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# Influence of phosphate dispersing agents on particle size distribution of soil fines

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**ABSTRACT:** The particle size distribution of the fine portion of soils is usually determined using sedimentation principles and the hydrometer method. Some hydrometer test methods, such as the South African Standard (SANS3001, 2014) and Guidelines laid by International Soil Reference and Information Centre (ISRIC, 2002) make provision for the effect of the dispersing agent in the solution on readings recorded during the test. Test methods that do not make such provision may be incorrectly yielding inflated fines (including clay) contents. This paper discusses the results of an investigation into the effect of dispersants when applying the TMH1 (1986) test method. The results have been compared using phosphate dispersants such as Calgon, sodium pyrophosphate decahydrate and sodium tetra pyrophosphate in various concentrations. The use of dispersants in higher concentrations produced anomalous increases in the hydrometer readings and the results indicated the importance of understanding the effect of dispersants on the fine soil particle size distribution analyses.

## 1 INTRODUCTION

The accurate determination of the particle size distribution of a soil is of utmost importance in the field of geotechnical engineering, as the clay content of the soil is used to determine the activity of a soil, which in turn is used for design purposes. Inaccurate clay content determinations have resulted in inappropriate design solutions which have even led to unacceptable damage to the structures. In South Africa, there is a problem with the accurate determination of the clay content of soils, basically due to the differences in the laboratory results (Jacobsz and Day, 2008). Therefore, there is a need for research into all the variables of the hydrometer test, with a view to improve its accuracy and thus standardize the test nationally and possibly, in future, internationally.

In the hydrometer analysis, an additive known as a dispersing agent or chemical dispersant is usually added to make the dispersion process more effective. Dispersants deflocculate solids and thus significantly reduce the viscosity of the dispersion paste. A variety of dispersing agents are used in different parts of the world for the sedimentation analysis. According to Lambe 1951, two commonly used deflocculants are sodium silicate and Daxad No. 23 (polymerized sodium salts of substituted benzoid alkyl sulfonic ac-

id). Sodium hexametaphosphate (Lambe, 1951 and ASTM D422-63, 1965), sodium silicate (TMH1, 1986), sodium oxalate (TMH1, 1986), sodium pyrophosphate (Schuurman & Goedewaagen, 1971), sodium tetra pyrophosphate (Yoo & Boyd, 1994) and Disodium Dihydrogen pyrophosphate (formerly used by the Soils Testing Laboratory of Department of Water Affairs of South Africa) have also been successfully used. Calgon which is a combination of sodium hexametaphosphate and sodium carbonate is one of the popular and considered to be the best dispersing agent. All over the world, Calgon has been used as a dispersing agent in varying concentrations (BS 1377 Part 2, 1990, IS 2720 Part IV, 1985 and International Soil Reference and Information Centre (ISRIC, 2002)). As mentioned earlier TMH1 (1986) uses a combination of sodium silicate and sodium oxalate as the dispersing agent. However, the dispersing agents prescribed in TMH1 (1986) do not yield the maximum clay percentage (Kaur & Fanourakis, 2016 (a) and (b)). For this reason, many national laboratories have adopted the TMH1 (1986) test method, with a deviation in the dispersing agent type, with the objective of obtaining more accurate results reflected by higher clay percentages. However, it has been observed that altering the dispersing

agent and following the TMH1 (1986) method gives inaccurately inflated results, especially with higher concentrations and volume in the case of certain dispersing agents (Kaur & Fanourakis, 2016 (a) and (b)). This is because of the aggregation of uniformed sized particles of dispersing agent (solids) in the hydrometer cylinder, which increases the density in the zones measured by the hydrometer. Some hydrometer test methods, such as the South African Standard (SANS 3001, 2014) and the guidelines laid by International Soil Reference and Information Centre (ISRIC, 2002) make provision for a correction for the effect of the dispersing agent in the solution on the readings recorded during the test. As TMH1 (1986) does not make provision for such a correction, it yields incorrectly inflated fines contents with dispersing agents other than the one recommended by the TMH1 (1986) method. Hydrometer test readings should be corrected by subtracting the readings obtained on companion “blank” solutions containing only water and dispersing agent (no soil), at the relevant time period, from the reading taken on the solution containing water, soil and dispersing agent.

In the current investigation, the effect of various dispersing agents on the hydrometer test results was considered, while following the TMH1 (1986) test method. The results were compared using phosphate dispersants such as Calgon (a combination of sodium hexametaphosphate and sodium carbonate), sodium pyrophosphate decahydrate and sodium tetra pyrophosphate in various concentrations.

## 2 TESTING PROGRAM

### 2.1 Tests Performed

A total of 155 hydrometer tests were conducted to explore the effects of using a high concentration and volume of the generally accepted most effective dispersing agents (Sridharan et al, 1991, Bindu & Ramabhadran, 2010 and Kaur & Fanourakis, 2016 (a) and (b)) on the hydrometer readings of water solutions (blank) containing no soil. The solutions comprised Calgon in concentrations of 35:7, 40:10, 60:10, 70:10, 80:10 and 90:10 and in the case of both sodium pyrophosphate decahydrate and sodium tetra pyrophosphate in the concentrations of 3.6%, 5%, 6%, and 7%.

The quantities of chemicals added for preparation of one litre of stock solution are given in Table 1.

When using Calgon as a dispersing agent, the minimum volume used was 100 ml and for both sodium pyrophosphate decahydrate and sodium tetra pyrophosphate, the minimum volume used was 20 ml. The justification for these quantities is that many methods (BS 1377 Part 2, 1990 and IS 2720 Part IV, 1985) recommend 125 ml of Calgon solution, so 100 ml volume was tried to confirm the recommended

volume, whereas many national laboratories use 20 ml of sodium pyrophosphate decahydrate.

Table 1. Quantity of chemicals added for preparation of Calgon, sodium pyrophosphate decahydrate, and sodium tetra pyrophosphate solution

Solution of Concentration (%)	Calgon		NaPP	NaTPP
	Quantity of NaHMP Added (g)	Quantity of Na <sub>2</sub> CO <sub>3</sub> Added (g)	Quantity of NaPP Added (g)	Quantity of NaTPP Added (g)
3.6	-	-	36	36
4.2	35	7	-	-
5	40	10	50	50
6	-	-	60	60
7	60	10	70	70
8	70	10	-	-
9	80	10	-	-
10	90	10	-	-

When using Calgon as a dispersing agent, the minimum volume used was 100 ml and for both sodium pyrophosphate decahydrate and sodium tetra pyrophosphate, the minimum volume used was 20 ml. The justification for these quantities is that many methods (BS 1377 Part 2, 1990 and IS 2720 Part IV, 1985) recommend 125 ml of Calgon solution, hence 100 ml volume was tried to confirm the recommended volume, whereas many national laboratories use 20 ml of sodium pyrophosphate decahydrate.

TMH1 (1986) recommended the use of 5ml of sodium silicate and 5 ml of sodium oxalate.

The stock solution of sodium silicate was prepared by dissolving sodium silicate in distilled water until the solution yielded a reading of 36 at a temperature of 20°C on the standard soil hydrometer and the stock solution of sodium oxalate consisted of a filtered saturated solution of sodium oxalate (Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>).

### 2.2 Testing procedure

For all the tests performed, the desired quantity of dispersing agent was mixed with about 400 ml of distilled water in a canning jar. This dispersing agent – water mixture was allowed to stand overnight. After the mixture had been allowed to stand, it was dispersed for 15 minutes with a standard paddle. The paddle was washed clean with distilled water allowing the wash water to run into the container. This mixture was then poured into the Bouyoucos cylinder and the canning jar was rinsed with distilled water from the wash bottle. The cylinder was then filled with distilled water to 1130 ml mark with the hydrometer (152H) inside. Thereafter, the hydrometer was removed and the cylinder was inverted a few times, using the palm of one hand as a stopper over the mouth of the cylinder to ensure that the temperature was uniform throughout. After bringing the cylinder to a vertical position, the stopwatch was started. The hydrometer was inserted and the readings

were taken at 18 seconds and 40 seconds without removing the hydrometer from the cylinder. The hydrometer was then taken out and rinsed with water and it was again inserted into the suspension when the elapsed time was 2 minutes. This reading was noted and the hydrometer was removed and placed in distilled water. This procedure was repeated for the 5 minutes, 15 minutes, 30 minutes, 1 hour, 4 hour and 24-hour readings. After taking each hydrometer reading, the temperature of the liquid was also recorded. Temperature corrections were appropriately applied to the readings.

For any soil sample, the percentages finer than 0.075 mm, 0.05 mm, 0.04 mm, 0.026 mm, 0.015 mm, 0.01 mm, 0.0074 mm, 0.0036 mm and 0.0015 mm were respectively calculated by the readings taken at 18 sec, 40 sec, 2 min, 5 min, 15 min, 30 min, 1 hour, 4 hours and 24 hours, by means by Equation 1.

$$P = \frac{C \times S_f}{S_m} \quad (1)$$

Where,  $P$  = Percentage finer than relevant size,  
 $S_m$  = Mass of soil fines used in analysis (50 grams),  
 $S_f$  = Percentage soil fines in total sample (<0.425 mm),  
 $C$  = Corrected hydrometer reading

The percentage clay content present in each sample (fraction finer than 0.002 mm) was obtained from the relevant particle size distribution curve. As, in the current study the hydrometer analysis was done on blank solutions (no soil), Equation 1 was not applicable.

### 3 RESULTS AND DISCUSSION

Semi-logarithmic graphs were plotted with grain size on the x-axis (log scale) and correction to be applied to (subtracted from) hydrometer readings (in tests where the soil is included) on the y-axis. Figure 1 shows the effect of the dispersing agent on hydrometer readings while following TMH1 (1986) guidelines.

It is clear from Figure 1 that in the case of all the dispersing agents except the Calgon, the corrections to be applied to hydrometer readings would be less than 1 g/litre. In the case of Calgon, the correction ranged from 5.3 to 5.8 g/litre (average 5.6 g/ litre).

Most of the national laboratories (in South Africa) are using 125 ml of Calgon (35:7) and TMH1 (1986). However, the hydrometer test results are not appropriately corrected (reduced) to allow for the above-mentioned contribution of the dispersing agent to the test readings.

In addition, hydrometer test analyses were conducted using Calgon, sodium pyrophosphate decahydrate and sodium tetra pyrophosphate in different concentrations and volumes to establish the effect of the increase in the volume and concentration of the solution on the hydrometer readings.

It is clearly seen from Figure 2, which is a plot between correction to the hydrometer reading and grain size for 9% Calgon (80:10) that, an increase in the volume of the dispersing agent used resulted in a concomitant increase in the hydrometer reading. Also, it is evident from the figure that there is almost no difference between the 18 sec reading and 24 hour reading which shows that the amount of dispersing agent particles in suspension remained almost constant with the time. A dispersing agent volume of up to 475 ml was included in the test program.

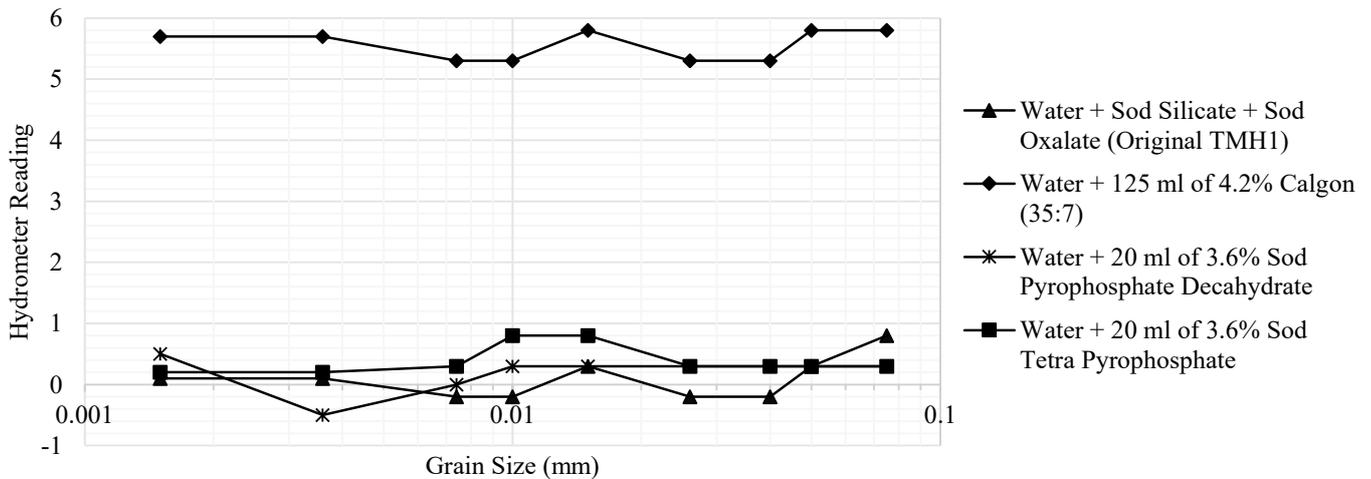


Figure 1. Effect of dispersing agent types on hydrometer readings.

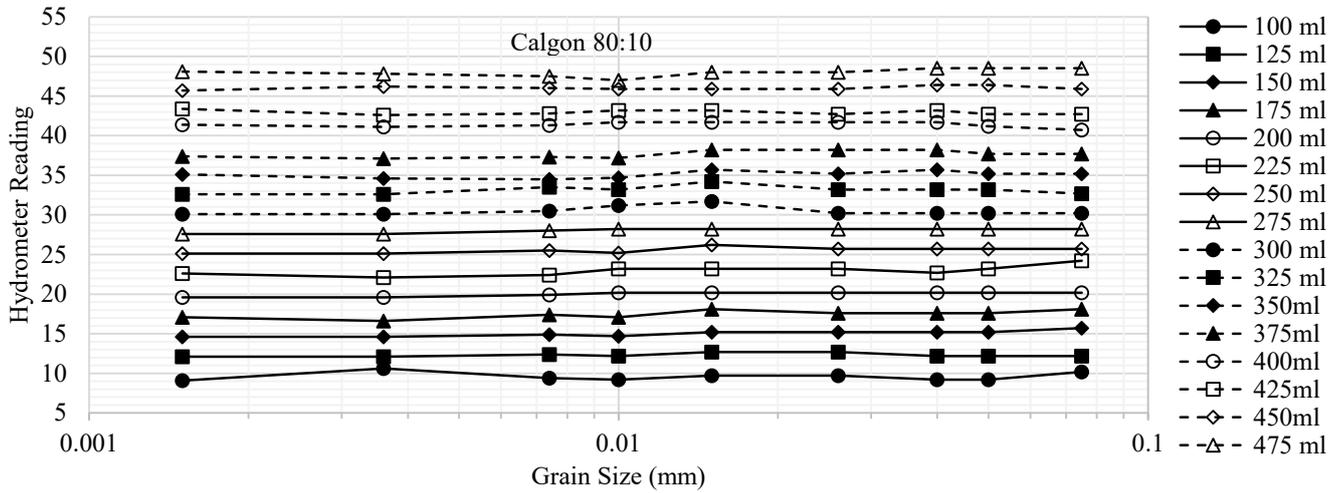


Figure 2. Hydrometer reading corrections for different grain sizes for Calgon (80:10).

Figures 3, 4 and 5 show the effect of volume and concentration of Calgon, sodium pyrophosphate decahydrate and sodium tetra pyrophosphate, respectively, on the hydrometer readings pertaining to the time at which the clay size (0,002 mm) reading would be taken. From all three figures it is clear that, with the increase in the concentration and volume of the dispersing agents, there is an increase in the hydrometer readings. With Calgon as a dispersing

agent, the hydrometer readings varied from 5 to 47 with an increase in volume and concentration while with sodium pyrophosphate decahydrate and sodium tetra pyrophosphate they varied from 0 to 20 and 0 to 9, respectively. The reason behind this increase is the aggregation of uniform sized solid particles of dispersing agent in the hydrometer cylinder, increasing the density of the solution in the zones measured by the hydrometer.

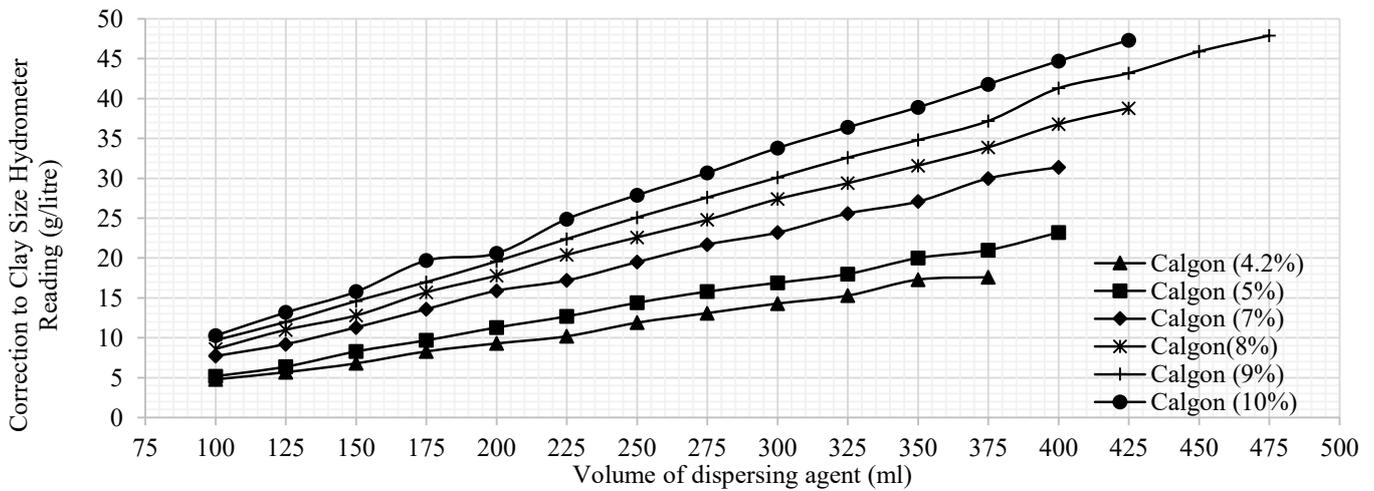


Figure 3. Effect of volume and concentration of Calgon on clay-sized period readings.

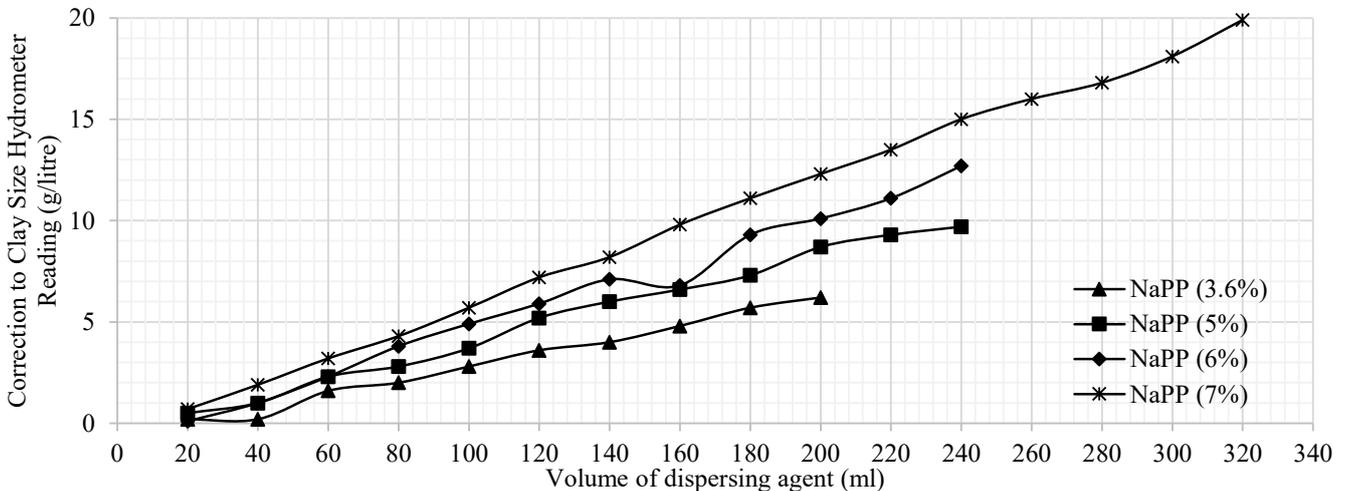


Figure 4. Effect of volume and concentration of sodium pyrophosphate decahydrate on clay-sized period readings.

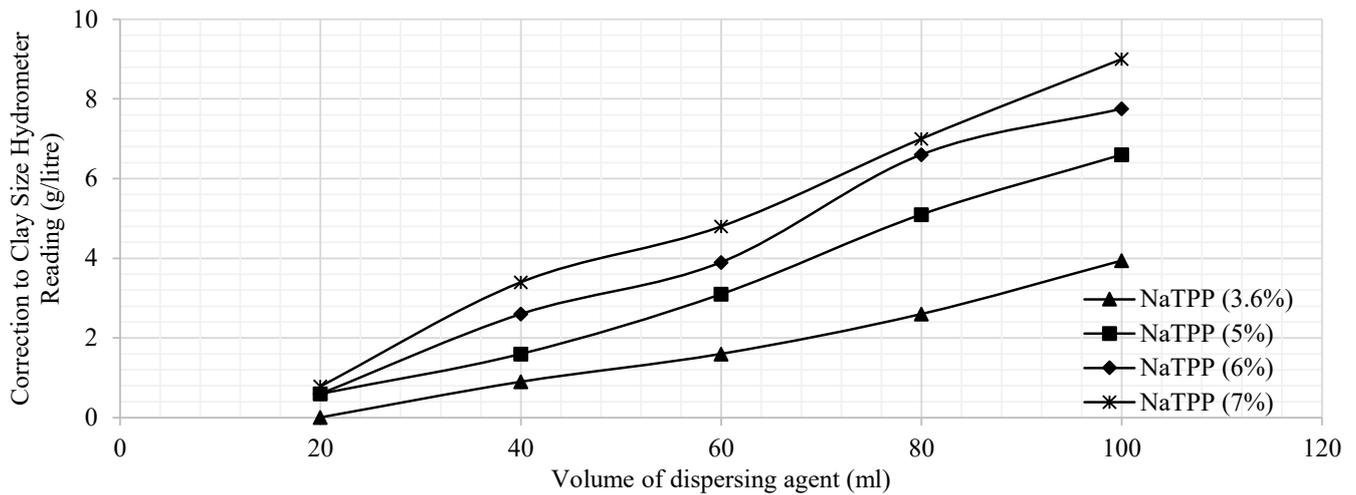


Figure 5. Effect of volume and concentration of sodium tetra pyrophosphate on clay-sized period readings.

In addition, the calculated soil activity is strongly affected if the dispersing agent corrections are not applied to the hydrometer readings. Table 2 shows the Atterberg's limits, activities and clay contents of the four soil samples used by Kaur and Fanourakis, 2016 (a) and (b). The activities tabulated in Table 2 were computed with and without applying the dispersing agent corrections and are referred to as A and A', respectively. The clay contents determined with and without the dispersing agent corrections are referred to as C and C', respectively.

Table 2. Activities of the soil samples with and without dispersing agent corrections

Properties	Light Yellow Soil	Black Soil	Light Brown Soil	Red Soil
Liquid limit (LL)	32	56	33	28
Plastic Limit (PL)	16	22	24	15
Plasticity Index (PI)	16	34	9	13
C	21.5	32	5.7	29
A	0.74	1.06	1.58	0.45
C'	32.4	44	11.8	40
A'	0.49	0.77	0.76	0.33
$\Delta$ % Activity	33.8	27.4	51.9	26.7

It is observed that as the blank readings are not subtracted from the hydrometer readings of the suspension thus yielding more clay content there is about 34 %, 27 %, 52 % and 27 % decrease ( $\Delta$  %) in the activity of light yellow soil, black soil, light brown soil, and red soil, respectively (Table 2). Therefore, the results discussed in this study indicate the importance of understanding the effect of dispersing agents in the water on the hydrometer test readings. Thus, hydrometer test readings should be corrected (reduced) to account for the contribution made by the type, concentration and volume of dispersing agent used in the test.

#### 4 CONCLUSIONS

The following conclusions were drawn from the study conducted:

- Tests with different dispersing agents clearly indicated that the hydrometer readings of blank solutions vary significantly depending upon the type of dispersing agent.
- The hydrometer readings for blank solutions increased with an increase in the concentration and volume of a dispersing agent. For Calgon, the hydrometer readings varied from 5 to 47 (g/litre) while for sodium pyrophosphate decahydrate and sodium tetra pyrophosphate, they varied from 0 to 20 (g/litre) and 0 to 9 (g/litre), respectively.
- The increase was attributed to the aggregation of uniform sized solid particles of dispersing agent in the hydrometer cylinder, increasing the density of the solution in the zones measured by the hydrometer.
- There is an average decrease ranging from approximately 27 % to 52 % (average of 36 %) in the activities of soils computed with the clay content determined without dispersing agent corrections.
- Readings of hydrometer tests where deviations from the prescribed type, volume, and concentration of the dispersing agent occur should be appropriately corrected.

#### 5 REFERENCES

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