

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 polyphos 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 polyphos 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)	$\text{Na}_6\text{P}_6\text{O}_{18}$ or $(\text{Na}_2\text{P}_6\text{O}_{18})_6$	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	$\text{Na}_4\text{P}_4\text{O}_{13}$	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 $(\text{NH}_4)_2\text{P}_2\text{O}_4 \cdot \text{NH}_4\text{NHP}_2\text{O}_5(\text{NH}_4)_2$ 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 $(\text{C}_{10}\text{H}_6\text{SO}_3\text{NaCH}_2)_2 \cdot \text{C}_{10}\text{H}_6\text{SO}_3\text{Na}$ 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	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 tetrakisphosphate	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)	7 (1)	7 (1)	28 (8)	43 (18)	5.8	
AASHO 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

PRACTICAL CONCENTRATIONS OF THE PHOSPHATES							
Dispersing agent		Practical concentration range (ml. of stock solution)					Practical concentration for podzolic soils 1to 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 tetrphosphate	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		Concentration (ml. of stock solution)	Dispersion value (% of <0.001 mm. group)											Deviation from maximum dispersion value (average for soils 7 to 14)
No.	Name		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	F	38 (0)	F (7)	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 tetrphosphate	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	F	F	28 (2)	37 (1)	68 (16)	39 (0)	F	
AASHO Method with sodium meta- silicate and 1 minute in the motor stirrer		20	8 (20)	7 (16)	9 (34)	34 (26)	14 (31)	F	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

No.	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		
		Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	Max.	Min.	Av.	#2 #6 Av.	#16	#1 #9 #14 Av.
2	Sodium hexametaphosphate (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
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
5	Sodium tripolyphosphate	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
6	Sodium tetraphosphate	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
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

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

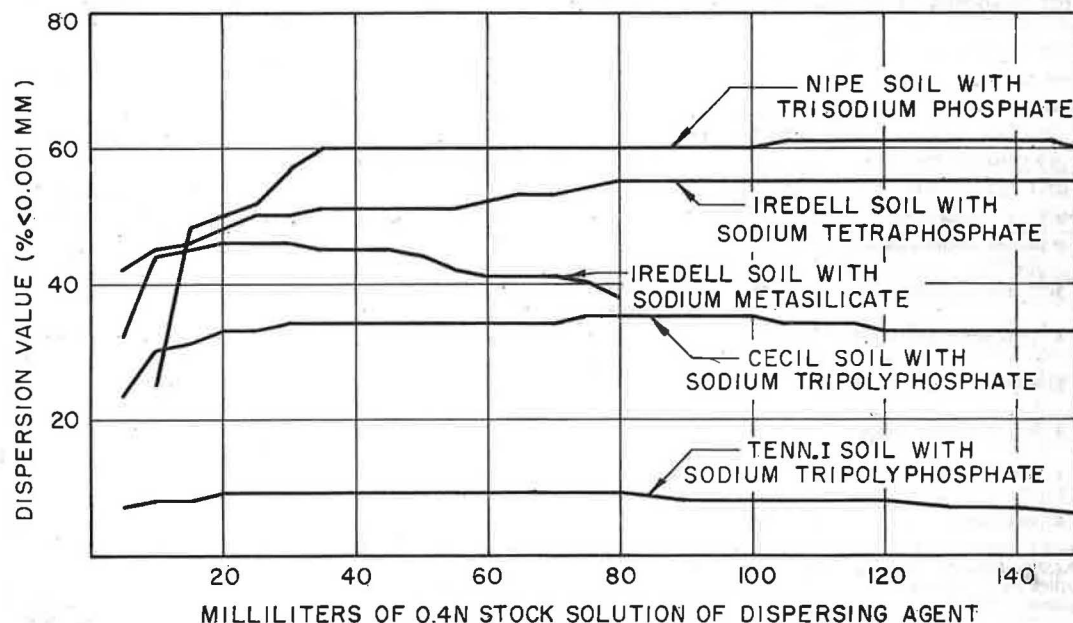


Figure 1. Effect of dispersing-agent concentration on dispersion value.

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 tetrphosphate, 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	0	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 tetrphosphate (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 tetrphosphate 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 tetrphosphate (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	Air-jet dispersion cup	Motor stirrer	Difference
	a	b	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.)

No.	pH of stock solution	pH ranges and deviations from the maximum dispersion values					
		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 percentage-points) of the dispersion value from the maximum value obtained for that soil.

^b F indicates flocculation at all concentrations 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-

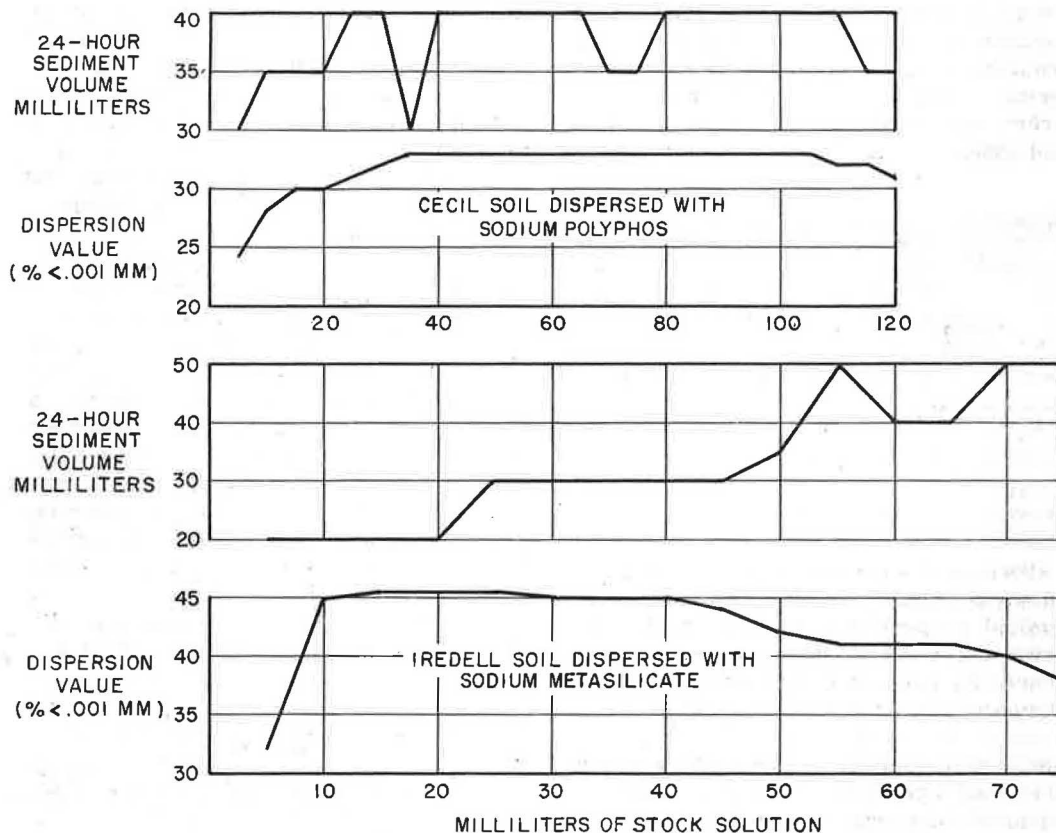


Figure 2. Effect of dispersion value on sediment volume with Cecil and Iredell soils.

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

measurements 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 polyphosph 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 polyphosph 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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