

QUICKLIME  
SPECIFICATION AND TEST METHODS RESEARCH

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by

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Producer Key

Producer

A	Chemical Lime - Clifton, Texas
B	Redland Worth - San Antonio, Texas
C	U. S. Gypsum - New Braunfels, Texas
D	Austin White Lime - Austin, Texas
E	Dravo Lime - Blum, Texas
F	Texas Lime - Cleburne, Texas

## Summary

Samples of pebble quicklime were obtained from each of the six major lime producers' plants in Texas. These samples were used for laboratory testing which determined chemical composition of the dry lime, physical properties of slurries prepared from the quicklime, and reaction properties of the lime with soil.

Preparation of slurries with the six materials using the same methods and material quantities yielded viscosities ranging from 13 to 165 poises at 77 F with solids contents between 28.5 and 32.4 percent solids. Adjustment of the high viscosity slurries to the range of 25-30 poises resulted in solids contents between 18.5 and 26.0 percent.

Wet sieve analyses of slurries prepared using two methods were performed. Residues retained on the No. 6 and No. 30 screens were weighed. Residues on the No. 30 ranged from 5.5 to 32.8 percent using the standard test method and 1.2 to 10 percent using a different slurring method.

Quicklime/soil mixes were prepared and subsequently compacted into 4 x 6 cylinders and cured under moist conditions. These cylinders were tested at intervals for compressive strength and unreacted lime content. These tests showed no consistent difference in the stabilizing performance of the different producers' materials. Samples prepared using quicklime passing the 1/4-inch sieve and hydrated lime showed more stabilizing effect than the coarsely graded quicklime.

Recommendations for changes to the existing lime specification and test procedures are made within this report. It is recommended that the residue requirement for the No. 30 sieve be removed and the limit for the No. 6 sieve be lowered. It is also recommended that a separate grade of quicklime be established solely for slurring purposes which has no limit on fines content.

## Background

Quicklime specifications in use by the Department at the time this work was performed were written for pebble quicklime which would be dry placed. With the increased amount of quicklime being hydrated in portable slurry plants at or near the job site, we now find the bulk of the quicklime being used by the Department is placed in slurry form.

The test procedures accompanying these quicklime specifications precluded the use of some highly reactive limes with which we had little job experience. During hydration after dry placement these quicklimes tend to form lumps with a putty-like consistency which might not mix well into the soil. In slurry application a gel-like mixture may result. As a result of these potential problems, our specification included a wet sieve analysis test in which the quicklime is reacted with a limited amount of water prior to the sieving. The specification limited the amount of residue on the No. 6 and No. 30 sieves.

With increased competition in the market for quicklime use by the Department, companies producing highly reactive materials have expressed greater interest in supplying quicklime. As a result of our limited experience using these materials and the increased interest, the question has arisen as to what problems might be encountered with their use and what benefits might be expected. A study was initiated to look into use of highly reactive quicklimes for dry and slurry placement.

## Objectives

The study investigated quicklimes produced by the six primary manufacturers of lime in Texas. Emphasis was on a comparison of the highly reactive quicklimes supplied by Producers B and C with the moderately reactive materials supplied by the other producers. Half of the study compared the hydration characteristics of pebble quicklime and of the lime products formed as a result of hydration with varying amounts of water. The other half studied the stabilization effects of dry pebble quicklime on high plasticity soil.

The purpose of both parts of this study was to determine the properties and benefits of the various quicklimes and to check the validity of the wet screen test requirements.

## Methods

In September of 1985, pebble quicklime samples were taken at six lime plants (Austin White Lime, Chemical Lime, Dravo Lime, Texas Lime, Redland Worth, and U. S. Gypsum). A sample of approximately five gallons of each was taken during a truck loading in order to obtain a sample representative of the plants' production.

Each of the six samples were quartered in the Soils Section by using a sample splitting device. Each quarter was placed into a plastic bag for protection against moisture. These bags were used one at a time over a period of several months so that a basis of comparison existed between the various tests run.

### Evaluation of Slurried Quicklime:

The equipment required for this testing was as follows:

1. Hobart Mixer, Model N-50 with mixing bowl, approximately one-gallon capacity.
2. Top-loading balance suitable for rapid weighing, with minimum capacity of 2000 grams.
3. Wire whip, Hobart brand, Type D.
4. Oven, mechanical convection type, 212 F.
5. Sieves, U. S. Standard Nos. 6 and 30.
6. Stopwatch, to register up to 30 minutes.
7. Spatula.
8. One-pint can with friction lid.

9. Tape - PVC type.
10. Plastic tubing and 0 to 30 psi water pressure gauge.
11. 250 milliliter plastic bottle.
12. Thermometer - range 0-200 F.
13. Tap Water ( $77 \pm 3$  F).
14. Brookfield Syncho-lectric viscometer of suitable range.
15. A six-ounce ointment can.

Samples of pebble quicklime weighing 800 grams were obtained by successively dividing by rod on a piece of paper the lime from one of the quartered bags. The wet sieve analysis was performed on each quicklime following the procedure outlined in Test Method Tex-600-J. A copy of the procedure is included in the appendix. In this procedure, 1700 milliliters of 74 to 78 F water is added to 800 grams of quicklime in one addition and then mixed with the Hobart mixer. A thick slurry results which is then wet sieved.

The quicklimes were then slurried by a procedure which would give a slurry typical of that produced in the field. After adding the 800 grams of pebble quicklime to different amounts of water, it was determined that 2600 milliliters would probably yield a slurry sample which would be in the 31 to 35 percent solids content which is typical of the most common grade of slurry placed in the field.

The following procedure for preparing slurry was developed and used for subsequent testing:

Twenty-six hundred milliliters of tap water, at a temperature of  $77 \pm 3$  F, is placed in the empty mixer bowl and the mixer with the wire whip attached

is placed on low speed. Eighty grams of the previously-weighed 800 grams of pebble quicklime is added to the water in the mixer, and the stopwatch started. At the end of three minutes of mixing, an additional 80 grams of lime is added to the lime-water mixture in the mixing bowl without interrupting the continuous mixing operation. This process is repeated until 10 minutes have elapsed, at which time, the mixer is stopped and a thermometer is placed near the center of the mixing bowl for 30 seconds and the temperature recorded. The thermometer is removed and mixing continued for an additional two minutes to complete the three minute cycle. The 80 grams of lime is continuously added every three minutes and the temperature of the slurry is taken for 30 seconds at 10-minute intervals throughout the 30 minute mixing time.

Upon completion of mixing, the mixing bowl is lowered and the wire whip is removed and rinsed over the No. 6 sieve which is stacked on the No. 30 sieve in a sink. A one-pint can of the mixed slurry is taken by means of dipping into the slurry with a six-ounce ointment can until the pint can is filled to within 3/4 inch from the top. The friction lid is then placed on the pint can immediately to prevent evaporation. Using the six-ounce ointment can, the 250 milliliter plastic bottle is filled with about 200 milliliters of slurry and the screw cap is then taped on to prevent evaporation.

At the completion of the mixing cycles, no more than two minutes should elapse before the plastic bottle is filled. It is important to follow these time limits because the slurry solids do tend to settle as soon as mixing is complete; also, evaporation is a problem throughout the entire process.

The 200 milliliters of slurry in the plastic bottle was allowed to cool to  $77 \pm 4$  F, and a chemical analysis was performed according to Test Method Tex-600-J, Type B, Lime Slurry. This test was performed primarily for the determination of the solids content.



The pint can of hot slurry was wrapped with foam insulation and allowed to cool to 140 F. This cooling required approximately 1 1/2 hours, at which time a viscosity test was performed. The material was cooled to  $77 \pm 3$  F by using an ice water bath and another viscosity was performed about three hours after the sample was taken. (All the viscosity tests performed in this research were done according to the procedure in Test Method Tex-614-J under Article A, Consistency.) Some of the lime producers' materials, after being slurried, had a viscosity in excess of 35 poises due to the nature of the pebble quicklime material. These slurried materials were diluted with water until their viscosity was in a range between 25 and 35 poises. The new solids contents were calculated on the basis of the amounts of water added to reduce the viscosity. The percent solids of a lime slurry is primarily the lime content.

The remainder of the slurried lime was screened through the Nos. 6 and 30 sieves, as described in Test Method Tex-600-J, Testing of Quicklime, Type C, Item 5, Wet Sieve Analysis, under Procedure. A No. 20 sieve was not needed in screening as described in the procedure because there was not an excess amount of material to be retained on either the No. 6 or No. 30 sieve.

The dried material remaining on the sieves was crushed in a Denver Fire Clay crusher and ground through a Braun Pulverizer, Type UA, with stainless steel plates. The pulverized material was screened to pass the No. 30 sieve and packaged in airtight plastic bags and returned to a desiccator to await chemical analysis.

The Lime Testing Procedure, Test Method Tex-600-J, Part II, Type A Hydrated Lime, was used to analyze the pulverized material for actual lime content. The procedure was modified by adding an excess of 1 normal hydrochloric acid to a pH of 1.5, after the 8.3 pH endpoint had been reached. The 1.5 pH excess was used because each sample varied considerably in total alkalinity and a measured excess as asked for by the procedure would not cover the range of alkalinity needed for these samples.

It was noted during the preliminary testing that there are different results on wet sieve analyses when water is added to the lime rather than lime being added to the water, as was done for this research.

The initial water temperature will affect the ultimate reaction temperature of the slurry which affects its viscosity, screen analysis, chemical analysis, etc. It is very important to check and record temperatures and keep the slurry in airtight containers throughout the procedure for reproducible results.

#### Evaluation of Stabilizing Effect of Dry-Placed Quicklimes:

It was desired to compare the reaction properties of the six pebble quicklimes by preparing soil-lime mixtures and studying the relative stabilizing ability. This was done primarily to check the validity of the 30-mesh wet sieve requirements in our specification. A method for determining the reaction rate of lime in soil was sought.

The method selected for analyzing lime content of soil-lime mixtures was ASTM D 3155, "Lime Content of Uncured Soil-Lime Mixtures." This analysis procedure follows:

Reagents used:

1. Ammonium Chloride Solution - 100 g/l
2. Disodium EDTA Solution - 0.1 M
3. Sodium Hydroxide Solution - 500 g/l
4. Triethanolamine Solution - 1:4 in water
5. Hydroxynaphthol Blue Indicator Powder

The cured or uncured soil-lime mixture is dried to constant weight and ground through the Braun Pulverizer to passing a 40-mesh screen. Three

hundred grams of the mixture is placed in a 2-liter jar and 600 ml of the ammonium chloride solution is added, the jar sealed, and the mixture well shaken. Unreacted lime, in the form of calcium oxide or calcium hydroxide, reacts readily with the ammonium chloride reagent to form ammonium hydroxide and calcium chloride, which is very soluble in water. Preliminary work showed that the reaction with calcium hydroxide is complete enough to allow quantitative analysis, while there is no apparent reaction with calcium carbonate or calcium-containing soil-lime reaction products. There is, however, some naturally occurring extractable calcium in the soil which must be corrected for by analysis of a blank soil sample. The reactions in this ammonium chloride extraction are complete within about five minutes.

After settling of the soil particles, 10 ml of the extraction solution is obtained by pipette, to which approximately 100 ml of deionized water is added. This solution contains soluble calcium proportionate to the lime content plus excess ammonium chloride. The pH of the solution is adjusted to 12.5 using the sodium hydroxide solution. This is the midpoint of the 12-13 pH range in which the hydroxynaphthol blue indicator provides its calcium specific color change. To this solution is added four drops of triethanolamine solution and 0.2 gram of powdered indicator. With pH probes in place, the solution is titrated with EDTA until a red-purple to blue endpoint color change occurs, maintaining the pH at 12.5 with sodium hydroxide as necessary. The results can be reported as ml of titrant or converted to weight percent of calcium hydroxide in soil.

Mixtures of hydrated lime and soil were prepared to test this method. This work showed the difficulty in obtaining a representative sample from a loose mixture and caused us to abandon the idea of obtaining core samples from an actual job using dry quicklime placement.

The method chosen for comparison of the slaking, mixing, and reaction characteristics involved preparation of 4 x 6 inch cylindrical compacted soil-lime specimens. This mixing and compaction procedure was intended

to simulate actual field methods such as the standard item requirements for quicklime placement which call for a slaking period of two to seven days between mixing and compaction.

One soil sample was used for preparing all the specimens. This sample had a plasticity index of 49 and an optimum moisture content of 20 percent. All of the specimens were prepared with 6.25 percent lime and optimum moisture unless otherwise noted. A separate mix was prepared for each specimen and all of that mix was used to prepare the specimen.

The dried soil was placed in a pan and the required amount of water to obtain optimum moisture was sprinkled on and well mixed into the soil. The quicklime was then spread evenly over the soil and the stoichiometric quantity of water needed for slaking was sprinkled evenly over the lime. After covering the pan the material was allowed to sit undisturbed for 24 hours at standard laboratory temperature. Using a trowel, the soil and lime were then mixed while taking care not to break up any lumps of lime which had not broken up on their own. This was done by running the trowel down the side of the pan, under the soil, and back up through the soil. A mellowing period of six days followed during which lost moisture was made up. After a total slaking time of seven days the mix was compacted into a 4 x 6 inch mold in four lifts according to the procedure outlined in Test Method Tex-113-E. Subsequent curing took place in a moist room according to the procedure for capillary absorption, Test Method Tex-117-E.

The above procedure was used to prepare specimens from each of the six limes using capillary cure times of two, five, and eight weeks for separate specimens. These specimens are identified by numbers 3 through 20. Inadvertently, specimens 3-14, with five and eight week cures, were allowed to slake unmixed for six days and had an uncompacted mixed cure of only one day. Specimens 15-20 were cured as originally planned.

Additional mixes were prepared with Brands A, B, C, and F limes. These mixes, Numbers 29, 30, 28, and 31 respectively were prepared to check

the short-term curing before compaction. After a 24-hour slake period, the soil and lime were mixed and allowed to cure covered and uncompacted. One-third of the material was removed at 4 hours, 24 hours, and seven days after initial mixing. These mixes were mixed and separated by hand.

Samples of Brands F and B quicklimes were sieved over a 1/4 inch screen and the material retained on the screen was discarded. One specimen was prepared with the minus 1/4 inch material from each lime using the standard procedure and the specimens were moist cured for five weeks. These specimens, No. 26 (Brand F) and No. 27 (Brand B), were intended for comparison against the standard gradation quicklime.

Specimens were also prepared using standard gradation Brand F and Brand B quicklimes but using moisture contents of 15 and 30 percent instead of the standard 20 percent. These specimens received a 24-hour slake time and six days of uncompacted cure and were analyzed after this time period.

In order to see the effects of slaking and mellowing time, specimens were prepared which were slaked, allowed to sit overnight, mixed, and cured uncompacted for two days before being analyzed. These mixes were prepared using Brand A (No. 25) and Brand C (No. 24) quicklimes.

One specimen was prepared using Brand D hydrated lime instead of quicklime. The uncompacted mix was stored seven days and the moist cure after compaction was five weeks. A moisture content of 20 percent was used.

All of the compacted specimens were tested for compressive strength immediately after being removed from the moist room at the end of their curing periods. The unconfined strength was determined using a crosshead speed of 0.15 in./min. After determining the strength, the specimens were broken up by hand using spatula and hammer to where the soil lumps were all passing a 3/4-inch sieve. The broken cylinders and the uncompacted mixes were dried by spreading evenly in a 22 by 13-inch pan and placing in a 140 F forced convection oven. Normally two to four hours of drying time provided a sample dry enough to be ground to pass the 40-mesh sieve. The dried material was then analyzed as explained previously.

## Results

When testing the various quicklimes according to Test Method Tex-600-J, Wet Sieve Analysis, problems occurred. Some of the quicklimes reacted rather violently with water, creating a gelling and/or lumps in the slurry. These characteristics have the potential to cause problems in the process of mixing and applying the slurry. It was found that if the quicklimes are mixed with an excess of water to produce an approximately 30 percent solids content slurry, no lumping occurs. Some plants' quicklimes take more water to make the slurry workable which means it may be necessary to go to a rather low solids content to produce a usable slurry.

The chemical analysis of the solids retained on the Nos. 6 and 30 sieves varied considerably between suppliers. For four of the six materials the residue was primarily calcium carbonate and inert material, and therefore of little soil stabilizing value. The other two materials showed a fairly high percentage of calcium hydroxide and calcium oxide in the screen residues.

In the work involving adding dry quicklime to the soil, the overnight slaking period produced a fluffy hydrated lime with relatively little lumping. None of the compacted cylinders showed cracking due to unhydrated material. After breaking the cylinders, the amount of undispersed lime clumps was very visible. The Brand B, C, and F specimens had the most visible lime, the Brand E the least.

An ammonium chloride extraction analysis was performed on a blank soil sample for a correction. The blank sample required 9.3 ml of EDTA for the titration. This adjustment was made to all samples analyzed.

Tabulated results follow:

EVALUATION OF SLURRIED QUICKLIME

Producer	% by wt of CaO in Quicklime Tex-600-J	Sieve Anal. of Orig. Dry Quicklime (% by wt ret'd on:)			Temp (F) at various time intervals in min. from start of Hydration	Wet Screen Residue of Quicklime % by wt*		Wet Screen Residue of Quicklime % by wt**		% Solids of Slurry*	Hydrate Alkalinity of "Solids Content" of Slurry % by wt*		
		1" Sieve	3/4" Sieve	100 Mesh Sieve		#6	#30	#6	#30				
						10	20	30					
A	94.9	0.0	0.5	86.0	115 114	149 143	172 156	0.8 1.3	1.2 1.7	2.2 -	5.5 -	31.1	96.7
B	89.7	0.0	0.0	96.4	120 118	149 137	165 153	7.2 8.2	9.6 10.0	11.9 -	32.8 -	28.5	96.4
C	87.7	0.0	1.8	99.0	119 117	148 145	163 166	2.7 3.4	3.8 4.2	4.1 -	21.0 -	29.7	95.2
D	94.2	0.0	2.8	99.1	119 126	149 153	166 173	1.2 0.3	4.1 2.4	1.0 -	8.7 -	32.4	95.7
E	87.8	0.0	7.5	98.3	109 103	142 129	154 149	2.9 3.9	5.2 5.6	3.0 -	10.9 -	28.6	94.4
F	91.7	0.0	0.8	98.1	119 118	148 147	167 165	0.9 0.9	5.3 4.5	1.1 -	8.2 -	31.0	87.8

\*800 g lime placed in 2600 ml of 74-78 F water in 80-gram increments of lime every three minutes.

\*\*1700 ml of 74-78 F water placed on 800 g quicklime in one 1700 ml addition.

Chemical Analysis of Material Retained on Nos. 6 and 30 Sieve  
Results in % by Wt of Dry Sample\*

Producer	Ca(OH) <sub>2</sub>	CaO	CaCO <sub>3</sub>	Inert	H <sub>2</sub> O	Total
A	13.49	6.19	76.92	3.40	0.00	100.00
B	12.41	0.00	70.68	16.10	0.80	99.99
C	4.76	-	98.70	3.20	-	100.00
D	77.16	1.88	14.64	6.30	0.00	99.98
E	12.05	19.77	63.16	5.00	0.00	99.98
F	70.08	8.86	14.46	6.60	0.00	100.00

EVALUATION OF SLURRIED QUICKLIME

<u>Producer</u>	<u>Viscosity of Slurry 77 F, Poises*</u>		<u>Viscosity of Slurry 140 F, Poises*</u>	<u>Bulk Density of Slurry 30 min after Start of Hydration lb/gal (US)</u>	<u>Water req'd (ml) to adjust vis of Slurry at 77 ± 2 F to 25-30 Poises*</u>		<u>% Solids of adjusted vis Slurry*</u>	<u>Characteristics of Original Slurry</u>
	<u>20 rpm</u>	<u>5 rpm</u>	<u>20 rpm</u>		<u>ml</u>	<u>Poises</u>		
A	73	80	43	10.01	100	28	26.0	Liquid - High vis, but no thixotropic properties
B	165	184	111	9.90	250	26	18.5	Gelled - Putty consistency
C	80	100	59	9.92	120	26	24.3	Gelled - Putty consistency
D	26	68	23	10.00	-	-	-	Liquid - Flows evenly
E	13	44	11	9.90	-	-	-	Liquid - Flows evenly
F	33	56	33	10.01	-	-	-	Liquid - Flows evenly

\*800 g lime placed in 2600 ml of 74-78 F water in 80-gram increments of lime every three minutes.



RESULTS FROM SOIL/LIME MIXES

<u>Producer</u>		<u>4 hr</u>	<u>24 hr</u>	<u>7 day</u>	<u>2 wk</u>	<u>5 wk</u>	<u>8 wk</u>	<u>2-day Slake 5 wk</u>	<u>-1/4" 5 wk</u>	<u>15% Moisture</u>	<u>30% Moisture</u>
A	Specimen No.	29	29	29	16	4	10	25			
	% Reacted	20.1	32.8	26.1	47.9	44.4	47.1	69.7			
	Comp. Str. (psi)	-	-	-	190.5	332.8	399.5	435.8			
B	Specimen No.	30	30	30	17	5	11		27	32	33
	% Reacted	19.2	22.4	24.4	47.1	42.3	55.0		54.1	23.9	28.9
	Comp. Str. (psi)	-	-	-	201.7	265.0	344.1		368.5	-	-
C	Specimen No.	28	28	28	15	3	9	24			
	% Reacted	24.9	25.7	15.1	53.5	38.4	49.8	61.4			
	Comp. Str. (psi)	-	-	-	174.3	284.8	408.1	404.1			
D	Specimen No.	-	-	-	19	7	13				
	% Reacted	-	-	-	43.9	42.4	46.2				
	Comp. Str. (psi)	-	-	-	190.2	340.7	439.8				
E	Specimen No.	-	-	-	18	6	12				
	% Reacted	-	-	-	49.5	42.0	45.5				
	Comp. Str. (psi)	-	-	-	178.0	292.7	392.2				
F	Specimen No.	31	31	31	20	8	14		26	34	35
	% Reacted	20.2	23.8	36.9	46.1	48.1	58.7		54.7	24.8	30.7
	Comp. Str. (psi)	-	-	-	154.5	309.0	348.7		360.5	-	-

<u>Producer</u>		<u>2 wk</u>	<u>5 wk</u>	<u>8 wk</u>
D	Specimen No.	21	22	23
Hydrated	% Reacted	48.3	54.0	69.9
Lime	Comp. Str. (psi)	178.3	328.3	487.3

DRY SIEVE ANALYSES OF QUICKLIMES USED TO PREPARE CYLINDERS, PERCENT BY WEIGHT

<u>Producer</u>	<u>+ 3/4</u>	<u>-3/4/+3/8</u>	<u>-3/8/+1/4</u>	<u>-1/4/+30</u>	<u>-30/+100</u>	<u>-100</u>	<u>-30</u>
A	1.3	78.4	10.0	3.7	1.7	5.0	
B		56.3	29.2	13.3			1.2
C		89.2	3.7	5.3			1.9
D	1.5	76.9	17.4	2.4			1.8
E	4.8	74.6	11.3	6.6			2.7
F		36.1	23.8	35.4			4.7

The preparation of slurries from the quicklimes showed variations in their workability and flow properties. The viscosities determined at 77 F show that standard 31 percent solids content slurries prepared from Brands A, C and especially Brand B are difficult to work with because of their high viscosities. Addition of water in order to obtain a workable mixture resulted in a solids content of only 18.5 percent for Brand B.

Residue from wet screening of the prepared slurries was the greatest for Brand B. The retained material was mostly carbonate and inert.

Strength results from the cylinders gave a better correlation than the unreacted lime content analysis. The strengths also correlated well with the amount of visible lime particles found after breaking open the cylinders. The lime content analysis showed discrepancies in the specimen preparation. The 4 hour, 24 hour, and 7 day results do not show the expected trend of reducing lime content, presumably due to inaccurate sample splitting. The 2 week standard cure results show a greater amount of reaction than do the 5 week results, apparently because of the duration of mixed uncompacted cure. The results from the 2-day slake specimens also come out differently than expected.

The strength results, which appear to be a truer indication of the relative stabilizing effects, do not show a correlation with the residue retained on the No. 30 screen. Neither the strength nor the lime content results shows any appreciable difference between the various producers. Results from the -1/4 inch quicklime and hydrated lime specimens indicate that smaller sized material provides better stabilization.

## Conclusions

The physical properties of the various quicklimes supplied to the State of Texas vary, depending on geographical source and therefore producer. The greatest differences observed between the different producers' materials were in the viscosities and thixotropic qualities of the slurries prepared from quicklime. This wide variation at a given solids content makes it undesirable to use certain of the quicklime slurries because of high viscosity. The Brand B quicklime, for example, might not be practical at any solids content because either the viscosity is too high or the solids content is too low.

Compressive strength of the cylinders seems to be a better indication of the stabilizing benefits of lime in soil than chemical analysis for unreacted lime. The wet screen analyses showed a much greater amount of Brand B material being retained on the No. 6 and No. 30 sieves. This would seem to indicate a problem with slaking and dispersion and therefore with stabilizing benefits. The cylinder strength and reaction results do not show, however, any consistent or major differences between the various dry applied quicklimes. The wet screen test appears to not be a true indication of the stabilizing benefits of dry placed quicklime.

The limited data gathered using hydrated lime and -1/4 inch quicklime indicates that a finely graded lime gives greater stabilizing benefit than a coarsely graded lime. The results from this work which are shown and previous work not shown both indicate that a rapid initial reaction takes place after mixing lime into soil. Possibly 15 to 20 percent of the lime reacts within the first four hours.

## Recommendations

Based on this work there are several changes to the existing state specifications which can be recommended. The specifications which govern quicklime are Special Provisions 260-001, 262-001, and 264-001, all dated 12-82. There are also changes to these Special Provisions which are recommended here and which are not necessarily a direct result of this research but relate to handling or purity.

Special Provision 264-001 establishes the requirement for the chemical and physical properties of quicklime (Type C). The purity, percent by weight CaO, can be raised from a minimum of 85 percent to 87 percent in order to better represent the typical product of established sources. The present test procedure and requirement on residue after slaking (wet sieve) should be eliminated. In order to preclude obtaining a slurry produced from quicklime containing large quantities of oversize material which might cause problems in handling and placement, it is recommended that a maximum limit of 5 percent for residue retained on a No. 6 sieve be included. The method of test would be modified to include the slurring procedure used in this research.

Pebble quicklime is used in dry form and for preparing slurry, either at a central plant or at the job location. The sizing requirements prevent an excessive amount of fines which, during dry placement, might present a safety hazard. For slurry use, however, this should not be the case. It is recommended, therefore, that a separate grade of quicklime be established which has no maximum limit on the fine material and which can be used for preparation of slurry only. The requirement that all material shall pass the one-inch sieve should be retained, however, along with the other requirements of Special Provision 264-001 and the changes recommended here. The standard Type C quicklime may still be used dry or as slurry. The new slurry grade quicklime should be so designated when shipped from its source.

Special Provisions 260-001 and 262-001 and Items 260 and 262 govern the methods of placement of lime for soil stabilization purposes. Along with the above changes, the following requirements should be added to these specifications. On-site slurring of quicklime, either grade, must be done in a manner to limit the amount of dusting so there is no hazard to personnel, the public, or adjacent property. Type C slurries must be of a consistency that can be handled and applied without difficulty.

## State Department of Highways and Public Transportation

## Materials and Tests Division

## LIME TESTING PROCEDURE

## PART I

## SAMPLING AND INSPECTION OF HYDRATED LIME, QUICKLIME AND COMMERCIAL LIME SLURRY

## Scope

## A. For Hydrated Lime

1. In powdered form as bulk hydrate.
  - a. From loaded tank trucks.
  - b. As discharged from tank trucks.
  - c. At other locations.
2. In powdered form as bagged hydrate.
  - a. From bag trucks being loaded.
  - b. From bagged shipments after delivery to warehouse or job site.

## B. For Quicklime

1. In crushed or pebble form.
  - a. As discharged from tank trucks.
  - b. At other locations.

## C. For Commercial Lime Slurry

1. As a mixture of hydrated lime solids in water.
  - a. From sampling port at plant site in the tank truck loading apparatus.
  - b. At other locations.

## Apparatus

## A. For Bulk Hydrate

1. Paint brush, 2 inch width.
2. Gallon bucket with double friction type lid, with bail.
3. Short sampling tube consisting of a 9-foot length of 1-1/2 inch IPS PVC 1120 plastic pipe of SDR 26, 160 psi, to meet ASTM 2241. This tube is fitted at one end with a rubber stopper drilled with a 1/4 inch diameter hole. The stopper may be cemented in place, using State Department of Highways and Public Transportation (SDHPT) standard grade epoxy designated as "B-25" marker adhesive. A 3 inch wide by 2 inch high, half-round plate of 12-gauge steel to which a hook is spot-welded should be riveted and cemented with epoxy to the rubber stopper end of the sampling tube.

The 3 inch long metal hook is a 1/2 inch wide by 3/4 inch doubled over section with 1/4 inch slot. It has the opening of the hook facing away from the rubber stopper. The hook is intended to serve as an aid to lowering the bucket off the top of the truck. The hook fastens to the open bucket bail so that the bucket and rod can be lowered to ground level. This allows the sampler to get down off the truck without having to concern himself with the sample bucket and the sample rod in addition to his own safety.

4. Long sampling tube consisting of two concentric plastic pipes with a pointed tip cap closure at one end and a 1-1/4 inch wide by 12 inch long sampling port cut through both pipes at a location some one and one-half inches from the lower end of the concentric pipe arrangement. The solid aluminum or solid epoxy cap end shall be inserted one inch into the tube and should rest as a solid bottom some 1/2 inch below the sampling port window. The outer pipe shall be 9 feet, 8 inches in length and shall consist of 1-1/2 inch IPS PVC plastic pipe fitted at the sample port end with cap made from a 7-1/2 inch length of solid aluminum round stock or cast epoxy plastic of 1-5/8 inch diameter tapered to a point along 6-1/2 inches of its length. This aluminum or plastic stock shall be of a diameter to fit inside the 1-1/2 inch IPS PVC plastic pipe and shall be fastened to it with two screws through the wall of the plastic pipe into two tapped holes on either side of the prepared cap. The point of the cap should be rounded off to a 1/2 inch diameter point for safety reasons. SDHPT standard grade epoxy designated as "A-103" adhesive may be used to mold up a cast epoxy tip in a metal, foil or cardboard mold. The inner pipe in the concentric pipe arrangement shall consist of a 1-1/4 inch IPS PVC plastic pipe 10 feet in length so as to extend beyond the outer pipe at the upper end so as to allow the sampler to rotate the inner tube within the outer tube so as to open and close the sampling port. Vertical index marks on the outside of the outer and inner tubes at the upper end should be marked "open" and "closed" so as to indicate the proper degree of rotation for effective bottom sampling of loaded tank trucks.

5. Catch pan, with handles, 8 inches deep by 12-1/2 inches wide by 18 inches long, SDHPT Catalog No. 2074500.

## B. For Bagged Lime

1. Paint brush, 2 inch width.

2. Gallon bucket with double friction type lid, with bail.

3. Bag sampling tube, 3/4 inch diameter steel electrical conduit cut to a three-foot length. The opening at one end shall be tapered by having that end cut off with a four-inch diagonal cut.

C. For Quicklime

1. Safety goggles for eye protection.

2. Respirator for respiratory protection.

3. Rubber gloves, for hand protection.

4. Paint brush, 2 inch width.

5. Gallon bucket with double friction type lid, with bail.

6. Bucket holder for quicklime sampling. This holder should be designed to permit the gallon sample bucket to be held in place between the wheel path of a bulk transport discharging quicklime without the bucket turning over when the sample is allowed to drop into it. After the sample is taken, the device shall be equipped with a means of removing the container from the quicklime windrow with a minimum of difficulty in a manner allowing the sample to be removed intact with a minimum of hazard to the sampler. One such device which has been used for this purpose has been constructed as follows: From 3/4 inch thick plywood, cut three square pieces. One piece is to be 18 inches square, another 14 inches square and the third 10 inches square. Cut a circular hole in the center of the 14 inch and 10 inch square boards. These holes should be of a size to closely fit a gallon bucket of the size being used as a sample container. Stack the three boards one atop the other. The 10 inch board is to be placed atop the 14 inch board, centered and fastened to it. The 10 and 14 inch boards should be placed atop the 18 inch board, centered and fastened to it. To the three board sampling device, fasten an 8 foot length of 1/8 inch diameter flexible steel cable to one of the corners. To the other end of the cable, attach a 10 inch length of steel conduit or wooden dowel to serve as a "T" handle to assist in pulling the bucket and sample from beneath windrowed quicklime.

7. Plastic sample bag, 18 inches by 9-1/2 inches (General Warehouse Catalog No. 2005000).

D. For Commercial Lime Slurry

1. Safety goggles for eye protection.

2. Polyethylene bottle, two and one-half gallon (10 liter) capacity, large mouth type with handles. The actual mouth opening is approximately 3-1/2 inches in diameter. The bottle closure is approximately 100 mm

and is fitted with a polypropylene or phenolic screw type cap. The bottle manufactured of conventional polyethylene and listed under Catalog Item No. 2234-0020 of Catalog No. 481 of the Nalge Company, a Division of Sybron Corporation, P.O. Box 365, Rochester, New York 14602, has been found to be satisfactory for this purpose.

3. Plastic electrical tape, PVC 3/4 inch width.

4. Cloth rag or shop towel.

Sampling Procedure

A. For Hydrated Lime

1. In powdered form as bulk hydrate.

From loaded tank trucks. In order to determine if the material being offered for acceptance does in fact meet specifications, as required, it is necessary that the material be made available in such a manner that a representative half-gallon portion can be obtained for purpose of analysis.

Since the purchase specification for hydrated lime limits the amount of moisture and other impurities which the product may contain, it is essential that the sample taken be representative of the overall quantity of the material contained in the truck. For this reason, the sample should be taken so as to avoid contamination by moisture or other road materials. Obviously, there is less likelihood of sample contamination occurring if the sample is taken from the truck itself prior to unloading. Since these tank trucks are pressurized for unloading, and attempts to open a pressurized top hatch might prove fatal, it is essential that the arrangements for on-truck sampling be such that the material in the truck be offered for inspection with top hatches "open." In effect, this requires the Contractor to make the load available for inspection prior to pressurization. If the truck is being offered for inspection in a "pressurized" condition, it shall be the responsibility of the Contractor to arrange to have the pressure bled off and the top hatches opened for inspection on any truck designated for inspection.

Preferred Sampling Method:

Method 1 - Rodding material from atop truck through open hatches.

Less desirable methods would be:

Method 2 - Scoop samples obtained through open top hatches.

Method 3 - As discharged from tank trucks.

If for some reason, the material in the truck cannot be sampled prior to unloading, then various optional sampling methods may be used provided the sample is truly representative and is not allowed to become



contaminated by moisture or mixing with base or other road material. Rodding the truck is the preferred sampling method. Other less desirable methods would be:

- Method 3a. Hose discharge.
- Method 3b. Dry application "catch-pan" method.

Samples should not be scooped from dry applied material spread on roadway due to likelihood of contamination by moisture and/or other road material.

Notes: Bulk hydrated lime should be sampled at the rate of one sample per approximate 200 tons, unless otherwise directed. This represents, roughly, one pneumatic trailer out of every ten shipped. The trailer to be sampled should be selected "at random" and identified on the sample ticket by seal number, name of producer and date sampled, in addition to the other information usually supplied on those forms. Samples shall not be "composited" or combined. The single sample from the truck selected to represent the ten or so trucks involved, shall be identified by seal number. The seal numbers of the other nine loads need not be listed on the sample ticket. The top rim of the sample bucket being of the double friction type may tend to collect lime in the top groove. A paint brush should be used to clear the rim of lime so that an effective seal may be obtained. Lime tends to become contaminated by exposure to the atmosphere. A tight seal is important in preserving the quality of the sample taken.

Two lengths of bulk sampling rods have been listed. The short sampling tube (9 foot length) is designed for routine sampling through the top hatches of a bulk transport. A sample obtained with this device represents, primarily, the upper portion of any given load. The tube is inserted with the air hole in the rubber stopper closed. When withdrawing the tube, hold the hole shut with a thumb. The long sampling tube (9 feet, 8 inches in length) is a concentric pipe arrangement designed to obtain samples at various levels in a bulk transport. It is designed primarily to obtain "bottom" samples occasionally, but may be used to obtain a representative sample at various levels and may be used for all routine sampling tasks. The long tube is inserted with the door closed, the door is opened at the sampling level desired. The tube is moved horizontally back and forth a bit and the door is then closed and the tube removed with the door closed. Lime may be removed by tapping the rod and allowing the lime to exit via the top end into a sample bucket.

2. In powdered form as bagged hydrate.

a. From bag trucks being loaded. If necessary, the sampler should ask plant personnel to make available the sacks which he desires to sample. Choice

of sacks should be made on the basis of obtaining a representative sample of the lot being shipped. The sampler should use the bag sampling tube to take through the sack loading spout sufficient diagonal rod-dings to insure a representative portion from each sack, but not a sufficient quantity to materially reduce the poundage being supplied in any individual sack. It is suggested that at least four to six sacks from each lot being inspected be sampled and the material combined to form a composite sample representing that lot of material. If the entire lot consigned for an individual truck is from several different warehouse lots, the sampler may, at his discretion, take samples from the separate lots which comprise the whole. Generally speaking, at least six sacks should be sampled to represent each truck being shipped.

b. From bagged shipments after delivery to warehouse or job site. It is suggested that the bag sampling tube described in the equipment list be used to obtain a total of a half-gallon sample from at least six sacks of material. Insert the bag sampling tube through the loading valve and take several through samples diagonally from each sack, taking care not to puncture the bottom or sides of the sack with the sharp sampling tube. It is suggested that the sacks chosen for sampling be selected from various points in the load or shipment so as to effect a reasonable representative sample.

Sampling Procedure

B. For Quicklime

CAUTION: Quicklime is extremely hazardous and capable of inflicting severe caustic burns to skin, lung damage and/or eye injury. It can possibly cause blindness if handled improperly. Personnel handling, sampling or testing and working around quicklime should wear proper protective clothing, respirators, dustproof goggles and waterproof gloves to prevent injury.

1. In Crushed or Pebble Form.

a. As discharged from tank trucks. Quicklime being somewhat soft and friable generates fines in transit. Since air-blown quicklime fines are hazardous, quicklime is usually unloaded at the job site by gravity feed through unbolted inspection ports at the bottom of each pot or compartment on the truck. Most trucks will be equipped with 3 or 4 such pots which are usually opened to discharge simultaneously.

It is recommended that sampling be accomplished by allowing the truck to pass over a sampling device while unloading. Quicklime, as unloaded, may create a pile of up to 8 or 10 inches in height as the truck travels along the unloading strip or land. Since the specification limits the amount of fines in the sample and includes sizing requirements, it is important that the sample taken be truly representative of the truck load as a whole and that sizing and gradation of the material

sampled be truly representative. Quicklime fines tend to settle to the bottom of the pots on the truck and the initial discharge usually contains a higher percentage of fines than the remainder of the load. The last of the load discharged tends to contain the coarser grade material. The center of the discharge run has been found to represent the most representative gradation to represent the entire truck load. For this reason, it is suggested that quicklime samples be taken from the midpoint of the discharge path, i.e., halfway between the initial discharge and the final emptying of the truck. The sampler should pick up the bucket by the bail from the sampling device in which it has been placed. The entire sample should be carefully transferred to a plastic sample bag. The bag should be closed and sealed with tape or a rubber band and placed in a clean gallon bucket with a double friction lid for transport. The bucket should be marked "caustic" and "quicklime" for transportation by motor freight. It should not be presented for shipment by bus, parcel post, air or railroad transportation. This is a hazardous material which, upon contact with water and combustibles, can cause fires. For this and other safety-related reasons, the carriers listed have refused to accept the material for shipment.

b. In the tank trucks. It is recommended that sampling be accomplished by opening the port at the top of the trucks and digging below the surface of the pebble quicklime at least 8 inches and then dipping a sample with a one-gallon bucket. A sample should be a minimum of 3/4 of a gallon. This material should be placed in a plastic bag previously placed in a one-gallon bucket and sealed to keep moisture out. The friction lid should be placed tightly on the bucket and all safety precautions as described in a. should be observed.

## Sampling Procedure

### C. For Commercial Lime Slurry

1. From sampling port in the tank truck loading apparatus at the plant. Lime slurry tends to settle quite rapidly. Upon settling, the bottom portion tends to be higher in percent solids and lower in purity than the top portion. In order to take a representative sample of lime slurry, it is recommended that the sample be taken from thoroughly agitated and extremely well-mixed storage tanks by means of a sampling valve placed in the vertical riser going to the loading spout. This sampling port is to be located between the storage tank and the loading spout. It is to be operated only after the storage tank material has been stirred by centrifugal pumps, compressed air, or vane stirrers or combinations of these stirring devices. The sample should be taken through a 1/2 inch, quick acting valve, fitted to a 3/4 inch diameter pipe and outlet spout. Half fill a 2-1/2 gallon sample container by quickly opening the valve to maximum open position and then quickly closing the valve. Small openings of the valve tend to prevent larger particles in the slurry from being sampled. Since there are sieve size requirements for lime slurry, just barely opening the valve does not always permit a truly representative sample to be taken. Take the sample during the truck loading operation at a point where the truck is about half loaded. Sample while the slurry is being pumped to the truck.

6. At other locations. The sampling of lime slurry from loaded tank trucks at the job site is not recommended without centrifugal pumps, compressed air, or a combination of these or other stirring devices. It is doubtful that a representative sample can be taken from a loaded tank truck. Spraybar samples taken from trucks without agitation will tend to be high in solids and lower in purity on initial discharge and lower in solids and higher in purity upon final discharge. It is preferred that samples for pay basis be taken at the plant site.

State Department of Highways and Public Transportation  
Materials and Tests Division

LIME TESTING PROCEDURE

PART II

TESTING OF HIGH CALCIUM TYPE HYDRATED LIME, TYPE A

Scope

This procedure is intended to serve as a reasonably accurate, fairly rapid means of testing hydrated lime in the powdered form.

Theoretical Considerations

This test procedure is based on the assumption that the principal constituents present in the product are calcium hydroxide, calcium oxide or water, calcium carbonate and inert matter as silica dioxide. Mixed alkali acid titration test data is integrated with test values obtained through the use of muffle furnace loss on ignition of the sample at elevated temperatures. Basically then it is this combination of data from acid titrations along with loss on ignition test data which is used to calculate the relative percentages of the most likely principal constituents present.

The acid titration portion of the test consists of the separate determination of hydrate alkalinity and carbonate alkalinity. The hydrate alkalinity is assumed to be due to calcium hydroxide plus any possible calcium oxide present. The carbonate alkalinity is assumed to be due to calcium carbonate. The total alkalinity of the sample by acid titration is the sum of the hydrate and carbonate alkalinities.

The loss on ignition is assumed to be solely due to the evolution of carbon dioxide and water from the sample at the elevated temperature of 2000 degrees Fahrenheit. The carbon dioxide is being derived from the calcination of the calcium carbonate to form calcium oxide and carbon dioxide gas. The water portion of the loss on ignition is either so called "free-mechanical moisture" or chemically combined water, or both. The chemically combined water is derived from the disassociation of calcium hydroxide at the elevated temperature to form calcium oxide and water vapor. The residue from the loss on ignition is assumed to be only calcium oxide plus "inert matter" in the form of silicon dioxide or sand.

The water portion of the loss on ignition is determined by subtracting the carbon dioxide content of the sample from the loss on ignition which is taken to be the sum of carbon dioxide and water. By difference then, we have the water portion of the loss on ignition. This water portion is converted to calcium hydroxide equivalent

gravimetrically. The lesser of the calcium hydroxide as determined by titration and the calcium hydroxide as determined by the loss on ignition is taken to be the true calcium hydroxide content of the sample.

The difference between the calcium hydroxide by titration and the calcium hydroxide by ignition is either "free water" or "free calcium oxide." When the titration has excess hydrate alkalinity that excess is converted to calcium oxide and so reported. When the ignition indicates alkalinity in excess of that indicated by titration the excess is converted to water and so reported. The total "active" lime content is the arithmetic sum of the calcium hydroxide and calcium oxide constituents in the sample.

The loss on ignition test serves primarily to determine the percent "inert matter" present in the sample. If the "inert matter" content of a sample is known with a reasonable degree of accuracy, then the loss on ignition test can be dispensed with and a so called "assumed inert" value may be used with the titration test data to calculate a range of probable calcium hydroxide contents. In effect by employing the "assumed inert" concept the titration alone serves as an extremely fast method of determining the probability of a particular sample having an "active" lime content in excess of the required minimum values required by a particular specification. For compliance testing, however, the complete analysis is to be performed.

Apparatus

1. Electric muffle furnace capable of sustained operation at a regulated temperature of  $2000 \pm 20^\circ\text{F}$ .
2. Platinum crucibles, low form wide bottom type, with reinforced rim and bottom, 30 cc capacity. These crucibles shall have a platinum content of at least 99.5 percent and should contain less than 0.3 percent iridium.
3. Tongs for use with muffle furnace, 20 inch length, made of nickel plated steel rod. Tongs are to be fitted with solid tips made of ruthenium-platinum alloy weighing approximately 40 grams.
4. Hard-surfaced asbestos board.

5. Vacuum desiccator.
  6. Indicating type silica-gel desiccant.
  7. Analytical balance, suitable for rapid weighing, with a minimum weighing capacity of 100 grams which meets the requirements of Test Method Tex-901-K, Class I-B.
  8. Top-loading balance, suitable for rapid weighing, with a minimum weighing capacity of 2000 grams which meets the requirements of Test Method Tex-901-K, Class II-E.
  9. Weighing boat, stainless steel, with tab type handle, for use with analytical balance. Size approximately 3 inches in length by 1-1/2 inches in width by 3/4 inch depth, fitted with a 1" x 3/4" tab, weight 20 grams.
  10. Spatula.
  11. Balance brush, camel's hair type.
  12. Bags, plastic, clear, with zip-lock type closures. Size approximately 4" x 6".
  13. Beakers, borosilicate type glass, Berzelius, Tall Form, without spout, graduated. Capacity 400 ml, similar to Corning Glassware Catalog Item No. 1040-400 or Kimble Glassware Catalog Item No. 0172165.
  14. Buret 100 ml capacity, of borosilicate glass, with a 50 ml bulb at the top and 50 ml in the graduated tube. With permanently color-etched scale, with 1/10 ml subdivisions. Similar to MCA Type No. 3 buret as marketed by Kimble Division of Owen-Illinois, under their catalog No. 17097-F, to comply with the requirements for this type buret in ASTM E 287. Buret may be modified for automatic zero and a side tube for filling may be utilized with a straight stopcock with Teflon plug, if so desired.
  15. Buret, 50 ml capacity, preferably of alkali-resistant glass, if available, otherwise borosilicate glass may be used subject to periodic replacement as necessary if used with alkaline solutions. The buret is to have 1/10 ml subdivisions with permanently color-etched scale and an automatic zero feature.
  16. Magnetic type stirrer, with a 1-1/2 inch diameter round teflon covered stirring bar 5/16 inch in thickness.
  17. Stopwatch with 60 second long hand to indicate 1/5 second and seconds. The long hand is to complete one turn in 60 seconds. The watch should also have a short hand to register up to 30 minutes. Operation should preferably be by successive depressions of the crown to accomplish starting, stopping, and returning to zero.
  18. Plastic tubing, 3/8 inch ID, 1/8 inch wall thickness, of suitable length, fitted with a Bunsen "fan-type" burner attachment at the outlet end of the hose.
  19. Zero to 30 psi water pressure gauge, 3 inch diameter face, with 3/8 inch IPS female thread fitting.
  20. Sieves. A set of Standard U.S. Sieves containing the following sizes: No. 6 and No. 30 which meets the requirements of Test Method Tex-907-K. All sieves are to be of 8 inch diameter. Sieve set is to be complete with one lid and one catch pan.
  21. pH meter having an accuracy of plus or minus 0.1 pH unit or better, with automatic temperature compensator jacks for automatic temperature control.
  22. pH meter electrode, standard combination type.
  23. Automatic temperature compensator for pH meter.
  24. pH meter reference electrode filling solution, KCl, 4M saturated with AgCl.
- #### Materials
1. Potassium hydrogen phthalate, National Bureau of Standards pH Standard, Standard Reference Material 185e or in lieu of this material, the following grade of the chemical may be utilized: potassium hydrogen phthalate, Acidimetric Standard, ACS, Fisher Scientific 1983 Catalog No. P-243, or J.T. Baker Catalog Item No. 2958 or an equal quality of this primary standard reagent grade chemical.
  2. Bromphenol blue, tetrabromophenolsulfonphthalein, to meet ACS specifications.
  3. Hydrochloric acid, assay (HCl) (by acidimetry) 36.5 to 38.0 percent, to meet ACS specifications.
  4. Phenolphthalein powder, to meet ACS specifications.
  5. Sodium borate, 10-hydrate, crystal or powder (sodium tetraborate, decahydrate) to meet ACS specifications.
  6. Sodium hydroxide, 50 percent solution, reagent grade, assay (NaOH) (by acidimetry) 50 to 52 percent. Baker Catalog No. 3727, or equal.
- #### Special Solutions Required:
1. 1.0 N Sodium hydroxide solution. This solution shall have a normality of  $1.0000 \pm 0.0005$  N, when the normality of the solution is accurately determined by direct standardization against the specified potassium hydrogen phthalate, acidimetric standard, suitably dried

if necessary. For the purpose of this standardization 8.1691 grams of the prescribed acidimetric standard shall be considered as equivalent to 40.00 ml of 1.000 N sodium hydroxide.

a. Preparation. A sodium hydroxide solution of the required normality may be prepared by measuring out the required volume of 50 percent sodium hydroxide solution and adding it to deionized or distilled water to bring the resulting solution to the desired volume.

It is important that the caustic be added to a sizable portion of the water to be used so that the heat evolved is dissipated without being concentrated in a small volume of liquid which can cause hazardous spattering of hot caustic, steaming, etc. The resulting mixture should be stirred at least four hours and preferably overnight, since it is extremely slow to become uniformly mixed. This caustic solution should be stored in a polyethylene or polypropylene container, since it will react with borosilicate and other glass over extended periods of time.

The volume of 50 percent sodium hydroxide solution required to prepare this solution is approximately 52.3 ml of 50 percent NaOH per liter of resulting solution.

For example, if 18.5 liters of solution is to be made up to fill a five-gallon plastic carboy, take  $18.5 \times 52.3$  ml or 967.6 ml of 50 percent NaOH and add it to 1/2 to 2/3 of a plastic carboy full of deionized or distilled water. Mix for 5 or 10 minutes and bring the total volume up to an 18-1/2 liter mark on the plastic carboy. Place carboy and contents on a magnetic stirrer and stir for at least four hours and preferably up to a total of 12 hours.

The factor of 52.3 is derived from the following relationships:

A concentration of 1.0 N NaOH = 40.0 g NaOH/liter.

In a 50 percent NaOH solution at 60°F, there is 0.7645 g NaOH per ml of solution; therefore:

$$\frac{40.0 \text{ g NaOH/liter of } 1.0 \text{ N NaOH}}{0.7645 \text{ g NaOH/ml of } 50\% \text{ NaOH}} = 52.3 \text{ ml of } 50\% \text{ NaOH/liter of } 1.0 \text{ N NaOH}$$

After this solution is prepared and stirred sufficiently, then the normality should be determined by standardization with the prescribed potassium hydrogen phthalate.

b. Standardization. Transfer 50 ml of the sodium hydroxide solution to be standardized to a well-rinsed 50 ml Class A buret by means of a pipette. Accurately weigh out from 7.5 to 8.5 grams of the specified grade of potassium hydrogen phthalate onto a

tared watch glass. Quantitatively transfer the weighed material from the watch glass to a 400 ml capacity tall-form beaker. Add 185 ml of carbon dioxide free distilled water at a temperature of 77 to 83°F and stir the mixture gently until the crystals dissolve. Then add five drops of described phenolphthalein indicator solution and proceed to titrate the prepared standard with the sodium hydroxide solution to be standardized. Titrate to a visual end point of light pink, which will persist for at least 60 seconds. Record the number of ml of sodium hydroxide solution required for the titration. Calculate the normality of the sodium hydroxide solution as follows:

$$\text{Normality of NaOH} = \frac{\text{Wt of potassium hydrogen phthalate in g}}{\text{ml of NaOH required} \times \text{factor of } 0.204228}$$

This preliminary standardization may reveal the necessity for either dilution with deionized or distilled water or the necessity to add 50 percent caustic solution in order to bring the solution concentration to within the normality specified. The final standardization must show the normality to be within  $\pm 0.0005$  N of the 1.0000 Normality specified.

If the normality shown on this preliminary standardization is not within these limits, it may be adjusted as follows:

If base is "too weak"

For example if 18.5 liters of 0.9907 N solution needs to be strengthened to 1.0000 N, calculate how much 50 percent NaOH solution to add as follows:

$$\begin{aligned} 18.500 \times 0.991 &= 18.334 \\ 18.500 - 18.334 &= 0.166 \\ 0.166 \times \text{factor of } 52.3 &= 8.68 \text{ ml of } 50 \text{ percent} \\ &\quad \text{NaOH solution needed} \end{aligned}$$

If base is "too strong"

For example, if the 18.5 liters of 1.021 N solution needs to be weakened to 1.0000 N, calculate how much deionized or distilled water to add as follows:

$$\begin{aligned} 18.500 \times 1.021 &= 18.888 \\ 18.888 - 18.500 &= 0.388 \text{ liters or } 388 \text{ ml of deionized} \\ &\quad \text{or distilled water needs to} \\ &\quad \text{be added.} \end{aligned}$$

After all anticipated or calculated additions have been made, the resulting well-mixed solution should be subjected to final standardization. To do so, continue to titrate individual portions of caustic solution until standardization values obtained agree to within 0.0002 Normal. If necessary, utilize ASTM E 178 "Standard Practice Dealing with Outlying Observations" to reject test results sufficiently out of agreement with other test values to warrant rejection.

2. 1.0 N Hydrochloric Acid Solution. This solution shall have a normality of  $1.0000 \pm 0.0005$  N when the normality of the solution is accurately determined by direct comparison with the previously standardized sodium hydroxide.

a. Preparation. A hydrochloric acid solution of the required normality may be prepared by measuring out the required volume of concentrated hydrochloric acid, ACS grade, and adding it to the required volume of deionized or distilled water to bring the resulting solution to the desired volume. To prepare 19.7 liters of solution in a five-gallon glass carboy, add 1625 ml of concentrated acid to 1/2 to 2/3 of a bottle of distilled or deionized water in the bottle and stir. Add sufficient water to the mixture to bring the total volume to the 19.7 liter mark. Acid mixed into water at the ratio of 82.5 ml of concentrated HCl per liter of water will give an approximate 1 N solution of HCl. It is important that the acid be added to a sizable portion of the water to be used so that the heat evolved is dissipated without being concentrated in a small volume of liquid. Such concentration can cause hazardous spattering of hot acid, fuming, etc. The prepared solution should be stirred by placing the carboy on a magnetic stirrer and stirring for at least four hours and preferable up to a total of 12 hours.

b. Standardization. Transfer an approximate 40 ml portion of the acid solution to be standardized into a 400 ml tall-form titration beaker. Use a burette to deliver this volume and accurately record the volume delivered. Add 140 ml of distilled water plus five drops of the specified phenolphthalein indicator to the portion of the acid solution contained in the beaker and proceed to standardize by titrating the contents of the beaker by adding from a burette some of the previously standardized sodium hydroxide.

Titrate to a visual end point of light pink, which will persist for at least 60 seconds. Record the number of ml of sodium hydroxide required for the titration. Calculate the normality of the hydrochloric acid solution as follows:

$$N \text{ HCl} = \frac{\text{ml of NaOH required} \times N \text{ of NaOH used}}{\text{ml of HCl used}}$$

This preliminary standardization may reveal the necessity for either dilution with deionized or distilled water or the necessity to add concentrated HCl in order to bring the solution concentration to within the normality specified. The final standardization must show the normality to be within  $\pm 0.0005$  N of the 1.0000 normality specified. If the normality shown on this preliminary standardization is not within these limits, it may be adjusted as follows:

If acid solution is "too weak"

For example, if 19.7 liters of 0.9908 N solution needs to be strengthened to 1.000 N, calculate how much concentrated HCl to add as follows:

$$\begin{aligned} 19.700 \times 0.991 &= 19.523 \\ 19.700 - 19.523 &= 0.177 \\ 0.177 \times \text{factor of } 82.5 &= 14.60 \text{ ml of concentrated HCl needed} \end{aligned}$$

If acid solution is "too strong"

For example, if 19.7 liters of 1.024 N solution needs to be weakened to 1.0000 N, calculate how much deionized or distilled water to add as follows:

$$\begin{aligned} 19.700 \times 1.024 &= 20.173 \\ 20.173 - 19.700 &= 0.473 \text{ liters or } 473 \text{ ml of deionized or distilled water needs to be added.} \end{aligned}$$

After all anticipated or calculated additions have been made, the resulting well-mixed solution should be subjected to final standardization. To do so, continue to titrate individual portion of hydrochloric acid solution in this fashion until standardization values obtained agree to within 0.0002 Normal. If necessary, utilize ASTM E 178 "Standard Practice Dealing with Outlying Observations" to reject test results sufficiently out of agreement with other test values to warrant rejection.

3. pH Buffer Solution. pH value 4.01 at 25°C, 0.05 M KH phthalate solution.

Preparation

Prepare this solution by dissolving  $10.21 \pm 0.05$  gram of primary standard reagent grade potassium hydrogen phthalate in sufficient water to make 1000 ml of solution.

The pH of the potassium hydrogen phthalate solution at various temperatures should be as follows:

Temperature		pH of 0.05 M KH Phthalate Solution
°C	°F	
15	59	3.999
20	68	4.002
25	77	4.008
30	86	4.015
35	95	4.024
40	104	4.035

4. pH Buffer Solution. pH value 9.18 at 25°C, 0.01 M NaB<sub>4</sub>O<sub>7</sub> (Borax) solution.

## Preparation

Prepare this solution by dissolving  $3.81 \pm 0.01$  gram of reagent-grade sodium tetraborate decahydrate in sufficient water to make 1000 ml of solution. Fresh-looking crystals of borax should be used. This buffer solution should be stored in a plastic container.

The pH of the borax solution at various temperatures should be as follows:

Temperature		pH of 0.01 M Borax Solution
°C	°F	
15	59	9.276
20	68	9.225
25	77	9.180
30	86	9.139
35	95	9.102
40	104	9.068

5. Phenolphthalein Indicator Solution. Prepare this solution by dissolving 0.5 gram of powdered phenolphthalein, ACS grade, in 50 ml of 200 proof (absolute) ethyl alcohol and add 50 ml of distilled or deionized water.

6. Bromphenol Blue Indicator Solution. Prepare this solution by dissolving 0.1 gram of powdered bromphenol blue in 7.5 ml of 0.02 N NaOH solution and diluting the mixture to 250 ml.

## Procedure

1. Sample Preparation. Take a representative half-gallon portion of the powdered lime to be analyzed and mix well for one to two minutes. Mixing should take place in a one-gallon paint bucket fitted with a double friction type lid. Thoroughly shake the sample contained in the bucket, invert the bucket and shake again, invert and shake again, etc. until the sample is well mixed. Remove the bucket lid and scoop out enough material to fill a sample vial 1/2 to 3/4 full. A 1-3/4 inch OD by 3 inch height vial made of crystal clear polystyrene with a tight fitting polyethylene cap has been found to be satisfactory for this purpose. After each sample withdrawal, the sample bucket and or container lid should be promptly replaced so as to minimize atmospheric contamination of the material to be analyzed. Moisture in the air tends to air slake calcium oxide in samples to convert it to calcium hydroxide which tends to recarbonate by reacting with carbon dioxide in the air to form calcium carbonate. Thus sample exposure to air tends to degrade the quality of the sample somewhat by reducing the active lime content in the manner described.

## 2. Titrimetric Analysis of Various Alkaline Components of the Sample.

a. Titration to pH 8.3 with HCl solution. Weigh out on an analytical balance  $2.804 \pm 0.0050$  g of the lime in the sample vial which had been reserved for analysis. Reseal the sample vial. The weighed sample should be transferred quantitatively to a 400 ml tall form beaker. Transfer material slowly so as to minimize loss of sample due to dusting. Slowly add 150 ml of distilled water to the sample contained in the beaker. Add a stirring magnet and stir the sample mixture on a magnetic-type stirrer for 30 to 90 seconds to disperse the lime mixture into a somewhat uniform suspension prior to starting the titration. Titrate with the 1.0 N hydrochloric acid solution to a prescribed end point of 8.3 on a pH meter of the type specified, which has been buffered in accordance with the pH meter manufacturer's directions using the pH 4.0 and 9.0 solutions described. The lime mixture should be stirred slowly while the titration progresses at the prescribed rate.

The rate of addition is dictated by the response or reaction time evidenced by the sample. Various lime samples will vary in response to additions of titrant. It is the intent of this procedure to titrate as rapidly as the reaction rate of a particular sample will allow. Large excess of titrant will cause localized pooling of titrant in which areas bubbling of carbon dioxide gas from the reaction of acid with calcium carbonate may be observed. This reaction occurs at a pH of less than 5.4. In the titration to pH 8.3, therefore, the titrant is added at a rather rapid rate, but sufficiently slow to keep the pH above or greater than 6.0 at all times. With addition of titrant the pH will drop and then in most cases recover or rise to a higher value as more solid material dissolves and enters into the reaction. After the first 50 ml of titrant has been added as described the pH indication may change upon addition of titrant to a momentary pH indication of less than 9.0. At this point, continued additions of titrant should be made in a "rapid drop-wise" fashion until a pH indication of between 6.0 and 9.0 is obtained which persists for 30 seconds or less. At this point, add titrant slowly in small increments to maintain a pH slightly less than 8.3 which will rise above 8.3 at which point more titrant is added until 8.3 or slightly less persists for 60 seconds. The prescribed end point for this titration to pH 8.3 is taken to be that point at which the addition of one two-drop increment of titrant produces a pH meter indication of 8.3, or slightly less than 8.3 when observed exactly 60 seconds after the addition of titrant. Record the volume of titrant required to reach this pH 8.3 end point before proceeding with the further addition of HCl to "measured excess."

b. Titration to "measured excess" of HCl. After the 8.3 end point has been reached, continue to add hydrochloric acid titrant to the sample contained in

the beaker to a point where a total of 100 ml of titrant has been added to the sample since the beginning of the titration. The sample mixture contained in the titration beaker at this point shall be considered as containing a "measured excess" of hydrochloric acid. Record the total volume of acid added to the sample since the beginning of the titration before proceeding with the back-titration. This should always be 100.00 ml.

c. Back-titration to pH 4.4 with NaOH.

Titrate the sample mixture plus the "measured excess" of hydrochloric acid contained in the beaker with the 1.0 N sodium hydroxide solution to a pH meter indication of 4.4, or slightly greater than 4.4 when observed exactly 10 seconds after a one-drop addition of titrant. The first occurrence of such an end point in the normal course of the back-titration shall be taken to be the prescribed end point for this portion of the analysis.

3. Determination of percent loss on ignition.

Weigh out a 2.9 to 3.1 gram portion of the well-mixed material contained in the gallon bucket. Transfer the material by means of a spatula from the sample vial to a previously ignited and weighed platinum crucible. This sample portion should be weighed accurately to the fourth decimal place on the described analytical-type balance after which the crucible and sample portion which it contains should be placed uncovered in an electric muffle furnace maintained at a temperature of  $2000 \pm 20^\circ\text{F}$ , where it should be allowed to remain for a required ignition period of three hours.

After the sample has been ignited in this fashion, it should be removed from the furnace and placed on a piece of hard-surfaced asbestos board such as "Transite" for a period of 10 to 30 seconds prior to placing the crucible in a vacuum desiccator for further cooling. If several samples are being run simultaneously, it has been found expedient to remove all of the samples from the furnace, place them on the asbestos board and then proceed to place them in the desiccator, replace the desiccator lid with the vacuum stopcock in the open position. The heated air will expand through the opening for a period of several seconds after which the stopcock should be closed. This, in effect, has been found to cause a moderate vacuum without necessitating the use of an aspirator, safety bottle, etc. The residue from the loss on ignition should be allowed to cool for a minimum of 20 minutes prior to weighing. This weighing operation should take place promptly after the cooling period so as to avoid possible contamination of the highly reactive quicklime residue. The actual weighing of this residue is to be accomplished as rapidly as possible so as to prevent hydration of the residue due to exposure to the atmosphere.

4. Screen Analysis. Weigh out the remainder of the sample contained in the one-gallon sample bucket

and screen all of it. Weigh this screen analysis sample to the nearest 0.1 g and then attempt to water-wash this material first through the No. 6 (3360 micron) sieve and then through the No. 30 (590 micron) sieve. The wash water to be used for this purpose shall be tap water applied in a fantail spray pattern from the 3/8 inch ID plastic tubing fitted with a Bunsen "fan-type" burner attachment at the outlet. The water pressure in the plastic tubing shall be adjusted to  $12 \text{ psi} \pm 2 \text{ psi}$  by a water valve. The pressure shall be monitored on a 0-30 psi water gauge "Teed" into the 3/8 inch inlet line past the valve.

In no case should the velocity of the stream of water being used to wash the residue be allowed to become sufficiently strong so as to cause loss of sample over the sides of the sieve. The washing shall be continued until the water coming through each sieve is clear. If milkiness persists, the washing operation may be continued up to a maximum of 30 minutes. In no case shall the washing operation be continued past the point where the wash water appears clear. When the washing is complete, the residue on each sieve shall then be oven-dried in a mechanical convection oven for a period of one hour at a temperature of  $212 \pm 10^\circ\text{F}$ . After drying, the residue retained on each of the screens shall be removed from the oven and allowed to cool for 20 minutes. The residue on each screen shall be transferred to a tared plastic bag and weighed separately on an analytical balance to the nearest 0.01 g.

#### Calculations

A form setting forth in proper sequence all the calculations necessary to determine the composition of the lime is presented in Appendix I along with a definition of each of the terms appearing on the form. Example calculations for three different samples are shown — one containing free water and two containing unhydrated lime (CaO). For referee testing, exact calculations, as presented in Appendix I must be made. For routine quality control, the calculations may be simplified by the following:

1. Assuming the normality of the acid and base, which has been standardized to  $1.000 \pm 0.0005 \text{ N}$ , is actually 1.0000 N.

2. Controlling the weight of the titration sample to within  $2.8040 \pm 0.0050$  grams, but assuming that it is actually 2.8040 grams. This results in the acid and base factors and the sample weight being the same numerically, thus each milliliter of titrant is equivalent to one percent by weight of hydrate alkalinity, as CaO, present in the lime.

Appendix II describes the procedure for rapid calculation of a lime sample's constituents.



State Department of Highways and Public Transportation

Materials and Tests Division

APPENDIX I

STANDARD CALCULATION METHOD

1. Definition of Terms

A = Apparent hydrate alkalinity of the sample indicated by titration and calculated as % CaO.

B = Carbonate alkalinity of the sample derived by titration and calculated as % CaO.

B' = Carbonate alkalinity of the sample derived by titration and calculated as % CaCO<sub>3</sub>.

C = Total alkalinity of the sample calculated as % CaO.

D = Apparent hydrate alkalinity of the sample indicated by titration and expressed as % Ca(OH)<sub>2</sub>.

E = Carbonate alkalinity of the sample derived by titration and calculated as % CO<sub>2</sub>.

F = Loss on ignition of the sample when heated for 3 hours at 2000°F, as % loss in weight and reported as % loss on ignition.

G = "Total Water" of the sample calculated as H<sub>2</sub>O.

H = Apparent hydrate alkalinity of the sample indicated by the "Total Water" portion of the loss on ignition and expressed as % Ca(OH)<sub>2</sub>.

I = Corrected value for the hydrate alkalinity of the sample calculated as % Ca(OH)<sub>2</sub>.

J = "Free Water" of the sample calculated as % H<sub>2</sub>O.

K = Unhydrated lime content of the sample calculated as % CaO.

L = "Inert Matter" content of the sample calculated as % "Inert Matter."

M = Screen analysis on the sample expressed as the % by weight of the total solids retained on the No. 6 sieve.

O = Screen analysis on the sample expressed as the % by weight of the total solids retained on the No. 30 sieve.

Values obtained by analysis: A  
C  
F  
M  
O

Values obtained by calculations: B  
B'  
D  
E  
G  
H  
I  
J  
K  
L  
Total "Active"  
Lime Content

Values to be reported: B'  
I  
J  
K  
L  
M  
O  
Sum I + K

$$I = N \text{ of acid used} \times \frac{56.08}{2000} \times 100$$

Note: I = a factor derived by multiplying the normality of the acid used by the product of the molecular weight of calcium oxide divided by 2000 (to convert to milli-equivalents) multiplied by 100 (so as to express the final unit to be calculated in terms of percent). Example: In a case where the normality of the acid being used is 1.0005 then the correct value for  $I = 1.0005 \times 2.804 = 2.8054$ .

$$II = N \text{ of base used} \times \frac{56.08}{2000} \times 100$$

$$III = \text{ml of acid to pH of 8.3}$$

$$IV = (I) \times (III)$$

$$V = \text{Total ml of acid used}$$

Note: This volume of acid should approximate 100 ml and represents that volume of acid used in the titration to reach a pH of 8.3 plus that volume of acid added to insure a "measured excess" before proceeding with the prescribed "back-titration."

$$VI = (I) \times (V)$$

VII = Ml of base used in the "back-titration" to proceed from the point of "measured excess" to the prescribed end point of a pH of 4.4.

$$VIII = (II) \times (VII)$$

$$IX = (VI) - (VIII)$$

X = Titration sample weight expressed in grams.

XI = Loss on ignition sample weight expressed in grams.

XII = Loss on ignition residue weight expressed in grams.

XIII = Weight of the screen analysis sample (weighed to the nearest 0.1 gram) screened on the No. 6 sieve.

XV = Weight of the screen analysis sample (weighed to the nearest 0.1 gram) screened on the No. 30 sieve.

XVI = Weight of solids retained on the No. 6 sieve (weighed to the nearest 0.01 g)

XVIII = Weight of solids retained on the No. 30 sieve (weighed to the nearest 0.01 g)

## 2. Basic Calculations and Remarks

A = Apparent hydrate alkalinity of the sample indicated by titration and calculated as % CaO

Calculations:

$$\%CaO = \frac{(IV)}{(X)}$$

Note: This equation is derived from the expression

$$\% CaO = \frac{ml \times N \times meq\ wt \times 100}{sample\ wt}$$

Where:

ml = net ml of acid to pH 8.3

N = N of acid used

meq wt = milli-equivalent weight of CaO

100 = factor for conversion to percent

sample wt = titration sample weight

B = Carbonate alkalinity of the sample derived by titration and calculated as % CaO

Calculations:

$$\% CaO = C - A$$

B' = Carbonate alkalinity of the sample derived by titration and calculated as % CaCO<sub>3</sub>

Calculations:

$$\% CaCO_3 = B \times (1.78479)$$

C = Total alkalinity of the sample calculated as % CaO

Calculations:

$$\% CaO = \frac{(IX)}{(X)}$$

Note: This equation is derived from the expression

$$\% CaO = \frac{ml \times N \times meq\ wt \times 100}{sample\ wt}$$

Where:

ml = net ml of acid to pH 4.4

N. = N of acid

meq wt = milli-equivalent weight of CaO

100 = factor for conversion to percent

sample wt = titration sample weight

D = Apparent hydrate alkalinity of the sample indicated by titration and expressed as % Ca(OH)<sub>2</sub>

Calculations:

$$\% Ca(OH)_2 = A \times (1.32126)$$

Note: 1.32126 is a gravimetric factor used to convert % CaO to % Ca(OH)<sub>2</sub>

E = Carbonate alkalinity of the sample derived by titration and calculated as % CO<sub>2</sub>

Calculations:

$$\% \text{CO}_2 = B \times (0.78479)$$

Note: 0.78479 is a gravimetric factor used to convert CaO to % CO<sub>2</sub>

F = Loss on ignition of the sample when heated for three hours at 2000°F.

Calculations:

$$\% \text{ Loss on ignition} = \frac{(XI) - (XII)}{(XI)} \times 100$$

Note: This equation is derived from the expression

$$\% \text{ loss on ignition} = \frac{(\text{sample wt}) - (\text{residue wt})}{(\text{sample wt})} \times 100$$

For the purpose of this test procedure, loss on ignition shall be taken to be the sum of the losses due to "water of dehydration," "free water," and chemically combined carbon dioxide.

"Water of dehydration" shall be defined as that portion of the loss on ignition due to the loss of water resulting from the forced dehydration or disassociation of calcium hydroxide at elevated temperatures.

"Free water" shall be defined as that portion of the loss on ignition which is not accounted for by the loss due to "water of dehydration" plus the loss due to chemically combined carbon dioxide.

"Total water" shall be defined as that portion of the loss on ignition due to "water of dehydration" plus "free water."

G = "Total water" of the sample expressed as % H<sub>2</sub>O

Calculations:

$$\% \text{H}_2\text{O} = F - E$$

Note: This equation is derived from the assumption that at 2000°F the principal losses to be encountered will be carbon dioxide and water, and that any gain in weight due to oxidation of other impurities such as sulfides to sulfates will be negligible. The basic assumption is

that "total water" expressed as % H<sub>2</sub>O = (loss on ignition) - (carbonate alkalinity of the sample derived by titration expressed as % CO<sub>2</sub>).

H = Apparent hydrate alkalinity of the sample indicated by the "total water" portion of the loss on ignition and expressed as % Ca(OH)<sub>2</sub>

Calculations:

$$\% \text{Ca(OH)}_2 = G \times (4.1128)$$

Note: 4.1128 is a gravimetric factor used to convert % H<sub>2</sub>O to % Ca(OH)<sub>2</sub>

I = Corrected value for the hydrate alkalinity of the sample calculated as % Ca(OH)<sub>2</sub>

Calculations:

% Ca(OH)<sub>2</sub> = H in Case I Where D > H, then unhydrated lime is indicated as being present in the sample.

= D or H in Case II Where D = H, then neither unhydrated lime nor "free water" is indicated as being present in the sample.

= D in Case III Where H > D, then "free water" is indicated as being present in the sample.

Note: In instances in which this test procedure is used to arrive at a value for "Hydrate alkalinity, percent by weight Ca(OH)<sub>2</sub>," then it is to be understood that it is to be the value "I" which shall be reported since this is the corrected value for the hydrate alkalinity of the sample.

Where the procedure is used to arrive at a value for "Total 'active' lime content, percent by weight" then it is to be understood that "active" lime content is to be the value "I" + value "K" and shall be reported as the sum of "I + K" or the "active" lime content of the sample.

J = "Free water" of the sample calculated as % H<sub>2</sub>O

Calculations:

- % H<sub>2</sub>O = zero in Case I      Where D > H, then unhydrated lime is indicated as being present in the sample.
- = zero in Case II      Where D = H, then neither unhydrated lime nor "free water" is indicated as being present in the sample.
- = (H - D) × (0.24314) in Case III      Where H > D, then "free water" is indicated as being present in the sample.

Note: 0.24314 is a gravimetric factor used to convert Ca(OH)<sub>2</sub> equivalents to H<sub>2</sub>O equivalents.

K = Unhydrated lime content of the sample calculated as % CaO

Calculations:

- % CaO = (D - H) × (0.75686) in Case I      Where D > H, then unhydrated lime is indicated as being present in the sample.
- = zero in Case II      Where D = H, then neither unhydrated lime nor "free water" is indicated as being present in the sample.
- = zero in Case III      Where H > D, then "free water" is indicated as being present in the sample.

Note: 0.75686 = the conversion factor to convert Ca(OH)<sub>2</sub> equivalents to CaO equivalents.

L = "Inert matter" content of the sample calculated as % inert matter

Calculations:

% "inert matter" = 100 - (C + F)

Note: For the purpose of this test procedure, "inert matter" shall be defined as those sample constituents which by

their chemical nature do not react under the conditions prescribed in acid titration nor do they volatilize under the conditions prescribed for the loss on ignition determination. It shall be assumed to consist essentially of acid insolubles plus possible metallic oxides.

M = Screen analysis on the sample expressed as % by weight of the total solids retained on the No. 6 sieve.

Calculations:

% solids retained on No. 6 sieve (3360 microns) =

$$\frac{(XVI)}{(XIII)} \times 100$$

O = Screen analysis on the sample expressed as % by weight of the total solids retained on the No. 30 sieve.

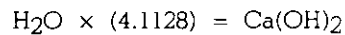
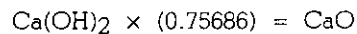
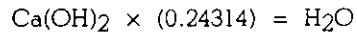
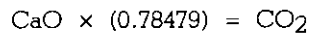
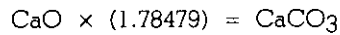
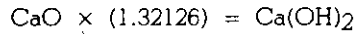
Calculations:

% solids retained on No. 30 sieve (590 microns) =

$$\frac{(XVIII)}{(XV)} \times 100$$

Summary of Calculations

- A =  $\frac{(IV)}{(X)}$
- B = C - A
- B' = B × (1.78479)
- C =  $\frac{(IX)}{(X)}$
- D = A × (1.32126)
- E = B × (0.78479)
- F =  $\frac{(XI) - (XII)}{(XI)} \times 100$
- G = F - E
- H = G × (4.1128)
- I = H if H < D  
D if D < H  
D or H if D = H
- J = zero if D > H  
zero if D = H  
(H - D) × (0.24314) if H > D
- K = zero if H > D  
zero if D = H  
(D - H) × (0.75686) if D > H
- L = 100 - (C + F)
- M =  $\frac{(XVI)}{(XIII)} \times 100$
- O =  $\frac{(XVIII)}{(XV)} \times 100$

Factors UsedBasic Assumptions Concerning the Loss on Ignition Portion of the Test Procedure

For the purposes of this test procedure, it shall be assumed that the loss in weight of sample during the loss on ignition portion of the test procedure is attributable solely to the volatilization of "free water," "water of dehydration" and of chemically combined carbon dioxide. The residue remaining in the platinum crucible, after the loss on ignition portion of the analysis, is assumed to be unhydrated calcium oxide plus "inert matter" which will not volatilize during the ignition at elevated temperatures nor react chemically during the acid titration of the principal alkaline constituents of the sample. This "inert matter" is usually termed "SiO<sub>2</sub>," "acid insolubles," "combined metallic oxides," etc.

Basic Assumptions Concerning the Titration Portion of the Test Procedure

The titration portion of the analysis involves the assumption that the principal acid neutralizable alkaline constituents are calcium hydroxide, calcium oxide and calcium carbonate. All of these constituents need not be present simultaneously, of course. This test procedure implies that under the conditions set forth for analysis, alkaline carbonates will not enter into the reaction during the first stage of the titration (to the end point of 8.3). The "measured excess" of acid plus the back titration with base is intended to cope with the bulk of the alkaline carbonates present and they are calculated as being present in the usual form of calcium carbonate. If unhydrated oxides are present in the sample, they will usually hydrate and/or react with the acid during the first stage of the titration (to the end point of 8.3) and will be calculated as calcium hydroxide. If they do not react under the conditions of the test, they will of course be considered as "inert matter" and calculated as such.

Possible Presence of Magnesium Compounds

This entire test procedure is intended to be used in connection with the testing of a high-calcium type lime hydrate. Magnesium compounds when present are assumed to be present to only a small degree and, as such, are either ignored or else calculated to the basis of calcium compounds. In instances where magnesium compounds are present to a significant degree, such a hydrate will usually encounter difficulty in meeting the requirements of the high-calcium type lime specifications for which this particular test procedure was designed.

HYDRATED LIME Page 1			ACID FACTOR	BASE FACTOR	ML ACID TO pH 8.3	PARTIAL ACID PRODUCT	TOTAL MLS OF ACID USED	TOTAL ACID PRODUCT	ML BASE USED	BASE PRODUCT	NET PRODUCT	TITRATION SAMPLE WEIGHT	TOTAL ALKALINITY	HYDRATE ALKALINITY	
PRODUCER <b>ZLC</b>			Nx(2.804)	Nx(2.804)		(I)x(III)		(I)x(V)		(II)x(VII)	(XI)-(VIII)		$\frac{(IX)}{(X)}$	$\frac{(IX)}{(X)}$	
DATE SAMPLED	SEAL #	LAB #	I	II	III	IV	V	VI	VII	VIII	IX	X	C	A	
07 Sep 82	13669	J82480001	2 8051 2 8051	2 8046 2 8046	71 80 71 55	201 41 200 70	100 00 100 00	280 51 280 51	26 40 26 80	74 04 75 16	206 47 205 35	2 8038 2 7992	73 64 73 36	71 83 71 70	
07 Sep 82	13673	J82480002	2 8046 2 8046	2 8048 2 8048	70 39 70 41	197 42 197 47	100 00 100 00	280 46 280 46	26 99 27 01	75 70 75 76	204 76 204 70	2 8037 2 8042	73 03 73 00	70 41 70 42	
09 Sep 82	13677	J82480006	2 8048 2 8048	2 8043 2 8043	70 31 70 28	197 21 197 12	100 00 100 00	280 48 280 48	26 91 26 88	75 46 75 38	205 02 205 10	2 7989 2 7993	73 25 73 27	70 46 70 42	

HYDRATED LIME Page 2			TOTAL ALKALINITY	HYDRATE ALKALINITY	CARBONATE ALKALINITY	CaCO <sub>3</sub>	CO <sub>2</sub>	LOSS ON IGNITION SAMPLE WT	LOSS ON IGNITION RESIDUE WT	IGNITION SAMPLE WT LOSS	% LOSS ON IGNITION	% INERT MATTER	TOTAL H <sub>2</sub> O	% Ca(OH) <sub>2</sub> by IGNITION	% Ca(OH) <sub>2</sub> by TITRATION	H <sub>2</sub> O as Ca(OH) <sub>2</sub>	CaO as Ca(OH) <sub>2</sub>	% Ca(OH) <sub>2</sub> CORRECTED VALUE	"Free" water as % H <sub>2</sub> O	% CaO	% Total active lime																					
PRODUCER <b>ZLC</b>			AVERAGE	AVERAGE	C - A	B x (1.78479)	B x (0.78479)			(XI) - (XII)	$\frac{(XI) - (XII)}{X} \times 100$	100 - (C+F)	F - E	G x (4.1128)	A x (1.5226)	H - D	D - H	LESSER OF D or H	(H - D) x (0.24514)	(D - H) x (0.75686)	I + K																					
DATE SAMPLED	SEAL #	LAB #	C	A	B	B'	E	XI	XII		F	L	G	H	D			I	J	K	Sum I + K																					
07 Sep 82	13669	J82480501	73	50	71	76	1	74	3	1	37	3	0010	2	2724	0	7286	24	28	2	22	22	91	94	22	94	81			0	59	94	22			0	45	94	67			
07 Sep 82	13673	J82480502	73	02	70	42	2	60	4	64	2	04	3	0034	2	2236	0	7798	25	96	1	02	23	92	198	38	93	04	5	34			93	04	1	30			93	04		
09 Sep 82	13677	J82480506	73	26	70	44	2	82	5	03	2	21	3	0035	2	2824	0	7211	24	01	2	73	21	80	89	66	93	07			3	41	89	66			2	58	92	24		

HYDRATED LIME Page 3								#6 & #30 SCREENS SAMPLE WEIGHT		# 6 SCREEN RESIDUE WEIGHT		#30 SCREEN RESIDUE WEIGHT		% RETAINED ON # 6		% RETAINED ON # 30
PRODUCER <b>ZLC</b>														$\frac{(XVI)}{(XIII)} \times 100$		$\frac{(XVII)}{(XIII)} \times 100$
DATE SAMPLED	SEAL #	LAB #						XIII		XVI		XVIII		M		0

07 Sep 82	13669	J82480001						487	6				0	00		0	14			0	00			0	03
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07 Sep 82	13673	J82480002						499	0				0	00		0	34			0	00			0	07
-----------	-------	-----------	--	--	--	--	--	-----	---	--	--	--	---	----	--	---	----	--	--	---	----	--	--	---	----

09 Sep 82	13677	J82480006						507	2				0	00		0	22			0	00			0	04
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State Department of Highways and Public Transportation  
 Materials and Tests Division

APPENDIX II

OPTIONAL RAPID CALCULATION METHOD

Requirements for using procedure:

Limits For Sample Wt And Titration Solutions

Sample Wt  $2.804 \pm 0.0050$  grams  
 Acid  $1.000 \text{ N} \pm 0.0005 \text{ N}$   
 Base  $1.000 \text{ N} \pm 0.0005 \text{ N}$

In conjunction with this procedure, a table is included for converting milliliters acid or base to component equivalents along with a work sheet which may be used.

Procedure:

1. Milliliters of HCl to pH 8.3 is equivalent to the hydrate alkalinity. Record this value and the corresponding uncorrected  $\text{Ca(OH)}_2$  from table shown.

2. Total milliliters HCl added, 100, less the milliliters of NaOH required to obtain a pH of 4.4 is equal to the total alkalinity. Record this value and subtract hydrate from total alkalinity to give carbonate alkalinity. From Table 1, obtain equivalent calcium carbonate and record this value.

3. Calculate the loss on ignition of the sample. The inert value can now be calculated by subtracting the sum of the loss on ignition and the total alkalinity from 100. This inert value (or an average or assumed inert value based on previous analyses of lime from a given source) is then recorded.

4. The uncorrected  $\text{Ca(OH)}_2$ ,  $\text{CaCO}_3$  and the inert value are then totaled. If the total is less than 100 percent, the difference is free water. If the total is greater than 100 percent, quicklime is present. The following examples demonstrate each situation.

Example where CaO is present:

Uncorrected $\text{Ca(OH)}_2$ :	94.8
$\text{CaCO}_3$ :	3.1
Assumed inert:	2.2
	<u>100.1</u>

Subtotal  $100.1 - 100 = 0.1$

$$\% \text{ CaO} = 0.1 \times 3.11 = 0.3$$

Uncorrected $\text{Ca(OH)}_2$	=	94.8
$(\text{Ca(OH)}_2 \text{ Correction Value})$	=	<u>0.4</u>
		94.4

Analysis is shown as:

$\text{Ca(OH)}_2$	=	94.4
$\text{CaCO}_3$	=	3.1
CaO	=	0.3
Total	=	<u>100.0</u>

Example where  $\text{H}_2\text{O}$  is present:

Uncorrected $\text{Ca(OH)}_2$ :	=	93.0
$\text{CaCO}_3$	=	4.6
Assumed inert:	=	<u>1.0</u>
		98.6

$$\% \text{H}_2\text{O} = 100 - 98.6 = 1.4$$

Analysis is shown as:

$\text{Ca(OH)}_2$	=	93.0
$\text{CaCO}_3$	=	4.6
Assumed inert	=	1.0
$\text{H}_2\text{O}$	=	1.4
Total	=	<u>100.0</u>

5. If free water is present the uncorrected hydrate alkalinity will be actual hydrate alkalinity, as  $\text{Ca(OH)}_2$ . If quicklime is present, the  $\text{Ca(OH)}_2$  must be corrected as shown above. The total of corrected  $\text{Ca(OH)}_2$ , CaO or free water,  $\text{CaCO}_3$  and inerts should equal 100 percent.

Table I

For Un-Corrected Calcium Hydroxide Value:

For Whole Milliliters:

ml of Acid and Hydrated Alkalinity	%Ca(OH) <sub>2</sub>
68.0	89.8
69.0	91.2
70.0	92.5
71.0	93.8
72.0	95.1
73.0	96.4
74.0	97.8
75.0	99.1
factor = 1.32126	

For 1/10 Milliliters:

Proportional Parts		
	0.1	0.1
	0.2	0.3
	0.3	0.4
ml	0.4	0.6 %Ca(OH) <sub>2</sub>
acid	0.5	0.7
	0.6	0.8
	0.7	1.0
	0.8	1.1
	0.9	1.3
factor = 1.32126		

For Calcium Carbonate Value As CaCO<sub>3</sub>:

Whole ml Difference Between Two End Points:

Carbonate Alkalinity	CaCO <sub>3</sub>
0.0	0.0
1.0	1.8
2.0	3.6
3.0	5.4
4.0	7.1
factor = 1.78479	

For 1/10 Milliliter Difference:

Proportional Parts		
Fractional Carbonate Alkalinity	CaCO <sub>3</sub>	
	0.1	0.2
	0.2	0.4
	0.3	0.5
	0.4	0.7
	0.5	0.9
	0.6	1.1
	0.7	1.3
	0.8	1.4
	0.9	1.6
factor = 1.78479		

For Correction For Calcium Hydroxide Value When CaO Is Present:

(Sub Total - 100%)	(Ca(OH) <sub>2</sub> Correction Value)
0.1	0.4
0.2	0.8
0.3	1.2
0.4	1.6
0.5	2.1
0.6	2.5
0.7	2.9
0.8	3.3
0.9	3.7
1.0	4.1
1.1	4.5
1.2	4.9

Use this chart if sub total (sum of uncorrected calcium hydroxide value + calcium carbonate value + assumed inert value) is greater than 100%. This means CaO is present and not free water.

If sub total is less than 100% then the uncorrected calcium hydroxide value is assumed correct. Calcium hydroxide and free water are present.

To use this chart to obtain a corrected calcium hydroxide value subtract the Ca(OH)<sub>2</sub> correction value obtained from this chart from the uncorrected calcium hydroxide value.

amount of sub total over 100%

For % Quicklime:

$$\text{When quicklime is present: } \% \text{ CaO} = (\text{Subtotal} - 100\%) \times (3.11)$$

For % Free Water:

$$\text{When free water is present: } \% \text{ free water} = 100\% - \text{Subtotal}$$

Rapid Calculation Method Work Sheet

<u>Titration:</u>		<u>Chemical Composition:</u>	<u>% by Wt:</u>	<u>Code</u>
Lime + Boat	_____g	Hydrate Alkalinity, Ca(OH) <sub>2</sub>	_____	I
Boat	_____g	Unhydrated Lime content, CaO	_____	K
Lime	_____g	Ca(OH) <sub>2</sub> + CaO	_____	I + K
		"Free Water" content, H <sub>2</sub> O	_____	J
<u>Muffle:</u>		<u>Residue:</u>		
Lime + Cruc.	_____g	Retained on No. 6 sieve	_____	M
Crucible	_____g	Retained on No. 30 sieve	_____	O
Lime	_____g			

<u>Titration:</u>		<u>Titration:</u>		
HCl to pH 8.3	_____ml	Uncorrected Ca(OH) <sub>2</sub>	_____	D
HCl, total	100.00 ml	Total alk.	_____	C
NaOH to pH 4.4	_____ml	Hydrate alk.	_____	A
		Carbonate alk. _____ B CaCO <sub>3</sub>	_____	B'
<u>Muffle:</u>		Total alk.	_____	
Lime + Cruc.	_____g	% Loss	_____	
Res. + Cruc.	_____g	100.0 - ( _____ ) = Inert	_____	L
Loss	_____g	Total	_____	
Weighed by: _____		Titrated by: _____		

Screen Analysis: \_\_\_\_\_ - \_\_\_\_\_ = \_\_\_\_\_  
 (Container + Sample) (Container) Sample Wt.

	<u>No. 6</u>	<u>No. 30</u>	
Pan + Res.	_____	_____	
Pan Residue	_____	_____	
	<u>_____ x 100 =</u>	<u>_____ x 100 =</u>	%

Screened by \_\_\_\_\_

## State Department of Highways and Public Transportation

## Materials and Tests Division

## APPENDIX III

Optional Indicator Method for the Titrimetric Analysis of Various Alkaline Components of the Sample

## Procedure

1. This method may be used in lieu of the prescribed pH meter method. However, for all referee tests, or in cases of dispute, the prescribed pH meter method shall be considered the official method to be used. In some cases impurities such as aluminum compounds will prevent or hinder the appearance of the color change in the back-titration portion of this optional method. If such a difficulty is encountered with a particular sample, then use the prescribed pH meter method. The procedure to be followed in employing this optional indicator method shall be exactly as outlined in the pH meter method with the following exceptions:

2. Titration to Phenolphthalein End Point with HCl. At the beginning of the titration to the phenolphthalein end point, add five drops of the prepared phenolphthalein indicator solution to the sample mixture and proceed with the titration to the point where the color of the solution changes temporarily from pink to clear. Before adding additional titrant, allow the pink color to return. At the return of the pink color, additional titrant should be added until the color change appears imminent then add acid in four-drop increments to where the pink coloration disappears for some 35 seconds. Then add two-drop increments until the prescribed end point of "failure to regain pink coloration for 60 seconds" is reached. The prescribed end point for this portion of the titration shall be taken to be that point at which the addition of one two-drop increment of titrant produces a condition in which the mixture in the titration beaker fails to regain its pink coloration when observed exactly 60 seconds after the last addition of titrant. The first occurrence of such an end point in the

normal course of the titration shall be considered as the end point for this portion of the analysis. Once the prescribed end point has been reached, the further re-appearance of pink color shall be ignored. The phenolphthalein end point shall be considered as being roughly equivalent to the pH 8.3 end point described in the official pH meter method of titration. For purposes of calculation, this phenolphthalein end point shall be considered to be the pH 8.3 end point when using this optional indicator method of titration. The volume of titrant required to reach this end point should be recorded before proceeding with the further addition of HCl.

3. Titration to "measured excess" of HCl. Consider the phenolphthalein end point as being the 8.3 end point and proceed to add a "measured excess" of hydrochloric acid by adding titrant to the 100 ml point and recording the total volume of acid added to the sample since the beginning of the titration before proceeding with the back titration. This should always be 100 ml.

4. Back titration to the bromphenol blue end point with NaOH. Add 2.0 ml of the bromphenol blue indicator to the sample mixture plus "measured excess" of HCl contained in the titration beaker. Proceed to titrate this mixture to the point where the color of the mixture changes from yellow to blue with a slight tinge of purple, and maintains this prescribed color of blue when observed exactly 10 seconds after a one-drop addition of titrant. The first occurrence of the prescribed end point in the normal course of the back titration shall be taken to be the bromphenol blue end point. For purposes of calculation, this bromphenol blue end point shall be considered to be the pH 4.4 end point when using this optional indicator method of back-titration.

## State Department of Highways and Public Transportation

## Materials and Tests Division

## LIME TESTING PROCEDURE

## PART III

## TESTING OF HIGH CALCIUM TYPE COMMERCIAL LIME SLURRY, TYPE B

## Scope

This procedure is intended to serve as a reasonably accurate, fairly rapid means of testing hydrated lime in the form of commercial lime slurry.

## Theoretical Considerations

This test procedure is based on the assumption that the principal constituents present in the product are calcium hydroxide, water, calcium carbonate and inert matter as silica dioxide. Mixed alkali acid titration test data is integrated with test values obtained through the use of oven drying and then muffle furnace loss on ignition of the sample at elevated temperatures. Basically then, it is this combination of data from acid titrations along with oven drying and loss on ignition test data which is used to calculate the relative percentages of the most likely principal constituents present.

The acid titration portion of the test consists of the separate determination of hydrate alkalinity and carbonate alkalinity. The hydrate alkalinity is assumed to be due to calcium hydroxide. The carbonate alkalinity is assumed to be due to calcium carbonate. The total alkalinity of the sample by acid titration is the sum of the hydrate and carbonate alkalinities.

The loss on ignition is assumed to be solely due to the evolution of carbon dioxide and water from the sample at oven temperatures and at the elevated temperature of 2000°F. The carbon dioxide being derived from the calcination of the calcium carbonate to form calcium oxide and carbon dioxide gas. The water portion of the loss on ignition is either so called "free-mechanical moisture" or chemically-combined water, or both. The chemically-combined water is derived from the disassociation of calcium hydroxide at the elevated temperature to form calcium oxide and water vapor. The residue from the loss on ignition is assumed to be only calcium oxide plus "inert matter" in the form of silicon dioxide or sand.

The water portion of the loss on ignition is determined by subtracting the carbon dioxide content of the sample from the loss on ignition which is taken to be the sum of carbon dioxide and water. By difference then, we have the water portion of the loss on ignition. This water portion is converted to calcium hydroxide equivalent

gravimetrically. The lesser of the calcium hydroxide as determined by titration and the calcium hydroxide as determined by the loss on ignition is taken to be the true calcium hydroxide content of the slurry. From this we calculate the calcium hydroxide content of the solids contained in the slurry. Excess calcium hydroxide equivalents of water present in the loss on ignition are due to "free water content" and so reported.

## Apparatus

All of the items of equipment listed under Part II for the Testing of Hydrated Lime, are required for the testing of slurry, with the following additional items also being required:

1. Eyedropper, large bore polyethylene type.
2. "Weight per gallon" cup with tare, to be made of stainless steel. This cup should be similar in all essential details to the cup and tare as manufactured by the Garner Laboratory, Division of Pacific, Scientific Co., P.O. Box 5728, 5521 Landy Lane, Bethesda, Maryland 20014.
3. Polyethylene bottles with caps, 16 ounce capacity, with 28 mm size screw cap.
4. Plastic (PVC) electrical tape, 3/4" wide. Scotch Brand No. 33, as manufactured by Minnesota Mining and Manufacturing Co., St. Paul, Minnesota, or equal.

## Materials

The chemical solutions required, buffers, etc., are the same as those listed for the testing of bulk hydrate or powdered lime in Part II of this test procedure and their preparation and standardization is the same.

## Procedure

1. Sample Preparation. The prime consideration in any attempt to obtain a representative sample of a slurried material is the possible settling out of heavy bodied components which may be present in the mixture. At the time and place that a lime slurry sample is taken, every attempt should be made to assure that all the solids present in the material are in an agitated state of relative suspension. The rapid settling of solids in some slurries make it absolutely essential that careful at-

attention be paid to the details of how to properly reduce a sample in size and still obtain a representative portion for final analysis. The following method of sample preparation has been found to be satisfactory for this purpose:

To resuspend a slurry sample contained in the recommended polyethylene bottle (see Part I slurry sampling equipment), it is suggested that the lid be untaped and removed and the sample and its container be placed under a drill press or 1/2" diameter chuck hand drill with variable speed fitted with a long-shafted three inch diameter propeller and that mechanical means be used to work out any possible lumps and bring the slurry material back to a uniform state of suspension before smaller samples are taken for purposes of analysis. A representative portion of the well-mixed slurry contained in the two and one-half gallon bottle should be poured immediately after mechanical agitation into each of two separate pint bottles so as to half fill each of the smaller bottles. Pour rapidly and make every effort to avoid settling. Store the slurry in these pint-sized containers until such time as test portions are to be withdrawn. In replacing the screw type caps, it is important that all traces of slurry be removed from the threaded portion of the bottles and from the threads, etc., inside the cap. If slurry is allowed to remain on these surfaces, there exists the possibility that an air tight seal will not be obtained and that the slurry samples may be subjected to some degree of dehydration pending withdrawal of test portions. The procedure for removal of test portions of slurry from the half filled polyethylene bottles shall be as follows: Shake both the bottle and its contents vigorously for approximately one minute prior to pouring the test portions into the appropriate final containers for test purposes such as "weight per gallon" cups, titration beakers, ignition crucibles, etc.

a. For bulk density determination, pour the slurry sample from one of the pint bottles into the gallon weight cup as rapidly as possible so as to minimize settling. If final increments of slurry are to be added to the gallon weight cup, recap the pint bottle and reagitate the sample before making the final addition.

b. For titrimetric analysis purposes, transfer the well mixed slurry directly from one of the pint bottles into a tall form titration beaker resting on the weighing pan of an analytical balance. In the case of viscous slurries of high solids content, some difficulty is usually experienced in weighing out sample weights within the minimum and maximum limits prescribed. In order to make additions in smaller increments than would normally result from a simple pouring operation, the following procedure is to be followed: Pour the greater part of the prescribed amount of well-mixed slurry directly from the polyethylene bottle into the 400 ml titration beaker; recap the polyethylene bottle and

reagitate the contents before pouring a small portion of the slurry into a 50 ml Pyrex beaker. The addition of final increments to the slurry already contained in the 400 ml titration beaker should be made from the material contained in this small 50 ml beaker. Swirl the contents of this small beaker prior to each withdrawal of a final increment. Accomplish the actual transfer of these small final increments through the use of a large bore polyethylene type eye dropper.

c. For the withdrawal of test portions of slurry to be used for the determination of loss on ignition, it may not be necessary to use the expedient of an eye dropper, etc. Usually, it will be found to be satisfactory to pour the well-mixed slurry sample directly from the polyethylene bottle into a platinum crucible resting on the weighing pan of an analytical balance.

2. Determination of Slurry Bulk Density at 77°F by Means of a "Weight per Gallon" Cup. Rapidly pour a portion of the well-mixed slurry from one of the polyethylene bottles into a previously tared "weight per gallon" cup of the type described in the list of necessary equipment. Fill the cup to within approximately 1/16 of an inch of the top rim. Figure 3. Tap the bottom of the filled cup against a hard surface in order to help remove any possible air bubbles or voids which may be present within the slurry portion contained in the cup. Then place the special cup top on the cup, gently pressing it down into place. Excess slurry should flow out the small center opening in the special top or lid. If this does not occur, then remove the top and add more well-mixed slurry from the pint bottle. After the top has been properly placed on the "weight per gallon" cup, carefully wipe off all of the excess slurry on the outside of the cup and lid and place the container on a suitable balance. Weigh the cup and its contents to the nearest 0.1 gram and subtract from this value the tare weight of the empty cup plus lid. The net weight in grams represents the weight of the 77°F slurry contained in the cup expressed in grams. This weight of slurry in grams divided by 10 shall be reported as value R and represents the bulk density of the slurry at 77°F expressed in pounds per U.S. gallon. The cup and special lid required for this determination are such that the "weight per gallon" cup will contain exactly 83.2 grams of distilled water when the cup, lid and water are at 77°F and the special lid of the cup has been placed on top of the cup and forced down to the extent of its travel. Calculate the bulk density of the slurry at 77°F as follows:

Bulk density of the slurry at 77°F. expressed in lbs./gal. (U.S.)	Wt. of slurry sample contained in "wt./gal." cup (expressed in grams) <hr style="width: 100%;"/> 10
---	--



Figure 3

3. Selection of the Appropriate Amount of Slurry Sample for Titrimetric Analysis. The portion of slurry selected for titrimetric analysis should be of such a quantity that it will contain the equivalent of 2.9 to 3.1 grams of dry lime. The previously determined value for the bulk density of the slurry at 77°F will, through the use of Figure Nos. 4A, 4B, and 4C, provide the analyst with a range of slurry sample weights within which any sample portion of slurry chosen for analysis would normally be expected to contain the prescribed equivalent dry lime content.

4. Selection of the Appropriate Amount of Slurry Sample for Determination of Loss on Ignition. The portion of slurry selected for this determination should be of such a quantity that it will contain the equivalent of 2.9 to 3.1 grams of dry lime. Use the bulk density value and Figure No. 4 to obtain an indication of the range of sample weights within which a sample of slurry might normally be expected to contain the prescribed equivalent quantity of dry lime.

5. Titrimetric Analysis of Various Alkaline Components of the Slurry Sample.

a. Titration to pH 8.3 with HCl. To the required weight of slurry sample contained in the tall form titration beaker, slowly add sufficient distilled water to bring the overall liquid level of the mixture to 150 ml. Titrate immediately with approximately 1.0 N hydrochloric acid to a prescribed end point of pH 8.3 on a

pH meter of the required type. Gentle stirring of the mixture to be titrated shall be accomplished through the use of a magnetic type stirrer. The first 50 ml of titrant may be added rather rapidly, after which continued additions of titrant should be made in a "rapid dropwise" fashion until the pH indication of 9 or slightly less is obtained. A suitable rate for this "rapid dropwise" addition has been found to be approximately 12 ml/min. After attaining a momentary pH indication of 9, or slightly less, proceed with the titration by adding additional titrant at the rate of approximately 2 ml/min until the pH is in the vicinity of pH 8.5, then add acid in four-drop increments to where the pH is 8.3 or less for approximately 35 seconds. Then add two-drop increments until the prescribed end point of 8.3 or slightly less for 60 seconds is reached. The prescribed end point for this portion of the titration shall be taken to be that point at which the addition of one two-drop increment of titrant produces a pH meter indication of 8.3, or of slightly less than 8.3, when observed exactly 60 seconds after the addition of titrant. The first occurrence of such an end point in the normal course of the titration shall be considered as the end point for this portion of the analysis. The volume of titrant required to reach this end point should be recorded before proceeding with the further addition of HCl.

b. Titration to "measured excess" of HCl. After the 8.3 end point has been reached, continue to add hydrochloric acid titrant to the sample contained in the beaker to a point where a total of approximately 100 ml of titrant has been added to the sample since the beginning of the titration. The sample mixture contained in the titration beaker at this point shall be considered as containing a "measured excess" of hydrochloric acid. Record the total volume of acid added to the sample since the beginning of the titration before proceeding with the back-titration.

c. Back-titration to pH 4.4 with NaOH. Titrate the sample mixture plus the "measured excess" of hydrochloric acid contained in the beaker with approximately 1.0 N sodium hydroxide to a pH meter indication of 4.4, or of slightly greater than 4.4, when observed exactly 10 seconds after a one-drop addition of titrant. The first occurrence of such an end point in the normal course of the back-titration shall be taken to be the prescribed end point for this portion of the analysis.

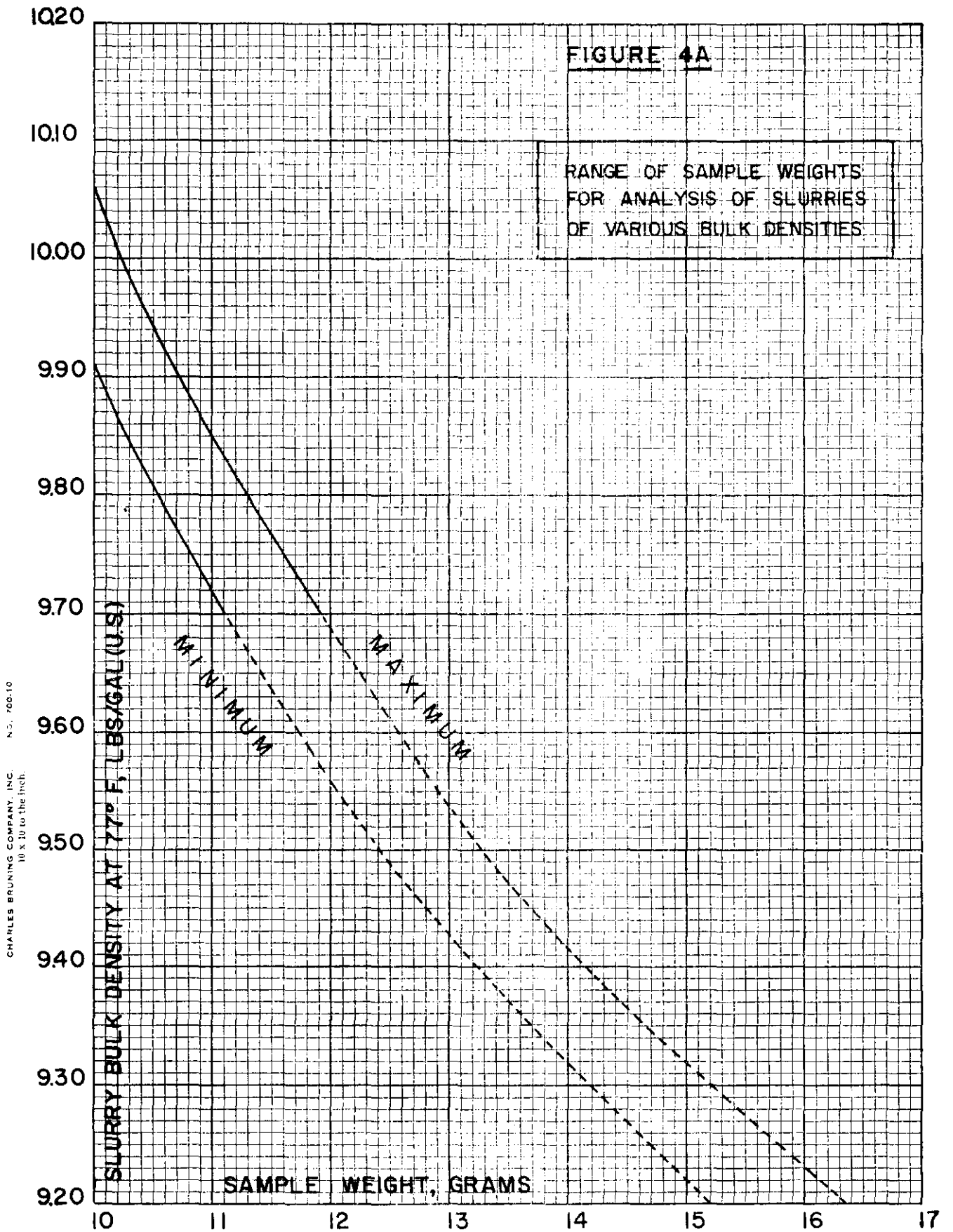
6. Determination of Percent Loss on Drying and Subsequent Ignition. Place the platinum crucible containing a carefully weighed appropriate amount of slurry into a 212°F drying oven for a period of some sixteen hours or until such time as the sample attains a state of apparent dryness. The initial weighing of the as received slurry should be made as rapidly as possible to the fourth decimal place. After the sample is brought to a state of apparent dryness at 212°F, it should be removed from the drying oven. Figure 9. Any contamina-

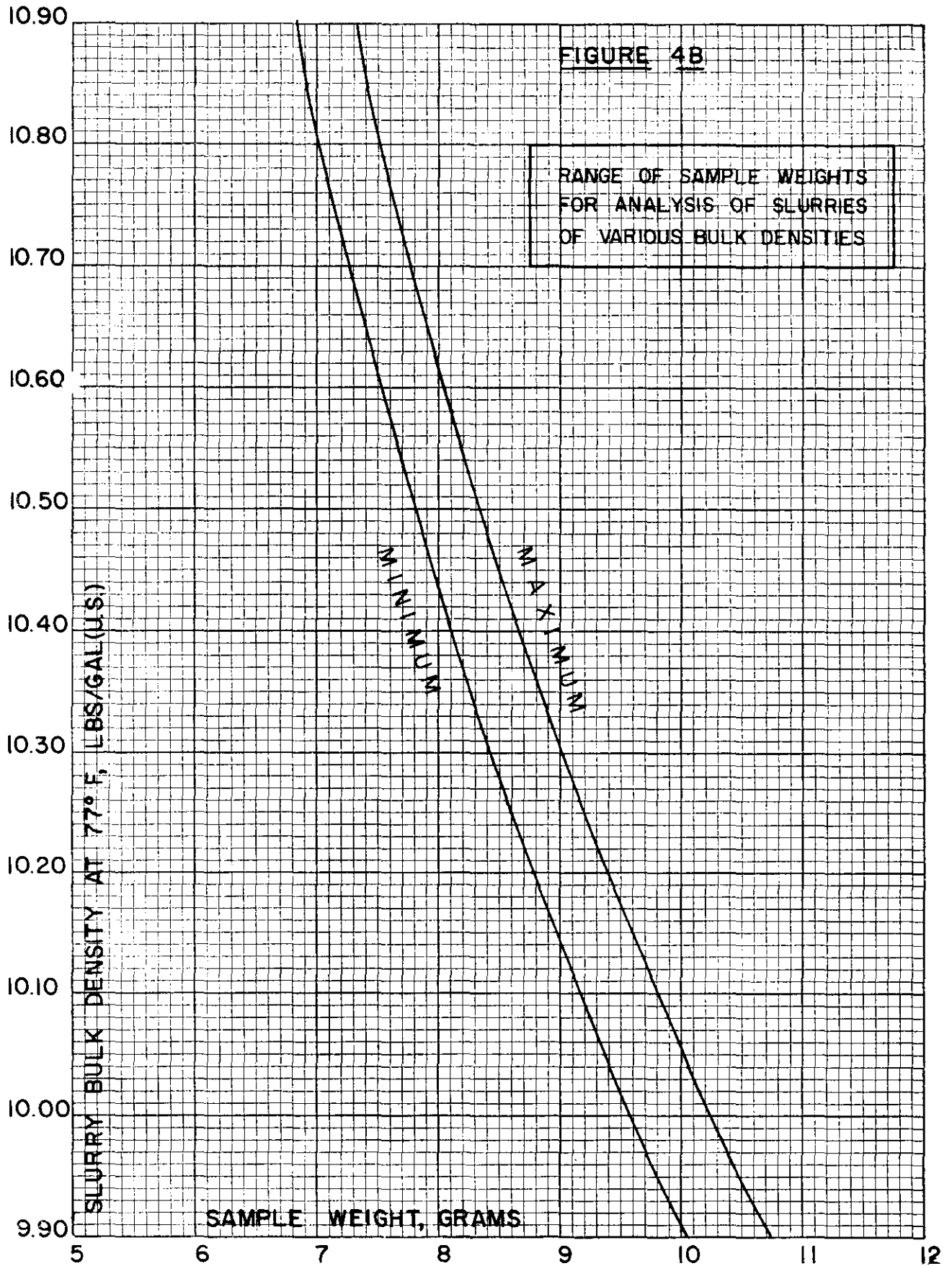
tion of sample due to exposure to carbon dioxide contained in the atmosphere may be neglected due to the subsequent forced disassociation of carbon dioxide during the period of loss on ignition at 2000°F. The crucible with unweighed lid and the dried sample portion which it contains should then be placed in an electric muffle furnace maintained at a temperature of 2000°F where it should be allowed to remain for a required ignition period of three hours. The crucible is to be fitted with a platinum lid during this ignition period so as to prevent loss of sample due to splattering. After the sample has been ignited in this fashion, it should be removed from the furnace and placed on a piece of hard-surfaced asbestos board such as "transite" for a period of 10 to 30 seconds prior to placing the crucible in a vacuum desiccator for further cooling. If several samples are being run simultaneously, it has been found expedient to remove all of the samples from the furnace, place them on the asbestos board, and then proceed to place them in the desiccator. After they have been placed in the vacuum desiccator, replace the desiccator lid with the vacuum stopcock in the open position. The heated air will expand through the opening for a period of several seconds after which the stopcock should be closed. This, in effect, has been found to cause a

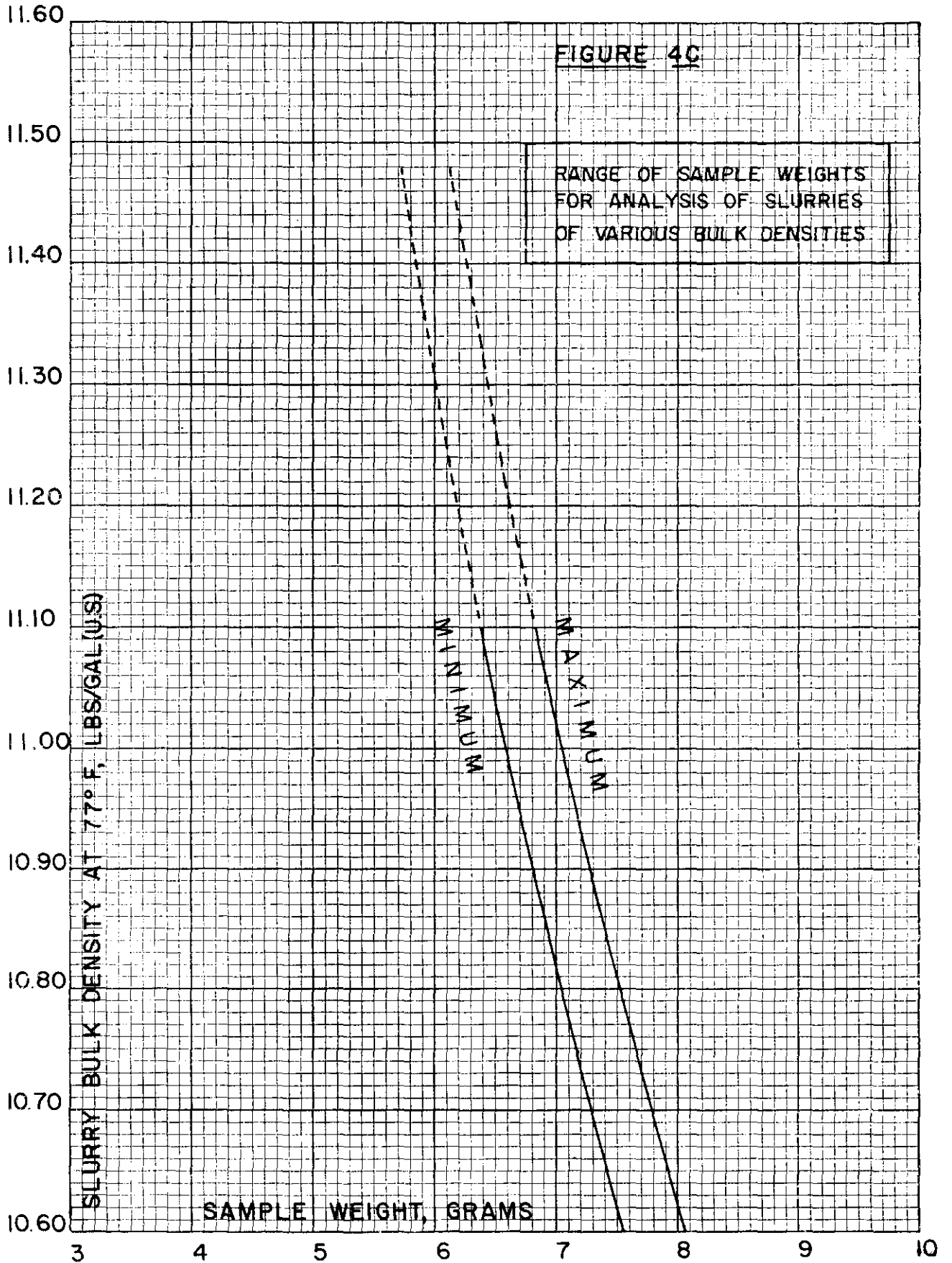
moderate vacuum without necessitating the use of an aspirator, safety bottle, etc. The residue from the loss on ignition should be allowed to cool for 30 minutes prior to weighing. Weigh the crucible and sample without lid. This weighing operation should take place promptly after the cooling period so as to avoid possible contamination of the highly reactive quicklime residue. The actual weighing of this residue to the fourth decimal place is to be accomplished as rapidly as possible so as to prevent hydration of the residue due to exposure to the atmosphere.

7. Screen Analysis. Pour approximately two liters of the well-mixed slurry contained in the two-gallon polyethylene bottle into a previously tared liter Pyrex glass beaker. Weigh this slurry portion to the nearest gram and then proceed to attempt to water wash it through each of the following screens in the order listed. First, screen the slurry on a No. 6 (3360 micron) sieve, and then on the No. 30 (590 micron) sieve. The wash water to be used for this purpose shall be applied in accordance with the wash water method and by the equipment described for the screening of dry lime samples.









State Department of Highways and Public Transportation  
Materials and Tests Division

APPENDIX I  
STANDARD CALCULATION METHOD

1. Definition of Terms

- A = Apparent hydrate alkalinity of the sample indicated by titration and calculated as % CaO.
- B = Carbonate alkalinity of the sample derived by titration and calculated as % CaO.
- B' = Carbonate alkalinity of the sample derived by titration and calculated as % CaCO<sub>3</sub>.
- C = Total alkalinity of the sample calculated as % CaO.
- D = Apparent hydrate alkalinity of the sample indicated by titration and expressed as % Ca(OH)<sub>2</sub>.
- E = Carbonate alkalinity of the sample derived by titration and calculated as % CO<sub>2</sub>.
- F = Loss on drying and subsequent ignition.
- G = "Total Water" of the sample calculated as % H<sub>2</sub>O.
- H = Apparent hydrate alkalinity of the sample indicated by the "total water" portion of the loss on drying and ignition and expressed as % Ca(OH)<sub>2</sub>.
- I = Corrected value for the hydrate alkalinity of the sample calculated as % Ca(OH)<sub>2</sub>.
- J = "Free Water" of the sample calculated as % H<sub>2</sub>O.
- K = Zero in the case of a slurry since the unhydrated lime content may be assumed to be nil.
- L = "Inert Matter" content of the sample calculated as % "Inert Matter."
- M = Screen analysis of the sample expressed as the % retained, by weight, of wt of total solids contained in the slurry portion screened on the No. 6 sieve.
- O = Screen analysis of the sample expressed as the % retained, by weight, of the wt. of the total solids contained in the slurry portion screened on the No. 30 sieve.

- P = Dry solids content of the slurry expressed as % by weight "solids content."
- Q = Hydrate alkalinity of an as received sample, calculated as % by weight of the solids content of the slurry as Ca(OH)<sub>2</sub>.
- R = Bulk density of the slurry at 77°F expressed in lb/gal (U.S.).
- S = Equivalent dry lime content of the slurry, expressed as lb of lime/gal (U.S.) at 77°F.
- T = Calcium hydroxide content of the slurry, expressed in lb/gal (U.S.) at 77°F.

Values obtained by analysis: A  
C  
F  
M  
O  
R

Values to be reported: M  
O  
P  
Q  
R  
S

Values obtained by calculations: B  
B'  
D  
E  
G  
H  
I  
J  
K  
L  
P  
Q  
S  
T

$$I = N \text{ of acid used} \times \frac{56.08}{2000} \times 100$$

Note: I = a factor derived by multiplying the normality of the acid used by the product of the molecular weight of calcium oxide divided by 2000 (to convert to milli-equivalents) multiplied by 100 (so as to express the final unit to

be calculated in terms of percent). Example: In a case where the normality of the acid being used is 1.0005 then the correct value for I =  $1.0005 \times 2.804 = 2.8054$ .

$$\text{II} = \text{N of base used} \times \frac{56.08}{2000} \times 100$$

$$\text{III} = \text{Ml of acid to pH of 8.3}$$

$$\text{IV} = (\text{I}) \times (\text{III})$$

$$\text{V} = \text{Total ml of acid used}$$

Note: This volume of acid should approximate 100 ml and represents that volume of acid used in the titration to reach a pH of 8.3 plus that volume of acid added to insure a "measured excess" before proceeding with the prescribed "back-titration."

$$\text{VI} = (\text{I}) \times (\text{V})$$

$$\text{VII} = \text{Ml of base used in the "back-titration" to proceed from the point of "measured excess" to the prescribed end point of a pH of 4.4.}$$

$$\text{VIII} = (\text{II}) \times (\text{VII})$$

$$\text{IX} = (\text{VI}) - (\text{VIII})$$

$$\text{X} = \text{Titration sample weight expressed in grams.}$$

$$\text{XI} = \text{Loss on drying and ignition slurry sample weight expressed in grams.}$$

$$\text{XII} = \text{Loss on drying and ignition residue sample weight expressed in grams.}$$

$$\text{XIII} = \text{Weight of the approximate 2 liter portion of slurry (weighed to the nearest 1 gram) screened on the No. 6 sieve.}$$

$$\text{XV} = \text{Weight of the approximate 2 liter portion of slurry (weighed to the nearest 1 gram) screened on the No. 30 sieve.}$$

$$\text{XVI} = \text{Weight of residue retained on the No. 6 sieve.}$$

$$\text{XVIII} = \text{Weight of residue retained on the No. 30 sieve.}$$

## 2. Basic Calculations and Remarks

A = Apparent hydrate alkalinity of the sample indicated by titration and calculated as % CaO

Calculations:

$$\% \text{CaO} = \frac{(\text{IV})}{(\text{X})}$$

Note: This equation is derived from the expression

$$\% \text{CaO} = \frac{\text{ml} \times \text{N} \times \text{meq wt} \times 100}{\text{sample wt}}$$

Where:

ml = net ml of acid to pH 8.3

N = N of acid used

meq wt = milli-equivalent weight of CaO

100 = factor for conversion to percent

sample wt = titration sample weight

B = Carbonate alkalinity of the sample derived by titration and calculated as % CaO

Calculations:

$$\% \text{CaO} = \text{C} - \text{A}$$

B' = Carbonate alkalinity of the sample derived by titration and calculated as % CaCO<sub>3</sub>

Calculations:

$$\% \text{CaCO}_3 = \text{B} \times (1.78479)$$

C = Total alkalinity of the sample calculated as % CaO

Calculations:

$$\% \text{CaO} = \frac{(\text{IX})}{(\text{X})}$$

Note: This equation is derived from the expression

$$\% \text{CaO} = \frac{\text{ml} \times \text{N} \times \text{meq wt} \times 100}{\text{sample wt}}$$

Where:

ml = net ml of acid to pH 4.4

N = N of acid used

meq wt = milli-equivalent weight of CaO

100 = factor for conversion to percent

sample wt = titration sample weight

D = Apparent hydrate alkalinity of the sample indicated by titration and expressed as % Ca(OH)<sub>2</sub>

Calculations:

$$\% \text{Ca(OH)}_2 = A \times (1.32126)$$

Note: 1.32126 is a gravimetric factor used to convert % CaO to % Ca(OH)<sub>2</sub>

E = Carbonate alkalinity of the sample derived by titration and calculated as % CO<sub>2</sub>

Calculations:

$$\% \text{CO}_2 = B \times (0.78479)$$

Note: 0.78479 is a gravimetric factor used to convert CaO to % CO<sub>2</sub>

F = Loss on drying and subsequent ignition of the sample, as % loss in weight and reported as % loss on ignition.

Calculations:

$$\% \text{ Loss on ignition} = \frac{(XI) - (XII)}{(XI)} \times 100$$

Note: This equation is derived from the expression:

% loss on ignition =

$$\frac{(\text{slurry sample wt}) - (\text{residue wt})}{(\text{sample wt})} \times 100$$

For the purpose of this test procedure, loss on ignition shall be taken to be the sum of the losses due to "water of dehydration," "free water," and chemically combined carbon dioxide.

"Water of dehydration" shall be defined as that portion of the loss on ignition due to the loss of water resulting from the forced dehydration or disassociation of calcium hydroxide at elevated temperatures.

"Free water" shall be defined as that portion of the loss on ignition which is not accounted for by the loss due to "water of dehydration" plus the loss due to chemically combined carbon dioxide. "Total water" shall be defined as that portion of the loss on ignition due to "water of dehydration" plus "free water."

G = "Total water" of the sample expressed as % H<sub>2</sub>O

Calculations:

$$\% \text{H}_2\text{O} = F - E$$

Note: This equation is derived from the assumption that when a slurry sample is dried in a 212°F oven for 16 hours, or longer, and then subjected to ignition at 2000°F for a period of some three hours, the principal losses to be encountered will be carbon dioxide and water. It is also to be assumed that any gain in weight due to oxidation of other impurities, such as sulfides to sulfates will be negligible. The basic assumption may be represented as follows:

"Total water" expressed as % H<sub>2</sub>O =

(loss on drying and subsequent ignition) - (carbonate alkalinity of the sample derived by titration, expressed as % CO<sub>2</sub>).

H = Apparent hydrate alkalinity of the sample indicated by the "total water" portion of the loss on drying and ignition and expressed as % Ca(OH)<sub>2</sub>.

Calculations:

$$\% \text{Ca(OH)}_2 = G \times (4.1128)$$

Note: 4.1128 is a gravimetric factor used to convert % H<sub>2</sub>O to % Ca(OH)<sub>2</sub>

I = Corrected value for the hydrate alkalinity of the sample calculated as % Ca(OH)<sub>2</sub>

Calculations:

$$\% \text{Ca(OH)}_2 = D$$

Note: This equation is derived from the assumption that in a lime slurry D < H and that D will not equal H. This assumption implies that "free water" is present in the slurry, unhydrated calcium oxide will not be present and that the definition of a slurry is such that the existence of a near perfect hydration exemplified by the condition wherein D = H will not exist. It is only a natural assumption, almost by definition of the term slurry itself, that a slurry will contain "free water" above and beyond the amount necessary to satisfy the chemical affinity of unhydrated calcium oxide in the hydration process. In instances in which this revised test procedure is used to arrive at a value for "hydrate alkalinity of an as received sample, calculated as % by

weight of the 'solids content' of the slurry, as  $\text{Ca}(\text{OH})_2$ ," then it is to be understood that it is to be the corrected value "I" which shall be used to calculate the reported value "Q" since this value "I" is the corrected value for the hydrate alkalinity of the sample.

J = "Free water" of the sample calculated as %  $\text{H}_2\text{O}$ .

Calculations:

$$\% \text{H}_2\text{O} = (\text{H} - \text{D}) \times (0.24314)$$

Note: 0.24314 is a gravimetric factor used to convert  $\text{Ca}(\text{OH})_2$  equivalents to  $\text{H}_2\text{O}$  equivalents.

K = Zero in the case of a slurry since the unhydrated lime content may be assumed to be nil.

L = "Inert matter" content of the sample calculated as % "inert matter."

Calculations:

$$\% \text{ "inert matter" } = 100 - (\text{C} + \text{F})$$

Note: For the purpose of this test procedure, "inert matter" shall be defined as those sample constituents which by their chemical nature do not react under the conditions prescribed in acid titration nor do they volatilize under the conditions prescribed for the loss on ignition determination. It shall be assumed to consist essentially of acid insolubles plus possible metallic oxides.

M = Screen analysis on the sample expressed as % retained, by weight, of the weight of total solids contained in the slurry portion screened on a No. 6 sieve (3360 microns).

Calculations:

$$M = \frac{(\text{XVI})}{(\text{XIV}) \times P} \times 100$$

O = Screen analysis of the sample expressed as the % retained, by weight, of the weight of total solids contained in the slurry portion screened on a No. 30 sieve (590 microns).

Calculations:

$$O = \frac{(\text{XVIII})}{(\text{XV}) \times P} \times 100$$

P = Dry solids content of the slurry expressed as a decimal, "solids content" by weight.

Calculations:

$$P = \frac{100 - J}{100}$$

Q = Hydrate alkalinity of an as-received sample, calculated as % by weight of the solids content of the slurry as  $\text{Ca}(\text{OH})_2$

$$Q = \frac{I}{P}$$

Note: This equation is derived from the expression:

$$\% \text{Ca}(\text{OH})_2 = \frac{\% \text{ by wt Ca}(\text{OH})_2 \text{ in the slurry}}{\text{dry solids content by wt (as a decimal)}}$$

Example: For a slurry which contains 30.60%  $\text{Ca}(\text{OH})_2$  and has a solids content by weight of 0.3169, the

$$\% \text{Ca}(\text{OH})_2 = \frac{30.60}{0.3169} = 96.6\%$$

R = Bulk density of the slurry at 77°F expressed in lb/gal (U.S.)

$$R = \frac{\text{wt in grams of slurry in the "wt/gal" cup at 77°F}}{10}$$

Note: This cup should contain exactly 83.2 grams of distilled water at 77°F with the lid properly fitted, etc.

S = Equivalent dry lime content of the slurry, expressed in lb of lime/gal (U.S.)

Calculations:

$$S = R \times P$$

Note: This equation is derived from the expression:

lb of lime/gal (U.S.) at 77°F = bulk density of slurry at 77°F in lb/gal (U.S.) multiplied by the dry solids content of the slurry as percent.

Example: For a slurry which has a bulk density at 77°F of 10.18 lb/gal (U.S.) and which contains 32.42% by weight dry solids, then the equivalent dry lime content of the slurry at 77°F = 10.18 lb/gal  $\times$  0.3242 = 3.30# lime/gal.

T = Calcium hydroxide content of the slurry expressed in lb/gal (U.S.) at 77°F.

$$T = S \times Q$$

Note: This equation is derived from the expression:

lb of 100% pure  $\text{Ca(OH)}_2$ /gal (U.S.) at 77°F = lb of lime/gal (U.S.) at 77°F multiplied by the % by weight of  $\text{Ca(OH)}_2$  to be found in the "solids" content of the slurry.

#### Summary of Calculations

$$A = \frac{(IV)}{(X)}$$

$$B = C - A$$

$$B' = B \times (1.78479)$$

$$C = \frac{(IX)}{(X)}$$

$$D = A \times (1.32126)$$

$$E = B \times (0.78479)$$

$$F = \frac{(XI) - (XII)}{(XI)} \times 100$$

$$G = F - E$$

$$H = G \times (4.1128)$$

$$I = D$$

Note: In a lime slurry we will assume that  $D < H$  and D will not equal H.

$$J = (H - D) \times (0.24314)$$

$$K = \text{zero}$$

Note: In a slurry we will assume the unhydrated lime content to be nil.

$$L = 100 - (C + F)$$

$$M = \frac{(XVI)}{(XIII) \times P} \times 100$$

$$O = \frac{(XVIII)}{(XV) \times P} \times 100$$

$$P = \frac{100 - J}{100}$$

$$Q = \frac{I}{P}$$

$$R = \frac{\text{Wt in grams of slurry in "wt/gal" cup at 77°F}}{10}$$

$$S = R \times P$$

$$T = S \times Q$$

#### Factors Used

$$\text{CaO} \times (1.32126) = \text{Ca(OH)}_2$$

$$\text{CaO} \times (1.78479) = \text{CaCO}_3$$

$$\text{CaO} \times (0.78479) = \text{CO}_2$$

$$\text{Ca(OH)}_2 \times (0.24314) = \text{H}_2\text{O}$$

$$\text{H}_2\text{O} \times (4.1128) = \text{Ca(OH)}_2$$





COMMERCIAL LIME SLURRY Page 2 PRODUCER <b>XCC</b>			TOTAL ALKALINITY	HYDRATE ALKA- LINITY	CARBO- NATE ALKA- LINITY	CaCO <sub>3</sub>	CO <sub>2</sub>	LOSS ON IGNITION SAMPLE WT.	LOSS ON IGNITION RESIDUE WT.	IGNITION SAMPLE WT. LOSS	% LOSS ON IGNITION	% INERT MATTER	TOTAL H <sub>2</sub> O	% Ca(OH) <sub>2</sub> by IGNITION	% Ca(OH) <sub>2</sub> by TITRATION	H <sub>2</sub> O as Ca(OH) <sub>2</sub>	CaO as Ca(OH) <sub>2</sub>	% Ca(OH) <sub>2</sub> CORRECTED VALUE	"Free water" as % H <sub>2</sub> O	% CaO
DATE SAMPLED	SEAL #	LAB #	AVERAGE	AVERAGE	C - A	B x (1.78479)	B x (0.78479)			(XI)-(XII)	$\frac{(XI)-(XII)}{(XI)} \times 100$	100 - (C+F)	F-E	Gx(4.1128)	Ax(1.32126)	H-D	D-H	LESSER OF D or H	(H-D)x (0.24314)	(D-H)x (0.75686)
C	A	B	B'	E	XI	XII	F	L	G	H	D	I	J	K						

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## State Department of Highways and Public Transportation

## Materials and Tests Division

## LIME TESTING PROCEDURE

## PART IV

## TESTING OF QUICKLIME, TYPE C

## Scope

This test procedure is intended to serve as a reasonably accurate, fairly rapid means of testing high calcium, crushed or pebble quicklime on a routine basis.

**CAUTION:** Quicklime is extremely hazardous and capable of inflicting severe caustic burns to skin, lung damage and/or eye injury. It can possibly cause blindness if handled improperly. Personnel handling, sampling or testing and working around quicklime should wear proper protective clothing, respirators and dust-proof goggles to prevent injury.

## Apparatus

1. Analytical balance, suitable for rapid weighing, with a minimum weighing capacity of 100 grams which meets the requirements of Test Method Tex-901-K, Class I-B.

2. Top loading balance, suitable for rapid weighing, with a minimum weighing capacity of 2000 grams which meets the requirements of Test Method Tex-901-K, Class II-E

3. Mill, laboratory model with fine grinding plates and a smooth feed worm, Straub Co. Model 4-E or equivalent.

4. Mechanical shaker with timer, to accommodate 8 inch diameter sieves.

5. Mixer, Hobart Brand, Model N-50 with mixing bowl and special lid, approximately one-gallon capacity.

6. Wire whip, Hobart Brand, Type D.

7. Oven, mechanical convection type 212  $\pm$  2°F.

8. Sample splitter, alternate slot type with 1-1/4 inch slots, six slots in each direction, slots measure 8-1/2 inches in length. Splitter frame size: 16"  $\times$  8-1/2". Splitter to be provided with two catch pans of appropriate size, plus one pour pan of 16 inch length.

9. Sieves. A set of standard U.S. sieves containing the following sizes: 1", 3/4", 3/8", 1/4", No. 6, No. 20, No. 30 and No. 100 which meets the requirements of Test Method Tex 907-K. All sieves are to be of 8 inch diameter. Sieve set to be complete with one lid and one catch pan.

10. Magnetic type stirrer with a 1 1/2 inch diameter round teflon covered stirring bar 5/16 inch in thickness.

11. Hot plate.

12. Burette, Class A, 50 ml capacity.

13. Stopwatch or timing device to register up to 30 minutes.

14. Stainless steel weighing boat.

15. Spatula.

16. Balance brush, camel's hair type.

17. Plastic sample bags approximately 4 mils in thickness, size 12"  $\times$  18", with tie closures.

18. Flasks, Erlenmeyer, 500 ml capacity.

19. Stoppers, rubber, No. 10 size, no hole.

20. Cans, ointment type with lids, 6 oz capacity.

21. Tape, electrical, PVC type.

22. Plastic tubing, 3/8 inch ID, 1/8 inch wall.

23. Zero to 30 psi water pressure gauge, 3 inch diameter face, with 3/8" IPS female thread fitting.

## Solutions

1. Sugar solution, freshly prepared. A 600 ml batch may be prepared as follows: Dissolve 20 grams of granulated cane sugar to every 40 ml of deionized or distilled water. Add two drops of phenolphthalein indicator to the entire batch and neutralize with 1.0 N

Sodium Hydroxide added drop-wise until a faint pink coloration is obtained. This solution should not be used if over two days have lapsed since preparation. Show time and date of preparation on solution bottle.

2. Phenolphthalein Indicator Solution prepared by dissolving 0.1 gram of powdered phenolphthalein, ACS Grade, in 50 ml of absolute ethyl alcohol and adding 50 ml of deionized or distilled water.

3. Deionized or distilled water.

4. Acid, hydrochloric, approximately 1.0 Normal, with actual normality determined to be 1.0000 Normal, plus or minus 0.0005 Normal when the normality of the solution is accurately determined by direct comparison with a 1.0 N sodium hydroxide solution prepared and standardized in accordance with Part II of Test Method Tex-600-J.

#### Procedure

1. Sample Preparation. Take a representative one-gallon portion of the quicklime to be tested and divide it into two representative portions through the use of the described sample splitter. One portion is to be used for the dry screen tests, the other portion is ground to completely pass a No. 30 sieve. The ground sample is to be used for chemical analysis. The dry screened portion is to be reserved for the wet screen analysis. All portions are to be suitably protected from contamination pending analysis. The use of plastic bags with tie closures is recommended for this purpose. Smaller portions reserved for analysis shall be removed from the plastic bags and stored in taped 6 oz capacity ointment cans. All grinding should be accomplished within a 15-minute time interval so as to minimize sample contamination by moisture from the air. Samples found to contain flint or other extremely hard materials should be crushed to pass a 1/4-inch sieve in a jaw type crusher prior to grinding.

2. Chemical Analysis. Accurately weigh out a representative  $2.8040 \pm 0.0050$  gram portion of the material which was ground to pass the 30 mesh sieve. Place this titration sample in a 500 ml Erlenmeyer flask which contains 10 ml of freshly-boiled deionized or distilled water which has been previously cooled to room temperature.

Stopper the flask with a No. 10 rubber stopper pending addition of 50 ml of boiling deionized or distilled water. Swirl the contents for a few seconds and then heat the contents of the flask to boiling for a timed interval of two minutes. Allow the flask and contents to cool to room temperature. Add 150 ml of the freshly prepared sugar solution to the flask, stopper once again and let stand for 15 minutes plus or minus 5 minutes with swirling at 5-minute intervals. After this reaction period, add 5 drops of phenolphthalein indicator solution and titrate the sample with the 1.0000 N Hydrochloric acid until the first pink to clear color is observed.

3. Calculations for Titration. The amount of acid used is taken to be the percent by weight of "Active CaO" contained in the sample and it is to be reported to the nearest 0.1 percent.

4. Dry Sieve Analysis. Take the representative half-gallon portion of the original sample which has been reserved for the dry sieve analysis and pass the entire portion in 500 g increments across a nest of sieves of the designated sizes. The following 8 inch diameter sieves shall be nested top to bottom in this order: 3/4", 3/8", 1/4", No. 6, No. 30 and No. 100. Please note that the 3/8", 1/4", No. 6 and No. 30 sieves have been added to the nest of sieves to prevent undue loading on any particular sieve. Since the shaker will accommodate only six sieves plus lid and catch pan, each portion to be sieved is manually passed across the one-inch sieve, which is not placed on the shaker. The entire 1/2 gallon of material is added to the sieve nest in approximate 500 gram portions, each of which is agitated on a mechanical sieve shaker for exactly three minutes, after which the amounts caught on each sieve are transferred into marked containers and later the fractions retained on a given sieve from each successive three minute shaking of a 500 gram portion are combined. This sieving operation is performed in stages so as to minimize undue loading and attrition within the three minute shaking period. At the conclusion of the dry sieving test, the half-gallon portion of the original sample used in the test is now contained in separate marked containers. A proportional part of the contents of each container is recombined so as to form the 800-gram sample needed for the wet sieve analysis. The recombined sample must be stored in a sealed container prior to sieving.

#### Calculations for dry sieve analysis:

$$\% \text{ by wt quicklime retained on sieve} = \frac{\text{wt in grams of quicklime retained on sieve}}{\text{wt in grams of } 1/2 \text{ gallon portion of quicklime being dry sieved}} \times 100$$

Sieve residues are reported on a cumulative basis as a percentage of the original sample used for the dry sieve analysis. Dry sieve analysis values shall be rounded off and reported to the nearest whole percent.

5. Wet Sieve Analysis. The proportionally recombined sample from the dry sieve analysis shall weigh 800 grams plus or minus one gram and is to serve as the test sample for this wet sieve analysis. Place this wet sieve analysis sample into the approximately one-gallon capacity stainless steel mixing bowl on the mixer. Add 1700 ml of tap water at a temperature of  $77 \pm 5^\circ\text{F}$  and cover immediately with a stainless steel lid of 10-3/4 inch diameter. The stainless steel lid is to have a 1"  $\times$  5-3/4" slot cut from the center to the edge of the lid. This slot allows for the mixer wire whip to be beneath the lid, but still be attached to the mixer. A 10-3/4 inch diameter lid of 3/32 inch or 1/4 inch thick rubber may be used atop the stainless steel lid to help

hold it down. The rubber weight or lid is also fitted with a 1" x 5-3/4" slot so as to allow for the whip head. When the metal and the rubber lid are rotated in relation to each other, the top of the bowl is effectively closed except for the area immediately adjacent to the whip head.

The quicklime sample and water is allowed to react for 10 minutes in the bowl with the wire whip in place and the lid in place, but without stirring.

After this initial 10 minute reaction period, remove the lid, mix the reacted material by turning on the mixer to a low speed setting which corresponds to a "no-load" setting of 140 rpm. In use, as loaded, this mixer will be mixing at a considerably slower speed than the 140 rpm "no-load" rating for the low speed setting.

CAUTION: The quicklime sample, upon addition of water, generates large amounts of heat and steam and due caution should be exercised.

Mix the hydrating sample for two minutes on the low speed setting, utilizing the wire whip agitator. Turn off the mixer and allow the reaction to proceed for an additional 18 minutes without supplying additional agitation. After this prescribed reaction time, turn on the mixer at the same low speed for one additional minute of agitation. Then, rinse all the slurry from the whip agitator, from the mixing bowl and lid and wash the reacted sample with water onto a stack of the following sieves nested top to bottom as follows: No. 6, No. 20 and No. 30. The wash water for this purpose may be ob-

tained from a cold water tap at an ambient temperature of between 60 to 80°F, through a 3/8 inch ID plastic tubing fitted with valve and water pressure gauge. Be sure that none of the slurry sample is lost over the sides of the sieves. Wash at a water pressure of 14 to 18 psi through the 3/8" ID plastic tubing. The end of the hose may be pinched somewhat if necessary to effect a more efficient washing operation. The sieving and wet washing of the sample should continue for a total time interval of 30 minutes from beginning of wet washing or until the wash water is clear, whichever occurs first. The No. 20 sieve in the middle position is used to prevent excessive loading on the No. 30 sieve in the bottom position.

At the completion of the prescribed wet wash, place the stack of sieves into a pan to catch the drippings and dry the residues and catch pan in the prescribed 212°F oven for a period of no less than one hour nor more than two hours.

After drying, cool the residue to room temperature and weigh the respective sieve residues on the top loading balance to the nearest 0.1 gram. Sieve residues are reported on a cumulative basis as a percentage of the original sample used for the wet sieve analysis.

Calculations for wet sieve analysis:

$$\% \text{ by wt of residue after slaking} = \frac{\text{residue retained on sieve in grams}}{\text{wt in grams of 1/2 gallon portion of quicklime being wet sieved}} \times 100$$

Wet sieve analysis values shall be rounded off and reported to the nearest whole percent.

State Department of Highways and Public Transportation  
Materials and Tests Division

TESTING EPOXY MATERIALS

Scope

This method covers the various test procedures for epoxy materials specified under the State Department of Highways and Public Transportation Departmental Material Specification D-9-6100. The test to be performed will depend upon the requirements set forth for each particular material. This method includes the following test procedures:

- A. Consistency
- B. Pot life
- C. Set time
- D. Thixotropy
- E. Adhesive shear strength
- F. Water gain
- G. Impact resistance
- H. Wet strength
- I. Grind
- J. Bonding of fresh portland cement concrete to cured portland cement concrete

The weight per gallon of each component shall be determined in accordance with ASTM D 1475, Density of Paint, Varnish, Lacquer, and Related Products. For all tests performed on the mixed epoxy, the proper weight ratio of resin and hardener components shall be determined based on the weight per gallon and the specified volume ratio, and the components proportioned by weight for all these tests.

A. Consistency

Apparatus

1. Synchro-Lectric viscometer of suitable range.
2. One pint or one quart can, friction top.
3. Thermometer, range 66 to 80°F, 0.2 division (ASTM 17 F).
4. Spatula, 3/4" wide x 4-1/2" blade.
5. A balance with a minimum capacity of 2000 grams which meets the requirements of Test Method Tex-901-K, Class II-E.
6. Stopwatch.

Procedure

The sample shall be placed in a container of such size that at least one inch of clearance is provided between the bottom and sides of the spindle and the

container when the spindle is immersed to the proper depth in the liquid. The temperature of the samples and the ambient temperature for all viscosity determinations shall be  $77 \pm 1^\circ\text{F}$ .

When making a viscosity determination, the proper spindle shall be attached to the lower end of the motor shaft. With disc type spindles, the spindle is first immersed in the liquid at an angle to eliminate air bubbles, then screwed onto the shaft. Adjust the height to bring the liquid level to the indentation in the spindle. Level the instrument, set the speed control at 20 rpm and start the motor. Allow the spindle to rotate for approximately two minutes before taking a reading. The scale reading obtained shall be converted to viscosity in poises by multiplying by the factor supplied by Brookfield for the combination of spindle and speed used. The spindle used shall be one which will result in a scale reading between 20 and 80.

For those specifications requiring viscosity determination on the mixed epoxy, an amount of resin and hardener component to result in a total quantity of 400 grams shall be weighed into a pint can from which the friction lip has been removed. The temperature of the components shall be such that the temperature after mixing will be  $77 \pm 1^\circ\text{F}$ . An initial temperature of  $76^\circ\text{F}$  is recommended. The properly proportioned components shall be mixed with a spatula for two minutes, the temperature checked, and then the proper spindle inserted and the test begun as indicated above. The viscosity reading shall be taken five minutes after initiation of mixing.

B. Pot Life

Apparatus

1. Thermometer, range 66 to 80°F, 0.2 division (ASTM 17 F).
2. Metal ointment can, 6 oz size.
3. One pint can with friction lip removed (for Type X epoxy only).
4. Metal spatula, approximate size: 3/4" wide x 4-1/2" blade.
5. Wooden block, minimum thickness of one inch.
6. Glass stirring rod.

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Procedure

The initial temperature of adhesive components and the ambient temperature shall be  $77 \pm 2^\circ\text{F}$  for this test. A total of 100 grams of the adhesive shall be weighed into a 6-ounce metal ointment can, the time recorded and the two components mixed for three minutes with a metal spatula. The sides and bottom of the can should be scraped periodically during the mixing. For Types I, I-M, II, II-M, II-MA, III, III-M, and IX epoxies, the following procedure shall be used:

The can shall be set on a wooden block and probed every minute with a glass stirring rod, starting five minutes from the initiation of mixing.

For Type IV, IV-M, V, VI, VII and VIII epoxies, the can shall be set on a wooden block and probed every two minutes with a glass stirring rod, starting 16 minutes from the initiation of mixing.

Procedure for Type X Epoxy

Samples of each of the coating components shall be brought to  $77 \pm 2^\circ\text{F}$ . Then 206 grams of the resin component and 100 grams of the hardener component shall be weighed into a pint can. The time shall be recorded and the two components mixed for five minutes, taking care to periodically scrape the walls and bottom of the can. The can shall then be set on a wooden block and probed every five minutes with a glass stirring rod, starting 40 minutes from the initiation of mixing. The time at which gelled material first forms in the container is recorded as the pot life.

C. Set Time

Apparatus

1. Metal spatula, approximate size: 3/4" wide x 4-1/2" blade.
2. Metal ointment can, 6 ounce.
3. Mortar briquettes.
4. Diamond-tooth saw.
5. Cold cabinet or room, capable of maintaining  $40 \pm 2^\circ\text{F}$ .
6. Riehle briquette tester, Tex-317-D.

Procedure

For the set time at  $77^\circ\text{F}$ , the ambient temperature and the initial temperature of the materials used in this test shall be  $77 \pm 2^\circ\text{F}$ .

Mortar briquettes shall be prepared according to Tex-317-D, Test for Relative Mortar Strength of Fine Aggregate for Concrete. The acid insoluble content of the fine aggregate used shall be not less than 95 percent by weight when tested in accordance with Tex-612-J.

The briquettes shall be cured for a minimum of seven days and then sawed at the center line perpendicular to the long axis. A diamond-tooth saw or other cutting tool capable of producing clean, smooth faces on the briquette halves shall be used. After cutting, the halves shall be washed with tap water and stored with cut faces exposed for a minimum of 72 hours at 70 to  $80^\circ\text{F}$  and  $50 \pm 15$  percent relative humidity. Sandblast the sawed faces as described under Tensile Shear Strength Procedure. The bonding faces shall be washed with methyl ethyl ketone and allowed to dry before use. Approximately 50 grams of the adhesive shall be mixed with a metal spatula in a 6-ounce ointment can for three minutes. Immediately coat the faces of the briquettes with the adhesive by placing a small amount on each face, then spread the material uniformly and place the faces together with light pressure. The excess adhesive shall be removed from the edges of the bonded area and the briquettes allowed to remain undisturbed until time for testing. No more than 10 minutes shall elapse during preparation of the specimens. A minimum of three briquettes shall be prepared. The amount of time allowed to elapse between initiation of mixing and testing shall be as follows:

Types:	I, I-M.....	40 minutes
	II, II-M, II-MA.....	2 hours
	III, III-M.....	4 hours
	IV,IV-M,V,VI,VII and VIII..	12 hours

The briquettes shall be subjected to tensile loading with the Riehle briquette tester and the load at failure recorded.

Set time at  $40^\circ\text{F}$ . The procedure is the same as specified under Set Time at  $77^\circ\text{F}$ , except that the initial temperature of the adhesive components and the briquette halves and the curing temperature shall be  $40 \pm 2^\circ\text{F}$ . The amount of time allowed to elapse between initiation of mixing and testing shall be three hours.

D. Thixotropy

Types I through IV Epoxy

Apparatus

1. Forced-draft oven, capable of maintaining  $120 \pm 2^\circ\text{F}$ .
2. Smooth, clean metal plate 3" x 6", approximately one-tenth inch thick.