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III. Some Practical Aspects

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●ALL OF THE USUAL testing procedures and identification of most of the chemicals are included in Parts I and II and will not be repeated here. Other equipment and procedures were as follows:

1. The Brabender Plastograph. This instrument, made by the C.W. Brabender Instruments Company, South Hackensack, N.J., is a mixing chamber equipped to measure the torque on the mixing shaft as a function of time. The mixing chamber used in this particular test was a sigma-blade mixer with a 1-qt chamber. The soil was brought to the proper moisture content in an ordinary muller-and-plow mixer and placed in the plastograph. Then the chemicals were added and the change of mixing torque observed with time. This was done at approximately optimum moisture content.

2. The penetration tests made on the field-test patches were conducted with a Soiltest CN-419 Proctor penetrometer. The results were obtained using several of the needles and converted to equivalent pounds required for penetration of $\frac{1}{4}$ in. on the No. 252 needle (0.25-in. diameter). Conversions of readings taken with larger needles were done by use of a factor representing the approximate ratio of surface areas of the respective needles.

3. Corrosion inhibitors were obtained from several companies as well as from experimental lots prepared at Monsanto Chemical Company. Rodine 201 and 203 are made by AmChem Products, St. Joseph, Mo.; Riley 22 is made by Riley Chemical Company, Indianapolis, Ind. MOKI corrosion inhibitor is under development by Monsanto Chemical Company's Corrosion Laboratory, St. Louis, Mo.

4. Sixty-eight test cylinders (2 by 4 in.) were prepared representing some 17 different conditions of chemical treatment or mode of preparation. Each condition was represented in quadruplicate. The order of preparation of these samples was randomized to eliminate operator deviations. The samples were placed randomly in the ground and covered with closely packed earth from the site. Because of the randomization of the sample burial, factors such as drainage and temperature from the nearby building were eliminated. The samples were removed in pairs from the ground at two different times—one set in mid-winter, the other in spring—so that the results are averages of two strength tests per variable.

5. The soil used in the experiment described as the Creve Coeur parking lot test was sampled from another location, tested in the usual manner in the laboratory, and then trucked in and dumped on the construction site. The soil was spread and aerated by means of a garden rototiller until the desired moisture content was attained. Water and phosphoric acid were added to the soil from water cans and the mass mixed by the rototiller. Phosphoric acid was added as a 50 percent solution in water. Compaction was done with pneumatic hammers of the type commonly used in back-filling ditches and the like. The compacted soil was tested for density by means of the Soiltest Volumeasure CN-980, determinations of amount of phosphoric acid present in the compacted soil were done by the usual wet chemical methods and also by X-ray fluorescence. Moisture content was determined by drying in an oven at 105 C overnight. Pen-

etration tests were made as previously described. The completed test patch (20 ft by 20 ft) was covered with road oil and then paved with hot-mix bituminous concrete.

RESULTS

Mixing Energy Requirements

The results of a brief study with the Brabender Plastograph are shown in Figure 1 for Keyport clay loam. In the figure, mixing torque is plotted vs time. In the upper plot, the mixing torque increased as the water was worked into the soil. Then 2 g or about 0.5 percent of N-tetrapropenyldiethylene triamine (a polyamine) were added and a dramatic drop in mixing torque was observed. The validity of this effect was reinforced by visual observation of a drier appearance resulting from the addition of the amine. In the second plot, 2 percent of phosphoric acid was added to the moist soil in the mixer and an immediate decrease in torque was observed that slowly built up again. Subsequently, 0.5 percent of the polyamine was added and again a dramatic drop in mixing torque was observed. In the lowest plot, the soil was mixed as in the upper plot for an extended period of time before addition of the phosphoric acid. Again, a decrease in torque was observed that returned to an intermediate level with further mixing. Torque decreased again by an addition of the polyamine. Therefore, Figure 1 seems to show that phosphoric acid by itself will decrease mixing torque (i.e., make field handling of the treated soil somewhat easier) and that a polyamine may be expected to improve dramatically the mixing qualities of clay soil.
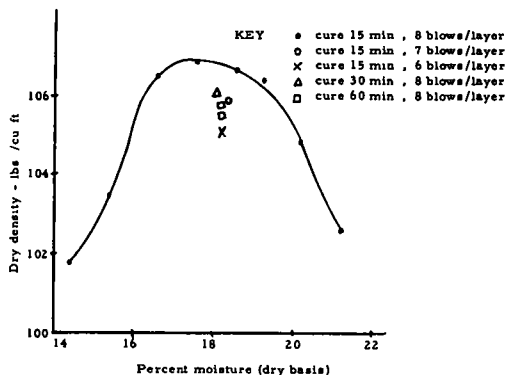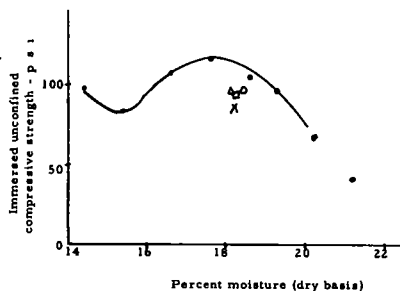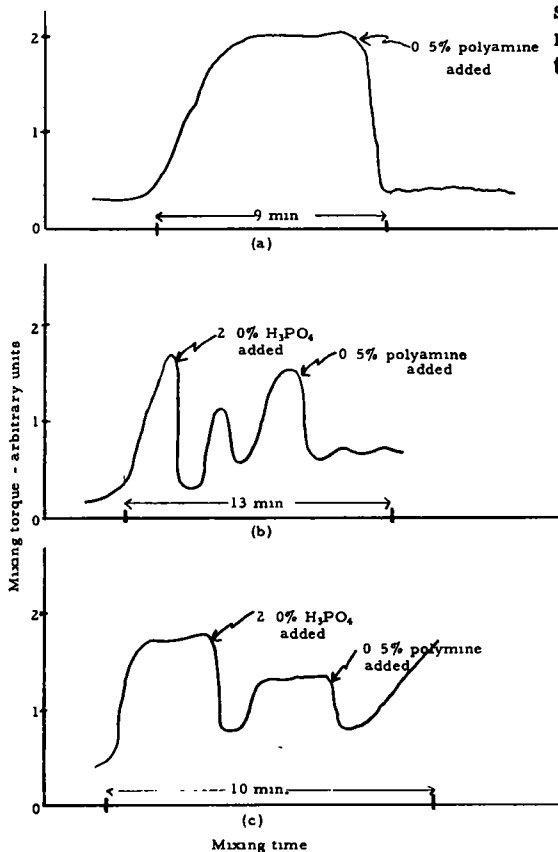

Figure 1. Mixing energy input for Keyport clay loam A-7-6 (12): (a) 392 g soil, 66 g H_2O; (b) 392 g soil 60.5 g H_2O; and (c) 392 g soil, 67.5 g H_2O.

Figure 2. Effect of curing time before compaction and compactive effort on density and strength for 2 percent H_3PO_4 in Cecil clay loam.

The effect with phosphoric acid is probably due to a slight dispersion of the clay solids and has been noted qualitatively by Michaels, Williams, and Randolph (1). The effect of the polyamine has been discussed previously in Part II of this paper.

Effect of Some Variables on Compaction

In the usual laboratory tests described in Parts I and II the soil is mixed for the specified length of time (5 min) and then allowed to damp-cure for 15 min before compaction in a specified manner. For field work such rigid control will not be possible. Rather, a dry density is specified that the contractor must achieve irrespective of curing time and, to a lesser extent, irrespective of moisture content. Moisture contents are usually not specified closer than $\pm$ 1-2 percent of optimum. In Figures 2 and 3, the effect of changing curing time before compaction is shown for two clay soils. In Figure 2, the effect on a Cecil clay loam is shown. The loss in density under fixed curing conditions and less compaction is shown. With increased curing before compaction, a greater number of blows is required to duplicate the density obtained with the standard condition. The important fact, however, is that the strength and density can be duplicated by a moderate increase in compactive effort. In Figure 3, the effect on another Cecil clay is shown. The figure shows the loss in both density and strength by allowing 1 hr to elapse before compaction with 7 blows per layer. Again, the compensatory effect of increasing the number of blows per layer is demonstrated. Figure 2 shows that, although compaction will be somewhat more difficult as more curing time elapses, it will be possible to make up for the effect of the hardening of the soil by increasing the compactive effort. It is interesting that in Figure 3 on the wet side of optimum, even though density may be increased by extra compaction above the baseline condition, the strength of the soil evidently may not be increased appreciably as evidenced by the points falling approximately on the control line on the strength curve. Evidently, increased strength from
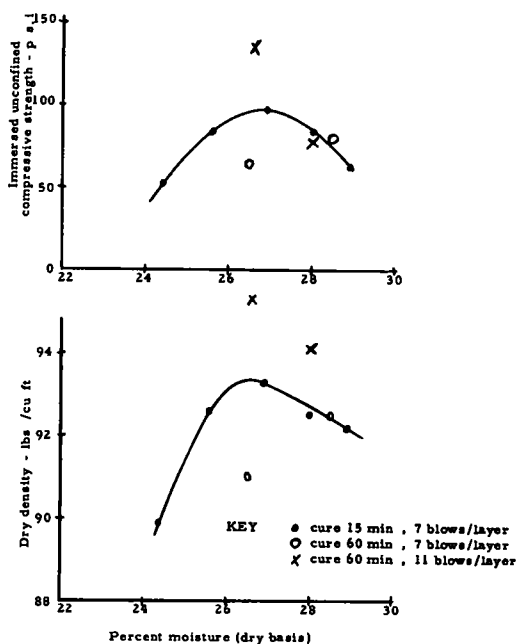

Figure 3. Effect of curing time before compaction and of compactive effort on density and strength for 2 percent H_3PO_4 in Cecil clay loam from Georgia.
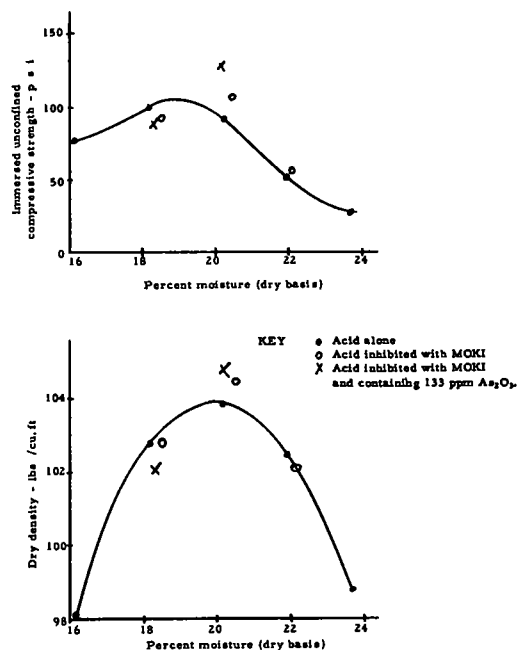
Figure 4. Effect of corrosion inhibitors on stabilization with 2 percent H_3PO_4 in Cecil clay loam A-7-5 (17).

extra compaction may be obtained at optimum moisture content or possibly at 1 or 2 percent less than optimum, but it will be very difficult to obtain increased strength on the wet side of optimum. The importance of maintaining close control on moisture as well as density is underscored.

Effect of Corrosion Inhibitors on Performance

Because phosphoric acid will normally be handled in mild-steel equipment by contractors, a corrosion inhibitor will often be added. These corrosion inhibitors are added in amounts of several tenths to 1 percent by weight of the acid. It was therefore required to know the effect of this amount of corrosion inhibitor on the performance of phosphoric acid as a soil stabilizer. Figures 4 and 5 show strength and density curves for phosphoric acid with several corrosion inhibitors. In Figure 4 the effect of adding Monsanto's MOKI inhibitor is shown to be negligible. Figure 4 also shows the effect of having 133 ppm As_2O_3 present in the acid. This represents one of the differences between food grade and non-food grade phosphoric acid. Again, it is seen that there is no significant change in either strength or density when either corrosion inhibitor or As_2O_3 is present in the acid. In Figure 5, results of a spot check are shown for three commercially available corrosion inhibitors. Little effect is found when Riley 22 or Rodine 201 is added. There seems to be some effect with Rodine 203 and this inhibitor should be tested in the soil and acid combination under study before use in the field. In general however, it is concluded that

KEY
- Acid alone.
- o Acid inhibited with 1 6% Reilly 22 (100% acid basis).
- X Acid inhibited with 0 6% Rodine 201 (100% acid basis).
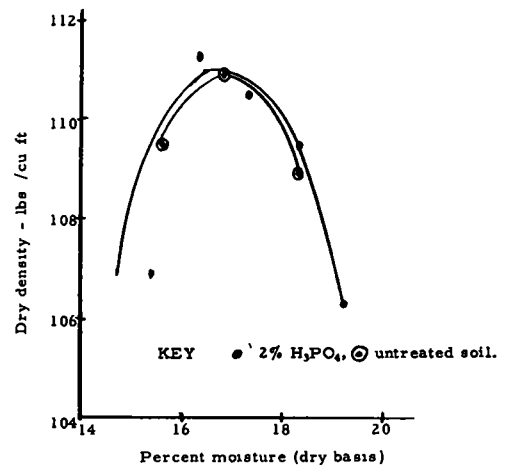- △ Acid inhibited with 0.4% Rodine 203 (100% acid basis).
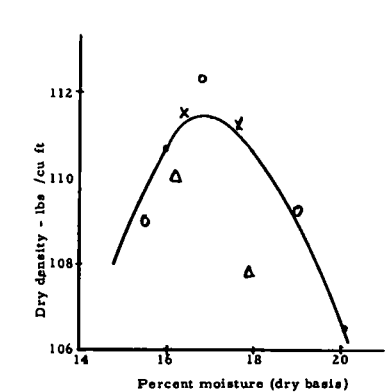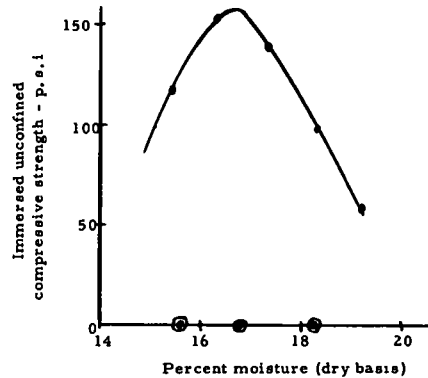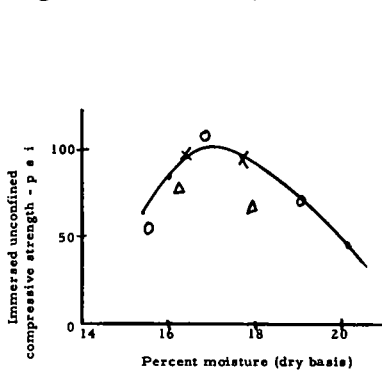
Figure 5. Effect of corrosion inhibitors on stabilization with 2 percent H_3PO_4 in Keyport clay loam A-7-6 (12).

KEY ● 2% H_3PO_4, ⊘ untreated soil.

Figure 6. Stabilization with 2 percent H_3PO_4 in clay soil from Creve Coeur, Mo.

corrosion inhibitors have little effect on stabilized strength or dry density of clay soils.

Outdoor Exposure of Test Cyliners

Of the test cylinders prepared and buried in a statistical fashion in reasonably well-drained soil at Everett, Mass., in the fall of 1956, one-half of the samples were removed in February 1957 and broken in unconfined compression. The other half of the samples were removed and broken in May 1957. The results are shown in Table 1. The compactive effort was 12 blows per layer and 24 blows per layer. It is clear in the table that the plain soil samples essentially failed during this test; that is, 9 and 21 psi of unconfined compressive strength are not considered adequate for most modern subgrades. The results for the phosphoric acid treatment show that straight reagent-grade phosphoric acid is the least effective of the phosphoric acids tested, although it is satisfactory and gains strength with age, even under these exposure conditions. As described in Part II, the best sample was that prepared with a wet-process phosphoric acid containing organic sulfates and sulfonates. Using a high compactive effort, strength exceeded 200 psi after exposure and no loss was noted due to the longer exposure time. This same acid with the lesser compactive effort still had strengths approaching 200 psi, whether or not mixing time was normal or reduced by a factor of two (labeled "poor mixing").

The wet-process acid made from pure sulfuric acid (containing no organic matter from the sulfuric acid) performed less effectively than the acid made from spent sulfuric. However, an attempt to duplicate this acid by adding an organic sulfonate to

TABLE 1

OUTDOOR EXPOSURE OF 2- BY 4-IN. TEST CYLINDERS[a]

Treatment	Blows per Layer	Unconfined Compressive Strength (psi)	
		65 Days	155 Days
Untreated	12	9	21
	24	9	24
2% Reagent grade H_3PO_4	12	79	86
2% Wet acid from spent alkylator H_2SO_4	12	154	190
2% Wet acid from pure H_2SO_4	12	166	132
2% Wet acid from spent alkylator H_2SO_4 poor mixing	12	192	180
3% Wet acid from spent alkylator H_2SO_4	12	190	187
2% Wet acid from spent alkylator H_2SO_4	24	202	215
2% Reagent grade H_3PO_4 + 0.5% polyamine[b]	12	134	153
2% Reagent grade H_3PO_4 + 0.2% dodecylbenzene sulfonic acid	12	126	88
1% Polyamine alone	12	21	20

[a]Keyport Clay Loam A-7-6(12), buried Oct. 1956, tested Feb. or May 1957 at Everett, Mass.
[b]N-tetrapropenyldiethylene triamine.

reagent-grade phosphoric did not succeed. The effect of adding 0.5 percent polyamine also was not particularly notable. The effect of adding the amine by itself in larger quantities was a distinct failure. Three general conclusions are drawn:

1. Reagent-grade phosphoric acid is inferior to the wet-process acid in this particular test, but it can be improved considerably by addition of either organic sulfonates or polyamine.

2. Increasing the acid level in this test was not as effective as increasing the compactive effort.

3. A reduction in mixing time does not effect the strength adversely within the limits of this test.

Part II showed that the various acids produced changes of about 10 percent on strength. Because the present results are subject to many more errors due to the outdoor exposure, it is not necessarily true that such a wide difference in stabilizing power exists between types of phosphoric acids. The main conclusion to be drawn is that phosphoric acid-treated clay soil will withstand severe weathering and cycling of freezing and thawing as typified by the climate at Everett, Mass., during the winter of 1956-57. Thus, frost resistance, etc., is deemed to be sufficiently good to proceed with extensive field testing in frost zones as well as in the southern parts of the U.S.

Parking Lot Test Section

The construction of the 20- by 20-ft test patch at Creve Coeur, Mo. in 1958 was

Figure 7. Mixing and compacting treated soil at Creve Coeur, Mo., August 1958.

described in the experimental section of this paper. Figure 6 shows the density and strength curves obtained in the laboratory for untreated soil and soil treated with 2 percent phosphoric acid. Excellent stabilization resulted. The untreated soil had zero strength after immersion in water. Figure 7 shows some of the construction methods used in this small test patch. The soil was aerated until the moisture was reduced to about $13\frac{1}{2}$ percent before beginning the work. Water was added to raise the moisture to the desired level at about 17 percent and phosphoric acid, as a 50 percent solution in water, was sprayed onto the site while mixing the soil with the rototiller. Screen size of the soil before the addition of acid was approximately 5 percent retained on a $\frac{3}{8}$-in. screen and 38 percent retained on a No. 5 screen. Mixing was continued with the rototiller until uniformity was achieved by visual inspection. The patch was then compacted by hammering with pneumatic hammers until no futher densification could be achieved. Sampling in each of four locations by means of in-place density testing, revealed that at least 95 percent of standard density was achieved and moisture was controlled at 16.2 $\pm$ 0.5 percent. Phosphoric acid level was 2.3 $\pm$ 0.6 percent. Thus,

TABLE 2

PERFORMANCE DATA ON 20- BY 20- BY 0.5 FT TEST PATCH,[a]
CREVE COEUR, MO.

Location	Date	Penetration Resistance (lb on No. 252 needle or equiv.)				H_2O (%)	H_3PO_4 (%)
		0-1 in.	1-2 in.	2-3 in.	3 in. + Lower		
N.W. Corner	Oct. 1958	74 $\pm$ 6	82 $\pm$ 4	92 $\pm$ 13	–	17.4	3.0
	May 1959	52 $\pm$ 6	44 $\pm$ 3	5 $\pm$ 0	0	20.8	1.23
Center	Oct. 1958	86 $\pm$ 13	84 $\pm$ 5	86 $\pm$ 12	–	17.9	2.26
	May 1959	35 $\pm$ 3	42 $\pm$ 2	41 $\pm$ 6	34 $\pm$ 3	19.4	1.73
S.E. Corner	Oct. 1958	71 $\pm$ 8	52 $\pm$ 8	37 $\pm$ 9	–	20.3	2.52
	May 1959	36 $\pm$ 6	44 $\pm$ 10	37 $\pm$ 4	32 $\pm$ 9	19.2	2.15

[a]Built Aug. 1958.

TABLE 3

PROFILE OF PENETRATION RESISTANCE AND ACID CONCENTRATION,
20- BY 20-FT TEST PATCH, CREVE COEUR, MO.[a]

Depth (in.)	Penetration Resistance (lb on No. 252 needle or equiv.)	H_3PO_4 (%)
0 - 1	37 $\pm$ 7	1.96
1 - 2	38 $\pm$ 6	1.87
2 - 3	34 $\pm$ 4	1.99
3 - 4	20 $\pm$ 5	1.95
4 - 5	$7\frac{1}{2}$ $\pm$ 2	1.43
5 - $6\frac{5}{8}$	4 $\pm$ $\frac{1}{2}$	0.61
$6\frac{5}{8}$ - 8	25 $\pm$ 7	0.43
8 - 9	5 $\pm$ $\frac{1}{2}$	0.28
9 - 10	–	0.34
10 - 11	4 $\pm$ $\frac{1}{4}$	0.33
11 - 12	6 $\pm$ 1	0.40

[a]Data taken May 1959.

there was considerable variation in density, moisture, and acid level across the test section indicating some inadequacy on the part of the mixing equipment and the method of distributing the acid.

Each section was evaluated principally by obtaining data with a Proctor penetrometer. At the time of sampling with the penetrometer small portions of soil were removed for water and phosphoric acid analyses. Table 2 presents the data obtained in the fall of 1958 and again in the spring of 1959. The data were obtained in different holes through the pavement and therefore are not wholly comparable due to the variation in phosphoric acid level from spot to spot. The numbers in the table represent pounds of force necessary to force a needle 0.252 in. in diameter into the soil to a depth of $\frac{1}{4}$ in. The actual face area of the needle is .05 sq in., so that the numbers in Table 2 may be converted to psi by multiplying by 20. In short, even a relatively low number in the table represents a fairly high strength. In the table, the comparison between fall and spring readings in the top layer indicates reduction of 35 to 50 percent in strength, but a very strong layer is still retained. A similar result was obtained for the layer down

TABLE 4

COST DATA ON PHOSPHORIC ACID STABILIZATION[a]

Item	Cost ($)
Cost/ton 75% H_3PO_4	72[b]
Cost/lb 100% H_3PO_4	0.048
Cost/ft^3 soil at 100 pcf	0.096
Cost/yd^2 0.5 ft thick	0.432
Processing cost/yd^2	0.22
Total cost/yd^2	0.65
Competitive cost/ton for rolled stone in place	2.05

[a] 2% H_3PO_4, dry soil basis, fertilizer grade
[b] Approx. plus freight.

TABLE 5

ECONOMIC DATA FOR PHOSPHATE ROCK PLUS SULFURIC ACID

Item	Cost ($)
Phosphate Rock f.o.b. mine:	7-9
Grinding cost	2
Ave. freight	8
Total/ton	17-19
H_2SO_4/ton (inc. freight)	25
Raw material cost/ton of 100% H_3PO_4 produced	77[a]
Cost/yd^2 0.5 ft thick	0.35
Processing cost/yd^2 [b]	0.32
Total/yd^2	0.67[c]

[a] Cf. $96 sale price for 100 percent acid.
[b] Includes extra $0.10 for premixing or double in-place mixing.
[c] Cf. $0.65/yd^2 for acid per se.

TABLE 6

RAW MATERIAL COSTS FOR ADDING AMINES TO STABILIZER

Concentration (%)	Amine (Cost/yd^2 0.5 ft thick)	
	at $0.20/lb	at $0.50/lb
0.1[a]	0.009	0.22
0.5	0.45	1.10

[a] Dry soil basis.

1 to 2 in. from the top. In the third layer from 2 to 3 in. down, the same conclusions hold except for the hole in the northwest section where apparently no acid was present in this 2- to 3-in. layer as evidenced by essentially no strength. Also, the water content had increased in the northwest and center sections from a starting point of around 16 percent when the job was built to 17.4 to 17.9 percent in the fall, and up to about 19.4 to 20.8 percent by spring. Conversely, in the southeast corner, which was wet by accidental conditions during the construction, a decrease in moisture was found during the winter season. Phosphoric acid analyses for these same holes show considerable variation.

Due to the rapid decrease in strength in the northwest sample hole in the spring of 1959 when descending from about the 2-in. level to about the 3-in. level, one large hole was opened up and sample readings carefully taken at 1-in. intervals down to a depth of 1 ft. These data are presented in Table 3. Penetration resistance is shown to be at a fairly high level down to the 4-in. mark and phosphoric acid content also at a reasonably high level down to the 4- to 5-in. mark. Below this point the acid level dropped sharply as did the penetration readings. The base-line phosphate reading in

TABLE 7

COST OF HYDROFLUORIC-PHOSPHORIC ACID MIXTURE AT EQUAL
PERFORMANCE LEVELS IN SILTY CLAY[a,b]

Acid Concentration	Raw Material (Cost/yd^2 0.5 ft thick)
2% H_3PO_4	0.43
1% Mixed acid (30% in F)	0.36
1.2% Mixed acid (19.35% in F)	0.38
1.33% Mixed acid (10% in F)	0.36
1.7% Mixed acid (1% in F)	0.41

[a]See Part II, Figure 7, for performance curves.
[b]70 Percent HF at $0.134/lb.

this soil is about 0.2 to 0.3 percent and it is concluded that no migration downward of the phosphoric acid occurred. This is a measure of the permanence of the treatment. The penetration resistance should fall off about 1 in. sooner than the acid level due to the nature of the test; i.e., there must be some support from beneath in order to get a good reading. The test at the 4- to 5-in. level is really a test of strength of the material somewhat below 5 in. which had very little phosphoric acid in it. Table 3 shows that the phosphoric acid level was about as expected down to 3 to 4 in. and began to fall off rather sharply thereafter. When the phosphoric acid was applied to the top surface of the soil, the rototiller did not fully distribute the liquid down to the full depth of mixing. The rototiller was actually combing the soil rather than thoroughly homogenizing it. Subsequent observations on the operations of large-scale pulvimixers of a similar design show that true homogenizing does occur with the latter mixers because of the much more violent action obtained.

The 20- by 20-ft test patch has performed very satisfactorily, is now 2 years old, and is judged to be in better condition than the adjacent surfaces, which are paved over a thin 3-in. layer of crushed stone.

ECONOMIC ASPECTS

Table 4 presents some cost figures for phosphoric acid stabilization. The table assumes the use of fertilizer-grade acid, a soil that compacts to a dry density of about 100 pcf, and the use of modern soil stabilization equipment. The cost table is developed for treatment with 2 percent of 100 percent H_3PO_4 based on dry soil weight. The raw material cost to treat 1 sq yd of road surface to a compacted depth of 6 in. is seen to

be $0.432 and the total cost assuming a processing charge of $0.22 per sq yd is then $0.65. The $0.22 figure for processing includes the cost of distributing the acid from a pressurized tank wagon, mixing with a high-speed rototiller or a pug-mill device, and shaping and compacting to grade. The figure does not include costs necessary to bring the road surface to a rough grade, nor does it include sealing with asphaltic material and ultimate surfacing. The figure used is a typical one given by a state highway organization. It is lower than the figures normally given for soil-cement or soil-lime stabilization in light of the advantages to be obtained by use of a water-soluble, liquid soil stabilizer. The assumption has been made in the table that the phosphoric acid can be satisfactorily handled in normal mild-steel equipment provided that a corrosion inhibitor is added to the acid. The figure of $0.65 per sq yd compares favorably with other methods of soil stabilization even in sandy-type, less intractable soils. Note that this method of stabilization will compete with a rolled stone base at $2.05 per ton compacted in place at the same thickness. $2.00 per ton is a commonly encountered figure that is often exceeded in those states that are experiencing shortages of aggregates. There will be other states where stone is available at a price considerably less than this figure. In this latter case, the approach of building a road foundation with stabilized soil might not be considered in any case.

Table 5 shows the economics for treating soil with a mixture of phosphate rock and sulfuric acid to produce, in situ, phosphoric acid. The figures are typical for costs of phosphate rock in this country, either in Florida or in Tennessee (for the eastern markets). An additional $0.10 to $0.15 per sq yd processing cost is added because of the difficulty of premixing phosphate rock and sulfuric acid in equipment made of materials other than rubber-lined or stainless steel or because of the additional processing step necessary in a two-step in-situ mixing operation. The over-all economics then become very similar to those for straight phosphoric acid soil stabilization and it would probably be easier in most cases, to use phosphoric acid itself rather than its components.

Table 6 shows cost figures for addition of amines to phosphoric acid at two price levels and two concentration levels and indicates that addition of amines will be prohibitively expensive. The table also gives costs for mixtures of phosphoric and hydrofluoric acids at equal performance levels in silty clay soils (Part II, see Fig. 7). From 1 to 10 percent HF based on phosphoric acid present will reduce the economics somewhat as compared to straight phosphoric acid in silty soils. Conversely, somewhat more than the amounts of acid shown may be used in silty soils at a cost equivalent to that for 2 percent phosphoric acid. The latter case will usually prevail because 2 percent phosphoric acid alone is not adequate in many silty soils. In some cases it may be necessary to go as high as 2 percent of the 30 percent mixture and the raw material cost will rise to over $0.70 per sq yd; the total cost will approach $1.00 per sq yd.

The commercially available Rodine corrosion inhibitors will add approximately $3.00 per ton to the cost of 75 percent phosphoric acid or about $0.002 per lb of acid used. Some of the experimental inhibitors now under study at Monsanto Chemical Company will add about $1.20 per ton, or less than $0.001 per lb of acid.

In sum, the method of stabilization described in this paper is an economical procedure that can be used in those states where aggregate shortages exist. The costs compare quite favorably with other soil stabilization methods now used in this country.

POTENTIALS

The technical feasibility of stabilizing clay soils with phosphoric acid has been demonstrated conclusively by these investigations and by those of many others now working in this field. The preliminary field-test work described herein indicates that there are no insurmountable problems pertaining to the field application of the method. Durability of the stabilized soil appears to be good. The attractive features of a liquid, water-soluble stabilizer that is economically priced and widely available have interested many organizations. Many laboratory investigations are now underway in laboratories of state highway organizations, and more than one field test installation has been in-

stalled. This method shows promise of becoming widely accepted as a means of stabilizing fine-grained, problem soils.

<div align="center">REFERENCE</div>

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