and made up to volume.

In accord with the details of the AASHO

method, hydrometer readings were made in the suspension at 2-, 60-, and 1,440-minute intervals for the silt, clay, and "colloid" size-groups and the mixture was then passed through standard sieves for separation of the sand groups. Sediment volumes were recorded at the time of the hydrometer readings and the suspensions were examined visually for evidence of

TABLE 2
EXPERIMENTAL SOILS

Soil No	Series Name	Soil group	Geographical source	Liquid Limit	Plasticity Index	pH (1-1 mix)	HRB classification and group index
1	Iredell	Yellow podzolic	Fairfax Co., Va.	88	62	5.7	A-7-6(20)
2	Cecil	Red podzolic	Lee Co., Ala.	63	22	4.7	A-7-5(16)
3	Tenn. I ^a	Red podzolic	Sevier Co., Tenn.	37	8	5.1	A-4(6)
4	Tenn. II ^a	Yellow podzolic	Sevier Co., Tenn.	38	9	4.9	A-4(6)
5	Sharkey	Alluvium from podzolic materials	Knox Co., Ind.	47	24	6.7	A-7-6(15)
6	Nipe	Laterite	Mayaguez, P.R.	44	13	4.9	A-7-5(10)
7	Miami ^a	Gray-brown podzolic	Tippecanoe Co., Ind.	33	18	7.6	A-6(10)
8	Washo ^a	Lake sediment in arid climate	Oneida Co., Idaho	35	9	7.8	A-4(8)
9	Houston	Rendzina (blackland)	Delta Co., Texas	54	32	7.0	A-7-6(19)
10	Yazoo	Alluvium of lower Miss. Valley	Hinds Co., Miss.	91	65	7.8	A-7-6(20)
11	Wabash	Alluvium of Mississippi Delta	Madison Parish, La	68	40	6.7	A-7-6(20)
12	Salt Flat ^a	Alkali (high Na ₂ SO ₄)	Lyon Co., Nev	58	35	8.6	A-7-6(20)
13	Salt Flat II ^a	Alkali (high NaCl)	Churchill Co., Nev	23	8	9.4	A-4(8)
14	Putnam	Planosol (clay layer accumulation)	Missouri	50	30	6.5	A-7-6(18)
15	Bentonite	Clay mineral montmorillonite	Wyoming	347	303	8.6	A-7-5(20)
16	Kaolin	Clay mineral kaolinite	Georgia	49	15	8.7	A-7-5(12)

^a Series name not known, soil occurs in unmapped area.

TABLE 3
EFFECTIVENESS OF 19 DISPERSING AGENTS WITH SOILS 1 TO 6

Dispersing agent		Dispersion values (% of the <.001-mm group) obtained with the air-jet dispersion cup at the optimum concentrations of the dispersing agents						Average deviation from the maximum dispersion value (percentage-points)	
No.	Name	Soil No						Podzolic soils Nos 1-5	
		1	2	3	4	5	6		
1	Sodium metasilicate	46 ^a (11)	33 (3)	7 (3)	6 (2)	28 (8)	51 (10)	5.4	
2	Sodium hexametaphosphate (Calgon)	55 (2)	36 (0)	10 (0)	7 (1)	34 (2)	52 (9)	1.0	
3	Trisodium phosphate	52 (5)	F ^b	F	F	30 (6)	61 (0)	F	
4	Tetrasodium pyrophosphate	53 (4)	34 (2)	8 (2)	5 (3)	33 (3)	60 (1)	2.8	
5	Sodium tripolyphosphate	54 (3)	35 (1)	9 (1)	7 (1)	35 (1)	56 (5)	1.4	
6	Sodium tetraphosphate	55 (2)	36 (0)	10 (0)	8 (0)	33 (3)	52 (9)	1.0	
7	Sodium polyphos	56 (1)	33 (3)	8 (2)	6 (2)	34 (2)	48 (13)	2.0	
8	Victamide	54 (3)	34 (2)	ND ^c	ND	ND	42 (19)	2.5	
9	Sodium oxalate	53 (4)	F	F	5 (3)	32 (4)	F	F	
10	Lithium oxalate	52 (5)	F	F	3 (5)	34 (2)	F	F	
11	Ammonium oxalate	49 (8)	F	F	4 (4)	34 (2)	F	F	
12	Sodium hydroxide	54 (3)	F	F	4 (4)	34 (2)	F	F	
13	Lithium hydroxide	53 (4)	34 (2)	F	3 (5)	36 (0)	59 (2)	F	
14	Sodium carbonate	50 (7)	F	F	3 (5)	33 (3)	57 (4)	F	
15	Lithium carbonate	57 (0)	F	F	3 (5)	33 (3)	41 (20)	F	
16	Gum acacia	F	16 (20)	5 (5)	4 (4)	21 (15)	F	F	
17	Gum ghatti	44 (13)	15 (21)	9 (1)	5 (3)	23 (13)	14 (47)	10.2	
18	Lomar PW	F	F	9 (1)	5 (3)	F	44 (17)	F	
19	Marasperse CB	52 (5)	22 (14)	9 (1)	7 (1)	28 (8)	43 (18)	5.8	
AASHTO Method with sodium metasilicate and 1 min. in motor stirrer		29 (31)	22 (14)	5 (5)	5 (3)	13 (23)	20 (41)	15.2	

^a All numbers in parentheses refer to the deviation (in percentage-points) of the given dispersion value from the maximum obtained for that soil

^b F - indicates failure of the test due to flocculation or other colloidal effect.

^c ND - value not determined.

flocculation or other interfering effects; pH measurements were obtained for each soil (1-to-1 soil-water mixture), dispersant solution, and soil-water-dispersant mixture, using a glass electrode and a Beckman Model G pH meter.

For the tests made with the motor-stirrer, the mixture was given a 1-minute dispersion in the prescribed cup of this device with 20 ml. of a 3-deg. -Baumé (approximately 0.2N) solution of sodium metasilicate. Otherwise, the details of the analysis were the same as those given above.

Stock solutions of those dispersing agents having a definite chemical formula were 0.4N, except that of Li_2CO_3 , which was limited to 0.1N by low solubility. Those of the gums were 0.13 percent and that of Marasperse CB was 0.33 percent by weight. Weights used for a liter of stock solution are shown in Table 1. The volumes used in individual tests ranged from 5 ml. to a maximum at which no further increase in dispersion was measured.

EXPERIMENTAL RESULTS AND DISCUSSION

General Evaluation of the Dispersants

Although percentage values of the usual size groups were determined in all of the tests, the present report is chiefly concerned with those of the $<0.001\text{-mm.}$ -size group, the dispersion values. In the majority of cases in the first series of tests, the dispersion values increased with increasing concentrations of the dispersant and reached a maximum. With further increases in concentration, varying for different soils and dispersants, the degree of dispersion either persisted at the maximum value or decreased to lower values. Several examples of these effects are illustrated by the curves in Figure 1. The concentrations at which maximum values occurred are referred to as "the optimum concentration range." Those yielding values within 1 percentage point of the maximum value are termed "the practical concentration range." Several other effects which also occurred in this series of tests were: flocculation at low and high but not at medium concentrations, gelling at certain concentrations, and failure of suspensions over the entire

concentration range.

The dispersion values obtained for Soils 1 to 6 at optimum concentrations of each of the dispersants are given in Table 3. The figure in parentheses adjoining each dispersion value represents the deviation of that value from the maximum of the 20 dispersion values obtained for that soil. The symbol F indicates failure of the suspension due to flocculation or other colloidal effects at all concentrations of the dispersant.

As indicated by the F symbols, ten of the dispersants failed at all concentrations with one or more soils. Two others, gum ghatti and Marasperse CB, maintained the suspensions of all six soils, but were highly ineffectual, as shown by their deviation values. For at least one soil, however, each of the twelve produced dispersion values equal to or reasonably near the maximum value. This explains why, despite their failure with certain soils, several of these have been considered satisfactory in laboratories where the soils tested are closely related or of limited variety.

For each soil, wide variations occur among dispersion values given by the several dispersing agents. This indicates that close agreement (± 1 percentage point) between the values from duplicate tests with the same soil does not insure that a specific dispersing agent has provided a high degree of dispersion, since other duplicate samples of the same soil dispersed with a different agent will also yield closely-agreeing dispersion values, though they are higher or lower in degree.

With the podzolic soils, Numbers 1 to 5, the majority of the phosphates provide a combination of high dispersion values (low deviations) and freedom of suspensions from flocculation or other colloidal effects. Of the phosphates, Numbers 2, 5, 6, and 7 were highly effective and Numbers 4 and 8 were only slightly less so. However, Number 3 failed at all concentrations with three of the five soils.

With the laterite (No. 6) a high degree of dispersion was obtained from only four of the dispersants, among which were two phosphates, (Nos. 3 and 4). Trisodium phosphate, (No. 3), which was ineffectual with the podzolic soils, gave the highest dispersion value obtained. This indicates that the dispersion of laterites presents a

TABLE 4
PRACTICAL CONCENTRATIONS OF THE PHOSPHATES

Dispersing agent		Practical concentration range (ml of stock solution)					Practical concentration for podzolic soils 1 to 5 (ml of stock solution)
		Soil number					
No.	Name	1	2	3	4	5	
2	Sodium hexametaphosphate (Calgon)	85-150	120-150	105-150	25-150	135-150	140
3	Trisodium phosphate	40-55	F ^a	F	F	20-100	55
4	Tetrasodium pyrophosphate	90-140	5-110	20-100	25-140	60-150	100
5	Sodium tripolyphosphate	25-100	30-115	10-120	15-100	45-150	80
6	Sodium tetraphosphate	75-150	50-150	55-150	75-150	65-150	140
7	Sodium polyphos	10-115	30-150	10-80	10-85	30-150	75
8	Victamide	20-95	10-140	ND ^b	ND	ND	90

^a F - Suspension failure due to flocculation or other colloidal effect.

^b ND. - Value not determined.

different problem from that of the podzolic soils.

For most soils, sodium metasilicate yielded low dispersion values at optimum concentrations in the air-jet dispersion cup (Line 1, Table 3) and at the single concentration with the motor-stirrer (bottom line of Table 3). It has, however, the advantage of preventing flocculation in tests of all six soils. The dispersion values for Soils 2 and 5 also provide data for a valid comparison of the effectiveness of the air-jet dispersion cup and the motor-stirrer for mechanical dispersion since the single concentration of sodium metasilicate employed in the AASHO method is within the optimum concentration range for both soils in the air-jet dispersion cup.

Dispersion values from the air-jet dispersion cup are higher by 11 percentage points with Soil 2, and 15 with Soil 5. For the other soils, values from the air-jet dispersion cup range from 1 to 31 percentage points higher, but concentrations of the dispersant are not equivalent.

FURTHER STUDIES OF THE PHOSPHATES

In the second series of tests, Soils 7 to 14 and the clay minerals bentonite and kaolin, 15 and 16, were dispersed at the "practical concentration" of each of the several phosphates. This concentration, expressed as a volume of stock solution, was determined individually for each phos-

TABLE 5
**EFFECTIVENESS OF PHOSPHATES WITH SOILS 7 TO 14 AND THE CLAY
MINERALS BENTONITE, NO. 15, AND KAOLINITE, NO. 16**

Dispersing agent			Dispersion value (% of <0.001 mm group)											Deviation from maximum dispersion value (average for soils 7 to 14)
No	Name	Concentration (ml of stock solution)	Soil or clay mineral sample number											
			7	8	9	10	11	12	13	14	15	16		
2	Sodium hexametaphosphate (Calgon)	140	25 ^a (3)	20 (3)	40 (3)	57 (3)	40 (5)	44 (4)	28 (2)	38 (0)	63 (21)	35 (4)	2.9	
3	Trisodium phosphate	55	25 (3)	18 (5)	39 (4)	F ^b (6)	39 (6)	F (6)	F (6)	38 (0)	F (6)	32 (7)	F	
4	Tetrasodium pyrophosphate	100	26 (2)	15 (8)	39 (4)	56 (4)	41 (4)	40 (8)	27 (3)	36 (2)	78 (6)	35 (4)	4.4	
5	Sodium tripolyphosphate	80	28 (0)	23 (0)	43 (0)	58 (2)	42 (3)	43 (5)	30 (0)	38 (0)	82 (2)	39 (0)	1.2	
6	Sodium tetraphosphate	140	25 (3)	20 (3)	41 (2)	57 (3)	43 (2)	45 (3)	27 (3)	36 (2)	74 (10)	35 (4)	2.6	
7	Sodium polyphos	75	26 (2)	20 (3)	41 (2)	60 (0)	45 (0)	48 (0)	29 (1)	38 (0)	84 (0)	39 (0)	1.0	
8	Victamide	90	22 (6)	20 (3)	30 (13)	F (13)	F (13)	F (13)	28 (2)	37 (1)	68 (16)	39 (0)	F	
AASHO Method with sodium metasilicate and 1 minute in the motor stirrer		20	8 (20)	7 (16)	9 (34)	34 (26)	14 (31)	F (31)	20 (10)	10 (28)	ND ^c	ND	23.6	

^a All values in parentheses are deviations (in percentage-points) of the given dispersion value from the maximum value obtained for that soil.

^b F = Suspension failure due to flocculation or other colloidal effects

^c ND = Not determined

TABLE 6
EFFECTIVENESS OF PHOSPHATES WITH ALL SOILS TESTED AND WITH CERTAIN GROUPS OF SOILS

Dispersing agent		Deviation of the dispersion values from the maximum (percentage-points)																				
		All soils (#1-14)			All soils except laterite (#1-5 & 7-14)			Podzolic soils (#1-5 & 7)			Illitic soils (#5,7,10 & 11)			Kaolinitic soils			Kaolinite	Montmoril- lonitic soils			Montmoril- lonite (Bentonite)	
		Max	Min	Av	Max	Min	Av	Max	Min	Av	Max	Min	Av	#2	#6	Av	#16	#1	#9	#14	Av	#15
2	Sodium hexameta- phosphate (Calgon)	9	0	2.5	5	0	2.0	3	0	1.3	5	2	3.2	0	9	4.5	4	2	3	0	1.7	21
4	Tetrasodium pyrophosphate	8	1	3.4	8	2	3.6	4	2	2.7	4	2	3.2	2	1	1.5	4	4	4	2	3.3	6
5	Sodium tripoly- phosphate	5	0	1.4	5	0	1.2	3	0	1.2	3	1	1.8	1	5	3.0	0	3	0	0	1.0	2
6	Sodium tetra- phosphate	9	0	2.4	3	0	1.8	3	0	1.3	3	2	2.5	0	9	4.5	4	2	2	2	2.0	10
7	Sodium polyphos	13	0	2.1	3	0	0.6	3	1	2.0	2	0	1.0	3	13	8.0	0	1	2	0	1.0	0

phate from the volume-ranges in tests of Soils 1 to 5 that gave dispersion values within a percentage point of the maximum. Practical concentration values and the practical volume ranges from which they were derived are listed in Table 4.

The results of the second series of tests (Table 5) agree in general with those pertaining to the phosphates in the first series and reveal several points of further information:

1. Sodium polyphos and sodium tripolyphosphate (Nos. 5 and 7) are highly effective for the soils and clay minerals, having six optimum dispersion values (zero deviations) out of a possible 10, maximum deviations of 5 and 3 percentage

points, and low average deviations of 1.2 and 1.0.

2. Sodium hexametaphosphate and sodium tetraphosphate (Nos. 2 and 6) are somewhat less effective, as shown by their average deviation values — 2.9 and 2.6. This is undoubtedly related to their higher deviations with the clay minerals: 21 and 10 for bentonite, and 4 in both cases for kaolin. Approximately the same degree of effectiveness is shown by tetrasodium pyrophosphate (No. 4) for most of the soils, but two deviations of 8 points raise its average to 4.4, which approximates its dispersion results with the clay minerals.

3. Trisodium phosphate and Victamide (Nos. 3 and 8) each failed to maintain the

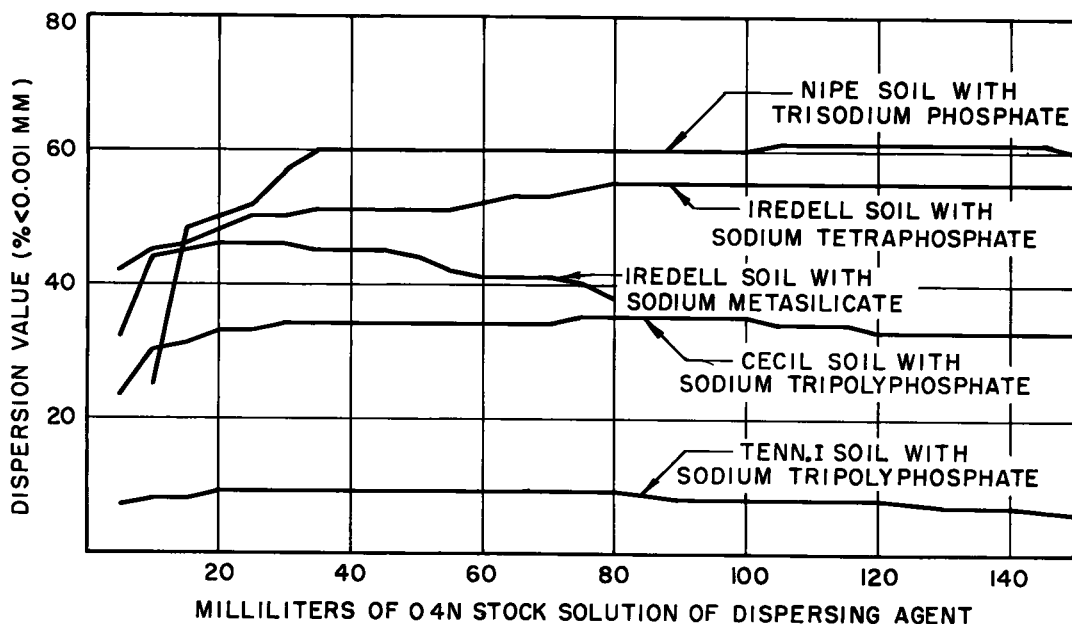


Figure 1. Effect of dispersing-agent concentration on dispersion value.

suspensions of three soils and was only moderately effective with the others. Victamide, however, produced the optimum dispersion value for kaolin but was ineffective with bentonite, and trisodium phosphate was ineffective with both of the clay minerals.

4. Sodium silicate was even less effective than in the previous series of tests, with a suspension failure with one soil and very high deviation values varying from 10 to 34 with the others.

5. Summarizing the results obtained with the clay minerals, bentonite was highly dispersed by only sodium tripolyphosphate and sodium polyphos (Nos. 5 and 7). Kaolin was highly dispersed by both of these

from Tables 3 and 5 and rearranged and summarized in Table 6. Due to their failure with several suspensions, Phosphates 3 and 8 were not included.

Comparison of the results of tests on all of the soils (1 to 14) shows sodium tripolyphosphate (No. 5) to be most effective, with an average deviation value of 1.4 percentage points, followed closely by sodium polyphos, sodium tetraphosphate, sodium hexametaphosphate and tetrasodium pyrophosphate (Nos. 7, 6, 2, and 4) in the order of their average deviation values. The maximum deviation values show that each dispersant gives a high deviation with at least one of the soils of the group.

TABLE 7

EFFECT OF LOW, MEDIUM, AND HIGH DEGREES OF DISPERSION (THREE LEVELS OF DISPERSANT CONCENTRATION) ON THE AMOUNT OF SPECIFIC SIZE GROUPS

Size group (mm.)	A. - Cecil (No. 2)						B. - Cecil (No. 2)						C. - Nipe (laterite, No. 6)					
	Dispersed with sodium tripolyphosphate a - 10 ml, b - 20 ml, c - 60 ml.						Dispersed with sodium polyphos a - 10 ml ; b - 30 ml , c - 100 ml.						Dispersed with trisodium phosphate a - 50 ml., b - 60 ml., c - 120 ml.					
	Amount found (percent)						Amount found (percent)						Amount found (percent)					
	a	b	c	b-a	c-b	c-a	a	b	c	b-a	c-b	c-a	a	b	c	b-a	c-b	c-a
2 - .42	4	5	4	+1	-1	0	4	4	4	0	0	0	6	5	3	-1	-2	-3
.42 - .25	8	7	8	-1	+1	0	8	8	8	0	0	0	2	1	1	-1	0	-1
.25 - .074	20	20	19	0	-1	-1	19	19	19	0	0	0	5	4	3	-1	-1	-2
.074 - .05	4	4	3	0	-1	-1	5	5	5	0	0	0	1	1	0	0	-1	-1
.05 - .005	25	21	22	-4	+1	-3	24	23	21	-1	-2	-3	18	15	15	-3	0	-3
.005 - .001	16	13	9	-3	-4	-7	16	11	10	-5	-1	-6	16	17	17	+1	0	+1
< .001	23	30	35	+7	+5	+12	24	30	33	+6	+3	+9	52	57	61	+5	+4	+9

Size group (mm.)	D - Nipe (laterite, No. 6)						E. - Iredell (No. 1)						F - Iredell (No. 1)					
	Dispersed with sodium polyphos a - 10 ml, b - 20 ml, c - 60 ml.						Dispersed with sodium hexa- metaphosphate a - 10 ml , b - 40 ml., c - 250 ml.						Dispersed with tetrasodium pyrophos a - 10 ml , b - 30 ml , c - 240 ml.					
	Amount found (percent)						Amount found (percent)						Amount found (percent)					
	a	b	c	b-a	c-b	c-a	a	b	c	b-a	c-b	c-a	a	b	c	b-a	c-b	c-a
2 - .42	13	10	8	-3	-2	-5	10	9	9	-1	+1	0	10	10	10	0	0	0
.42 - .25	5	5	3	0	-2	-2	2	2	2	0	0	0	2	2	2	0	0	0
.25 - .074	10	9	6	-1	-3	-4	8	9	9	+1	-1	0	8	8	8	0	0	0
.074 - .05	4	3	2	-1	-1	-2	2	2	1	0	-1	-1	2	2	1	0	-1	-1
.05 - .005	24	21	20	-3	-1	-4	19	18	16	-1	-2	-3	19	18	17	-1	-1	-2
.005 - .001	10	12	13	+2	+1	+3	16	10	8	-6	-2	-8	18	13	9	-5	-4	-9
< .001	36	40	48	+4	+8	+12	43	50	55	+7	+5	+12	41	47	53	+6	+6	+12

and Victamide (No. 8) and moderately well by sodium hexametaphosphate, tetrasodium pyrophosphate, and sodium tetraphosphate (Nos. 2, 4, and 6). It is significant that only sodium tripolyphosphate and sodium polyphos (Nos. 5 and 7) were highly effective for both of the clay minerals.

Dispersion of All Experimental Soils with the Five Effective Phosphates

In order to compare the essential results obtained by using the most-effective phosphates with all of the experimental soils and with certain special groupings of soils, some of the data were selected

For the soils in Groups 1 to 5 and 7 to 14, from which the laterite is excluded, the best dispersant is sodium polyphos (No. 7) which has a very low average deviation value of 0.6. Sodium tripolyphosphate, sodium tetraphosphate and sodium hexametaphosphate (Nos. 5, 6, and 2) follow in the order given. The average deviation of No. 4 (sodium pyrophosphate), 3.6 percentage-points, is significantly higher than that of the others.

For the podzolic soils, sodium tripolyphosphate, sodium hexametaphosphate, and sodium tetraphosphate (Nos. 5, 2, and 6), are almost equal in effectiveness, with closely agreeing average deviation values — 1.2, 1.3, and 1.3. Those of No. 7, sodium

polyphos, are slightly higher but probably not significantly. Although No. 4, tetrasodium pyrophosphate, is again the least effective, it is noteworthy that in this group of soils none of the phosphates produced an inordinately high maximum deviation value.

As previously noted, an illite clay mineral, per se, was not available in sufficient quantity for testing. However, data for Soils 5, 7, 10, and 11, which are reported as highly illitic (1 and 4), are combined in Table 6 for an examination of dispersion results where illite is the principal (in percentage) clay mineral. These soils may also be considered as a group containing mixtures of the three chief clay minerals, since the reported analyses (referred to above) indicate that in addition to illite, each contains about 10 percent of kaolinite and 25 percent of montmorillonite. Sodium polyphos (No. 7) again gave highest dispersion, with an average deviation of 1.0; sodium tripolyphosphate (No. 5) is next, with 1.8 percentage-points; and sodium tetraphosphate, sodium hexametaphosphate and tetrasodium pyrophosphate (Nos. 6, 2, and 4) follow as a less-effective group. Here, again, maximum deviations are not high.

Since only two examples of kaolinitic soils, Cecil and Nipe (2 and 6) are included in the experimental list and since these also contain considerable amounts of iron oxides (2 and 5), the specific influence of kaolinite in soils is not precisely defined by the data. However, deviation values given in Table 6 for Cecil soil and kaolin, 2 and 16, indicate that the five phosphates listed are relatively highly and about equally effective in dispersing these kaolinitic materials. Generally higher deviations for the laterite (Nipe) may be due to the iron oxides present in this soil. However, the moderately high iron-oxide content of Cecil soil has not similarly influenced its dispersion characteristics.

Results of the dispersion of bentonite show little resemblance to those of Iredell, Houston, and Putnam soils (1, 9, and 14), in Table 6, which have been reported (5 and 9) to be highly montmorillonitic. These three soils are highly dispersed by each of the phosphates, though only sodium tripolyphosphate and sodium polyphos (Nos. 5 and 7) are highly effective

with bentonite; tetrasodium pyrophosphate (No. 4) is moderately so, and sodium hexametaphosphate and sodium tetraphosphate (Nos. 2 and 6) are unsatisfactory. All of the phosphates were very highly effective with Putnam (No. 14), a claypan soil with a clay fraction consisting largely of beidellite, a species of montmorillonite (7).

Miscellaneous Results and Observations

Several examples of the specific size-groups affected during the later stages of the dispersion process are given in Tables 7 and 8. Those in Table 7 are of soils dispersed in the air-jet dispersion cup with three dispersant concentrations, yielding low, medium, and high degrees of dispersion. The size groups sustaining losses when the dispersion is increased are found to vary from one soil to another. Losses in Cecil and Iredell (Tables 7A, B, E and F) occur chiefly in the 0.05-to-0.005-mm. and 0.005-to-0.001-mm. groups, and Nipe (Tables 7C and D) sustains losses in all of the groups above the <0.001-mm. size. Although a certain amount of attrition of prime particles undoubtedly occurs with any soil, it is believed that little of this effect is reflected in these data.

Since dispersant concentration is the only variable involved in each set of figures, decreases in the coarser sizes are attributed to the breakdown of true aggregates. When the dispersant concentration is increased, the disaggregation increases until a maximum dispersion is reached at the optimum concentration. Differences in the degree of dispersion of the same soil with different dispersants are attributable, accordingly, to the greater ability of the better dispersant to disintegrate the aggregate structures and to maintain a condition of deflocculation in the resulting suspension.

The data in Table 8 show the effects of the two mechanical dispersion devices on the percentages of the several particle-size groups in two soils, Cecil and Sharkey. The values given are for dispersion treatments of 15 minutes with the air-jet dispersion cup and 1 minute with the motor-driven stirrer, using, for both treatments, 20 ml. of 0.2N sodium metasilicate solution, in accordance with the Standard AASHTO Method T 88-49. This amount of

the silicate solution also corresponds to the optimum concentration for both soils. The degree of dispersion produced by the air-jet dispersion cup treatment is much greater than that by the motor-driven stirrer, as indicated by the values of the <0.001-mm. group. In the Cecil soil, the gain of this group is derived from small reductions of all of the coarser groups, whereas most of the reduction in the Sharkey soil occurs in the 0.05-to-0.005-mm. range.

TABLE 8

COMPARISON OF GROUPS FROM DISPERSIONS WITH AIR-JET CUP AND MOTOR STIRRER USING THE OPTIMUM CONCENTRATION OF SODIUM METASILICATE

Size group (mm)	Amount found (percent of total weight of soil)					
	Cecil (No. 2)			Sharkey (No. 5)		
	Air-jet dispersion cup	Motor stirrer	Difference a-b	Air-jet dispersion cup	Motor stirrer	Difference a-b
	a	b		a	b	
2-0 42	8	9	-1	6	6	0
0 42- .25	4	6	-2	3	3	0
25- .074	17	20	-3	14	16	-2
.074- .05	4	5	-1	3	6	-3
.05- .005	23	24	-1	26	35	-9
.005- .001	11	14	-3	20	21	-1
<.001	33	22	+11	28	13	+15

Although an extensive study of the mineralogical nature and the physical and chemical properties of the difficultly-dispersed soil constituents was beyond the scope of the present investigation, studies of this sort offer interesting possibilities. It would be of value to know: (1) the identity and quantity of the clay or other active fine material which is "bound" in difficultly-dispersed aggregates of silt and sand sizes; (2) whether physical properties such as plasticity and volume change of the aggregated clay material are inhibited, and if so, to what extent the properties of the whole soil are affected; and (3) the basic nature of the binding forces in inorganic soils and why and to what extent they are effective in soils of different origin.

Observations on Sediment Volumes and pH Values

During the sedimentation of the soil suspensions, a large number of measurements of sediment volumes and pH values (indicating degree of acidity) and observations of colloidal activities were made. Since the measurements do not constitute a thorough and specific study of any of these effects, the bulk of the data is therefore not included in this report. However,

some of the findings warrant a brief mention.

1. Sediment volumes measured in suspensions (after a 24 hr. sedimentation period prepared with various concentrations of a dispersing agent are not a good index of the degree of dispersion. In the two examples given in Figure 2, contrary to what might be expected, the sediment volume is small at a low degree of dispersion, and increases as dispersion improves. Although the relationship varied with different soils and dispersing agents, similar results were obtained in many of the other tests.

2. Colloidal effects, such as flocculation and gelation, that interfere with the analytical results are often unnoticed unless they are strongly manifested. However, less-evident ones can often be detected by simple tests. The absence of a sharp line of demarcation between the sediment and the suspension, after a brief settling period, invariably indicates an abnormality of the deflocculation process. This observation can be made long before any pronounced flocculation or gelation occurs. A gelled volume in the lower part of the suspension is sometimes observed as a dark layer but is often ob-

TABLE 9

pH VALUES IN SUSPENSIONS OF SOILS 1 TO 6 WITH PHOSPHATES

(The two values shown for each soil-dispersant combination are those which occurred at the high and low extremes of the practical concentration range of the dispersing agent when used with that soil.)

Dispersing agent		pH ranges and deviations from the maximum dispersion values					
No	pH of stock solution	Soil No.					
		1	2	3	4	5	6
2	6.8	6.6-7.0 (2) ^a	7.3-7.3 (0)	7.4-7.4 (0)	7.1-7.2 (1)	7.2-7.5 (2)	7.4-7.4 (9)
3	11.3	8.7-9.2 (5)	6.7-10.7 F ^b	7.6-11.1 F	7.2-11.4 F	9.4-10.9 (6)	9.5-11.3 (0)
4	10.0	8.2-8.7 (4)	6.2-9.0 (2)	8.6-9.5 (2)	8.5-9.3 (3)	8.8-9.3 (3)	9.2-9.4 (1)
5	8.8	7.2-7.6 (3)	6.8-8.4 (1)	7.3-9.1 (1)	7.2-8.9 (1)	7.9-8.8 (1)	8.1-9.0 (5)
6	7.6	6.7-7.2 (2)	7.8-8.1 (0)	8.1-8.2 (0)	7.3-8.1 (0)	7.4-7.8 (3)	7.9-8.1 (9)
7	6.7	6.6-6.8 (1)	5.8-6.8 (3)	7.2-7.3 (2)	6.6-7.3 (2)	7.0-7.3 (2)	7.3-7.4 (13)
8	4.3	5.6-6.1 (3)	6.0-6.2 (2)	ND ^c	ND	ND	6.2-6.7 (19)
pH of soil (1-1 mix)		5.7	4.6	5.1	4.9	6.7	4.9

^a All values in parentheses are deviations (in r of the dispersion value from the maximum obtained for that soil)

^b F indicates flocculation at all concentr. and over the pH range cited

^c ND indicates pH not determined

scured by the color of the soil. It can more readily be detected by the pearly iridescence which results when the suspension is caused to rotate in the cylinder by means of a glass rod. With normal deflocculation, stirring produces a pearly

requirement a routine examination of each suspension under bright illumination so that the appearance of the demarcation line and any pearly-iridescent effect can be observed.

3. Some of the data from pH measure-

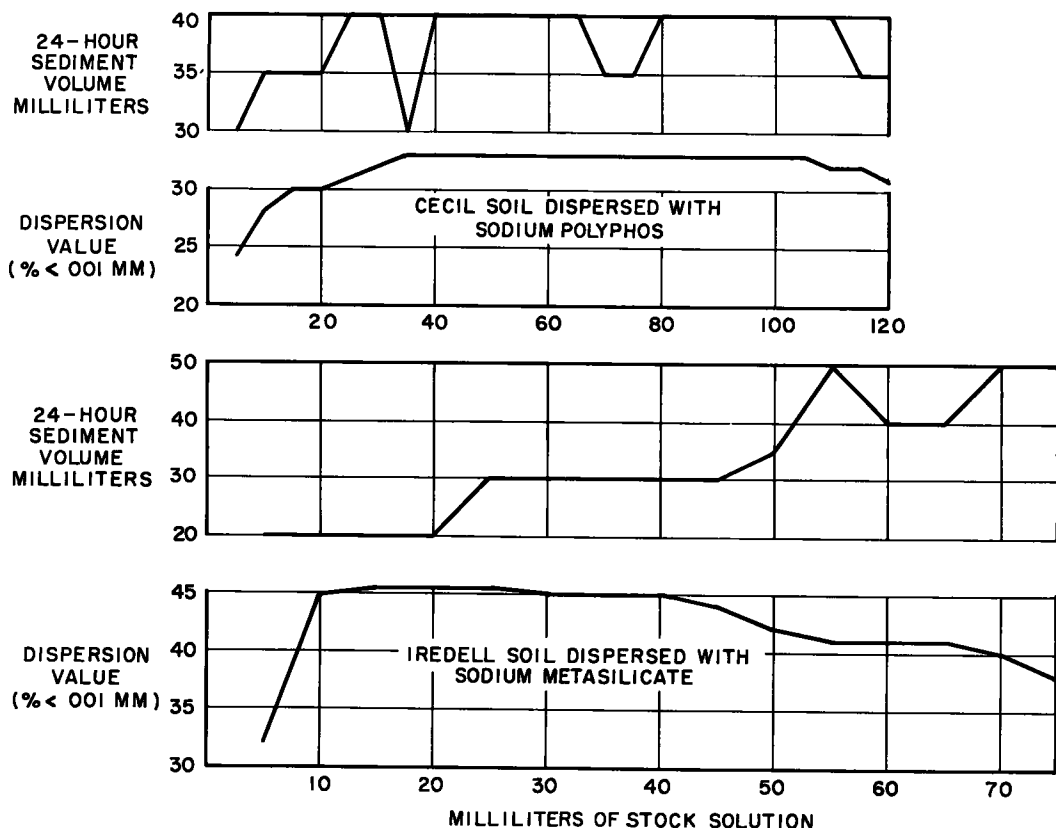


Figure 2. Effect of dispersion value on sediment volume with Cecil and Iredeil soils.

effect of light and dark streaks throughout the suspension from the reflection of light by the flat sides of platy particles, which are highly oriented by the rotation. If gelation is present in any part of the suspension, this phenomenon is reduced or obscured in the affected area. Gelation is also indicated when (1) after a reading has been made and the hydrometer removed from the suspension, the liquid remaining on the hydrometer flows off as a film, or (2) upon pouring the suspension into a No. 200 or No. 270 sieve for separation and washing of the coarse soil fractions, a restriction of the flow of the suspension through the sieve is noted. Although any of these qualitative tests may be used to advantage, the analytical procedure should include as a minimum

ments of suspensions obtained by the dispersion of each of Soils 1 to 6 with each of the phosphates are given in Table 9. The two pH values shown for each soil-dispersant combination are those which occurred at the high and low extremes of the practical concentration range of the dispersing agent when used with that soil. The figure in parentheses adjacent to each pH range is the deviation (in percentage points) of the dispersion value obtained in that range from the maximum obtained for the soil.

As in the previous discussions, the data of Soils 1 to 5 are considered separately from those of Soil 6, the laterite. With the group of Soils 1 to 5, the pH range in which each dispersing agent is most effective, is different for different soils.

For example, the pH ranges for Dispersant 2 are within narrow limits, 6.6 to 7.5, and are nearly identical; those for Dispersant 5 consist of somewhat higher values (up to 9.1), and are broader in scope, 6.8 to 9.1. It is further observed that for any one of the soils of this group, both a high degree of dispersion and good stability of the suspension can occur at several different pH ranges, depending on the dispersant used. However, suspension failure (flocculation of Soils 2, 3, and 4 with Dispersant 3) can occur also over broad pH ranges which include values at which suspensions are successfully maintained by other dispersants. The stock solutions of the better dispersants (Nos. 2, 5, 6, and 7) for Soils 1 to 5, have pH values between 6.7 and 8.8; that of No. 4, which is less effective, is 10.0; and those of Nos. 3 and 8, which are least effective, are 11.3 and 4.3.

In contrast to Soils 1 to 5, the laterite (No. 6) is effectively dispersed only under conditions of high pH; its deviations are lowest (best dispersion) at pH values of 9.2 and above with Dispersants 3 and 4, intermediate in the range from 8.1 to 9.0 with Dispersant 5, and highest (poorest dispersion) in the range from 6.2 to 6.7 with Dispersant 8.

Puri has presented data (8) showing that many soils are most highly dispersed at a pH of 11.2 when sodium hydroxide is used as the dispersant and the soil samples are pretreated to convert them to the H-form. The data in Table 9 are at variance with Puri's findings, since they were obtained using (1) soil samples in their natural cationic state, and (2) phosphate materials which are more effective at lower pH values. The present investigation was concerned with dispersion techniques which provide a high degree of dispersion without time-consuming pretreatments of the soil samples.

SUMMARY AND CONCLUSIONS

Nineteen chemical dispersing agents were tested to determine their effectiveness in the dispersion of soils and the stabilization of soil suspensions for particle-size analysis. Dispersions were made in air-jet dispersion cup and with

a high-speed motor stirrer. In all, 14 soils and two clay minerals were tested. Both soils and dispersing agents were selected to include a wide range of properties and conditions of the respective materials. In all tests, complete particle-size analyses were made and the usual size groups were determined by the hydrometer method and with standard sieves. Except for variations in kind and amount of dispersant, details of the analyses conformed to the Standard AASHTO Method T 88-49 (10). Sodium metasilicate, of the AASHTO Method, was among the dispersants tested.

Principal findings and conclusions from the investigation are:

1. Thirteen of the dispersing agents were found to be generally unsatisfactory. Several failed to stabilize the suspensions of one or more soils, others frequently produced low dispersion values, and some were ineffective in both respects.

2. Sodium metasilicate was among the less-satisfactory dispersants. Although it failed to stabilize the suspension of only one soil, many of its dispersion values were seriously low. Its use as a general soil dispersing agent should be discouraged.

3. Widely effective dispersing agents were found only among the phosphate materials. Two of these, sodium polyphos and sodium tripolyphosphate were highly effective with all soils except one, a laterite. Sodium hexametaphosphate and sodium tetrakisphosphate were only slightly less effective.

4. The highest dispersion values for a laterite soil were obtained from trisodium phosphate and tetrasodium pyrophosphate. For most other soils, however, the first was unsatisfactory and the second produced dispersion values that were only moderately high.

5. From the standpoint of special groupings, such as, podzolic, illitic, kaolinitic, and montmorillonitic soils, the four phosphates noted in Item 3 (above) were consistently highly effective, though their relative ratings varied somewhat among the groups. For general use, sodium polyphos is rated best and sodium tripolyphosphate is a close second. Their unsuitability for the dispersion of a true laterite soil does not extend to related, partially laterized soils.

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Deflocculating Agents for Mechanical Analysis of Soils

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Soil samples from different parts of the United States were used in mechanical analysis experiments for comparing the effectiveness of several chemicals as deflocculating agents. Sodium metaphosphate was found most promising. Since different varieties of this chemical are manufactured, experiments were conducted to evaluate compounds available through chemical supply houses.

The recommended compound was further studied to determine the effect of amount and age of the deflocculating solution used on efficiency of dispersion. Experiments were also performed to determine the change in viscosity and density of a soil suspension due to the use of sodium metaphosphate.

The viscosity of the soil suspension in the standard methods of mechanical analysis is assumed to be that of water. Viscosity measurements in this study indicate that correction factors for the influence of both the deflocculating agent and soil fines are desirable. The effect of sodium metaphosphate on the density of the soil suspension was also found to be appreciable, and a method for determining a correction factor is presented.

● **ADEQUATE** and stable dispersion of a soil sample is important for an accurate mechanical or particle-size analysis. Since most soils are difficult to disperse in water and tend to flocculate after being dispersed, the chemicals used as deflocculating or dispersing agents are added to the soil-water mixture to obtain satisfactory dispersion.

The addition of a deflocculating agent to a soil-water mixture affects the degree of dispersion of the soil sample and may also affect the specific gravity of the soil particles and the viscosity and specific gravity of the suspending medium. The experiments described in this paper were conducted: (1) to compare the effectiveness of several chemicals as deflocculating agents for the dispersion of soils and (2) to determine the effect of one of the deflocculating agents on the specific gravity of the soil dispersed and on the viscosity and specific gravity of the suspending medium.

Soil samples from different parts of the United States were used in the experiments. Table 1 gives the sources and some properties of the soil samples.

EFFECTIVENESS OF DEFLOCCULATING AGENTS

The theory of soil dispersion has been

discussed in a previous paper (1). The effectiveness of a deflocculating agent can be rated on the degree of dispersion of a soil sample with the deflocculating agent. The degree of dispersion can be determined especially by particle-size measurements of the fractions finer than 0.005 mm. and 0.001 mm. For example, the higher the content of material finer than 0.005 mm. and 0.001 mm., the higher the degree of dispersion.

Two types of dispersion apparatus were used in the mechanical analysis experiments reported in this paper. The first is the mechanical stirrer specified by both the American Society for Testing Materials and the American Association of State Highway Officials; the second is the Soil Dispersion Tube (S. D. T.). The dispersion procedure with the mechanical stirrer is given in the ASTM and AASHTO standard methods of mechanical analysis (2, 3). The S. D. T. apparatus and its use have been described (4).

Hydrometer tests were performed essentially according to the standard methods of mechanical analysis, except that corrections were applied to hydrometer readings, to compensate for the change in the specific gravity of the suspending medium due to the addition of a deflocculating agent.

The determination of correction constants is discussed later in this paper. Particle-size measurements reported in this paper are the average of results from duplicate tests.

COMPARISON OF SODIUM SILICATE, SODIUM PYROPHOSPHATE, AND SODIUM METAPHOSPHATE AS DEFLOCCULATING AGENTS

Sodium silicate is specified as the deflocculating agent in the present ASTM and AASHTO standard methods of mechanical analysis. Sodium pyrophosphate and sodium metaphosphate have been found effective as deflocculating agents for many types of soil (1, 5, 6). These three chemicals were evaluated as deflocculating agents for the soil samples listed in Table 1.

TABLE 1
SOURCE AND SOME PROPERTIES OF SOIL SAMPLES

Sample No.	Source	Textural Classification	Plasticity Index	Organic Matter Content, Percent	Content of Carbonates, Percent	pH
1	Iowa	Clay	51.7	1.2	2.5	5.3
2	Virginia	Clay	35.3	0.7	2.5	6.7
3	California	Clay	38.7	0.3	6.8	8.5
4	New York	Clay	13.1	0.6	14.9	8.1
5	Texas	Clay loam	3.6	0.2	81.3	8.2
6	Iowa	Silty loam	6.2	0.3	11.6	8.3
7	Virginia	Sand	N.P.	0.3	40.8	7.4
8	Texas	Clay	42.4	0.3	13.6	7.5

Note Only material passing No. 10 sieve was used in this study. Textural classifications are based upon the Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction.

In all experiments reported in this paper, deflocculating agents in solution were added to the soil-water mixture. The concentration of sodium silicate solution prepared from sodium metasilicate crystals was 3 deg. Baumé. The concentration of sodium pyrophosphate¹ solution and of sodium metaphosphate solution² was 0.5 N.

The degree of dispersion of a soil sample usually varies with the amount of deflocculating agent used. The trend of variation depends on the type of soil dispersed, the type of deflocculating agent used, and the apparatus and procedure of dispersion. Soil Samples 1 and 6 were used to determine the relation between degree of dispersion and amount of each deflocculating solution.

¹ The chemical used is also known as tetrasodium pyrophosphate ($\text{Na}_2\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$)

² Sodium metaphosphate B was used to prepare the solution. The description of this chemical is given in Table 3

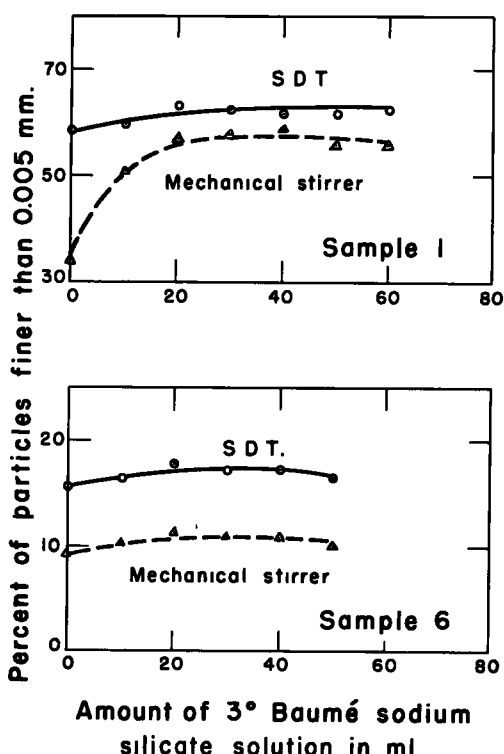


Figure 1. Relation between amount of sodium silicate solution and degree of dispersion obtained with different dispersion apparatus.

Both the mechanical stirrer and the S. D. T. were used in dispersing the soils.

As mentioned before, the degree of dispersion obtained by different methods of dispersion can be compared by means of particle-size measurements. This approach was followed in comparing the degree of dispersion obtained with different amounts of the three deflocculating solutions. The relations between the amount of each deflocculating solution and the degree of dispersion of the two samples, as represented by the percent of particles finer than 0.005 mm., are shown in Figures 1, 2 and 3. The curves for percentages finer than 0.001 mm. are similar.

Note that for equal amounts of solution the S. D. T. gave a higher degree of dispersion than the mechanical stirrer and that the amount of deflocculating agent used in the S. D. T. procedure was of less importance than in the mechanical stirrer procedure. For example, Figure 3 shows that the degree of dispersion of Sample 1 varied only slightly with the amount of sodium metaphosphate solution when the soil

TABLE 2
COMPARISON OF EFFECTIVENESS OF DEFLOCCULATING AGENTS IN SOIL DISPERSION

Dispersion Apparatus	Deflocculating Agent			Sample 1		Sample 2		Sample 3		Sample 4	
	Type	Concentration of Solution	Amount ^a (ml)	Percent ^b of Particles Finer Than							
				0.005mm.	0.001mm.	0.005mm.	0.001mm.	0.005mm.	0.001mm.	0.005mm.	0.001mm.
				No deflocculating agent			58.2	38.6	54.9	34.6	Flocculated
S D. T.	Sodium Silicate	3° Baume	20	63.1	51.1	58.3	45.9	53.7	40.8	56.9	29.8
	Sodium Pyrophosphate	0.5N	40	62.7	51.5	62.4	49.6	53.5	44.1	53.7	28.9
	Sodium Metaphosphate	0.5N	40	63.7	53.3	62.9	50.6	56.0	52.1	57.1	30.2
Mechanical Stirrer	No deflocculating agent			34.0	13.5	35.0	9.8	Flocculated		Flocculated	
	Sodium Silicate	3° Baume	20	57.0	34.8	51.8	25.4	43.9	16.8	55.3	27.3
	Sodium Pyrophosphate	0.5N	40	59.6	48.4	59.5	48.2	54.2	42.7	53.3	28.4
	Sodium Metaphosphate	0.5N	40	62.8	51.0	59.9	48.0	54.8	43.0	56.3	29.6

^a Refers to the amount of deflocculating solution used in dispersing a sample of 50g or 100g into a one liter soil suspension

^b All percentages are the average of results from duplicate tests

Dispersion Apparatus	Deflocculating Agent			Sample 5		Sample 6		Sample 7		Sample 8	
	Type	Concentration of Solution	Amount ^a (ml.)								
				0.005mm.	0.001mm.	0.005mm.	0.001mm.	0.005mm.	0.001mm.	0.005mm.	0.001mm.
S D T	No deflocculating agent			Flocculated		15.7	6.0	Flocculated		Flocculated	
	Sodium Silicate	3° Baume	20	28.8	7.1	17.8	10.4	4.8	2.7	Flocculated	
	Sodium Pyrophosphate	0.5N	40	30.1	7.4	20.6	13.6	5.5	3.5	65.6	50.6
	Sodium Metaphosphate	0.5N	40	30.7	8.8	18.8	12.7	6.6	4.0	65.7	52.3
Mechanical Stirrer	No deflocculating agent			Flocculated		9.4	4.1	Flocculated		Flocculated	
	Sodium Silicate	3° Baume	20	20.1	2.6	11.3	4.2	3.8	1.8	Flocculated	
	Sodium Pyrophosphate	0.5N	40	29.6	7.5	17.9	11.0	5.5	3.5	63.6	47.7
	Sodium Metaphosphate	0.5N	40	29.4	9.1	16.2	9.9	6.0	3.8	64.6	51.3

was dispersed with the S. D. T. As a contrast, when dispersed with the mechanical stirrer, the degree of dispersion changed substantially as the amount of sodium metaphosphate solution was varied from 10 to 30 ml.

The data in the three figures further indicate that, regardless of the type of dispersion apparatus used, the degree of dispersion of each sample practically remained unchanged when the amount of deflocculating solution used was about 20 ml. or more, as is shown in Figures 1 and 2, and was about 40 ml. or more in Figure 3.

The three deflocculating agents were further compared in the dispersion of samples 2, 3, 4, 5, 7 and 8 with both types of dispersion apparatus. The amounts of deflocculating solutions used for these samples were 20 ml. of sodium silicate solution³, 40 ml. of sodium pyrophosphate solution, and 40 ml. of sodium metaphosphate solution. Results are given in Table 2. With most of the samples, sodium sili-

cate was found inferior to the other two deflocculating agents, and sodium metaphosphate gave slightly better results than sodium pyrophosphate. Therefore sodium metaphosphate was chosen for more detailed studies.

COMPARISON OF DIFFERENT VARIETIES OF SODIUM METAPHOSPHATE

The sodium metaphosphate used in the foregoing experiments is one variety of the complex chemical also known as sodium hexametaphosphate or Graham's salt. The nomenclature of this group of chemicals is discussed in the appendix.

Since the different varieties of sodium metaphosphate sold by chemical supply companies may differ in their dispersing actions, experiments were made to compare the six varieties listed in Table 3. Variety B was used in the previously discussed experiments to compare sodium metaphosphate with sodium silicate and sodium pyrophosphate. The source and

³ As specified in ASTM Method D422-51 and AASHTO Method T88-49

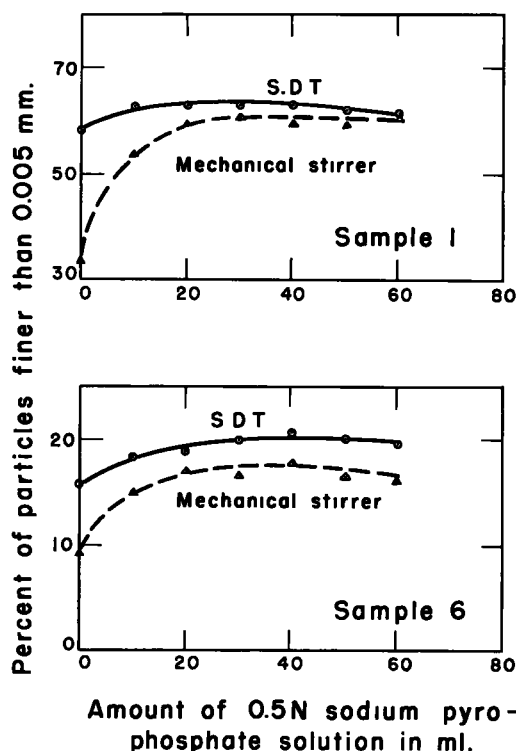


Figure 2. Relation between amount of sodium pyrophosphate solution and degree of dispersion obtained with different dispersion apparatus.

structure or crystallinity of each variety are given in the table. The significance of the structure of sodium metaphosphate with respect to its dispersing action is discussed by Tyner (5), who suggests that only the glassy form be used for soil dispersion purposes.

Soils 1 and 6 were chosen for the experiments. The S. D. T. was the only dispersion apparatus used. Since the amount of deflocculating solution needed for maximum dispersion may be different for different varieties of sodium metaphosphate, a number of solutions in the range from 30 to 60 ml. of 0.5N solution were tested to compare the six varieties (see Table 4).

The mechanical analysis results given in Table 4 indicate that the degree of dispersion varies only slightly with the varieties and amounts of sodium metaphosphate tested. The type of structure of sodium metaphosphate appears to be of little consequence.

Because the mechanical analysis data for Samples 1 and 6 showed no significant difference in the effectiveness of the six

TABLE 3
SOURCE AND STRUCTURE OF DIFFERENT VARIETIES OF SODIUM METAPHOSPHATE

Variety of Sodium Metaphosphate	Source	Structure ^a
A	Made by Calgon, Inc., Pittsburgh, Pa. and distributed under the trade name "Calgon"	Partially Microcrystalline
B	Made by Blockson Chemical Co., Joliet, Ill., available at Fisher Scientific Co., St. Louis, Mo. as sodium metaphosphate, C P	Glassy
C	Distributed by Fisher Scientific Co., St. Louis, Mo. as sodium hexametaphosphate	Crystalline
D	Prepared at Iowa Engr. Exp. Sta. Laboratory from sodium dihydrogen phosphate according to Tyner (5)	Glassy
E	Made by Rumford Chemical Works, Rumford, R. I., and sold under the trade name "Quadratos"	Glassy
F	Made by Rumford Chemical Works, Rumford, R. I., and sold under the trade name "Metafos"	Glassy

^a Based on examination with a petrographic microscope varieties of sodium metaphosphate, only two types, B and F, were used with the other six soil samples. Types B and F were selected mainly because of their comparative purity. The amounts of these two de-

TABLE 4
COMPARISON OF EFFECTIVENESS OF DIFFERENT VARIETIES OF SODIUM METAPHOSPHATE IN SOIL DISPERSION

Variety	Deflocculating Agent (Sodium Metaphosphate) Amount ^a (ml)	Sample No. 1		Sample No. 6	
		Percent of Particles Finer Than ^b			
		0.005mm	0.001mm	0.005mm	0.001mm
A	30	61.9	51.1	18.2	12.3
	40	63.1	52.1	19.0	13.3
	50	62.9	51.5	19.7	14.2
	60	63.6	51.3	18.9	13.5
B	30	62.4	51.3	19.1	11.7
	40	63.7	53.3	18.8	12.7
	50	61.7	52.0	18.4	11.9
	60	62.0	51.7	18.9	13.3
C	30	63.0	51.7	20.3	11.8
	40	62.6	52.2	18.9	12.2
	50	62.8	52.6	19.2	11.3
	60	62.9	52.1	18.2	11.7
D	30	62.5	50.8	20.3	11.2
	40	63.6	52.3	19.6	10.6
	50	62.8	51.4	19.3	10.6
	60	63.5	52.3	20.8	10.0
E	30	63.4	51.8	19.1	12.2
	40	63.0	51.6	18.7	14.3
	50	62.2	50.8	18.4	11.9
	60	64.4	53.2	18.4	12.8
F	30	62.6	51.5	18.1	15.0
	40	63.4	52.0	19.0	13.0
	50	61.5	51.6	18.5	13.4
	60	64.4	52.3	18.3	15.2

^a Refers to the amount of 0.5N deflocculating solution used in dispersing a sample of 50g into a one liter soil suspension

^b All percentages are the average of results from duplicate tests

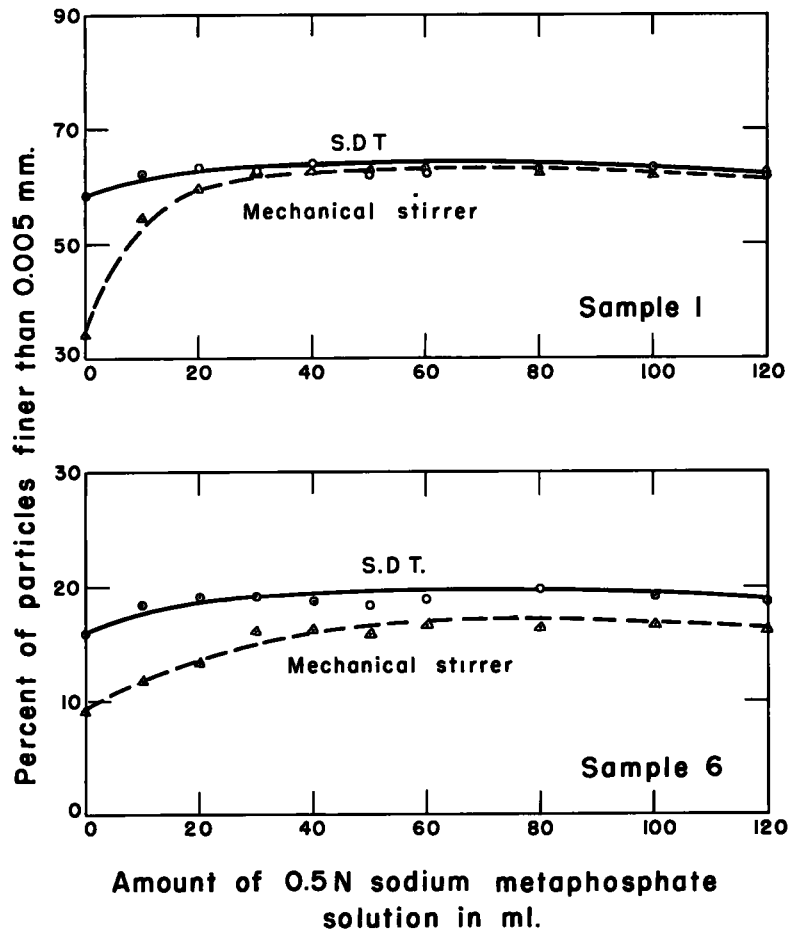


Figure 3. Relation between amount of sodium metaphosphate solution and degree of dispersion obtained with different dispersion apparatus.

flocculating solutions to be used with Samples 2, 3, 4, 5, 7 and 8 were chosen on the basis of the data shown in Figures 3 and 4. The curves representing dispersion with the S. D. T. show that when the amount of either B or F solution is within the range from 20 to 120 ml., the degree of dispersion is nearly independent of the amount of deflocculating solution. For this reason,

two amounts, 40 ml. and 100 ml., were used to cover this comparatively wide range. In these experiments only the S. D. T. dispersion apparatus was used.

Mechanical-analysis data representing the degree of dispersion obtained with the two varieties of sodium metaphosphate are presented in Table 5. Types B and F appear equally effective. An analysis of the

TABLE 5
COMPARISON OF TWO VARIETIES OF SODIUM METAPHOSPHATE IN SOIL DISPERSION

Deflocculating Agent (Sodium Metaphosphate)		Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6		Sample 7		Sample 8	
		Percent of Particles Finer Than ^b															
Variety	Amount ^a (ml)	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm	0.005 mm	0.001 mm
B	40	63.7	53.3	62.9	50.6	56.0	52.1	57.1	30.2	30.7	8.8	18.8	12.7	6.6	4.0	65.7	52.3
	100	63.0	52.0	62.5	52.9	55.2	48.2	57.0	30.3	30.2	10.3	19.1	12.5	6.5	4.3	66.2	54.0
F	40	63.4	52.0	59.0	46.0	56.3	48.1	56.6	32.0	30.8	8.3	19.0	13.0	7.4	4.0	67.1	52.1
	100	62.2	53.1	60.8	50.3	55.7	51.0	57.8	30.5	31.5	10.0	19.3	14.5	6.6	4.0	67.6	54.6

^a Refers to the amount of 0.5N deflocculating solution used in dispersing a sample of 50g. or 100g. into a one liter soil suspension.
^b All percentages are the average of results from duplicate tests

data in the table indicates that the use of 100 ml. of both kinds of sodium metaphosphate gives slightly better results than 40 ml.

Although all varieties of sodium metaphosphate were equally effective in dispersing the soils used in the preceding experiments, this might not be true with a greater variety of soils. Because of this, it seems desirable to recommend one kind of sodium metaphosphate in a standard method of mechanical analysis. Among

soils is desirable. On the basis of the experiments described in this paper, 100 ml. of 0.5N sodium metaphosphate (B) solution seems to be a safe amount to recommend.

EFFECT OF AGE OF SODIUM METAPHOSPHATE B SOLUTION ON ITS DISPERSIVE ACTION

Tyner (5) points out that sodium metaphosphate solutions may slowly revert

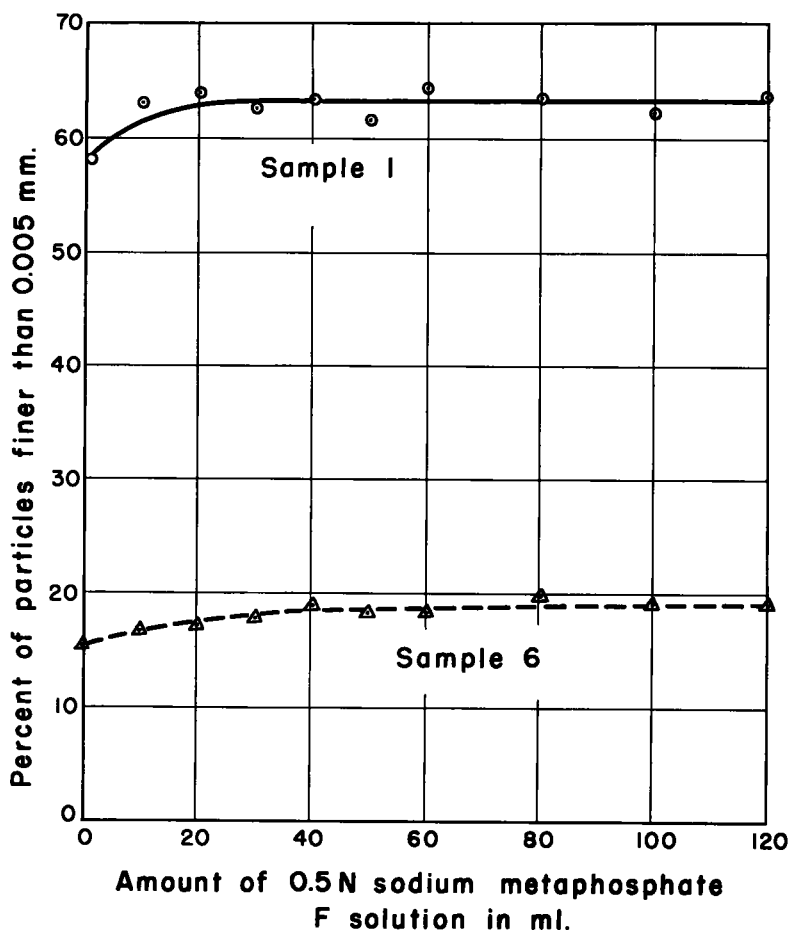


Figure 4. Relation between amount of sodium metaphosphate F solution and degree of dispersion obtained with the S.D.T. apparatus.

sodium metaphosphates of equal effectiveness as deflocculating agents, preference should be given to a kind, such as B, which is comparatively pure and readily available.

In a standard method of mechanical analysis, the recommendation of an amount of deflocculating solution that will give adequate dispersion to a great variety of

or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Because of this possibility, experiments were made to determine the effect of aging on the dispersing action of 0.5N sodium metaphosphate (B) solution. The pH of the solution used was 6.8, and its temperature during storage was about 70 to 80F. Both pH and temp-

erature may affect the rate of reversion.

Two amounts of the solution, 40 and 100 ml., were used in dispersing Samples 1 and 6 with the S. J. T. apparatus. Results given in Table 6 indicate that aging over a period of eight weeks had no appreciable effect on the dispersing action of the B solution. Because of the limited extent of this experiment, no definite conclusion should be drawn. It appears safe to say, however, that aging of a solution up to one month will not decrease the dispersive action of sodium metaphosphate Type B.

SECONDARY EFFECTS OF A DEFLOC-CULATING AGENT ON RESULTS

In addition to affecting the degree of dispersion of a soil sample, a deflocculat-

of soil particles remaining in suspension after a given sedimentation period are computed by the following equations:⁵

$$P = \frac{Ra}{W} \times 100 \quad (1)$$

Where:

P = percentage of originally dispersed soil remaining in suspension.

R = hydrometer reading (temperature correction should be applied if necessary).

W = weight in grams of soil originally dispersed minus the hygroscopic moisture.

a = constant depending on the specific gravity of soil dispersed and the specific gravity of the suspending medium.

$$d = \sqrt{\frac{30 \text{ nL}}{980 (G - G_1) T}} \quad (2)$$

TABLE 6

EFFECT OF VARIATION IN AGE OF SODIUM METAPHOSPHATE B SOLUTION ON ITS EFFECTIVENESS IN SOIL DISPERSION

Soil	Deflocculating Agent (sodium metaphosphate B)		Percent of Particles Finer Than ^c	
	Amount ^a (ml)	Age ^b (weeks)	0.005mm	0.001mm.
Sample 1	40	0	63.7	53.3
		2	62.1	52.0
		4	63.0	52.0
		6	62.8	51.4
		8	62.6	51.0
	100	0	63.0	52.0
		2	62.2	51.5
		4	62.7	53.1
		6	64.3	52.5
		8	62.3	51.3
Sample 6	40	0	18.8	12.7
		2	20.2	13.8
		4	18.4	14.2
		6	20.4	13.3
		8	19.5	13.1
	100	0	19.1	12.5
		2	19.7	14.3
		4	18.9	15.0
		6	20.0	13.9
		8	18.9	13.5

^a Refers to the amount of 0.5N deflocculating solution used in dispersing a 50g sample into a one liter soil suspension

^b Age refers to the time period after the solution is prepared

^c All percentages are the average of results from duplicate tests

ing agent may have other important effects on mechanical-analysis results. These effects, which will be referred to as secondary effects, include the changes in the specific gravity of the soil particles and in the viscosity and specific gravity of the suspending medium.

In the standard methods⁴ of mechanical analysis, the percentage and the diameter

⁴ASTM Method D422-51, AASHTO Method T88-49

TABLE 7

COMPARISON OF SPECIFIC GRAVITIES OF SOILS BEFORE AND AFTER DISPERSION WITH 100 ML 0.5N SODIUM METAPHOSPHATE (B) SOLUTION

Sample No	Specific Gravity ^a , 20C/20C	
	Before Dispersion	After Dispersion
1	2.714	2.711
6	2.729	2.724

^a Specific gravity values are the average of the data from triplicate tests

Where:

d = maximum particle diameter in mm.
n = coefficient of viscosity of the suspending medium in poises.

L = distance in cm. through which soil particles settle in a given period of time.

T = time in minutes, period of sedimentation.

G = specific gravity of soil particles.

G₁ = specific gravity of the suspending medium

In computing the percentage and the diameter of soil particles remaining in suspension from Equations 1 and 2, it is usually assumed that values of R and a in Equation 1 and of n, G, and G₁ in Equation 2 are not significantly affected by the use of a deflocculating agent. Actually these values may be substantially affected,

⁵Equation 1 is for tests using Bouyoucos hydrometer. When specific gravity hydrometers are used, the equation for computing the percentage of soil in suspension will be slightly different but the same variables are involved

especially when a relatively large quantity of deflocculating solution is used.

Experiments described in this part of the paper were conducted to determine the secondary effects resulting from the use

TABLE 8

COMPARISON OF VISCOSITIES OF DISTILLED WATER, DISTILLED WATER WITH DEFLOCCULATING AGENTS, AND SOIL SUSPENSIONS

Liquid	Viscosity at 68 F. (centipoise)
A (Distilled water)	1.004
(20 ml. 3 deg. Baume sodium silicate solution mixed with 981 ml. distilled water)	1.018
(40 ml. 0.5N sodium metaphosphate B solution mixed with 941 ml. distilled water)	1.025
(100 ml. 0.5N sodium metaphosphate B solution mixed with 881 ml. distilled water)	1.038
Soil suspension ^a prepared from fraction finer than 0.001 mm. in Sample 1 with the following suspending medium	
Liquid A	1.026
Liquid B	1.031
Liquid C	1.056
Liquid D	1.097
Soil suspension ^a prepared from fraction finer than 0.001 mm. in Sample 6 with the following suspending medium	
Liquid A	1.116
Liquid B	1.195
Liquid C	1.251
Liquid D	1.274

^a The soil suspension prepared from the fraction finer than 0.001 mm. in Sample 1 contains about 20 g. in 1000 ml. suspension, that prepared from the fraction finer than 0.001 mm. in Sample 6 contains about 3 g. in 1000 ml. suspension

of sodium metaphosphate(B). In these experiments the S.D.T. dispersion apparatus was used for Samples 1 and 6.

EFFECT ON SPECIFIC GRAVITY OF SOIL DISPERSED

Winterkorn, et. al. (7), in experiments with homoionic soils, found that the specific gravity of a soil varies with the kind of adsorbed cation. Since exchange of cations, as well as other chemical changes, may take place when a deflocculating agent is added to a soil-water mixture, there is a possibility for a change to occur in the specific gravity of the soil particles.

The exchangeable cations in Sample 6 are mainly sodium, potassium, and calcium, with the latter cation occupying about 80 percent of the exchange positions. Those in Sample 1 were not determined analytically but are estimated to be mainly hydrogen. The cation exchange capacities of Samples 6 and 1 are 40.0 and 13.4 m.e. per 100 g., respectively.

Experiments to determine the effect of sodium metaphosphate (B) on the specific gravity of Samples 1 and 6 consisted of specific gravity measurements (ASTM Standard Method D854-45T) before and after dispersion. Following dispersion with 100 ml. of 0.5N solution, the soil suspension was left undisturbed for 24 hours, filtered, washed with distilled water, and then dried. Results given in Table 7 indicate that no significant change in specific gravity occurred.

EFFECT ON VISCOSITY OF SUSPENDING MEDIUM

The relation between the viscosity of the suspending medium and the diameter of soil particles in suspension is shown by Equation 2. In the AASHO and ASTM methods of mechanical analysis, the viscosity of distilled water is taken as the viscosity of the suspending medium. Actually, when a deflocculating agent is used, the suspending medium will be a combination of water and the deflocculating solution, and the viscosity of the resulting suspending medium may be appreciably different from that of water. This is illustrated by the viscosity measurements given in Table 8 of Liquids A, B, C, and D representing different kinds of suspending medium. A Cannon-Fenske-Ostwald-type viscometer was used, and the test procedure recommended in ASTM Method D445-46T was followed. Note that the viscosity of Liquid D is about 3.4 percent higher than that of distilled water (Liquid A). Such a difference in viscosity will result in a relative difference of about 1.7 percent in the diameter of soil particles computed by Equation 2.

Theoretically, neither the viscosity of water nor that of water mixed with a deflocculating agent should be used as n in Equation 2. Consider a soil suspension containing silt-size and clay-size particles. Since the silt-size material settles much faster than the clay-size, especially the sizes finer than 0.001 mm., the medium through which silt-size material settles is a soil suspension comprised of clay-size material, deflocculating agent, and water. The viscosities of soil suspensions prepared from the fraction finer than 0.001 mm. in Samples 1 and 6 are also given in Table 8. Note that the viscosity of such suspending mediums may be

as much as 27 percent higher than that of pure water. In using Equation 2, this would result in a difference of about 13 percent in the diameter of soil particles.

From the somewhat-limited experimental results discussed above, for an accurate mechanical analysis it appears that the viscosity value used in computing particle diameters should be as nearly as possible that of the actual suspending medium. The most-practical approach in routine tests might be to apply corrections to the diameters as computed in the conventional manner. The correction will vary not only with the temperature of the soil suspension but also with the particle size composition of the soil sample and the value of the particle diameter. One way to obtain the correction values for routine testing purposes is to arbitrarily divide the common types of soil into several groups and to determine the corrections needed for the different particle-size ranges in each group. The temperature correction can either be included in these correction values or can be applied separately.

EFFECT ON SPECIFIC GRAVITY OF SUSPENDING MEDIUM

The addition of a deflocculating agent to a soil-water mixture will change the density or specific gravity of the suspending medium, which will affect particle-size determinations in two ways. The value of a in Equation 1 and that of G_1 in Equation 2 may be significantly affected. The hydrometer reading R in Equation 1 is influenced in the following manner. Regardless of the type of hydrometer used, hydrometer readings give the difference between the specific gravity of the soil suspension and that of water. If the suspending medium is water only, the hydrometer reading R represents the increase in specific gravity due to the presence of suspended soil particles. If water mixed with a deflocculating agent is the suspending medium, the hydrometer reading R represents the increase in the specific gravity due to the presence of both the suspended soil particles and the deflocculating agent.

The change in the values of a and G_1 can be determined by measuring the specific gravity of the actual suspending medium, water containing a deflocculating agent.

For example, when 100 ml. of 0.5N sodium metaphosphate (B) solution is contained in one liter of soil suspension, the specific gravity of the suspending medium at 67 F. will be about 1.003, which is approximately 0.5 percent higher than the specific gravity of water at the same temperature. A difference of this amount will result only in a change of about 0.3 percent in the percentage values and of about 0.2 percent in the diameter values obtained from Equation 1 and 2, respectively. These small changes can probably be overlooked in routine mechanical analyses.

The idea of correcting the hydrometer reading R for the presence of a deflocculating agent is not new. Many laboratories apply such a correction when the quantity of deflocculating solution used is relatively large.

Hydrometer readings may be corrected by subtracting the hydrometer reading⁶ of the suspending medium (water plus a deflocculating agent) from the reading taken in the soil suspension. The hydrometer reading of the suspending medium can be determined by a hydrometer measurement in water containing the amount of deflocculating agent in the soil suspension. The correction constant can be determined from the hydrometer reading (in the use of the Bouyoucos hydrometer, the reading is the constant).

When different amounts of a given deflocculating solution are being investigated, the following equations may simplify the determination of correction constants.

For Bouyoucos hydrometer

$$C = \frac{m R_d}{1000 - \frac{W}{G}} \quad (3)$$

For specific gravity hydrometer

$$C = \frac{m (R_d - 1)}{1000 - \frac{W}{G}} \quad (4)$$

Where:

C = correction constant.

m = amount of deflocculating solution in ml. contained in one liter of soil suspension.

R_d = hydrometer reading of deflocculating solution at specified temperature.

W = weight in grams of soil originally dis-

⁶With specific gravity hydrometers, only the decimal portion of the hydrometer reading will be subtracted

TABLE 9

COMPARISON OF EXPERIMENTAL AND COMPUTED CORRECTION CONSTANTS FOR HYDROMETER READINGS

Soil	Deflocculating Agent		Hydrometer reading of soil suspension at the elapsed time indicated ^b		Difference in hydrometer readings of soil suspension with and without deflocculating agent ^c		Correction constant determined by Equation 3
	Type	Amount ^a (ml)	15 min	60 min.	15 min	60 min.	
Sample 1, fraction finer than 0.0005 mm	No deflocculating agent		14.5	14.5	0	0	--
	Sodium meta-phosphate B	40	17.0	17.0	2.5	2.5	2.5
		100	21.0	21.0	6.5	6.5	6.4
Sample 6, fraction finer than 0.005 mm.	No deflocculating agent		6.5	Not taken	0	--	--
	Sodium meta-phosphate B	40	9.0	Not taken	2.5	--	2.5
		100	13.0	Not taken	6.5	--	6.4

^a Refers to the amount of 0.5N deflocculating solution used in preparing one liter of soil suspension^b Bouyoucos type hydrometer was used in all tests. The temperature of soil suspension was maintained at 67°F. during the hydrometer test^c Example: Hydrometer reading of suspension without deflocculating agent = 14.5, hydrometer reading of suspension with 40 ml. deflocculating solution = 17.0, difference in hydrometer readings = 17.0 - 14.5 = 2.5.

persed minus the hygroscopic moisture.

G = specific gravity of soil dispersed.

In deriving Equations 3 and 4, the Bouyoucos and the specific gravity hydrometer readings for water at the specified temperature (usually 67°F. or 68°F.) are assumed to be zero and one respectively. For practical applications of the two equations, the specific gravity G can be assumed as 2.65 because it has little effect on the correction constant computed. It should also be mentioned that the correction constant computed from either Equation 3 or 4 is always positive in value and should be subtracted from hydrometer readings taken in the soil suspension.

The method of determining correction constants discussed above is valid only if it can be assumed that chemical changes caused by the addition of a deflocculating agent to a soil-water mixture do not significantly affect hydrometer readings taken in the soil suspension.

A direct test of the validity of this assumption is to compare the correction constant determined by Equations 3 or 4 with the required correction as determined experimentally. The required correction equals the difference between the hydrometer reading taken in a soil suspension containing a deflocculating agent and that taken in a similar soil suspension without the deflocculating agent. Since the degree of dispersion of a soil sample may be greatly affected by the use of a deflocculating agent, the experimental determination of the required correction must be accomplished in such a way that any change in the degree of dispersion of the soil sample will not significantly affect hydrometer readings taken in the soil

suspension. This can be done by using clay-size soil samples. A description of two such determinations follows.

The clay-size material was collected by the layer method (8) from Samples 1 and 6. The clay separated from Sample 1 was finer than 0.0005 mm. in size, and that from Sample 6 was finer than 0.005 mm. Samples of each separated clay material were soaked for over 18 hours in either distilled water or distilled water containing sodium metaphosphate B (Table 9) before being dispersed with the S.D.T. for the hydrometer test. The hydrometer tests were conducted in essentially the same manner specified in the AASHTO and ASTM standard methods of mechanical analysis.

As mentioned, the purpose of using such clay-size material was to eliminate the degree of dispersion variable from the tests. Thus, any variation in the degree of dispersion of a sample consisting of particles finer than 0.0005 mm. will not significantly affect hydrometer readings taken within an hour after the beginning of sedimentation. Similarly, hydrometer readings in a soil suspension consisting of minus-0.005-mm. material will not be significantly affected by a variation in the degree of dispersion, if the readings are taken within 15 minutes after the beginning of sedimentation.

Carbonates in a soil sample may influence chemical changes taking place in a soil suspension in which sodium metaphosphate is the deflocculating agent. The minus-0.0005-mm. clay material used in the tests contained a negligible amount of carbonates; the 0.005-mm. clay contained about 8 percent of carbonates, principally calcium carbonate.

Hydrometer readings of the prepared soil suspensions taken at different sedi-

mentation times are given in Table 9. The readings taken before 15 minutes are not shown in the table, since they were the same as those taken at 15 minutes due to the smallness of the particle sizes contained in the suspensions. To eliminate the effect of variation in degree of dispersion on hydrometer readings, no readings were taken after 60 minutes of sedimentation in the soil suspensions prepared with minus-0.0005-mm. material and none after 15 minutes in suspensions prepared with the minus-0.005-mm. material.

The required correction constant for each suspension containing sodium metaphosphate (B), obtained by subtracting the hydrometer reading of the suspension from the hydrometer reading of a similar suspension containing no deflocculating agent, is given in the table together with the correction constants computed by Equation 3. Considering that hydrometer readings were taken to the nearest half division, the required and the computed corrections are practically in complete agreement. According to this experiment, the assumption made in developing Equations 3 or 4 seems valid.

CONCLUSIONS

1. Among the three chemicals compared, sodium metaphosphate is the most-

promising deflocculating agent.

2. Among the different varieties of sodium metaphosphate compared, variety B appears to be well suited for adoption as a deflocculating agent in standard methods of mechanical analysis.

3. On the basis of the results with the soils tested, the use of 100 ml. of 0.5N Sodium Metaphosphate B solution in making one liter of soil suspension is recommended.

4. To avoid a possible decrease in its dispersive action, it seems advisable to make up fresh B solutions about every 30 days.

5. The effect of B solution on the specific gravity of soils tested was found to be insignificant.

6. The viscosity of water is used as that of the suspending medium on the present standard methods of mechanical analysis in computing particle diameters. A correction should be applied to the computed diameter for accurate mechanical analysis results to compensate for the difference between the viscosity of water and that of the actual suspending medium.

7. The suggested method for determining the correction constant to compensate for the change in specific gravity of the suspending medium due to the use of a deflocculating agent appears to be valid.

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Appendix

NOMENCLATURE OF SODIUM POLYPHOSPHATES

There is no universally adopted nomenclature for the sodium polyphosphates at the present time. The following is one method of classifying the different sodium polyphosphates.

The sodium polyphosphates may be classified according to their structure into two groups: the crystals and the glasses. The crystalline sodium polyphosphates include sodium metaphosphate, sodium pyrophosphate, and sodium tripolyphosphate. The glassy sodium polyphosphates include sodium tetraphosphate, sodium hexametaphosphate, and also sodium metaphosphate. The two names, sodium hexametaphosphate and sodium metaphosphate, are often used interchangeably. Manufacturers use trade names (e.g. Calgon, Sodium Polyphos, Quadrafos, Metaphos) in referring to their glassy sodium polyphosphate products.

As mentioned, sodium metaphosphate

can be either crystalline or glassy. Different varieties of this chemical are available from chemical supply companies. The complicated properties of this group of chemicals were discussed by Thorne and Roberts (9) as follows:

"Metaphosphoric acid, HPO_3 , and its salts possess the most complicated properties of all the acids of phosphorus, because, on the one hand, they have a strong tendency to polymerize, when varied products of high molecular weight can be formed, while, on the other hand, these products show isomerism through different arrangements within the individual molecules, so that varying constitutions may appear with the same molecular complexity. The relations of the metaphosphates are still so confused, in spite of numerous investigations, that the practice of giving definite formulae for the products must be given up for the present."

Rapid Methods for Determining Liquid Limits of Soils

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Engineers responsible for the routine testing of large numbers of soil samples for highway construction and maintenance need rapid methods for testing. In this study, two simplified methods of determining the liquid limit of soil are compared with the standard method to indicate their reliability and accuracy. Both are one-run methods, i. e., require a single determination of moisture and number of blows for groove closure to calculate the liquid limit. The methods discussed—(1) a so-called chart method developed by the Washington State Highway Department and (2) a modification of the chart method by use of a slide rule—have been compared for a wide range of soils found in many of the states. Both of these methods are rapid and reduce the time normally required for testing as much as 30 to 70 percent. They were found to have good reproducibility and to have sufficient accuracy, within limits, to be acceptable as alternate test procedures for the standard method used for the determination of the liquid-limit constant.

● IN 1926 the lower liquid limit, developed by Albert Atterberg,¹ and described in the International Reports on Pedology, 1911, was suggested as one of the soil constants that could be used to evaluate a soil as a road-building material.

This soil constant is now widely used by highway and airport engineers to evaluate soils for use as subgrade, base course, and embankment material. High liquid limits usually indicate heavy clays and low values indicate friable soils, silts, and clayey sands.

The lower liquid limit, now known as the liquid limit, is defined as the percent of moisture, based upon the dry weight of soil, at which a soil will just begin to flow when jarred slightly, and according to this definition, soils at their liquid limit have a small but definite shear resistance which can be overcome by the application of very little force. The cohesion of the soil-water mixture at the liquid limit is practically zero.

The determination of this soil constant as originally proposed was made by a hand method. In this method, the operator adjusted the moisture content by trial and error until exactly 10 blows closed a standard width groove made in the soil sample. The moisture content for this groove closure

was taken as the liquid limit of the soil. This method required considerable skill and judgment by the operator to obtain duplicate test results.

As soil testing became more widely used, the need for a more practical test procedure became apparent and a mechanical device² was developed which eliminated personal judgment in estimating the intensity of the blows which cause closure of the groove. Although the liquid limit machine was found to give good reproducibility of results and has been adopted as a standard test procedure³, it requires more time than the hand method.

For example, the standard method required that three random trials be made, each at a different moisture content and number of blows; that the percent moisture and number of blows be plotted on semilog paper and a flow curve drawn; and that the liquid limit be taken as the moisture content corresponding to the point where the flow curve intersects the 25-blow line. This procedure has less probable error than a single determination by the hand method and the machine can be used by operators of less skill, but the additional trials, weigh-

² Research on the Atterberg Limits of Soils, by Arthur Casagrande, PUBLIC ROADS, Vol. 13, No. 8, Oct. 1932.

³ Standard Method of Determining the Liquid Limit of Soils, AASHTO Designation T89-49, Part II, Standard Specifications for Highway Materials and Methods of Sampling and Testing, published by The American Association of State Highway Officials, 1950.

¹ Adaptation of Atterberg Plasticity Tests for Subgrade Soils, by A. M. Wintermyer, PUBLIC ROADS, Vol. 7, No. 6, Aug. 1926.

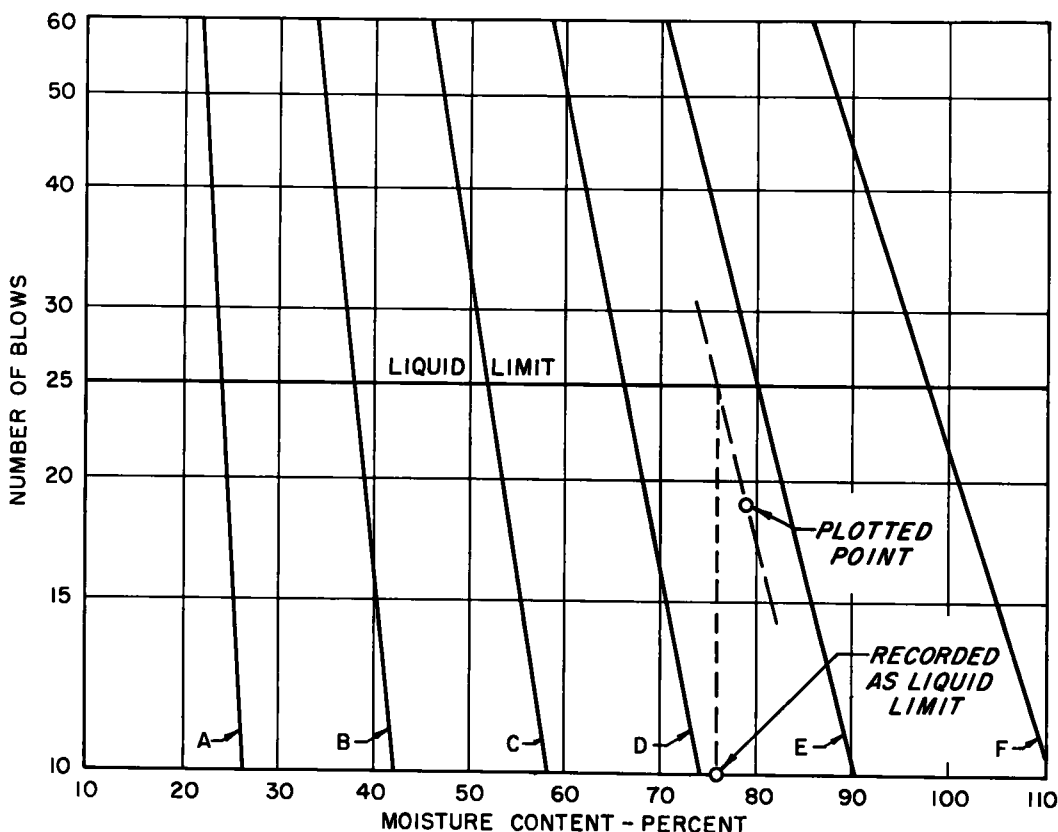


Figure 1. Relation between slope of typical flow curves and liquid limit.

ings, and computations require considerably more time than that required by skilled operators familiar with the use of the one-point hand method.

It is reasonable to expect that engineers responsible for routine testing of soils would be reluctant to accept a longer test procedure without carefully examining the method to determine if modifications could be made which would permit more rapid testing of soils.

One of the first modifications to be tried was the use of the mechanical liquid-limit device to furnish the standard force used in the hand method. This was accomplished by adjusting the soil-water consistency by a trial-and-error method until exactly 25 blows by the liquid-limit machine closed the standard-width groove.

This modification of the machine method, usually referred to as the "moisture-adjustment method," can be developed into an accurate rapid method by skilled operators if calibrated with the standard meth-

od, and it can be used effectively to reduce the time required for the determination of the liquid limit of a soil. However, it is difficult for inexperienced or less-skilled

TABLE 1

COMPARISON OF DEVIATIONS IN VALUES OF LIQUID LIMIT OBTAINED BY THE COOPER AND JOHNSON CHART METHODS FROM THOSE OBTAINED BY STANDARD TESTS^a

Range in deviations ^b	Original Chart Method ^c # tests	% total	Cum %	Revised Chart Method ^d # tests	% total	Cum %
0	4	6	6	8	9	9
0 1 - 0 5	28	38	44	41	48	57
0 6 - 1 0	19	26	70	26	31	88
1 1 - 1 5	6	8	78	7	8	96
1 6 - 2 0	6	8	86	3	4	100
2 1 - 2 5	6	8	94	-	-	-
2 6 - 3 0	1	1	95	-	-	-
3 1 - 3 5	2	3	98	-	-	-
3 6 - 4 0	0	0	98	-	-	-
over 4 0	2	2	100	-	-	-
Total	74	100		85	100	

^a Duplicate samples tested, one at random blows and the other by the standard test procedure used in the laboratory

^b Deviations \pm from the value of liquid limit determined by test

^c Chart (Fig 1) based on groove closure within range of 15 to 40 blows

^d Chart (Fig 2) based on groove closure within range of 17 to 36 blows

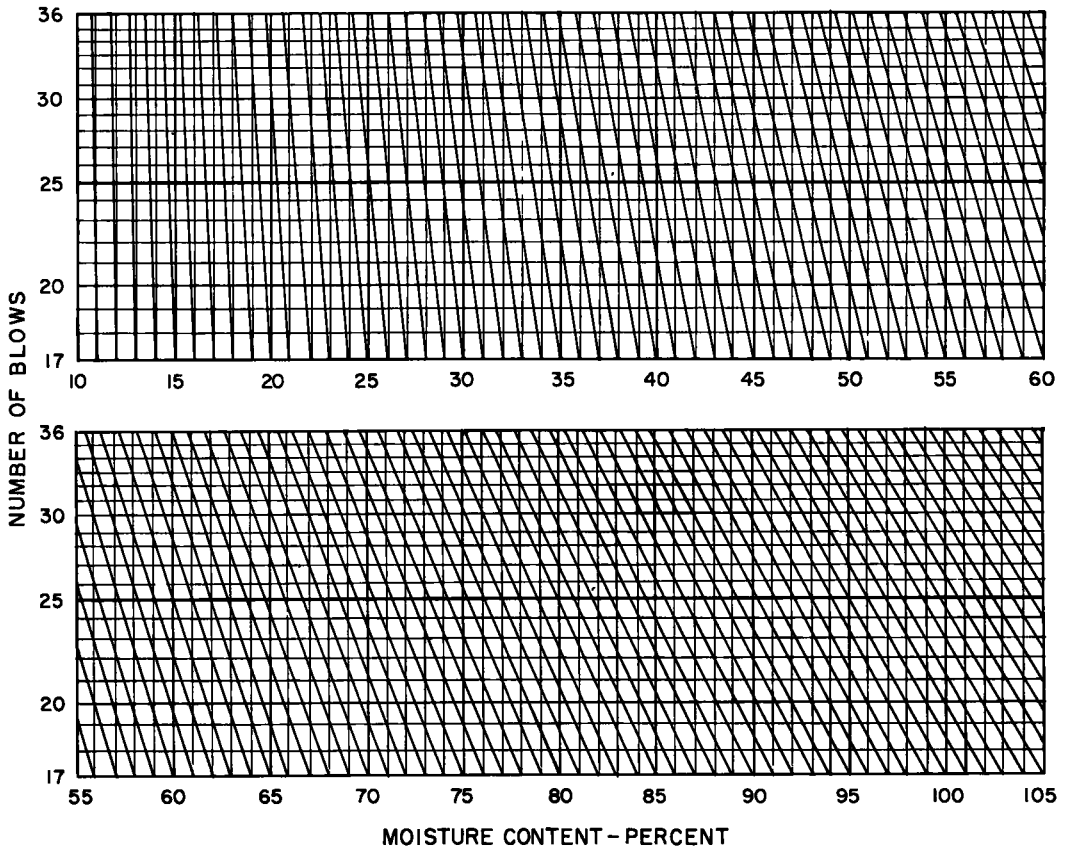


Figure 2. Chart developed by Washington State Highway Department for the calculation of the liquid limit.

operators to adjust the consistency of the soil-water mixture to obtain groove closure at exactly 25 blows.

CHART METHOD FOR DETERMINING LIQUID LIMIT

A rapid method for determining the liquid limit has been devised by Cooper and Johnson, of the Washington State Highway Department,⁴ for the routine testing of soils in their laboratory. It is called the "chart method" and is based upon the relation between the slopes of flow curves obtained for soils with different liquid-limit test values.

Cooper and Johnson observed, in their initial study of soils taken from various

parts of Washington, that the flow index⁵ computed from the flow curves obtained by the Standard AASHTO Method of Test increased uniformly with the liquid limit of the soils. From these data, six typical flow curves, A through F, shown in Figure 1, were developed.

Cooper and Johnson established the validity of the six typical curves by plotting the number of blows and moisture contents for single points representing random trial runs on each of 27 different soils. In this series of tests, the number of blows for groove closure was varied within a range of 15 to 40. The liquid limit was determined for each soil by drawing a line through the plotted point, which is parallel to the nearest typical flow curve (A through F) and recording as the liquid limit, the moisture content at the intersection of this line with the 25-blow line.

⁴A Rapid Method of Determining the Liquid Limit of Soils, by John H. Cooper and Kenneth A. Johnson, Materials Laboratory Report No. 83 March 1950, Washington State Highway Department

⁵Flow index is the range in moisture content represented by the number of blows or shocks in one cycle of the logarithmic scale of a flow curve

TABLE 2

FREQUENCIES OF DIFFERENT SIZE DEVIATIONS OF CHART AND SLIDE RULE LIQUID LIMITS FROM THOSE BY THE STANDARD AASHO METHOD

Range of liquid limit	Number of one-point tests	Deviation from Standard LLA	Frequency of deviations	
			Chart ^b Percent	Slide rule ^c Percent
(1)	(2)	(3) ^d	(4)	(5)
15 - 25	171	0	76.6	84.8
		1	23.4	15.2
		2	0	0
26 - 40	209	0	76.1	77.0
		1	23.4	23.0
		2	0.5	0.0
		3	0	0
41 - 55	129	0	69.0	70.6
		1	27.1	27.9
		2	3.9	1.6
		3	0	0
56 - 70	82	0	58.6	58.6
		1	40.2	40.2
		2	1.2	1.2
		3	0	0
71 - 85	94	0	44.7	45.7
		1	47.9	48.9
		2	7.4	5.3
		3	0	0
86 - 100+	74	0	39.2	35.2
		1	37.8	43.2
		2	20.2	18.9
		3	2.7	2.7
Total No. of tests	759	4	0	0

^a Determined by AASHO Standard Method T 89-49

^b Determined by the Washington State Highway Dept Chart Method

^c Determined by the Bureau of Public Roads Slide Rule Method

^d Liquid limit values are reported to the nearest whole number, therefore, deviations from the standard values within the ranges of $\pm(0$ to $0.49)$, $\pm(0.50$ to $1.49)$, $\pm(1.50$ to $2.49)$, $\pm(2.50$ to $3.49)$, and $\pm(3.50$ to $4.49)$ are grouped under the nearest values of 0, 1, 2, 3, and 4, respectively.

These values were compared with the liquid limit determined by the standard laboratory method and good agreement was found between the computed values obtained by this chart method and those determined by the standard test procedure.

Cooper and Johnson made a further simplification of the chart method by subdividing the original chart to show typical flow curves for each percentage of moisture so that any point selected from a single moisture content and corresponding number of blows for groove closure could be projected visually to the 25-blow line to obtain the liquid limit.

This improved chart, shown in Figure 2, was checked by Cooper and Johnson by using 84 duplicate tests of 73 soil samples. In each case, the liquid limit was determined by the "moisture-adjustment method" and by the chart method. This

comparison between the two methods of test showed that discrepancies ranged from 0.0 to 1.8 percentage points. However, because of the low frequency of errors in excess of 1.0 percent, they considered the accuracy of the chart method adequate for soil-classification purposes. They pointed out that two separate closures of the soil-water mixture should be observed to check its consistency before selecting a moisture sample for the determination of the liquid limit by this revised chart method. It was their opinion that the operator should, after completing the second closure, be sufficiently familiar with the flow characteristics of the material to detect any erroneous results.

Although this method is used in their routine identification and classification of soils, Cooper and Johnson recommend that it should not be used for the acceptance or rejection of materials which have borderline values of liquid limit or plasticity index. In these cases, they recommend the use of the standard mechanical method (AASHO Designation T 89-49).

The results of a statistical analysis of the basic data made by Cooper and Johnson in the preparation of their original and revised chart methods are shown in Table 1. These data show that the reduction from the 15-to-40 to the 17-to-36 range in the number of blows for groove closure increased the accuracy of the chart method. The maximum deviation in the revised chart method for the 17-to-36 blow range was two points. For 88 percent of the tests, the deviation was less than one as compared to 70 percent for the 15-to-40 range.

TABLE 3

VALUES OF THE DENOMINATOR OF EQUATION 1 CORRESPONDING TO THE NUMBER OF BLOWS REQUIRED FOR GROOVE CLOSURE IN THE LIQUID LIMIT TEST

S Blows	1 419 - 0.3 log S ^a	S Blows	1 419 - 0.3 log S ^a
15	1.066	28	0.985
16	1.059	29	0.980
17	1.050	30	0.976
18	1.043	31	0.972
19	1.036	32	0.968
20	1.029	33	0.964
21	1.023	34	0.960
22	1.017	35	0.956
23	1.011	36	0.952
24	1.005	37	0.948
25	1.000	38	0.945
26	0.995	39	0.942
27	0.990	40	0.939

^a Value of the denominator of Equation 1

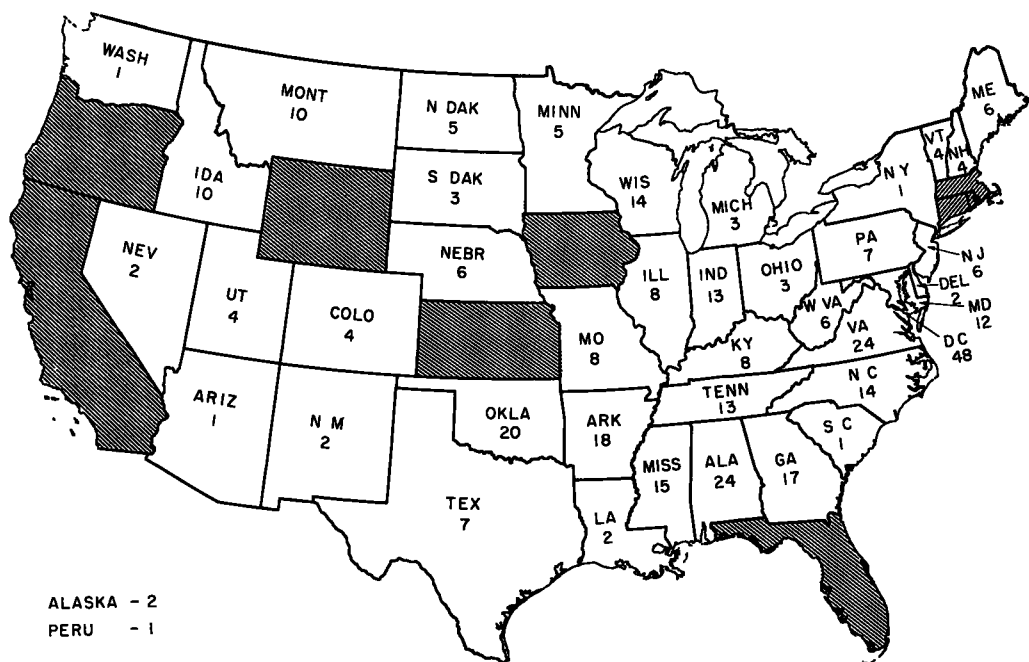
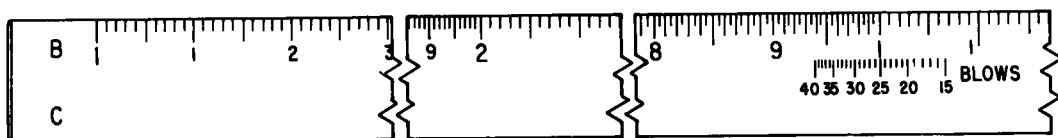
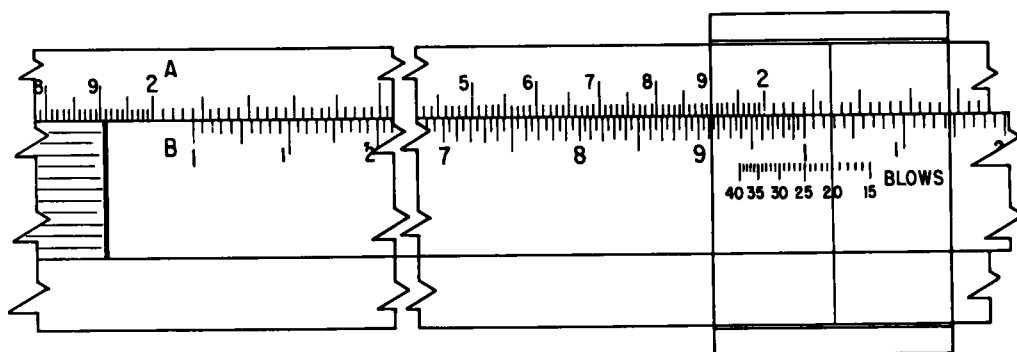


Figure 3. Map showing the number of soil samples from various areas used by the Bureau of Public Roads in testing the relation between the one-point liquid limit tests and the standard AASHTO method (Designation T 89-49).



A - LOCATION OF SPECIAL SCALE (BLOWS) WITH RESPECT TO B SCALE OF SLIDE RULE



B - SLIDE RULE SET FOR 21.4 PERCENT MOISTURE AT 20 BLOWS, INDICATING CALCULATED LIQUID LIMIT OF 20.8

Figure 4. Slide-rule with special scale for the calculation of liquid limit.

CHART METHOD INVESTIGATED AND COMPARED WITH STANDARD METHOD

Since only soils from Washington had been studied by Cooper and Johnson, and the moisture-adjustment method was the yardstick of comparison, the Bureau of Public Roads widened the scope of their investigation by checking the new method against the standard method of liquid limit (AASHTO Designation T 89-49) for 364 soil samples selected to represent soils from all sections of the United States. Figure 3 shows the areas from which these samples were obtained. The soils selected have liquid limits ranging from 15 to 104.

Data for checking the accuracy of the chart method were taken from laboratory records and two points with the highest and lowest number of blows were selected from each standard flow curve for each of the 364 soil samples. The moisture contents corresponding to these two numbers of blows for these two points were used for calculating the liquid limit from the revised chart.

To compare the accuracy of these results with those obtained by the standard test method, the data were subdivided according to their liquid limit values into six groups. The first group was terminated at 25 since it is the maximum liquid limit allowed for base course materials by the standard specifications of AASHTO, and it is also the upper limit for A-1 soils in the AASHTO system of soil classification.⁶

The second group was terminated at 40, the maximum liquid limit for A-4 and A-6 soil groups in the AASHTO system of soil classification. The liquid limit values above 40 were arbitrarily subdivided into four 15-point ranges. A tabulation of the deviations of 759 liquid-limit values calculated by the chart method from those determined by the Standard AASHTO Machine Method is shown in Table 2. Since the groups do not contain the same number of tests, the frequencies of the deviations, Column 4, are expressed in the form of percentages to facilitate comparisons.

These data obtained for 364 soil samples tested by the Bureau of Public Roads show a range in deviations from the standard values of 0.0 to about 2.0. This range

of deviations compares quite favorably with the range 0 to 1.8 reported by Cooper and Johnson for test data obtained for 73 soil samples taken from Washington.

SLIDE-RULE METHOD DEVELOPED

The preceding investigation of the accuracy of the chart method for calculation of the liquid limit suggested the possibility of eliminating the use of the chart by the development of a special scale which could be inscribed on a slide rule.

This was accomplished by deriving an equation for the family of typical curves used by Cooper and Johnson in the preparation of their chart. A study of the typical curves in Figure 1, indicated a point of convergence near the zero-moisture-content axis, and while it would be possible to derive an equation for such a family of curves, the problem was simplified by arbitrarily moving the point of convergence to the zero axis. This point is so far from the 25-blow line that the consequent changes in the slopes of the flow curves are negligible within the 17-to-36 blow range used. The formula derived for this new family of curves is:

$$LL = \frac{W}{1.419 - 0.3 \log S} \quad (1)$$

where

LL = liquid limit

S = number of blows (or shocks) required to close groove

W = percent moisture contained by the soil when S-blows close the groove

The use of this formula is facilitated by computing a table of the denominator $(1.419 - 0.3 \log S)$ for different values of S. The data in Table 3 show values of this denominator for values of S and the liquid limit for any moisture content at groove closure between 15 and 40 blows is obtained by dividing the percent moisture by the value of the denominator in Table 3 corresponding to the number of blows required to close the groove.

A more-convenient method for using the data in Table 3 is to inscribe a special scale in the blank space below the B scale of a slide rule to indicate the number of blows required for groove closure corresponding to the values of the denominator of Equation 1.

Figure 4a shows a section of a 20-inch

⁶The Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes, AASHTO Designation M 145-49, Part I, Standard Specifications for Highway Materials and Methods of Sampling and Testing, 6th Edition 1950, published by the American Association of State Highway Officials

polyphase slide rule with this special scale inscribed for the number of blows between 15 and 40. A study of this special scale with respect to the normal B scale of the slide rule shows that it is constructed by making a mark in the blank section located below the B scale of the slide rule for each value of the denominator of

COMPARISON OF THE SLIDE RULE AND CHART METHOD

The reproducibility of the slide-rule method was checked against the standard method for the same data that were used to check the revised chart method developed by Cooper and Johnson (see Table

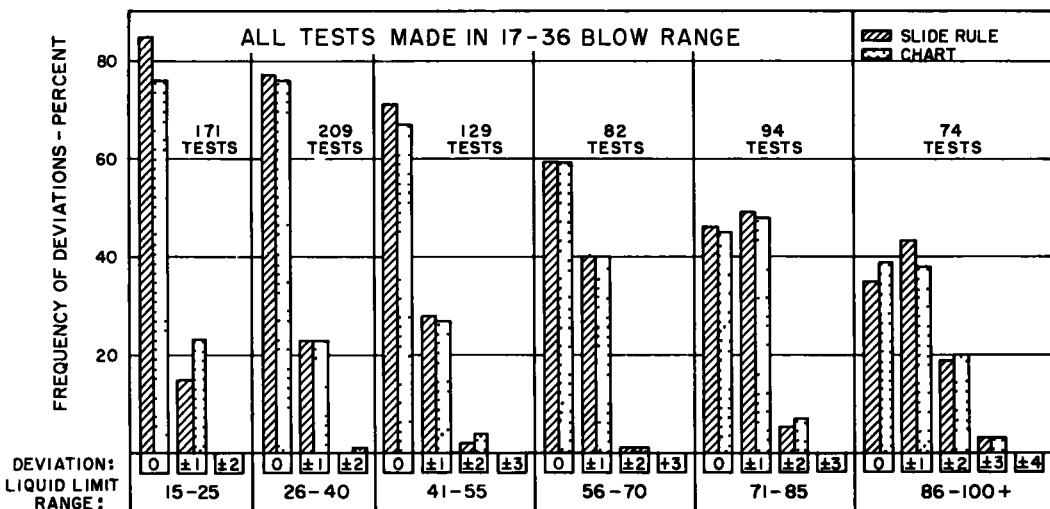


Figure 5. Percentage of chart and slide-rule tests deviating from the AASHTO standard liquid limit by 0, ± 1 , ± 2 or more, in six ranges of liquid limit. (All tests made between 17 and 36 blows and liquid limit values reported in whole numbers).

Equation 1. For example, the 20-blow mark on the special scale corresponds to a B-scale reading of 1.029, the 25-blow mark to a value of 1.000 (the midpoint of the B scale) the 30-blow mark reading of 0.976, etc.

The calculation of the liquid limit by the slide-rule method is easily made by setting the indicator line over the percent of moisture found in the soilpat on the A scale of the slide rule and adjusting the special scale until the corresponding number of blows coincides with the indicator line. With this setting of the slide rule, the liquid limit is read on the A scale above the end index of the B scale, or on the A scale directly above the 25-blow mark on the special scale.

In the example, shown in Figure 4b, 20 blows were required to close the groove and the corresponding moisture content was 21.4 percent. The calculated liquid limit shown above the end index of the B scale, on the A scale, is 20.8.

2, Column 4). The percentage frequency for the same deviations, Column 3, is shown in Column 5, Table 2, so that comparisons can be made with the chart method. Figure 5 shows the comparison of the accuracy of the slide rule and the revised chart methods in the form of a bar graph for the ranges of liquid limits selected for this study. The number of tests deviating by ± 0 , ± 1 , and ± 2 from the liquid limits determined by the standard method is expressed on a percentage basis to facilitate the comparison of the accuracy of the two methods.

It is shown by these comparisons that within the range of liquid limits most generally encountered, the chart and slide-rule methods may deviate from correct values by as much as 2 percentage points. Therefore it is concluded that dependence should not be placed on results obtained by the chart or slide-rule methods for the acceptance or rejection of materials when the liquid limit as determined by either of

these methods is within 2 percentage points of the specification limits.

The data in Figure 5 show that for the liquid limit range of 15 to 40, over 75 percent of the calculated values for both methods are within ± 1 point and practically all are within ± 2 points of the values determined by the standard test procedure.

However, for the acceptance or rejection of soil-aggregate materials based on a specification having a maximum liquid limit of 25, a deviation of +0.5 from that value, or 2 percent of 25, is the maximum that can be permitted. In order to provide this accuracy, the one-point method needed further improvement. It was thought that this could be accomplished if the range of blows for groove closure was decreased.

ACCURACY OF METHODS IMPROVED BY NARROWING THE RANGE IN BLOWS

A statistical analysis was made of the test data to determine the range in number of blows for groove closure which would give results within 2 percent of those obtained by the standard AASHTO method. The results of this analysis are shown in Table 4 and a study of these data indicates that the desired level of accuracy can be accomplished by narrowing the range of the acceptable number of blows for groove closure from a range from 17 to 36 to a range from 22 to 28.

Three ranges in number of blows for

groove closure 29 to 35, 22 to 28, and 17 to 21 are indicated in Column 1 of Table 4 to show the reproducibility obtained by chart and slide rule methods for six ranges in liquid limit, Column 2. The number of tests studied in each of these ranges is shown in Column 3.

To facilitate comparison of the accuracy of the methods for each of the three ranges in blows for groove closure, the number of cases having less than the indicated deviations is expressed on a percentage basis for each of the six ranges in liquid limit.

Within each of the ranges of liquid limit, the number of cases having less than each consecutive larger deviation is expressed on a cumulative percentage basis. For example, under the chart method for 29 to 35-blow range, Column 1 of Table 4, and a liquid limit range of 15 to 25, Column 2, the percentages of test results deviating less than 0.3, 0.5, 0.8, 1.1, 1.4, 1.7 and 2.0+ from the true values, (see Columns 4, 5, 6, 7, 8, 9 and 10 of Table 4) are 42, 74, 90, 100, 100, 100 and 100, respectively. The underlined values of 42 and 74 are the percentages of test results deviating less than 0.3 and 0.5, and are within 2 percent of the maximum and minimum values of liquid-limit range of 15 to 25. Similarly, other comparative values for any designated range in number of blows or liquid limits can be determined for either the chart or slide-rule methods.

A study of these comparative data in

TABLE 4
COMPARISON OF ACCURACY OF CHART AND SLIDE RULE METHODS

Range of blows for LL tests (1)	Liquid limit ranges (2)	No of tests (3)	Frequency deviations from std test values in cum percentages													
			Chart Method							Slide Rule Method						
			Deviations \pm							Deviations \pm						
			3-	5-	8-	1 1-	1 4-	1 7-	2 0+	3-	5-	8-	1 1-	1 4-	1 7-	2 0+
			(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)
29-35	15-25	31	42	74	90	100	100	100	100	81	97	100	100	100	100	100
	26-40	37	51	84	95	100	100	100	100	85	97	100	100	100	100	100
	41-55	37	38	54	92	97	100	100	100	43	73	95	100	100	100	100
	56-70	22	32	41	88	96	96	100	100	23	50	86	100	100	100	100
	71-85	31	23	29	61	81	90	97	100	23	36	74	87	97	97	100
	86-100+	20	15	25	30	40	85	95	100	20	25	35	50	70	85	100
22-28	15-25	34	88	100	100	100	100	100	100	91	100	100	100	100	100	100
	26-40	27	85	100	100	100	100	100	100	93	100	100	100	100	100	100
	41-55	23	65	91	100	100	100	100	100	65	98	100	100	100	100	100
	56-70	13	62	92	100	100	100	100	100	69	92	100	100	100	100	100
	71-85	28	68	82	96	98	100	100	100	82	96	100	100	100	100	100
	86-100+	20	30	45	85	85	95	100	100	35	45	70	85	95	100	100
17-21	15-25	37	51	81	97	97	100	100	100	73	95	97	100	100	100	100
	26-40	43	56	77	100	100	100	100	100	58	84	98	100	100	100	100
	41-55	25	52	80	88	96	100	100	100	40	68	84	88	96	100	100
	56-70	22	36	50	73	81	100	100	100	36	50	68	82	91	100	100
	71-85	29	38	41	66	88	93	93	100	38	59	69	90	93	93	100
	86-100+	22	9	23	41	50	64	73	100	9	18	36	46	55	59	100

Note. Underlined values are the percentage of tests with deviations less than 2 percent of the minimum and maximum liquid limit range shown in Column 2.

Table 4 clearly shows that the greatest degree of accuracy for both methods is obtained in the 22-to-28-blow range and the least accuracy occurs in the 17-to-21 blow range. Within the 22-to-28 blow range there are no test values (see underlined results in Table 4) calculated by either the chart or the slide-rule methods which exceed the maximum deviations based upon 2 percent limit of error in the liquid-limit values determined by the standard AASHTO method. The only exception was the deviation of less than 0.3, which is 2 percent of the minimum value (15) in the 15-to-25 liquid-limit range, which shows 88 percent for the chart and 91 percent for the slide-rule methods. However, this is insignificant, since it is the usual practice to report the liquid-limit value to the nearest whole number. For this reason, the 22-to-28-blow range was selected as the limits to be used to obtain test results meeting the tolerance specified.

SUMMARY

Thus, on the basis of the data obtained in this investigation, both the chart and slide-rule methods should be acceptable

as alternates for the standard methods, provided: (1) procedure for the preparation of the soil pat for test be that prescribed for the standard method, (2) the acceptable number of blows for groove closure be within the 22-to-28 blow range, and (3) at least two consecutive closures be observed before selecting the moisture sample for calculation of the liquid limit.

Under these conditions, the results of the slide-rule method have been consistently checked with those obtained by the AASHTO Standard Method in the Bureau of Public Roads Soils laboratory for more than 2 years. There appears to be no difficulty for technicians in the adjustment of the consistency within the 22-to-28 blow range.

It is suggested that other laboratories make similar comparative check studies, using local soils, to determine whether the accuracy of this improved one-point method of calculation can be established for a range of conditions wider than those included in this study. If comparable accuracy is obtained by these proposed check tests, it would warrant the use of this method as a standard AASHTO and ASTM method.

Discussion

ROGER V. LeCLERC, Senior Materials Engineer, Washington Department of Highways — The one-shot method of liquid-limit determination, as described in the Washington Highway Department Laboratory Report 83, "A Rapid Method of Determining the Liquid Limit of Soils," by Cooper and Johnson, has been used in routine testing in our laboratory for approximately 4 years. It is not used as a basis of acceptance or rejection of materials, the mechanical method of ASTM Designation D423 being used in these cases. We find that the rapid method is quite acceptable for classification purposes and that it promotes an appreciable saving in time.

For quite some time it had been our contention that it would be possible to convert the chart developed by Cooper and Johnson into an equation which could be solved by means of a slide rule. The paper by Olmstead and Johnston has ably demonstrated that this is so and, also, that the one-shot method of liquid-limit determination is applicable to soils else-

where throughout the United States. The equation that they have developed to represent the relationship between flow curves and liquid limits and their suggestion for converting an ordinary slide rule into a liquid-limit slide rule are most commendable.

Some investigation along similar lines was begun in our laboratory in the spring of 1953, but the investigation was short-lived, due to the press of an extra-heavy work load and did not, therefore, proceed to any conclusion. The results of our minor investigation and comments are offered for whatever worth they may be in supplementing the work so ably presented by the authors.

The work done in our laboratory was primarily a re-analysis of data presented in our Laboratory Report 83, the report by Cooper and Johnson, in an attempt to find an equation for the chart. No new data were involved. Our first efforts indicated two possible approaches. The following equation was developed from

that approach which appeared more accurately to fit the data:

$$(LL - 10) = \frac{(W - 10)}{1.48 - .343 \log S} \quad (A)$$

The terminology is the same as given in the paper by Olmstead and Johnston. This equation would argue that the curves as presented in the chart tend to converge at the 10-percent-moisture-content line. It was at this point that our work had to be abandoned with only a cursory attempt to check the accuracy of the equation.

After receipt of the authors' paper, curiosity led us to follow through on the second possibility for an equation. We found that the equation which developed from this latter approach was identical with that developed by the authors. It is quite apparent that we were somewhat amiss in our evaluation of which approach would give the better results.

It might be pointed out that work of a similar nature on the liquid limit determination has been reported by the Corps of Engineers, U. S. Army, Waterways Experiment Station at Vicksburg, Mississippi, in their Technical Memorandum 3-286, entitled "Simplification of the Liquid Limit Test Procedure." They, too, developed an equation which was based on the fact that a logarithmic plot of moisture content versus number of blows in the liquid-limit test produced flow curves with approximately the same slope, at least within a limited range of blows. They stated that their equation, which is shown below, was considered strictly applicable only to those soils which they tested (inorganic clays from the Alluvial Valley of the Mississippi River and the East and West Gulf Coastal Plains).

$$LL = W (S/25)^{0.121} \quad (B)$$

The terminology, once again, is that used by the authors in their paper.

Analysis of the liquid limit data contained in the report by Cooper and Johnson in a manner similar to that used by the Army Engineers showed that their liquid limit chart may also be represented quite closely by the equation:

$$LL = W (S/25)^{0.130} \quad (C)$$

Within a 17-to-36 range in the number of blows, the above equation and that of the authors give close results. Both of these equations may easily be converted to slide-

rule form in the manner demonstrated by Olmstead and Johnston. We found that a 10-inch polyphase duplex slide rule with the special scale inscribed below the folded C scale (CF) works well. The C, D, CF, and DF scales are used and the accuracy should be comparable to that of the A and B scales on the 20-inch slide rule used by the authors.

A comparison of deviations in values of the liquid limit as determined by the chart method, by the authors' equation, and by Equations A and C given previously shows no significant difference in accuracy for a range of blows between 17 and 36 when applied to data on Washington soils. We would be curious to know if this same comparison of accuracy would prevail on a wider range of soils.

In conclusion, we feel that Olmstead and Johnston have contributed greatly to simplifying the routine task of liquid limit determination in soils testing. The data they have presented should provide the impetus for further substantiation of their conclusions and the eventual use of a rapid method as a standard for the determination of the liquid limit of soils.

W.J. EDEN, Division of Building Research, National Research Council, Ottawa, Canada — The authors are to be commended in their effort to summarize the results of liquid-limit determination of soils in the United States. Perhaps it would be of interest to add to the data presented, the results of 150 tests on two Canadian soils. The two soils in question are the "Leda" clay, a marine clay, which occurs in the vicinity of Ottawa, and varved clay occurring at Steep Rock Lake in northwestern Ontario.

The data were originally treated in the manner suggested by the U. S. Army Corps of Engineers, Waterways Experiment Station. Briefly, this method assumes that (1) flow lines plotted on logarithmic paper will be straight lines and (2) soils of the same geological origin will have a constant flow lines slope. With these assumptions, the flow line may be expressed by the equation:

$$L. L. = W_n \frac{N}{25}^{\tan B}$$

where:

N = no. of blows

W_n = water content at N blows

TABLE A
VALUES OF TAN B FOR VARIOUS SOILS

Soil Type and Location	No. of Tests	Tan B
Leda Clay - Ottawa, Canada	100	0.1003
Dark Laminae, varved clay - Steep Rock Lake, Ontario, Canada	31	0.1400
Light Laminae, varved clay - Steep Rock Lake, Ontario, Canada	19	0.0982
Average for Three Canadian Soils	150	0.1082
Alluvial and Coastal Soils - Southern USA (Reported by Vicksburg Water- ways Exp. Station)	767	0.121
Soils from Various Locations in USA (Reported by Olmstead)	759	0.1351

TABLE B
DATA WITHIN RANGE OF TAN B ± 6
(STANDARD DEVIATION)

Soil Type	Tan B	Standard Deviation ± 6	% of Total Observations Within Tan B ± 6
Leda Clay	0.1003	.0516	82%
Dark Laminae - Varved Clay	0.1400	.0404	74%
Light Laminae - Varved Clay	0.0982	.0265	84%
Average	0.1082	.0497	78%

tan B = slope of the flow line when plotted on logarithmic paper

At any particular number of blows N, the equation may be expressed as $L. L. = Wn \times K$. This is a reciprocal relation to the equation expressed by the authors.

$$L. L. = \frac{Wn}{1.419 - 0.3 \log N} = \frac{Wn}{K}$$

Data for 100 tests on Leda clay, 31 tests on the dark laminae of varved clay, and 19 tests on the light laminae of varved clay were collected, and values of tan B shown in Table A were computed.

As can be seen from the values of tan B, the values obtained are close. In the writer's opinion, the geological origin of the soil need not be considered, because it was found in the analysis, that the variation in values of tan B for soils of any particular geological formation was greater than the variations shown in Table A.

To show the errors inherent in this method, take for example a soil with a water content of 50 percent at 20 blows. If the liquid limit is calculated using the various values of tan B shown in Table A, the results are as follows:

Tan B	L. L.
0.1003	48.9
0.1400	48.4
0.0982	48.9
0.1082	48.8
0.1210	48.7
0.1351	48.7

In conclusion, the writer suggests that the rapid methods for determining liquid limit are sufficiently accurate for all but special correlative purposes, certainly for classification purposes. Before a one-point method is adopted, it would be well as suggested by the authors, to make an effort to collect existing data in order to arrive at a truly representative value of tan B or other constant. In this regard, since the authors have already done so much toward this to date, the effort towards standardization of the method could well be sponsored by the Bureau of Public Roads.

F. R. OLMSTEAD and C. M. JOHNSTON,
Closure - It is gratifying to note (from information on the three Canadian soils) that all six of the liquid-limit values Eden used to illustrate the inherent error of the method differ from one another by less than the tolerance we specified as the maximum limit consistent with the reproducibility of the test.

We have conducted many cooperative check tests with a large number of state highway laboratories using different soils at various times. The results of these tests have shown that experienced operators working in the same or different laboratories cannot be expected to check closer than ± 2 percent of the liquid limit of the soil involved, even when using the standard method. Of course, some operators check perfectly, but not every time; therefore, the 2-percent rule was selected to embrace as large a number of operators as possible while still maintaining safe classification of soils for highway use.

Based on this evidence, we feel that a search for a more-perfect series of slopes for the family of typical flow curves would serve no practical purpose, unless we find some soils that invariably fall outside the ± 2 -percent limit.

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