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INVESTIGATION OF FAILING CONCRETE IN HOUSTON, TEXAS, CAUSED BY UNSOUND CEMENT



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5-20-72-057

TEXAS HIGHWAY DEPARTMENT

INVESTIGATION OF FAILING CONCRETE IN HOUSTON, TEXAS,

CAUSED BY UNSOUND CEMENT

by

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 Materials and Tests Division Texas Highway Department

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PREFACE

New designs and increasing numbers of structures requiring portland cement concrete, coupled with the increasing practice of using cement stabilized soils and base in construction programs, are reflected in the consumption trends of over 4 million barrels of cement annually by the Texas Highway Department for the past 10 years. It stands to reason that with greater demands placed on the use of portland cement, the greater the importance of quality control over its manufacturing and specification requirements. The sampling and testing of portland cement in accordance with standard specifications are the responsibility of the Materials and Tests Division. In light of rigorous requirements and the demands of a competitive market, only on rare occasions have portland cement samples destined for Texas Highway projects failed to meet the soundness requirement as reflected by the routine autoclave test. This report is the result of studies made on samples of unsound cement and the failing concrete which resulted from its use not only for the benefit of concerned departmental personnel but to serve as an unprecedented documentation of an engineering problem heretofore unobserved on such a scale involving Texas Highway projects.

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ABSTRACT

Physical, chemical and petrographic studies, together with on-site inspections, have been utilized in delineating the conditions present in structural concrete which has failed as a result of unsound cement. Concrete samples representing three structures at two separate highway projects in Houston, Texas, had irreparable damage and evidence of internal distress soon after being placed. Portland cement, routinely sampled from shipments supplied to these projects and submitted to the Materials and Tests Division for physical testing, failed to meet the soundness requirements. A total of seven samples, all produced at Trinity Portland Cement Company's Houston Plant during April and May of 1972, exhibited expansion which ranged from 2.5 to 20 percent by the autoclave test. Chemical analysis showed that these unsound cements contained excessive amounts of uncombined calcium oxide. Additional testing demonstrated that the relative unsoundness of the cement decreased with aeration; however, briquettes cast with the cement which showed the highest range of expansion were found to be unsound by the autoclave and steam-bath tests even after 5 months of open-air storage. Petrographic studies on 4-month-old concrete found no free lime present; however, evidences of unaccommodative chemical reactions were observed along with an extensive system of microcracks in the paste.

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I. SUBJECT

Several job samples of portland cement, produced at the Trinity Portland Cement Company Houston plant and submitted during April and May 1972 to the Materials and Tests Division Laboratories for routine physical tests, failed to meet the soundness test as outlined in ASTM Designation: C 150. In addition, failing concrete made with this unsound cement has been removed from highway projects located in the Houston area. The formal documentation of the studies made on the cement and samples of the concrete removed from structures is the subject of this report.

II. PURPOSE

The purpose of this report is to make available to departmental personnel the results of a series of laboratory studies, which include physical, chemical and optical as well as field observations, made on unsound cement and associated failing concrete from Texas Highway projects in Houston.

III. CONCLUSIONS

- The extensively-cracked concrete observed on three structures at two separate highway projects in Houston failed as a result of using unsound portland cement.
- 2. A total of seven cement samples produced at Trinity Portland Cement Company's Houston plant contained uncombined calcium oxide and exhibited autoclave expansion results ranging from 2.5 to 20 percent.

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3. Petrographic studies on the concrete removed from the structures identified extensive systems of microcracks in the paste fraction as well as exudations of secondary mineral compounds within the internal void system; however, traces of free calcium oxide could not be determined in the concrete by conventional optical methods.

IV. MATERIALS AND METHODS

A. Location, Field Observations and Samples.

The two construction sites from which concrete was removed and examined for this report are located within the city of Houston as indicated by the general highway map in Figure 1.

One site, a District 12 project, located in the western part of Houston on US 59 (C-27-13-65, PD0028) consisted of a foundation for a retaining wall situated at an approach to the West Belfort Overpass. The other site, a Houston Urban project (I-610-7(189) 782, 271-15-8) in the eastern part of the city consists of a center slab of a 3-span continuous unit on structure 351, ramp "A," at the multilevel interchange on Loop 610 and SH 225. Concrete samples taken from a pier on structure 346, ramp "C," of the same interchange were also collected for this study.

Field observations at the project sites were conducted by District 12 and Houston Urban personnel. Photographs and pertinent data, along with concrete samples removed from the retaining wall foundation and both cores and large pieces from the bridge deck, were

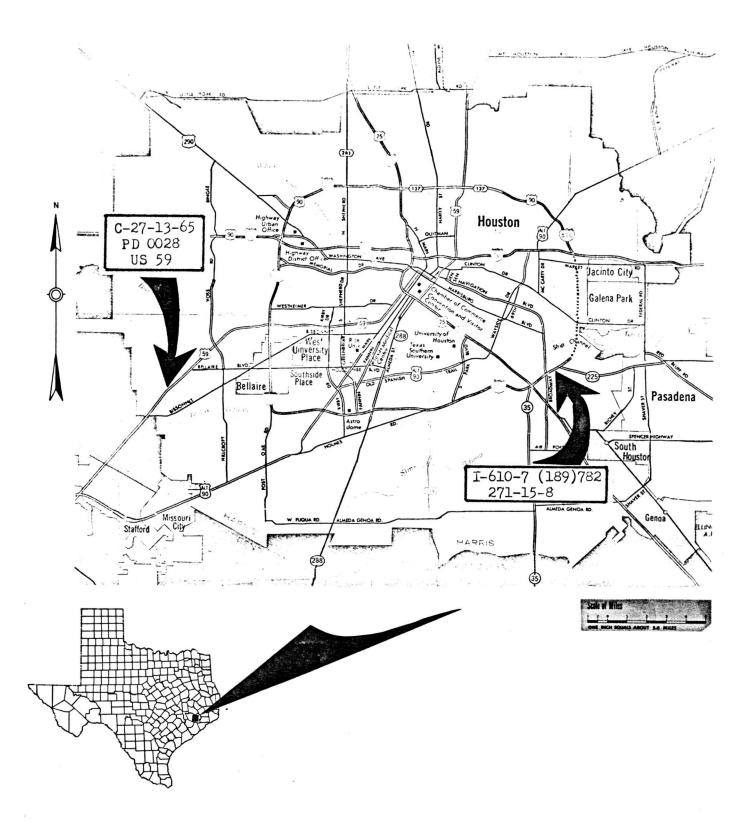


Fig. 1 General highway map of Houston showing location of structures examined for this investigation.

submitted to the Materials and Tests Division for examination. Results of compression tests conducted by a commercial testing laboratory on cores taken from the bridge deck and on test cylinders, in addition to the Concrete Design Worksheet that was used in the batching operation, have been included in Appendix I.

Portland cement samples taken according to standard procedures by departmental personnel and submitted for routine physical tests have been retained after testing by the Materials and Tests Division for extended physical testing, chemical analyses and petrographic studies. The cement samples taken from transport trucks by inspectors and shipped in 1-gallon buckets are identified under Laboratory Numbers 72-2321-D, 72-2349-D, 72-2420-D, 72-2423-D, 72-3137-D and 72-3603-D, all of which are Type I, and 72-2816-D, a Type III. Each were produced at Trinity Division of General Portland Cement's Houston plant during April 1972.

B. Chemical Analyses.

When routine physical tests indicated that a cement sample from Trinity-Houston was failing to meet specification requirements for soundness, a representative amount was submitted to the Materials and Tests Division Chemical Section for chemical analyses. Sample 72-2321-D, which exhibited significant expansion in the Autoclave Test, and a "normal" sample from the same producer were chemically analyzed for the free lime content as outlined in ASTM Designation: C 114, "Chemical Analysis of Hydraulic Cement." In addition, the

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percent loss on ignition (LOI) and the percent magnesium oxide were determined; the first by the ASTM method, the latter by a Materials and Tests Chemical Section procedure (See Appendix II). The sulfur trioxide (SO₃) content of the unsound cement was determined by the Materials and Tests Cement Section by means of a Wagner turbidimeter (ASTM C 114). For comparative studies a duplicate sample was chemically analyzed at the Trinity's Fort Worth laboratory by means of X-ray methods.

C. Physical Tests.

A number of routine physical tests are performed on all cement samples submitted to the Materials and Tests Division and, if a sample shows abnormal results, additional tests are often applied.

One of the basic routine tests performed on the subject cement samples, which are required on all cements proposed for use in concrete pavements or structures; is outlined in ASTM Designation: C 190, "Tensile Strength of Hydraulic Cement Mortars." This test employs the briquette specimen cast with the standard Ottawa sand.

Another routine test method utilized for this report is the "Fineness of Portland Cement by the Turbidimeter," ASTM Designation: C 115. This standard method covers the determination of the grind size as represented by a calculated measure of specific surface, expressed in square centimeters of total surface area per gram of cement, using the Wagner turbidimeter.

The third, and no doubt the most significant, physical test applied

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for this study, provides an index of potentially delayed expansion caused by the hydration of calcium oxide (CaO) and/or magnesium oxide (MgO). This method, outlined in ASTM Designation: C 151, "Autoclave Expansion of Portland Cement," involves subjecting a neat-cement prism to steam pressure at 295 psi (about 420°F) for 3 hours. The prisms are cast and placed in moist chambers for one day, then taken out and measured, after which they are placed in the autoclave and subjected to the test. After initial cooling, the pressure was released, the specimens were removed and placed in 194°F water, cooled gradually to 74°F, then dryed and measured. Calculations were determined and expressed as a percentage of length-change.

Two additional tests on the Trinity-Nouston cement were used for informational purposes; namely, the pat-boiler test and aeration. The former is actually a discontinued (Since 1947) ASTM Standard Method, "Soundness of Hydraulic Cement Over Boiling Water," ASTM Designation: C 189-44, and the latter is simply a technique utilized to gain information on relative hydration rates of cement samples containing unhydrated components. This observational test involved allowing the raw cement to be exposed to the air for various lengths of time and repeating some of the above listed tests and noting any differences in results.

D. Petrographic Studies.

A number of petrographic techniques and methods were utilized in

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examining the samples obtained for this investigation and each employed the use of a binocular stereoscopic microscope and/or a polarizing microscope.

Representative pieces of concrete from each examined structure were sliced, ground to a smooth finish and examined under a microscope by the linear traverse technique as outlined in ASTM Designation: C 457, "Microscopical Determination of Air-Void Content, Specific Surface and Spacing Factor of the Air-Void System in Hardened Concrete." During this examination, a survey of microcracks in the paste was taken in addition to noting the presence of bleed channels and secondary mineral deposits within the air voids.

Highly-polished sections processed from the concrete samples were examined at magnification ranges up to 1000X in order to determine the relative extent of cement hydration. The general condition of the aggregate-paste bond and other physical features of the cement paste such as hardness, granularity, porosity and color were observed from freshly broken pieces.

Powder mounts of the cement using immersion oils of different refractive indexes were devised for making additional observations with polarized light under high magnification. The cement was also examined by means of thin-sections of the concrete (ground to less than 25 microns), however, this technique was found to be inferior

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to the polished-section method of preparing sliced pieces of the concrete. The latter method included polishing the smoothed surface to a mirror finish for noting the composition, fineness, extent of hydration and the dispersement of relic cement particles. Transmitted light was utilized for examining thin-sections and oilimmersion slides; whereas, reflected light was necessary to examine the polished sections.

Additional powder mounts of the unsound cement were subjected to a "quickie-test" treatment as an additional optical verification of free lime. The test, described by Lea (1956)*, consists of placing a small amount of the cement on a glass microscope slide and adding a drop or two of White's reagent; a solution of 5 g of phenol in 5 cc of nitrobenzene to which two drops of water are added. After stirring, a cover-glass is pressed lightly onto the mixture. The slide is then examined under crossed polarized light at a magnification of about 125X. If free lime is present in the cement, long needles of calcium phenoxide form within a few minutes.

V. RESULTS

The condition of the deck surface and cores examined for this investigation, documented by personnel from the Houston Urban Office, can best be seen in the photographs taken during their on-site inspections

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^{*} Lea, F.M. "The Chemistry of Cement and Concrete," Edward Arnold Publishers LTD., London (Revised Edition). 637 pp., 1956.

during May 1972. The photos, shown as Figures 2-7, illustrate the distress cracks, which occurred in the unstable concrete as a result of the incorporated unsound cement, as well as show the condition of the cores removed from the deck. As can be seen, the crack patterns occur in localized areas a few feet across and sometimes show lineation when influenced by the underlying reinforcement.

Photographs of the affected retaining wall foundations at the West Belfast Overpass and US 59, taken by District 12 personnel, are shown as Figures 8-13. In addition to showing the cracked and spalled condition of the concrete, Figures 9, 10 and 12 show that a weak aggregatepaste bond is prevalent. Two of the 24' long sections cast with the unsound cement were removed soon after these photos were taken.

Photographs were not taken of the failing column at the I-610 interchange; however, visible cracks were observed when the samples were received in the Materials and Tests Laboratory. Details of the microscopic studies are included in the petrographic analysis.

Results of partial chemical analyses obtained on the initial cement sample (Lab #72-2321-D) which showed to be unsound by the autoclave expansion test are summarized in the following Table I. For comparative purposes, another sample of Type I cement from Trinity-Houston, which showed only 0.078% autoclave expansion, was analyzed and the results are also listed.

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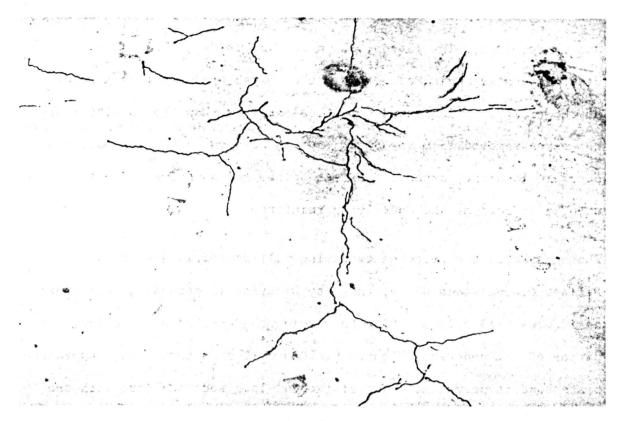


Fig. 2



Fig. 3

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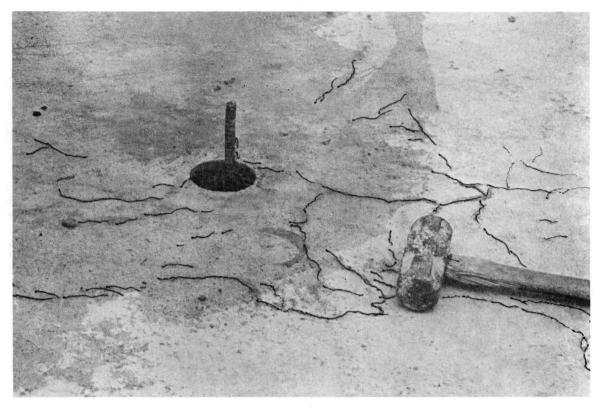


Fig. 4

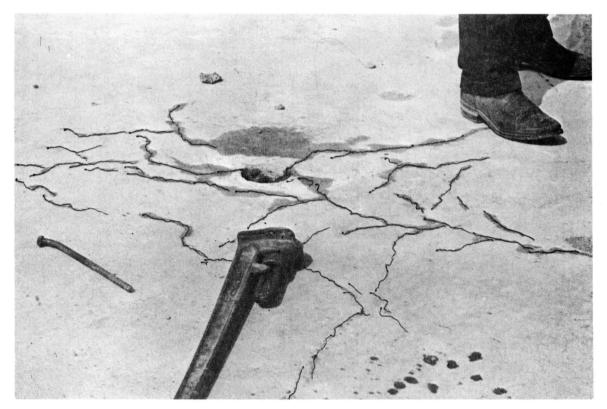


Fig. 5



Fig. 6



Fig. 7

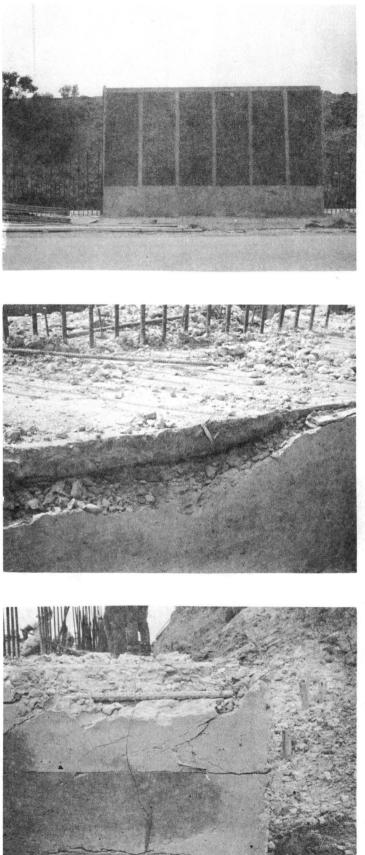


Fig. 8 View of retaining wall at the west Belfast overpass and US 59.

Fig. 9 View of retaining wall foundation before removal showing distress cracks. Scale is indicated by pocket knife.

Fig. 10 End of foundation section which measured 24' long, 8½' wide and 2½' deep. Note distress cracks and lack of aggregate-paste bond. Scale is indicated by pocket knife.

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Photos showing retaining wall foundations also shown in Figures 9 and 10. Distress cracks are indicated. Scale is indicated by pocket knife.

Fig. 11

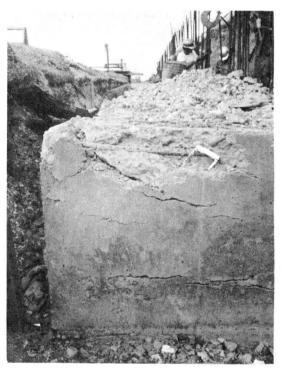


Fig. 12



Fig. 13

| Sample # | <u>72-2321-D</u> | <u>72-1827-D</u> |
|--|------------------|------------------|
| Free Lime (CaO) - Run #1 | 8.31% | 0.77% |
| Run #2 | 8.34% | 0.77% |
| Magnesium oxide (MgO) - Run #1 Run #2 | 1.08% 1.14% | 1.64% |
| Loss on Ignition (LOI) | 2.62% | .0.08% |
| Sulfur Trioxide (SO ₃) | 2.41% | * |

*Results Undetermined

Additional chemical data was collected when a portion of sample #72-2321-D was taken to Trinity's Fort Worth plant and analyzed by X-ray methods. The oxides of silica, aluminum, iron and magnesium were found to be normal; whereas, the calcium oxide content was found to be too high.

During the course of this investigation a total of seven cement samples from the Trinity-Houston plant were found to be unsound. A number of routine physical tests were performed on these samples and the results are listed in Table II.

Table I

| Table | II |
|-------|----|
|-------|----|

| | Sample No. | | | | | | |
|--|-------------|-------------|-------------|------------|-------------|----------------------|-------------|
| | <u>2321</u> | <u>2349</u> | <u>2420</u> | 2423 | <u>3137</u> | <u>3603</u> | <u>2816</u> |
| Surface Area (cm ² /gm) | 1910 | 1815 | 1855 | 1815 | 1915 | 1780 | - |
| Tensile Strength (psi) | | | | | | | |
| 1-day | - | - | - | æ | 610 | - | 323 |
| 3-day | 187 | 173 | 260 | 263 | 303 | 290 | 413 |
| 7-day | 273* | 250* | 337 | 343 | 400 | 410 | - |
| Autoclave Expansion - % | 20* | 20* | 10* | 10* | 3.0* | 3.0* | 2.5* |
| Sulfur Trioxide - % | 2.39 | æ | - | 4 7 | 2.56 | - | 1 9 |
| Setting Time | О.К. | 0.K. | 0.K. | 0.K. | 0.K. | 0.K. | О.К. |
| * Failing Results - No Results Determined | | | NOTE : | 275 ps | i | v streng ved expa | |

Of the seven cement samples listed in Table II, two exhibited approximately 20% expansion when subjected to the autoclave test. Figures 14 and 15 show prisms cast with normal and the unsound cement (#72-2321-D) as observed in the autoclave and after removal. Figures 16 and 17 i1lustrate results of the autoclave test on a number of unsound prisms. The relative amounts of expansion can be easily compared with a "normal" prism. Figure 17 shows a prism made with cement from sample #72-2349-D



Fig. 14

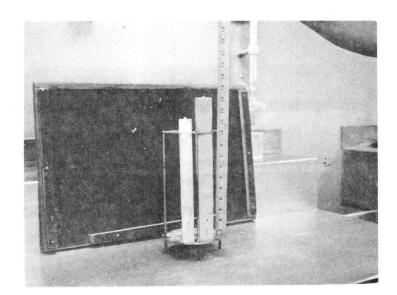


Fig. 15

| - 2321 | |
|--|---|
| *2420 | (All and a second s |
| 3137 | |
| Auto # 2 2 Ab NO. 5-23 | -22 2816 D |
| A REAL | |
| and a state of the | |

Fig. 16

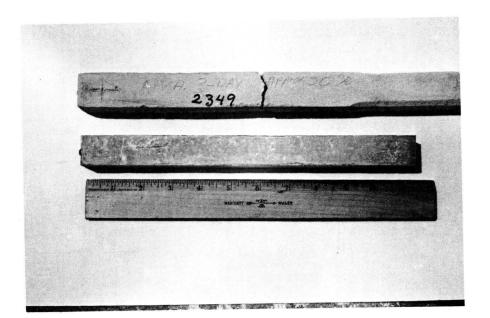


Fig. 17

which still exhibited 20% expansion even after the cement was aerated for three days.

Results from additional aeration on two separate cement samples containing the free lime are summarized in Table III. The two samples (#2349 and #3137) were placed in a pan, stirred frequently and exposed to the air (in the laboratory). Subsequent autoclave tests show that a loss occurs in the cement's expansive properties which reflects the chemical changes taking place as the free calcium oxide is converted to the hydrated form upon exposure.

Table III

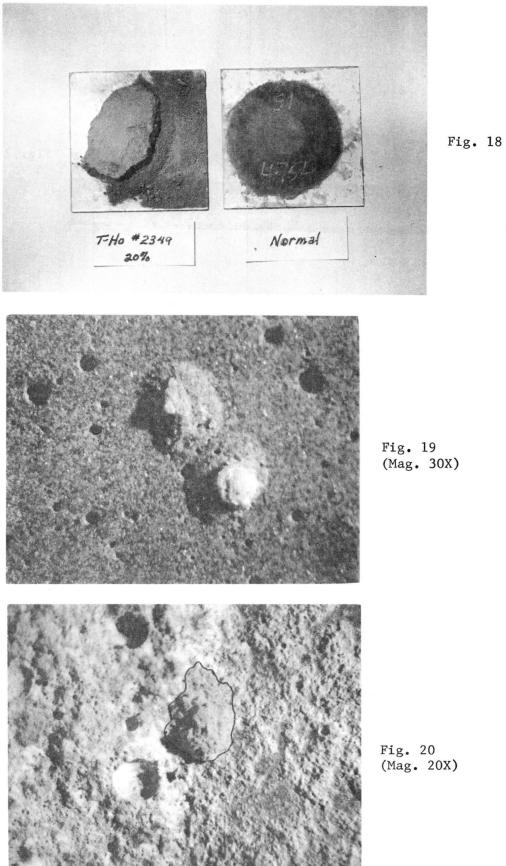
| Sample # | Aeration Time | Percent Expansion |
|-----------|----------------------|-------------------|
| 72-2349-D | As received | 20.0 |
| 72-2349-D | 1-week | 20.0 |
| 72-2349-D | 4-weeks | 0.1 |
| | | |
| 72-3137-D | As received | 3.0 |
| 72-3137-D | 2-days in closed can | 0.24 |
| 72-3137-D | 1-day in open air | 0.16 |
| | | |

Results from samples being subjected to the pat-boiler test are best

illustrated by Figures 18, 19, 21 and 22. A neat-cement sample made with the unsound cement (#2349) completely disintegrated when it was subjected to the pat-boiler (steam bath) for 5 hours as shown in Figure 18. The sample was cured in a moist chamber for 24 hours. A normal control sample which exhibited no expansion is also shown for comparison. Figure 19 shows exudations that were observed on the bottom of one of the neat-cement samples (#3137) after being subjected to the steam bath. Figure 20 shows a similar type of exudation observed on the surface of a piece of concrete (from the bridge deck) which had been subjected to the autoclave test.

Figures 21 and 22 illustrate the effect of 5 hours of steam on briquettes made with the unsound cement. An untreated briquette is shown on the far right for comparison in Figure 21. Photomicrographs taken at 4X show details of the crack pattern as observed on the curved sides of specimen #2321 in Figure 22; a close-up of the untreated sample showing a crackfree condition is illustrated as Figure 23. Briquettes containing the bad cement (#2321 and #2349) cured 7 days in water and left exposed in the laboratory for 5 months still showed to be unsound by the autoclave and steam-bath tests.

Other than the identification of the expected component phases of the cement, results from the petrographic studies were limited to indirect evidence of the presence of free lime. The major significant evidence that the failure of the concrete examined for this investigation was



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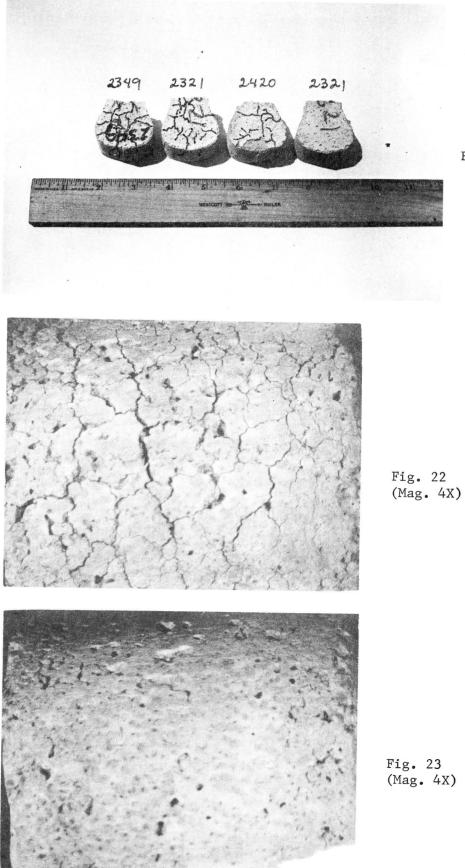




Fig. 21

caused from internal instability comes from the association of secondary chemical exudations formed within the air-void system (as those shown in Figures 24 and 25) and the presence of a pronounced network of microcracks. Secondary compounds formed on the surface of neatcement pats subjected to the steam-bath were similar to those observed within the air-void system of the concrete as well as those formed on the surface of concrete samples subjected to the autoclave test. Powder mounts of these secondary materials indicate that they are composed of calcium hydroxide being partially converted to calcium carbonate. The calcium hydroxide occurs in abundant quantities in concrete; however, these exudations suggest that an abnormal condition exists. These secondary deposits may very well indicate that "unaccommodative" chemical reactions have occurred as a result of the accelerated conditions (steambath and autoclave). Many of these exudations are in the form of circular mounds that are often cracked or collapsed as illustrated in Figures 24 and 25. Some are donut-shaped.

The crack patterns range in size from those shown in Figures 2-5 of the bridge deck, to hair-line cracks as those covering the entire surface of the column (Figures 26 and 27), down to the microcracks noted in the paste from all the examined structures. Especially, unusual are the "septa-like" cracks found in several air voids of the column. These unusual microcracks shown as Figures 28, 29 and 30 appear to have been formed before final-set of the paste and "healed" as calcium hydroxide solutions were forced into the void space and solidified. Microcracks were also noted throughout the retaining-wall samples. Large

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Fig. 24 (Mag. 15X)



Fig. 25 (Mag. 60X)



Fig. 26 (Mag. 7.5X)

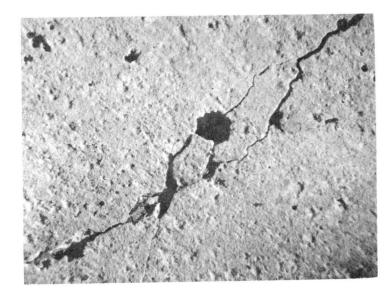


Fig. 27 (Mag. 7.5X)

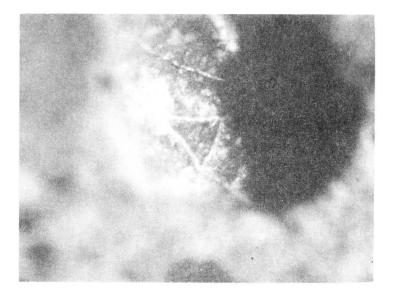


Fig. 28 (Mag. 50X)



Fig. 29 (Mag. 50X)

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Fig. 30 (Mag. 75X)

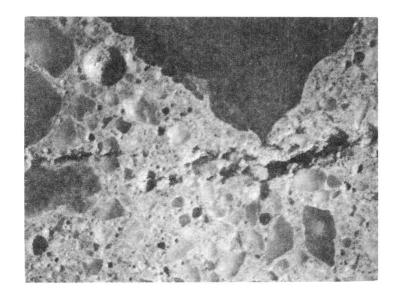


Fig. 31 (Mag. 10X)

cracks visible on the top of Core VI-A removed from the bridge deck were easily traced to depths of about 10 inches. Several pronounced horizontal separation cracks were found some 6 inches from the bottom of the core, but were not associated with the rebar (Figures 31 and 32).

Extended efforts were made to identify any free calcium oxide component within the remaining unhydrated cement grains comprising the paste fraction of the concrete samples. Highly-polished concrete samples were examined at 1000X under polarized reflected light but failed to reveal any abnormal compounds. Although the primary cement components (C₃S, C₂S, C₃A and C₄AF) were easily identified, no positive determination of CaO was made in any of the samples examined. Relative density of cement particles was noted, however, and indications were that mix-water contents for the bridge deck and the column were normal but may have been too high for the concrete in the retaining wall foundation. It was also noted that the relative extent of carbonation was much higher for the latter concrete samples. This condition is indicative of higher porosity which is generally related to high water/ cement ratios. Numerous bleed channels were also noted and the paste appeared chalky and powdery.

Linear traverse data was obtained from the bridge deck samples and the column; however, efforts were unsuccessful in preparing samples from the retaining wall for this type of examination. From the first two

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structures samples were analyzed for comparative purposes and the results are listed in Table IV.

Table 1V

Results of Linear Traverse

| | Bridge-Deck Samples | | | Column Samples | | |
|----------------------------|---------------------|-------|-------|----------------|-------|--|
| | A | В | C | A | B | |
| • 9 | - | | | ÷ | | |
| Air Content (%) | 6.38 | 7.42 | 6.98 | 2.75 | 2.11 | |
| Paste Content (%) | 27.25 | 24.53 | 25.30 | 29.01 | 21.41 | |
| Surface Area (in^2/in^3) | 570 | 541 | 597 | 412 | 391 | |
| | | | | | | |
| | | | | | | |

Although powder mounts of the Trinity-Houston cement proved to be inferior to polished sections of concrete for examining the cement, they did provide some information that suggests the presence of free lime more so than the latter technique. Under plane and polarized light at intermediate magnification ranges (200-400X) when emersion oils with refractive indexes of 1.71-1.73 were used, a number of "high relief" particles (those with refractive indexes which differ from the oil used) which resembled free lime were noted. However, these particles could not be differentiated from other cement components which also have a higher relief (about 1.83). On the other hand, when a small quantity of the cement was placed on a microscope slide with about 2 drops of White's solution, very delicate needle-like crystals of phenoxide appeared in 2-3 hours (Fig. 33). Many crystals were observed when the slide was observed a day later. Reportedly, this solution will react within a few minutes if appreciable quantities of free lime are present; however, the samples of cement examined (#2321 and #2349) had been retained in the lab for about 5 months before this observation was made. The delay in reaction time no doubt reflects a loss in reactive or uncombined free lime due to its gradual hydration. The same applies to the absence of free lime in the polished concrete pieces. By the time these pieces were prepared in the laboratory the original properties of the free lime had long disappeared.

VI. DISCUSSION

Departmental personnel concerned with the quality control of concrete and durability of structures have long recognized that volume stability is one of the basic desired characteristics of structural concretes. Regardless of how well the aggregates have been selected or the quality of entrained air, it cannot be expected to produce structural concrete that will be free from objectionable volume changes and cracking, unless the cement paste has a high degree of volume stability. Often these changes contribute to at least a gradual, and sometimes sudden, deterioration.

During April and May 1972 several cement samples, produced at Trinity-Portland's Houston plant, and shipped to the Materials and Tests Division

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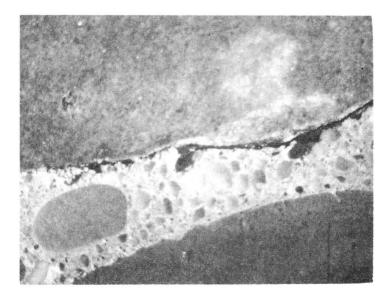


Fig. 32 (Mag. 7.5X)

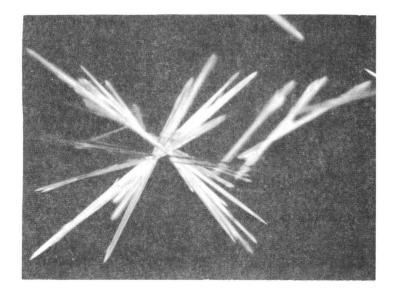


Fig. 33 (Mag. 500X)

laboratory in Austin, were found to be unsound as indicated by the autoclave expansion test. It was determined that these cements exhibited expansion ranges from 2.5 to 20 percent compared to the maximum allowable of 0.8 percent. Two samples, which had a 20 percent autoclave expansion, passed the 3-day tensile-strength test only by a narrow margin but failed the 7-day test. Project engineers both at the Houston Urban Office and District 12 were quickly alerted so that highway projects using the unsound cement could be closely watched and precautions made. Fortunately, use of the cement on highway projects was limited; however, it was found that three structures at two separate projects in Houston had severe damage and had to be removed.

Both uncombined calcium oxide and magnesium oxide are chemically unstable in portland cements. Although the chemical analysis of the cement samples revealed that the calcium oxide content was abnormally high, the percentage of the latter compound was found to be normal. In addition, results of the loss-on-ignition test were also high indicating that some of the free lime had converted to the hydrated form when the sample was tested. No doubt that when the cement was used on the Houston projects it contained excessive amounts of calcium oxide in the unhydrated form, and with an attendant volume increase of almost 100 percent when free CaO hydrates to Ca(OH)₂, the resulting concrete failed with irreparable damage.

Additional laboratory studies, which involved subjecting neat-cement pats and briquettes to the steam-bath test, showed that a decrease in

- 32 -

potential reactivity occurred with aeration. The samples which exhibited 10 percent autoclave expansion or less showed a rapid loss in potential reactivity; whereas, the ones with 20 percent expansion still showed a 0.1 percent expansion even after 4 weeks. Extensive cracking occurred when these latter briquettes containing the "20%" cement were cured 7 days in water, stored 5 months in the laboratory and finally subjected to the autoclave and steam-bath tests.

The significant petrographic evidences for unstable cement in the examined concrete samples were exudations of calcium hydroxide within the air-voids and the associated network of microcracks. Cracks ranged in size from those shown in the photographs of the bridge deck and retaining wall foundation to microscopic in size as with those found in the paste of all samples examined. Exudations also occurred on the surface of neat-cement pats subjected to the steam bath and on concrete samples subjected to the autoclave test. Both the cement pat samples and briquettes showed varying degrees of disintegration when tested in the steam bath after extended periods of aeration. Microscopic examinations of those samples reveal a pronounced loss of bonding strength, loss of color and a change from a dense glassy appearing paste to one that looks porous and chalky. The concrete samples appeared the same way after treatment in the autoclave.

Thin-sections made of the concrete samples proved to be inadequate for examining the cement as compared to the successfully used polished sections. Although the polished sections provided a means of determining a number of features of the paste and cement particles such as the relative

- 33 -

extent of hydration, no traces of free lime were found at the time of this study (about 4-5 months after the concrete was placed). However, powder mounts of the cement after being treated with a solution of phenol and nitrobenzine showed that minute traces of the free lime were present.

Microscopic analysis indicates that mix-water contents for the bridge deck and column were not excessive; however, the lighter-colored paste, higher porosity and the presence of bleed channels suggest that the concrete batched for the retainer wall may have had some extra water added. Linear traverse data shows that the air content of the deck concrete was about 6.5 percent and had a paste content of some 25 percent. Air content for the column was measured at about 2.5, indicating that no air-entraining admixtures were added to the mix. The aggregatepaste bond was so poor in the retaining wall samples that efforts were unsuccessful for analysis by linear traverse methods. APPENDIX I

HOUSTON URBAN EXPRESSWAYS

URBAN LABORATORY

| | | | | DATE | 5/1/72 |
|------------------|-------------|--------------|--------|-----------------------------------|-----------------------|
| ONTROL NO |). <u> </u> | 5-8 | | FEDERAL PROJECT NO. I 610-7(189) | 782 |
| ^ONTRACTO | Trinity C | onstr. Co. | | RESIDENT ENGINEER Frank Ge | eron |
| MATERIAL | Class "C" | Conc. | SOURCE | Parker Bros., Old Galveston Rd. | TRUCK NO. 361 |
| TEM NO | 421 | STRUCTURE NO | 351 | STRUCTURE LOCATION Bridge Slab(C: | ontinuous Uait)Four 6 |

BROKEN BY Shilstone Testing Laboratory

| CORE | | | | | | | | | LOAD | |
|-------------------|---|----------------|--------------|-----|--------------|--------------|----------|-------------|---------------|-------------------|
| NO. | MADE BY | DATE MADE | TIME MADE | C/F | DESIGN | SLUMP IN. | AIR % | AGE DAYS | RRITAR REA | AVERAGE STRESS |
| 1-A Top 1/2 | Lindsay | 4/24 | 13:35 | 6.0 | PB -2 | 3-1/2 | 5.0 | 7 | 22500 | 1791 |
| Bottom 1/2 | · | | | | - - | | | | 33000 | 2627 2209 |
| REMARKS: | Air Temp. = Conc Temp. = MB-VR = 5 o Pozz. 100-X | 830 F z./CY | ./CY | | | | | <u>_</u> | | Letter 1 |

INSTRUCTIONS:

1. Complete all columns of this report.

- 2. Report any abuse, unnecessary handling, improper curing, etc., of cylinders under "REMARK." Be specific about location, e.g., Column Bent Structure . If more than one (1) set of cylinders is listed on this report, indicate location for each set. 3.
- Report admixtures (brand and dosage) and air temperature under "REMARKS". 4.
- 5. Make only six (6) test cylinders from a truck.

El

HOUSTON URBAN EXPRESSWAYS

URBAN LABORATORY

| | DATE 5/1/72 |
|--------------------------------|--|
| (NTROL NO. 271-15-8 | FEDERAL PROJECT NO. 1 610-7(189)782 |
| CONTRACTOR Trinity Constr. Co. | RESIDENT ENGINEER Frank Garon |
| MATERIAL Class "C" Conc. | SOURCE Parker Bros., Old Galveston Rd. TRUCK NO. 361 |
| TEM NO. 421 STRUCTURE NO. 35 | SI STRUCTURE LOCATION Bridge Slab(Continuous Unit)Pour 6 |

BROKEN BY Shilstone Testing Laboratory

| CORE | | | | | | | | | LOAD LES. | |
|-------------------|---|---------------|--------------|-----|--------|--------------|----------|-------------|---|-------------------|
| NO. | MADE BY | DATE MADE | TIME MADE | C/F | DESIGN | SLUMP IN. | AIR % | AGE DAYS | XXX XXX XXX XXX XXX XXX XXX XXX XXX XX | AVERACE STRESS |
| 2-A Top 1/2 | Lindsay | 4/24 | 13:35 | 6.0 | PB-2 | 3-1/2 | 5.0 | 7 | 52000 | 4140 |
| Bottom 1/2 | | | | | | | | | 43000 | 3424 3782 |
| Cor ME | r Temp. = nc Temp. = 3-VR = 5 oz ozz. 100-XR | 83° F ./CY | - - | | | | | | | <u>LEB</u> |

INSTRUCTIONS:

1. Complete all columns of this report.

- 2. Report any abuse, unnecessary handling, improper curing, etc., of cylinders under "REMARSS". 3. Be specific about location, e.g., Column Bent Structure . If more than one (1) set of cylinders is listed on this report, indicate location for each set.
- 4. Report admixtures (brand and dosage) and air temperature under "REMARKS".
- 5. Make only six (6) test cylinders from a truck.

Tell

HOUSTON URBAN EXPRESSWAYS

URBAN LABORATORY

| | | | | | |] | DATE | 5/1/72 | |
|------------|-----------|--------------|---------|-------------|-----------|----------|------------------|-----------|-------------|
| (NTROL NO | 271-1 | 5-8 | _ | FEDERAL PR | OJECT NO. | <u> </u> | L 0-7 (18 | 9)782 | |
| CONTRACTOR | Trinity C | onstr. Co. | | RESID | ENT ENGIN | EER | Frank G | eron | |
| MATERIAL | Class "C" | Conc. | SOURCE_ | Parker Bros | s., 01d G | alvesto | on Rd. | TRUCK | NO. 361 |
| EM NO. | 421 | STRUCTURE NO | 351 | STRUCTURE | LOCATION | Bridge | Slab(C | ontinuous | Unit)Pour 5 |

BROKEN BY Shilstone Testing Laboratory

| CORE | | | | | | | | | LOAD LBS. | |
|----------------------|---|---------------|--------------|-----|--------|--------------|----------|-------------|-------------------|-------------------|
| NO. | MADE BY | DATE MADE | TIME MADE | C/F | DESIGN | SLUMP IN. | AIR % | AGE DAYS | XXXIIIXX XXXII | AVERAGE STRESS |
| 3-A Middle 1/3 | Lindsay | 4/24 | 13:35 | 6.0 | PB-2 | 3-1/2 | 5.0 | 7 | 27500 | 2 189 |
| | | | | | | | | | | |
| · | | | | | | | | | | |
| | | | | | | | | | | I.B. |
| C c | Air Temp. = onc Temp. = B-VR = 5 oz Pozz. 100-XR | 83° F ./CY | /сч | | | | | | | |

1. Complete all columns of this report.

- Report any abuse, unnecessary handling, improper curing, etc., of cylinders under "REMARUS".
 Be specific about location, e.g., Column _____ Bent _____ Structure _____. If more than
- one (1) set of cylinders is listed on this report, indicate location for each set.
- 4. Report admixtures (brand and dosage) and air temperature under "REMARKS".
- 5. Make only six (6) test cylinders from a truck.

HOUSTON URBAN EXPRESSWAYS

URBAN LABORATORY

| | | | | | DA | TE | 5/1/72 | | |
|------------|-----------|---------------------------|--------|------------------------|---------|----------------|-----------|----------------|----------|
| ONTROL NO | 271-3 | 15-8 | | FEDERAL PROJECT NO | 1 610 |)-7(1 8 | 9)782 | | |
| CONTRACTOR | Trinity (| Constr. Co. | | RESIDENT ENGINE | ER Fr | ank C | Beron | | <u> </u> |
| ATERIAL | Class "C" | Conc. | SOURCE | Parker Bros., Old Galv | veston | Rd. | TRUCK | NO. <u>361</u> | |
| TEM NO | 421 | STRUCTURE NO. 351 | | STRUCTURE LOCATIONEr | idge Sl | ab(Co | ontinuous | Unit) Pour | 6 |
| BROKEN BY_ | Shilston | <u>- Testing Laborato</u> | ry | | | | | | |

| | 1 | | | | · | | | | LOAD LOS. | |
|-------------------|---|----------------------|-------------------------|-------------------|------------------------------|-------------------------------------|-------------------|--------------------|----------------------------|-------------------------------------|
| CYLINDER NO. | MADE BY | DATE MADE | TIME MADE | C/F | DESIGN | SLUMP IN. | AIR % | AGE DAYS | RITERIA REA | AVERAG STRES |
| 643 644 645 | Lindsay Lindsay Lindsay | 4/24 4/24 4/24 | 13:35 13:35 13:35 | 6.0 6.0 6.0 | PB-2 PB-2 PB-2 PB-2 | 3-1/2 3-1/2 3-1/2 | 5.0 5.0 5.0 | 7 7 7 7 | 101000 107000 107500 | 3572 3785 3802 3720 |
| Cone MB | r Temp. = 2 c Temp. = 2 -VR = 5 oz. zz. 100-XR = | 83° F /CY | CY | | | HOUST MATERIAL DATE SIGNED | MG.18 | BAN DA Steche v | 302.414.23 1.085 | |

- 1. Complete all columns of this report.
- 2. Report any abuse, unnecessary handling, improper curing, etc., of cylinders under "REMARNS" 3. Be specific about location, e.g., Column ____ Bent ____ Structure ____. If more than
- one (1) set of cylinders is listed on this report, indicate location for each set.
- 4. Report admixtures (brand and dosage) and air temperature under "REMARKS".
- 5. Make only six (6) test cylinders from a truck.

Texas Highway Department Construction Form 309

| County: Project: | Harris |
|---------------------|---------|
| Date: | 9-7-71 |
| Design No | · PB-2. |

CONCRETE DESIGN WORK SHEET (NATURAL AGGREGATES)

| | (INTURAL AUGREDATES) | Design N | h. F | ロース | |
|--|--|--|---------------|---------------|-------------|
| AGGREGATE CHARACTERISTICS: | | | | | |
| SSD SP. GR Lbs. | Unit Wt. <u>/Cu. Ft.</u> <u>% SOLIDS</u> Soutenberry Ca <u>5.8</u> <u>13.9</u> <u>04.5</u> <u>64.3</u> W | sie fabre |) | | |
| Fine Aggregate (FA)6510 | 5.8 63.9 | 2 Gravel | A (| A | |
| Coarse Aggregate (CA) 2.60 18 | 04.5 64.3 W | Air & | K=+ | arde | Y |
| Water | | / | | | |
| Cement | | | | | |
| DESIGN FACTORS: | | BATCH FACTOR: | | | |
| Cement Factor (CF), 6.0 sacks per d | cubic yard of concrete | Size of Batch (Full Size) | 27 | 0 | - |
| Coarse Aggregate Factor (CAF), | | Size of Batch (Full Size) Yield for I-Sk. Batch | = | 5 = - | 0,0 |
| Water Factor (WF), 4.8 gal. per sa | ck of cement | | 4 | | |
| Air Factor (AF),5.0_% | | | | | |
| | | VOL. TO WT. (LB.) | 1-SK. | FULL | |
| BATCH DESIGN (ONE SACK) | VOLUMES: 1-SK. BATCH (CU. FT.) | VOL. X 62.5 X SP. GR | BATCH WTS. | BAT Factor | WTS. |
| I. Concrete Yield = $\frac{Cu. Ft. per Cu. Yd.}{CF}$ | $\frac{27}{6.0} = \frac{4.5}{}$ | | | | |
| 2. Volume CA = Yield X CAF X Solids | 4.5 × .70 × .643 2.0 |)26 x 62.5 x 2.62 = | 329 | 6 | lany |
| 3. Volume Mortar = Yield — Vol. CA | 4.5 - 2.026 = 2.474 | | | | |
| 4. Volume Water $= rac{{\sf WF}}{{\sf Gal.~Water~per~Cu.~Ft.}}$ | <u> </u> | $\frac{40}{\times 62.5 \times 1.00} =$ | 40 | 6 | <u>•240</u> |
| 5. Volume One Sk. Cement | = 0.4 | 485 X 62.5 X 3.10 = | 94.0 | 6 | 534 |
| 6. Volume Entrained Air = Yield X AF | | 225 | | | |
| 7. Volume Paste = Vol. Cem. + Water + Air | 0.485 + 640 + ,225 = 1.350 | | | | |
| 8. Volume FA = Vol. Mortar — Paste | $\begin{array}{r} 0.485 + .640 + .225 = 1.350 \\ 2.414 - 1.350 \\ \end{array} = 1.1 \\ \end{array}$ | 124 × 62.5 × 2.55= | 18% | 3 | 110 |
| 9. Yield (Summation of 2, 4, 5, 6 & 8 to Check No | L | .500 | T | 1:1 | 3894 |
| 10. Fine Aggregate Factor = Vol. FA FA Solids X Vol. N | $\frac{1.124}{400000000000000000000000000000000000$ | 7/ | | | |

* Correct For Free Moisture or Absorption.

REMARKS: Volumes in Above Are Absolute Unless Otherwise Noted. Water Added at Mixer Must Include the Liquid of the Admixtures.

| exas Highway Department Construction Form 309 | | 0 | NCRETE DESIG | N WORK | SHFFT | • | Project: | Har 27 - | 12 - | |
|--|---|----------------------------|---------------------------|----------------|--------------------------------------|-----------|---|--------------------------|------|------------|
| | | 001 | (NATURAL A | | UNELI | | | 1o:2 | | ł |
| AGGREGATE CHARAC | S | SD Unit Wt. bs./Cu. Ft. | % SOLIDS | | · | | , | Bros. | | |
| Fine Aggregate (FA) | 2.62 | 97_60 | 59.60 | Parker | Bros. | Eagle L | ake, Tex | 1 5 | | |
| Coarse Aggregate (CA)_ | 2.59 1 | 05.74 | 65.32 | * | * | | tt tr | | | |
| Nater City of Staf | ford, Texa | 8 | | | | | | | | |
| Cement Ceme AIR : MB * VR DESIGN FACTORS: Cement Factor (CF),6 Coarse Aggregate Factor Water Factor (WF),1=3 Air Factor (AF),5=0 | : Master 0sacks pr (CAF),78 gal, per | er cubic yard | of concrete | ERDER 3 | | BATCH F | ter Buil ACTOR: Ich (Full Size I-Sk. Batch | <u>) _ 27</u> 4.50 | | 6.0 |
| BATCH DESIGN (ON | ié sacki | | VOLUMES: 1-SK. | BATCH (CU. F | T.) | , | . TO WT. (LB.) (62.5 X SP. G | 1-SK. Batch R Wts. | | SIZE |
| I. Concrete Yield $=$ $\frac{Cu. Ft.}{Cu. Ft.}$ | per Cu. Yd. CF | 27 | -=_4.500 | - | | | | | | |
| 2. Volume CA = Yield X C | | | . × <u>•78</u> × | < <u>6532</u> | = 2.29 | 3X 62. | 5 x 2 <u>.5</u> 9 = | -371-2 | 6.0 | • 2227 |
| 3. Volume Morter — Yield - | - Vol. CA | 4.500 | 2_293 = | <u>2 2 207</u> | | | | | | |
| 4. Volume Water = Gal. W. | WF ater per Cu. Ft. | 4 7. | 3 5 | | = 0.57 | 3X 62 | .5X i.00 = | -35.8 | 6_0_ | • - 215 |
| 5. Volume One Sk. Cement | | | | | - 0.4 | 85 X 62 | .5 X 3.10 = | - 94.0 | 6.0_ | -564- |
| 6. Volume Entrained Air = 1 | Yield X AF | 4.500 | x0_5 | <u>.</u> | = <u>0.22</u> | 5 | • | | | |
| 7. Volume Paste — Vol. Cen | n. + Water + A | Vir 0.485 + 0 | <u>-573</u> +0-22 | 5_=1_28 | 3 | | | | | |
| 8. Volume FA = Vol. Morta | r Paste | 2.207 | 1_283 | | -0.92 | x 62 | .5 x 2 <u>+62</u> - | -151-3 | 6.0 | 908 |
| 9. Yield (Summation of 2. 4, | 5, 6 & 8 to Checi | No. I Above) | | | - 4.5 | 00 | | | | |
| 10. Fine Aggregate Factor = | Vol. F/ FA Solids X Vo | A ol. Morter | 0 <u>934</u> •5960 x 2 | •207 | $= \frac{0_{\bullet}}{1_{\bullet}3}$ | 924 15 | •70 | | | |
| * Correct For Free Moisture o | r Absorption. | | | | | | | | | |

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REMARKS: Volumes in Above Are Absolute Unless Otherwise Noted. Water Added at Mixer Must Include the Liquid of the Admixtures.

FORXOOATENS FOR DRUM COATING ADD : 282 # Sand 94 # Coment Design for retaining cuell with CX posed aggregate (21/2")

(Exp. Agg. for Ret. Walls)

2 = GRAVEL

W.A.S.

| Texas Highway Department Construction Form 309 | 9 | 9 | County: Harr | <u>is</u> |
|---|---|--------------|---|-------------------------------------|
| | CONCRETE DESIGN WORK S | UCCT | Project: 27-1 Date: 1-10 | |
| | (NATURAL AGGREGATES) | | | |
| AGGREGATE CHARACTERISTICS: | | | Design No: 2 | |
| SSD | Unit Wt. /Cu. Ft. % SOLIDS | | Class "C" C Parker Bros Staîford, T | • |
| Fine Aggregate (FA) 2.64 | <u>104.16 63.13</u> Parker | Bros. Eagl | e Lake, Texa | S |
| Coarse Aggregate (CA) 2.60 | 102.64 63.19 Parker B | ros. Eagle | Lake, Texas | |
| Water City of Staff | ord, Texas | | | |
| Cement 🏭 🌛 🦛 🗺 🕬 Cemen | t Co. | | | |
| Air MB-VR DESIGN FACTORS: | | BATCH F | ACTOR: | |
| Cement Factor (CF), 6.0 sacks_per of | cubic yard of concrete | Size of Bate | ch (Full Size) 27 | 6.0 |
| Coarse Aggregate Factor (CAF), | - | Yield for | I-Sk. Batch 4.50 | 10 = |
| Water Factor (WF), 49 gal. per sa | ck of cement | | | |
| Air Factor (AF), <u>5.0</u> % | | | | < |
| BATCH DESIGN (ONE SACK) | VOLUMES: 1-SK. BATCH (CU. FT.) | | TO WT. (LB.) 1-SK. BATCH 62.5 X SP. GR WTS. | FULL SIZE BATCH FACTOR WTS. |
| I. Concrete Yield = $\frac{Cu. Ft. per Cu. Yd.}{CF}$ | 4.500 | | | |
| 2. Volume CA = Yield X CAF X Solids | <u>4.500</u> × <u></u> | 2.161 × 62.5 | x <u>2.60_351.1</u> | 6 6.0 2107 |
| 3. Volume Morter — Yield — Vol. CA | $\frac{4.500}{2.161} = \frac{2.339}{2.339}$ | | | |
| 4. Volume Water = WF Gal. Water per Cu. Ft. | $\frac{4.9}{7.5}$ = | 0.653 x 62 | $5 \times 1.00 = \frac{40.81}{100}$ | 6.0 245 |
| 5. Volume One Sk. Cement | = | 0.485 × 62.9 | 5 X 3.10 = 94.0 | 6.0 564 |
| 6. Volume Entrained Air = Yield X AF | <u>4.500 x 0.5</u> = | 0.225 | | |
| 7. Volume Paste = Vol. Cem. + Water + Air | 0.485 + 0.653 + 0.225 = 1.363 | | | |
| 8. Volume FA = Vol. Morter Peste | 2.339 - 1.363 = | 0.976 × 62. | s x <u>2.64</u> = <u>161.0</u> | 4 6.0 966 |
| 9. Yield (Summation of 2, 4, 5, 6 & 8 to Check N | o, I Above) = | = | | |
| 10. Fine Aggregate Factor = Vol. FA FA Solids X Vol. 1 | 0.976 Morter 0.6313 × 2.339 | .66 | | |
| * Correct For Free Moisture or Absorption. | | | | |

REMARKS: Volumes in Above Are Absolute Unless Otherwise Noted. Water Added at Mixer Must Include the Liquid of the Admixtures.

FOR COATING BATCH TO EACH LOAD ADD: 282 Lbs. Sand 94 Lbs. Cement

Conc design for footing Conc.

FIL

APPENDIX II

DETERMINATION OF MAGNESIUM OXIDE*

Procedure

Weigh out approximately 3 grams of the cement to be analyzed. Transfer the sample to a platinum dish and moisten with a little distilled water. Then add approximately 25 ml of 6 N. HCl and heat until the sample dissolves. Evaporate the sample to dryness by baking in 212°F oven and then redissolve in 25 ml of 6 N. HCl. Transfer the sample from the platinum dish to a 250 ml small-form beaker. Carefully add 15 ml of 20% ammonium hydroxide. Then, using the pH meter, adjust the pH to between 5 and 7 using 1 Normal NaOH. A precipitant of Aluminum and Iron hydroxides should form. After adjusting the pH, carefully wash off the probes so as not to lose any sample. To the sample, add 30 ml of the 20% Na₂SO₃ solution to precipitate calcium and any other heavy metals that might be present. At this point, the solution must be filtered. A quantitative filter paper, #42, should be used. The sample may be filtered directly into a 250 ml volumetric flask. After filtering out the precipitant, wash it several times with distilled water. The precipitant may be discarded at this point. The filtrate in the 250 ml volumetric flask contains the Magnesium which is to be determined titrimetrically. Add enough distilled water to the flask to bring the total sample volume up to the

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^{*} This method is primarily used for analyzing lime samples but can be appropriately applied to portland cements.

total sample volume up to the 250 ml mark. Mix the contents of the flask thoroughly, and pipet out a 50 ml portion into a 400 ml tall-form beaker. Add 10 ml of a pH 10 buffer and 10 ml of a 10% NaCN solution. Add distilled water to bring the total volume up to about 150 ml. Then add 0.100 to 0.150 gm Eriochrome Black-T indicator. When viewed with a light behind it, the solution should have a bright red color. Titrate the sample rapidly with a 0.01 molar solution of EDTA until the solution changes from red to a reddish blue color. Then add the EDTA slowly until the last trace of red disappears. This is the end point. Duplicate samples should be run. To calculate the Magnesium present in the sample as MgO:

Note: The size of the original sample and the fraction of solution titrated were designed for a sample containing approximately 0.5 to 3.0% MgO. Sample size, dilution, and fraction titrated may be adjusted to compensate for MgO content outside these limits. In event this is done, the formula for calculating percent MgO must also be changed.