Louisiana Highway Research

CORRELATION OF RAPID HYDROMETER ANALYSIS FOR SELECT MATERIALS TO EXISTING PROCEDURE LDH-TR-407-66

Louisiana Department of Highways – Research & Development Section

CORRELATION OF RAPID HYDROMETER ANALYSIS FOR SELECT MATERIAL TO EXISTING PROCEDURE LDH-TR-407-66

Final Report

by

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SYNOPSIS

Conditions arise during construction of bases with Portland cement stabilized soils which require close programming of work. Therefore, time is of significant importance.

That is the objective of this report; to evaluate a method by which considerable savings in time can be made. Under accepted procedures for issuing cement contents to the project engineer, a maximum of 2-½ days can elapse before laboratory testing is completed. However, utilizing the tentative rapid hydrometer test investigated in this report, this maximum time can be reduced to 24 hours with no detrimental affect on the accuracy of the results.

This report contains the laboratory results and statistical evaluation of a rapid hydrometer analysis as compared to L. D. H. TR 407-66, Method of Test for Mechanical Analysis of Soils.

After a statistical analysis of the results obtained from 180 samples run by three separate laboratories, it has been determined that the accuracy and repeatability of the rapid method is at least equal to the conventional test. Therefore, it is possible to replace TR 407 with the rapid method for soils with plasticity indices less than 15.

In view of the fact that sufficient samples have been run to statistically prove that it is possible to interchange the two tests, it is recommended that the Department consider the adoption of the rapid method as a valid test. It is further recommended that each laboratory run a series of soils from their respective districts to ascertain if the soils react in an acceptable manner and personnel are sufficiently consistent to perform the rapid method satisfactorily. Each laboratory that can complete this series, obtaining results equal to the conventional test, should then be able to interchange the two tests for select materials without difficulty.

ACKNOWLEDGMENTS

Appreciation is extended to Mr. Billy C. Sharp, District 58 Laboratory Engineer, and Mr. Cecil M. Watson, District 61 Laboratory Engineer, each responsible for running one-third of the samples for this research study.

Special thanks are offered to Mr. Shashikant C. Shah, Data Analysis Engineer, who analyzed all the results obtained during this study and who also contributed the section of the report Statistical Analysis of Data.

The above people's time and energy were a great aid in completing this study.

INTRODUCTION

In stabilization of soil with Portland cement it is necessary for the project engineer to submit samples from the roadway to the district laboratory for determination of cement content. The cement content provided to the field is based on a soil classification obtained in part by hydrometer analysis which can require a maximum of 2-½ days to complete. This time lapse has, at times, proven to be detrimental to smooth field operations.

During the past several years some of the district laboratories have been running what has become known as a "quickie" hydrometer analysis as a replacement for the conventional test, L. D. H. TR 407, on soils to be utilized in soil-cement stabilization.

The "quickie", a more rapid test requiring a maximum of 24 hours, was created under guidelines established by the Department in 1962. This test was a result of needing soil classification information on which to base cement content as soon as possible to meet the needs of field construction forces involved in cement stabilization.

Since there is no established test procedure in the L. D. H. Testing Procedures Manual dealing specifically with this test, some testers have been reluctant to utilize the rapid method for official determinations of cement content, thus requiring additional amounts of time to issue results. The purpose of this study is to evaluate the 24 hour test and, if feasible, present a workable test procedure to supplement our present test method for use with select materials.

It is evident that a considerable savings in time is possible, which directly relates to economy, using this tentative procedure.

SCOPE

This program was initiated December 1, 1967, as a cooperative research project between the Louisiana Department of Highways and the Bureau of Public Roads.

The principal objective of this investigation was the establishment of the acceptability, as to accuracy and repeatability, of a rapid method of hydrometer analysis when compared with existing procedure L. D. H. TR-407-66. This was accomplished by a statistical analysis of results from both methods obtained from comparative samples.

This study was confined to select materials suitable for cement stabilization. No attempt was made to evaluate the possibility of replacing TR-407 for soils with plasticity indices greater than 15.

METHOD OF PROCEDURE

At the inception of this program a series of 15 samples were run by District 07 Laboratory to determine the guidelines to be followed during the research. These first 15 samples were run under both the rapid and conventional methods utilized in the actual research program.

Based upon the analysis of these preliminary samples, the sampling and testing portion of the program was designed to be completed by three separate district laboratories as discussed below. The testing results would then be subjected to statistical analysis as a phase of the study.

A. Sampling

Each district laboratory obtained a series of 60 samples which represented the range of natural occurring select soils represented in their respective districts. These select soils are those that are suitable for cement stabilization without lime treatment in AASHO Classifications A-1-a, A-1-b, A-3, A-2-4, A-4 and/or A-6 with a maximum plasticity index of 15.

These individual samples were brought to the laboratory and handled in the same manner as select samples that are brought in from a construction site by project personnel. That is, the samples were placed in an oven and were dried (in several pans as necessary) in sufficient time to complete both the experimental and conventional tests.

B. Testing

During running of the preliminary samples, preparation was found to be a very vital operation in the attempt to obtain correlative results. During this part of the investigation it was found that the most reliable results were obtained when a sample splitter was used to reduce the original samples into 100 gram hydrometer samples. Therefore, a precision sample splitter was used in each laboratory to reduce the samples for the hydrometer test. All preparation was carried out under L. D. H. TR-411, Method of Dry Preparation of Disturbed Soil Samples for Test.

Another problem encountered during the early stages of the investigation was the breakdown of particles, mostly sand, during dispersion. The guidelines used since 1962 stated a dispersion time of 10 minutes for the rapid method as did the research proposal. Mr. J. R. Blystone's technical comments attached to a letter by Mr. F. E. Hawley, dated June 17, 1967, notifying the Department of approval of the project pointed out this possibility. A series of soil samples run specifically to verify the 10 minute dispersion time definitely indicated excessive abrasion. Further testing indicated that a shorter time of 5 minutes was satisfactory in that it closely correlated with results obtained from conventional testing with less danger of excessive particle breakdown.

The actual testing of the research samples involved running two separate sequences, conventional and experimental. After the sample from the field was prepared, six 100 gram hydrometer samples were separated using the precision splitter. Three of the 100 gram samples were run according to the rapid method contained in the appendix. These tests were completed within 24 hours after receipt from the field. The other three samples were run in the conventional manner following L. D. H. TR-407.

Both test methods are similar in that many techniques are common, and the equipment utilized in each is identical. However, major variations do exist, some necessary to increase the speed of testing in the rapid method. These differences are:

1. Soaking of the sample in calgon solution for 12 hours is eliminated.
2. Sample dispersion is increased from one minute to 5 minutes.
3. Sample size is maintained at 100 grams regardless of soil type.

C. Treatment of Data

Data were analyzed using standard statistical procedures and a Programma 101 desk computer.

The data shown in Tables 4, 5, and 6, representing the averages of the clay fraction for the three conventional and three rapid tests completed on each sample, was utilized in the final analysis.

All data not shown is on file and is readily available for any interested party.

STATISTICAL ANALYSIS OF DATA

Investigators in many fields are frequently confronted with the necessity of comparing two tests, processes, analytical methods or two different materials. One phase of statistical analysis that is of most value to the engineer studying and analyzing data is the t-test. The purpose of the t-test is to determine whether there is a significant difference between the two items under test in terms of the measurements involved or whether the mean difference is significantly different from zero.

In the two test methods under study, we are interested in determining whether one of the two methods, viz, the experimental or the quickie method, can replace the standard conventional method for determining soil particle distribution (clay content). Let us tentatively adopt an attitude that there is no difference between the test methods. The statistic "t" is then computed by the following expression:

$$t = \left| \frac{\bar{d} - O}{S/\sqrt{n}} \right| \qquad \text{where,}$$

$\bar{d}$ represents the average difference within paired measurements, i. e. $\sum_{i=1}^{n} \left[X_{i1} - X_{i2} \right] /n$ and S/ is the standard deviation of the mean difference.

The purpose of taking paired measurements is to overcome the extraneous effect of different soil types. In other words, we are typing to make sure that the two members in any pair are alike in all respects except that which we are trying to measure.

If the calculated value of "t" exceeds the tabulated value at the proper degrees of freedom and the significance level decided upon, then the null hyopthesis that the mean was equal to zero can be rejected with the accepted risk of error. We say, in effect, there is a significant difference between $\bar{d}$ and zero.

Table 1 below shows the calculated "t" value for the mean difference between the two test methods. Clearly, the difference is statistically significant at the 0.05 probability level. This means that there is less than 0.05 chance of being wrong, that the null hypothesis is false, since the occurrence of a "t" of 5.00, 3.01 and -9.35 is an event which has less than 0.05 chance of happening if the hypothesis was true.

TABLE 1

Laboratory		Calculated "t"	t.05
A	0.71	5.00	2.00
B	0.52	3.01	
C	-1.71	-9.35	

It should be pointed out that although the difference may be significant in a statistical sense, it should not be construed that the experimental test would not be a good replacement for the standard conventional one. The value of the parameter $\bar{d}$ should be scrutinized before final evaluation of the test method is made. Laboratory A and B indicate this value to be less than one percent and clay content is generally reported to the nearest one percent. However, the values reported by laboratory C seems too high to have occurred due to chance. Positive sign for $\bar{d}$ is indicative of higher clay content values for the experimental test. The negative sign, as for laboratory C, is indicative of lower values.

In order to investigate how the two methods correlate, correlation coefficients were computed for the three sets of data. These are recorded in Table 2 below. A value of one for this coefficient indicates perfect relationship and that of zero, none. The slope of the regression line in all cases is approximately one. Since we have shown the average difference between the test methods to be less than one percent, the slope of one would mean that on the average, a unit change from a given clay content for any one method would show a corresponding unit change for the other method to within one percent of the given value.

TABLE 2

Laboratory	Corr. Coeff.	Slope
A	0.9906	0.9772
B	0.9866	0.9931
C	0.9723	0.9757

As a final analysis, testing variance as the variation within measurements was computed for the two test methods. These values are recorded in Table 3. For laboratory A, the standard deviation of a single measurement for a single soil sample using the experimental test is 0. 44 (variance 0. 20). The corresponding value using the conventional test is 0. 52. Laboratory C indicated the largest variation of the three laboratories for both the tests. The experimental test, however, exhibited smaller variation than the conventional one.

TABLE 3

Laboratory	Testing Variance	
	Conventional	Experimental
A	0. 27	0. 20
B	0. 14	0. 19
C	0. 56	0. 42

Summing up the above analysis, the t-test indicates significant statistical difference between the test methods. However, the mean difference between paired measurements is of such a small magnitude in two cases out of three, that it would seem justified to use the experimental test method in lieu of the standard one. The argument in favor of such replacement is further demonstrated by the correlation coefficients presented in the second analysis and the extent of the testing variance in the third analysis.

CONCLUSIONS

(1) On the basis of the above analysis it is possible to replace the conventional test with the rapid test for select materials intended to be stabilized with Portland cement.

(2) There is a direct savings in time in that the rapid method produces results in 24 hours, while the conventional method may require up to 2. 5 days.

(3) Dispersion of a hydrometer sample for 10 minutes can cause excessive breakdown of soil particles.

(4) A dispersion time of 5 minutes, in general, closely correlates with conventional results.

(5) A workable test procedure has been developed.

RECOMMENDATIONS

In view of the results of this program it is recommended that the Department consider accepting the rapid method as a valid test procedure. This method would be used only on select materials with plasticity indices less than 15.

It is also recommended that each district laboratory run a series of soils in a manner similar to this research program. This will determine if all the soils statewide will react satisfactorily to the rapid method. It will also determine if the personnel of each laboratory are consistent enough to obtain satisfactory results by using the rapid method.

APPENDIX

SUMMARY OF HYDROMETER ANALYSIS
LABORATORY A

TABLE 4

	TEST METHOD			TEST METHOD			TEST METHOD	
SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE
1	9	8	21	2	3.3	41	15	17.7
2	19.3	21	22	16.7	14.3	42	30	28
3	22	22	23	18	17.7	43	13	12
4	14.7	14.3	24	19	16.7	44	14	12
5	11.3	11	25	8	7.7	45	16.3	15.3
6	20	18	26	8.3	8.3	46	2.3	1.7
7	8	8	27	24	24	47	15	13.7
8	12	10	28	9.7	9.7	48	6	4
9	21.7	21	29	26	27	49	16	15
10	8	8	30	25.3	24.7	50	6.7	6
11	23.3	23.7	31	9	9.7	51	14.3	13
12	37.3	36.7	32	16	15.3	52	19	17
13	17.7	16	33	21.7	21	53	4.3	2
14	7.7	7.7	34	14.7	13.3	54	0	.33
15	15.3	13	35	20	19	55	.33	0
16	25	25	36	6	5	56	22	22
17	23.7	23	37	24.3	24	57	11.3	9.7
18	4	3	38	6	4	58	14	12
19	26	26	39	20	22	59	26.3	25.3
20	16.3	15.3	40	11.7	11.7	60	11	9.7

SUMMARY OF HYDROMETER ANALYSIS
LABORATORY B

TABLE 5

	TEST METHOD			TEST METHOD			TEST METHOD	
SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE
1	17.3	16.7	21	15.3	15	41	16.3	15
2	17	14.3	22	17	15.7	42	17	15.3
3	33	32.3	23	5.7	5	43	17	15
4	28	27	24	6.7	6.3	44	16.7	15.7
5	17.7	14	25	5	5	45	17	16
6	17	13.7	26	6.3	6	46	4	5
7	17.7	13.7	27	6.3	6	47	5.7	6.3
8	12	12	28	6	5	48	6	7
9	14	14	29	6.3	6	49	28	30
10	8	7.7	30	5.7	6	50	27	28.3
11	8.3	8.3	31	14.7	13	51	4	5.3
12	9	9.3	32	13.7	12	52	5.7	6.7
13	10	11	33	10.7	10	53	3.7	5
14	12.3	13	34	13.7	12	54	3.3	5
15	13	13.7	35	12.3	11	55	32.7	34
16	10	11	36	19	18	56	30.7	30.7
17	12	12.3	37	15	13	57	30	30.3
18	14.7	12.7	38	18	16.7	58	31	31
19	13.3	12	39	15	12	59	30	30
20	14	12	40	17	16	60	28.7	29

SUMMARY OF HYDROMETER ANALYSIS
LABORATORY C

TABLE 6

	TEST METHOD			TEST METHOD			TEST METHOD	
SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE	SAMPLE NO.	EXPERIMENTAL AVERAGE	CONVENTIONAL AVERAGE
1	17.3	17.3	21	16.7	18	41	27.3	30
2	16	16.7	22	28.3	29	42	24	25.3
3	19	21.3	23	28.7	30.3	43	26.7	27.7
4	20.3	24	24	18.3	23	44	23.3	25
5	7	10	25	25.7	25.3	45	14	16.3
6	4	5	26	18	19.7	46	25.7	27
7	25	26	27	34	34.7	47	23.3	24
8	30	30.3	28	21.3	27.7	48	14.7	15
9	16.7	19	29	27.3	29	49	27.3	27.7
10	19	20.3	30	14.3	12.7	50	17.3	19
11	7.3	10	31	28	29	51	21.3	23
12	18.7	21	32	17.7	20	52	26.7	31
13	23	23.3	33	21	23.3	53	26	29
14	21.3	20.7	34	21.7	23.7	54	22.7	26.3
15	12	15	35	21	23	55	33	36.7
16	21	23.7	36	26	26.3	56	14.3	16.7
17	12	13	37	19	20.7	57	23.7	24
18	26	24.7	38	21	22	58	18.7	22
19	21.7	23	39	23	25	59	14.3	17
20	16.7	20	40	21	22.3	60	26	27.3

METHOD OF MECHANICAL ANALYSIS OF SOILS INTENDED FOR CEMENT STABILIZATION

SCOPE

1. This method is intended to describe a standard procedure for determining the distribution of particle sizes of soils intended for cement stabilization.

APPARATUS

2. The following apparatus will be necessary:

(a) Balances - A balance sensitive to 0. 1 gm. for weighing the material passing the No. 10 sieve, and a balance sensitive to 0. 1% of the weight of the sample to be tested for weighing the material on the No. 10 sieve.

(b) Stirring Apparatus - Stirring apparatus shall consist of a mechanically operated stirring device in which a suitable mounted electric motor turns a vertical shaft at a speed of 10, 000 rpm without load. Attached to the shaft shall be a paddle made of metal, plastic, or hard rubber. Special dispersion cups shall be used to contain the soil fraction while it is being dispersed.

(c) Hydrometers - The ASTM Hydrometer 152H must be used for this test.

(d) Sedimentation Cylinders - Glass cylinders approximately 18" high and 2-½" in diameter marked for a volume of 1000 ml. will be necessary to contain the sample while under test.

(e) Thermometer - A Fahrenheit thermometer accurate to 2° F.

(f) Sieves – A series of standard testing sieves to include at least the following:

No. 10
No. 40
No. 200

(g) Beakers - Beakers of sufficient capacity for slaking samples.

(h) Timing Device - A common stop watch.

PREPARATION

3. (a) Prepare sample in accordance with LDH Designation TR 411. To facilitate drying, it will be necessary to separate the soil into several pans and stir occasionally.

(b) Record that portion retained on the No. 4 and No. 10 sieves as percent gravel.

4. (a) Before a determination of grain sizes can be made, each hydrometer must be corrected to compensate for temperature variation of water without soil particles in suspension. Temperature variations of water cause changes in its density and specific gravity. Compensations for these changes must be made so that no matter what the temperature, the hydrometer will always effectively read zero.

(b) The following steps will enable the construction of a tabular or graphical chart showing the correction factors to be applied to each hydrometer reading for the testing temperature.

(1) 125 ml. of stock solution of sodium hexametaphosphate buffered with sodium carbonate (trade name Calgon) is placed in a sedimentation cylinder and sufficient distilled or demineralized water is added to bring the entire solution to 1000 ml. at 68°F.

Note: A stock solution consists of 40.00 grams of calgon plus sufficient distilled or demineralized water to make 1000 ml. This solution should not be kept longer than two weeks.

(2) The entire solution is then cooled to the minimum temperature expected to be encountered during any test and a hydrometer reading is recorded at that temperature.

(3) Sufficient temperature and corresponding hydrometer readings shall be taken as the solution slowly warms to room temperature to form a smooth correction curve, plotting hydrometer reading against temperature. For those temperatures which will be warmer than room temperature, the solution should be heated and allowed to cool.

Note: Care must be taken to insure uniform temperature throughout the solution.

(4) A tabular chart may then be lifted from the correction curve for every whole degree which will be encountered. This chart of graph will eliminate the constant temperature bath for a limited temperature variation on routine tests.

ALTERNATE HYDROMETER CALIBRATION

5. (a) A blank sample is made with the same water used in 6(b). Pour 125 ml. of 5(a) stock solution into the standard 1000 ml. sedimentation cylinder and add sufficient water to make a mixture of 1000 ml.

(b) The blank sample is made as the first sample of 6(b) to insure the same temperature as the test series.

(c) The correction factor of 7(a) is read directly from the blank sample with hydrometer before the start of the regular readings of 6(e).

PROCEDURE

6. (a) Use 100 grams of soils per sample. Thoroughly mix the soil in 125 ml. of stock solution calgon by hand. This soil should have been prepared in accordance with LDH. Designation TR 411 and be oven dry.

(b) After mixing, wash soil into dispersing cup with distilled or demineralized water, adding water until about 2/3 full and stir for 5 minutes with mechanical stirring apparatus.

(c) Pour from stirring cup into the standard 1000 ml. sedimentation cylinder and add sufficient distilled or demineralized water to make a mixture of 1000 ml.

(d) Covering the open end with the hand or rubber stopper, shake the cylinder for 45 seconds minimum using a rocking motion so that the cylinder is completely inverted numerous times. (Approximately 45 turns, counting upside down and back as two turns).

(e) At the conclusion of shaking, the timing is started. For routine samples, hydrometer readings are recorded on form 824-A at 60, and 120 minutes. Record the temperature of the mixture before each reading. Hydrometer insertion and withdrawal should be done so as to disturb the suspension as little as possible.

(f) Upon completion of the necessary readings, the sample is then washed over the 40 and 200 mesh sieves. The material retained on these sieves shall be dried, weighed, and calculated as percent coarse and find sand respectively.

CALCULATIONS

7. Form 2099 incorporates factors which include the (a) effects of a change of viscosity and buoyancy of the solution due to the soil particles remaining in suspension, and (b) the change of viscosity and other properties resulting from temperature changes of the mixture. The chart assumes a constant specific gravity for all soils at 2.65.

Form 2099 is used in the following manner:

(a) Apply the correction factor as obtained in steps 4 or 5 above to each hydrometer reading and compute the percent finer.

(b) The percent finer is then located on the form as the vertical coordinate, and is followed across the sheet until it intersects the temperature at which the reading was taken in the proper time group (i. e. 60 minutes, 120 minutes). Interpolation between each 10 degree line must be made as accurately as possible. This intersection is one point on the "grain size distribution curve."

(c) In each of the 60 minutes and 120 minutes groups, one point is plotted as in (a) above. A straight line is constructed between the points, and the point where this constructed line intersects the .005 mm. vertical line is taken as the percent finer than .005 mm. or the percent clay and colloids.

(d) To determine the Grain Size Distribution, the percent finer than .005 mm. is used as percent clay, the percent retained above the 200 mesh screen is considered sand, and the sum of the percent sand and clay subtracted from 100 is the percent silt.

(e) The percents as determined above are then plotted on the triangular chart and the textural name given to the section into which the plot falls is assigned to that soil. Cases of "border line" soils (i. e., soils that fall exactly on the border line between two or more groups) frequenly occur. In such cases, the "to the right and up" rule should apply. In other words, when a soil falls on a line which separates two textural names which are adjacent horizontally, the name on the right should be used. When a soil falls on a line which separates two textural names which are adjacent vertically, the topmost name must be used. In the extreme case, when the soil falls on an intersection of two border lines, the soil should assume the name of the section which is in the upper right hand quadrant.

(f) In cases where the presence of gravel exists, some adjustments must be made. The word "gravelly" is inserted in front of the textural name assigned to the minus 10 material. Since the hydrometer analysis considers the material passing the No. 10 sieve as 100%, a readjustment must be made so that percent gravel present in the sample will be included as a portion of the 100%. For example, 10% gravel was obtained from separation of the No. 10 sieve and from the hydrometer analysis on the material passing No. 10, 20% sand, 50%, silt, and 30%, clay and colloids were obtained. (Total 110 percent) Using the "to the right and up" rule, the name assigned to this sample will be Gravelly Silty Clay. Now it is necessary to readjust the percentages so that 100 percent includes all portions of the sample. Thus, the percentages reported should read: 10% gravel, 18% sand, 45% silt, and 27% clay and colloids.

REPORTS

8. The results of this test are usually incorporated with reports of other tests. They are at least accompanied by the "physical characteristics" of the same sample. A form similar to Form 3024 is recommended for reporting the results obtained from mechanical analysis.

Normal testing time 24 hours.