DEFECTIVE CONCRETE IN BRIDGE DECK POUR
I 94 over US 131 Southwest of Kalamazoo (BI 507 of 39024B, C1)

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DETECTIVE CONCRETE IN BRIDGE DECK POUR
I 94 over US 131 Southwest of Kalamazoo (BI 507 of 39024B, C1)

This investigation of a defective concrete pour on the subject structure was initiated at the request of R. S. Fulton, Field Testing Division, on September 25, 1962. The pour in question is designated as pour A3, at the east end of a structure carrying westbound I 94 over southbound US 131 (relocation), about 4.5 miles southwest of Kalamazoo.

The following pertinent information relating to this pour was obtained from R. S. Fulton's field notes and from a complete letter description by K. F. Whelton and E. B. Baga of the Bridge Construction Division, dated September 27, 1962. Pouring operations on pour A3 began at 9:30 a.m. and finished at about 11:00 a.m. on September 17, 1962, proceeding east from a steel expansion dam. A total of about 20.8 cu yd of transit-mix concrete from the Statler Ready Mix plant in Kalamazoo was used in this pour. An adjacent pour, B3, was started immediately after pour A3 was finished. Both pours were designed to contain Plastiment retarder, added by hand at the transit mix plant at the rate of 3 fluid ounces per sack of cement.

Apparently the first 5-cu yd load of concrete differed in setting properties from the other three loads used in pour A3. This became obvious when 5 to 7 days after pouring this first load, covering approximately
the west 12 ft, had not completely hardened. Because of the abnormal setting properties of the concrete on this part of pour A3, four cores were taken on September 24, 1962. Single cores were drilled near each end and near the middle of pour A3, and also near the east end of pour B3. The core from the west end of pour A3 completely disintegrated because the concrete had not completely hardened. The other three appeared to contain normal concrete. The entire pour A3 was removed on September 25 and 26 and re-poured with specification concrete on September 28, 1962.

**Physical and Chemical Tests on Cores**

The core samples removed September 24 were transmitted to the Research Laboratory Division on September 25, and were identified as samples 62 CR-50 through -53. Air content and compressive strength tests were run on the three sound cores, with the results given in Table 1. The results were quite normal for 11-day old concrete for both air content and strength. The loose material from sample 62 CR-50 and the broken portions of 62 CR-51, -52, and -53 were crushed and ground to a minus-140 mesh size for chemical tests. These tests were suggested to check for the presence of an adulterant such as sugar, salt, Noxcrete (a form release and cleaning compound), or to determine possible cement content variations or Plastiment over-dosages. Sample 62 CR-50 consisted of crumbled chunks of concrete with occasional patches of a white
powder on their surfaces. Infrared analysis of the powder indicated it was a carbonate similar to dolomite, which had crystallized when the concrete eventually dried. Infrared examination of 140-mesh samples from each of the four cores gave little information because carbonates present in the concrete (the coarse aggregate was limestone) masked out the spectral peaks of other materials. Results of this nature are usually expected from infrared spectra of inorganic materials, although the presence of organic materials can occasionally be determined.

**TABLE 1**
SUMMARY OF TEST DATA ON DECK CORES

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Core No.</th>
<th>Location</th>
<th>Air Content, percent*</th>
<th>Compressive Strength, psi**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pour A3, East Span</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>62 CR-50</td>
<td>1</td>
<td>8 ft east of expansion dam</td>
<td>—</td>
<td>4470</td>
</tr>
<tr>
<td>62 CR-51</td>
<td>2</td>
<td>15 ft east of expansion dam</td>
<td>7.2</td>
<td>3780</td>
</tr>
<tr>
<td>62 CR-52</td>
<td>3</td>
<td>3-1/3 ft from east end</td>
<td>7.4</td>
<td>5005</td>
</tr>
<tr>
<td>Pour B3, East Span</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>62 CR-53</td>
<td>4</td>
<td>7-1/2 ft from east end</td>
<td>7.5</td>
<td>5005</td>
</tr>
</tbody>
</table>

* Linear traverse method on the top 1/2-in. of 4-in. diam cores.

** Corrected to a height-to-diameter ratio of 2:1. Cores broken September 28, 1962, at age of 11 days.

An analysis for cement content according to ASTM method C 85-54 indicated that the same amount of cement was present in all four samples. To check for the presence of salt, sugar, or other materials, portions
of the finely ground sample cores were extracted with water and the dried extracts prepared for infrared examination. The spectra showed that sample 62 CR-50 enhanced an unidentified absorption band near 9 microns. The presence of sugar or form oil was not indicated. A subsequent chemical test confirmed the absence of sugar. Traces of salt or chlorides were found to be present in comparable amounts in both normal and defective concrete.

**Tests on Laboratory Mixes**

The use of Plastiment retarder in excess of the recommended 3 oz per sack of cement was considered another possible cause for the defective concrete. A sample of Plastiment was obtained so that concrete with known amounts of the retarder could be prepared in the laboratory. Concrete specimens containing 0, 3, and 18 oz of Plastiment per sack of cement were prepared, using the same materials and mix design as pour A3. The 18-oz figure was chosen since 12-oz over-dosages were reported recently by Grieb, Werner, and Woolf (1) as causing an 80-hour delay in initial and final set. The specimens containing 18 oz of Plastiment did not attain final set until 7 days. After physical testing, with the results shown in Table 2, one cylinder from each mix was ground to 140 mesh for chemical testing.

Plastiment was added to a basic solution of calcium chloride to test for the formation of insoluble calcium salts of the organic acids that are the active ingredients in Plastiment. Insoluble salts did not form, which indicated it would be possible to extract Plastiment residues from concrete rich in calcium compounds. Water extracts were prepared from the laboratory specimens and again from the bridge samples. Infrared spectra of these extracts showed that the laboratory specimens containing excess Plastiment enhanced a band near 9 microns as did the defective concrete of sample 62 CR-50.

**TABLE 2**

**SUMMARY OF TEST DATA ON LABORATORY MIXES**

<table>
<thead>
<tr>
<th>Mix*</th>
<th>Plastiment Content, fluid oz/sack of cement</th>
<th>Proctor Penetration Test, hr (time to reach given pressure)</th>
<th>14-Day Compressive Strength, psi**</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>500 psi</td>
<td>4000 psi</td>
</tr>
<tr>
<td>A</td>
<td>0</td>
<td>4:00</td>
<td>7:00</td>
</tr>
<tr>
<td>B</td>
<td>3</td>
<td>8:00</td>
<td>10:00</td>
</tr>
<tr>
<td>C</td>
<td>18</td>
<td>27:00</td>
<td>Approx. 7 days</td>
</tr>
</tbody>
</table>

* Same materials and mix design as used on September 17, 1962, for pour A3:

Mix Design (6 sacks cement per cu yd)
- 94.0 lb cement, Huron AE, Alpena
- 230.5 lb 2NS sand, American Aggregates (Pt 39-1)
- 271.0 lb 6AA coarse aggregate, Materials Service (limestone)
- 49.7 lb water (approximate)

** Average of three cylinders each, cured 7 days moist and 7 days in air.

An attempt was made positively to identify the 9-micron absorption band as characteristic of Plastiment. The water extracts from the concrete samples were acidified and extracted with ether to separate the
Plastiment organic acids from the soluble carbonates of the concrete. The dried extracts were insoluble in common organic solvents, but it was found that films could be cast on salt plates for infrared analysis from dimethylformamide solutions. Infrared spectra indicated the acids underwent random changes during isolation which did not allow definite identification of these products as Plastiment.

Since conclusive identification of the organic acids in Plastiment could not be obtained by infrared analysis, a color test for this type of organic acid was investigated. The literature revealed such a test, which depends on B-naphthol and sulfuric acid to produce a green fluorescence in the presence of some hydroxy acids. No color formed when Plastiment-free concrete was tested but a weak test was obtained on concrete core samples 62 CR-51, -52, and -53. A strong green fluorescent color was obtained from the defective concrete 62 CR-50 and a laboratory control prepared with 18 oz of Plastiment per sack of cement. The test does not give quantitative results, but definitely indicated excess Plastiment in core 62 CR-50.

Conclusions

It has been concluded that the defective bridge deck concrete in the west end of pour A3 was due to the addition of excess Plastiment retarder. The dosage was probably four to six times the 3-oz figure. This conclusion is supported by infrared spectra which showed an enhancement of
a 9-micron band in both the absorption spectrum of the actual defective concrete, and that of a laboratory concrete specimen prepared with excess Plastiment. Color tests also gave strong positive reactions with both the defective concrete and the laboratory concrete containing excess Plastiment. The setting properties of the latter laboratory mix compared closely with details of pour A3 in Whelton and Baga's summary. No evidence was found to indicate low cement content or the presence of sugar, excessive amounts of salts, or form release oil.