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TECHNICAL REPORT C-69-7

INVESTIGATION OF COLORLESS AND WATER-BASED CONCRETE CURING COMPOUNDS

by

C. F. Derrington



June 1969

Sponsored by

Office, Chief of Engineers
U. S. Army

Conducted by

U. S. Army Engineer Waterways Experiment Station
CORPS OF ENGINEERS
Vicksburg, Mississippi

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FOREWORD

The investigation reported herein was authorized by the Office, Chief of Engineers, by first indorsement dated 17 January 1967 to a U. S. Army Engineer Waterways Experiment Station (WES) letter dated 21 December 1966, subject: "Project Plan for Investigation of Water-Based and Colorless Curing Compounds." The work was conducted under Engineering Studies (ES) Item 623, "Investigation of Concreting Materials Other than Aggregates and Cementitious Materials," as Projects 623.2 and 623.5.

The investigation was conducted during the period February 1967 through July 1968 at the WES Concrete Division under the supervision of Messrs. Bryant Mather, R. V. Tye, and Leonard Pepper, and Mrs. Clara F. Derrington. This report was prepared by Mrs. Derrington.

Directors of the WES during this investigation and the preparation of this report were COL John R. Oswalt, Jr., CE, and COL Levi A. Brown, CE. Technical Directors were Messrs. J. B. Tiffany and F. R. Brown.

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CONVERSION FACTORS, BRITISH TO METRIC UNITS OF MEASUREMENT

British units of measurement used in this report can be converted to metric units as follows:

<u>Multiply</u>	<u>By</u>	<u>To Obtain</u>
inches	2.54	centimeters
square feet	0.092903	square meters
gallons	3.785412	cubic decimeters
miles per hour	1.609344	kilometers per hour
Fahrenheit degrees	5/9	Celsius or Kelvin degrees*

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

SUMMARY

Eleven concrete curing compounds designated as colorless and two compounds designated as water-based were evaluated for their suitability for curing of concrete and for their lack of color. All of the compounds met present specification requirements for sprayability, drying time, and flash point. Only ten of the materials met the moisture-retention requirements. The three materials that failed the moisture test were one colorless type (styrene-acrylate resin) and both water-based compounds.

An infrared spectrophotometer was used to identify and characterize each of the materials. Infrared analysis revealed that the 11 colorless materials represent five different chemical classes as follows: four chlorinated rubbers, two modified styrene-butadienes, one phthalic alkyd, three carboxylated hydrocarbons, and one styrene-acrylate. Both water-based materials were identified as sodium silicate liquids. Infrared measurements were used to detect chemical and physical changes of the colorless materials when exposed to laboratory conditions and natural sunlight.

The materials ranged in color from light yellow to dark reddish-brown and were rated from 1 to 18 on a standard color comparator. Generally, the mortar specimens coated with the curing compounds lightened in color when subjected to both a controlled temperature-humidity cabinet and ultraviolet radiation in a weatherometer. However, upon being subjected to alternate cycles of rainfall and ultraviolet radiation in the weatherometer, some of the coated surfaces continued to lighten and the curing compounds washed off, while some became mottled and unattractive. There appeared to be no correlation between original color of the curing compound and color of the coated mortar surfaces either before or after exposure to controlled conditions. Coating materials in the same chemical classes responded to weathering in a similar manner. Mortar surfaces coated with two of the carboxylated hydrocarbons possessed a dull finish and appeared more natural looking with fewer signs of mottling than any of the other coated surfaces.

For comparison purposes, a mortar specimen coated with white-pigmented carboxylated hydrocarbon curing compound was subjected to the same weathering tests as the colorless and water-based materials. The weathered pigmented specimen remained white throughout the tests, and the film adhered rather tenaciously to the mortar, with no mottling effects.

INVESTIGATION OF COLORLESS AND WATER-BASED
CONCRETE CURING COMPOUNDS

PART I: INTRODUCTION

1. Liquid, membrane-forming compounds that aid in the curing of concrete or in preventing the loss of water during the early ages of freshly placed concrete have been used quite extensively for many years. The U. S. Army Corps of Engineers has specification requirements for a liquid material that, when applied to the surface of concrete, forms a membrane that prevents the loss of appreciable water. Before the material may be used for a particular project, it should be tested for compliance with the following physical requirements:

<u>Property</u>	<u>Requirement</u>	<u>CRD-C Test Method¹</u>
Unit moisture loss	Must not lose more than 0.031 g of water per sq cm after 7 days exposure to a 10-mph* current of air at 100 F and 30% RH when the compound is applied at a coverage of 200 sq ft per gal	302
Sprayability	Must be sprayable at temperatures above 40 F	302
Drying time	Must be dry to touch in not more than 4 hr	311
Flash point	Must be not less than 75 F	303, 308
Reflectance	Must reflect not less than 65% of the incident infrared radiation	311

2. Conventional curing compounds vary greatly in composition but usually are resin- or wax-based materials in suitable solvents with added pigments. Pigments that are added to these compounds serve as a visual aid to the operator so that an adequate amount of material can be applied to the concrete surface. Pigments that are white enough to cause a reflection of

* A table of factors for converting British units of measurement to metric units is presented on page ix.

65% or more of the incident infrared radiation, as specified in the tabulation above, will lower the surface temperature of concrete. It has been reported that weathering of these pigmented compounds sometimes produces a mottled, unattractive concrete surface. Fugitive dyes may be used in place of pigments as a guide for application, but often the dyes do not completely fade. A need exists for colorless curing compounds for some jobs because of aesthetic reasons. The colorless material should be colorless in both fresh and weathered conditions, should leave a smooth unmottled appearance when sprayed and allowed to weather on concrete, and should be efficient in curing the concrete.

3. In addition to the conventional resin- or wax-based, pigmented or nonpigmented curing compounds, water-based liquid compounds that are purported to be suitable for curing concrete are also available commercially. These compounds do not contain petroleum or hydrocarbon distillate solvents and are safer to use than the conventional compounds in the field or in poorly ventilated areas such as tunnels and other closed areas.

4. Prior to the investigation reported herein, letters were written to several companies inquiring about the availability of both colorless and water-based compounds that might be suitable for use in the curing of concrete. As a result of the survey, 11 samples of material designated as colorless curing compounds and 2 samples designated as water-based compounds were received. These materials were identified by infrared analysis and were evaluated for moisture-retention properties, weatherability, color characteristics, stability, and other pertinent physical parameters. In addition, infrared techniques were used to detect changes of the materials upon exposure to the atmosphere.

PART II: MATERIALS, PHYSICAL TESTS, AND TEST RESULTS

Materials

5. Thirteen liquid colorless concrete curing compounds were obtained for evaluation in this study. The compounds were assigned laboratory symbols, which are CRD-CC-8 through CRD-CC-20.* Categorization of the compounds according to chemical classes and types as determined by infrared analysis, and the cost of 5 gal of each material are as follows:

<u>Sample Symbol</u> <u>No. CRD-CC-</u>	<u>Chemical Class</u>	<u>Manufacturer's</u> <u>Type</u>	<u>Cost per</u> <u>5 gal</u>
8	Chlorinated rubber	Colorless	\$15.75
9	Chlorinated rubber	Colorless	17.75
15	Chlorinated rubber	Colorless	†
16	Chlorinated rubber	Colorless	†
14	Modified styrene-butadiene	Colorless	†
18	Modified styrene-butadiene	Colorless	†
13	Phthalic alkyd	Colorless	25.00
10	Carboxylated hydrocarbon	Colorless	11.25
11	Carboxylated hydrocarbon	Colorless	12.50
12	Carboxylated hydrocarbon	Colorless	15.00
17	Styrene-acrylate	Colorless	†
19	Sodium silicate	Water-based	†
20	Sodium silicate	Water-based	13.75

† The costs of these materials are not known since they were furnished at no charge for this investigation.

General Description of Tests

6. All materials were tested according to the appropriate methods stipulated in CRD-C 300 of the Handbook for Concrete and Cement.¹ These

* For brevity, the prefix CRD will be used in this report only when its omission could possibly cause confusion. When samples are listed in a series, the CC will also be omitted for all items after the first.

tests included specific gravity, flash point, dry to touch, sprayability, drying time, and unit moisture loss. In addition, investigations involving color and color changes of each material and infrared studies were made. Details of these tests are discussed in the following paragraphs.

Moisture Retention

7. One of the essential characteristics that a membrane-forming material must possess is the ability to prevent loss of excessive water when applied to freshly placed concrete. In order to evaluate this property, the 13 materials were sprayed at a coverage of 200 sq ft/gal on freshly prepared 8-in.-diam mortar specimens, which were weighed and then placed in a cabinet controlled at 100 F and 30% RH, and subjected to a wind velocity of 10 mph for seven days. After seven days, the mortar specimens were again weighed, and the loss in weight due to moisture evaporation was calculated in grams per square centimeter. These weight losses of the specimens sprayed with the 13 curing compounds, and the calculated standard deviations (σ) are presented in the following tabulation.

Sample Symbol	Curing Compound Chemical Class	Unit Moisture Loss			
		Run 1		Run 2	
		g/cm^2	σ	g/cm^2	σ
8	Chlorinated rubber	0.021	0.007	0.018	0.004
9	Chlorinated rubber	0.015	0.002	0.018	0.004
15	Chlorinated rubber	0.017	0.003	0.020	0.003
16	Chlorinated rubber	0.023	0.003	0.031	0.002
14	Modified styrene- butadiene	0.009	0.001	0.011	0.002
18	Modified styrene- butadiene	0.029	0.017	0.014	0.001
13	Phthalic alkyd	0.029	0.011	0.031	0.008
10	Carboxylated hydrocarbon	0.014	0.009	0.007	0.006
11	Carboxylated hydrocarbon	0.026	0.005	0.030	0.007
12	Carboxylated hydrocarbon	0.009	0.005	0.007	0.003
17	Styrene-acrylate	0.064	0.009	0.052	0.005

(Continued)

Sample Symbol No. CRD-CC-	Curing Compound Chemical Class	Unit Moisture Loss			
		Run 1		Run 2	
		g/cm^2	σ	g/cm^2	σ
19	Sodium silicate	0.198	0.024	0.162	0.018
20	Sodium silicate	0.196	0.015	0.172	0.018

8. Each of the unit moisture loss values in the tabulation above is the average of five individual mortar specimens. For each material, the variances for each run were compared and found to be statistically the same except for CC-18, a modified styrene-butadiene material. The results obtained for run 1 of CC-18 are much greater for both unit moisture loss and σ than for run 2 and also than for both runs of CC-14. The results for run 1 of CC-18 were therefore discarded as erroneous.

9. The variances for each class of materials were examined and found to be homogeneous within each class. The homogeneity of variances between chemical classes was also examined, and only the carboxylated hydrocarbons and chlorinated rubbers were found to be the same. These two variances were pooled.

10. An examination of the moisture loss averages within the chemical classes revealed that the averages were statistically the same for the chlorinated rubbers, sodium silicates, and styrene-butadienes. The averages of the carboxylated hydrocarbons were not statistically the same within that chemical class. Average unit moisture losses and σ 's are shown below:

Curing Compound Chemical Class	Unit Moisture Loss (Pooled Results)	
	g/cm^2 (Average)	σ
Modified styrene-butadiene* (CC-14 and -18)	0.011	0.0014
Carboxylated hydrocarbon (CC-10 and -12)	0.009	0.0049
Chlorinated rubber (CC-8, -9, -15, and -16)	0.020	0.0049

(Continued)

* Excludes round one results of CRD-CC-18.

Curing Compound Chemical Class	Unit Moisture Loss (Pooled Results)	
	$\frac{g}{cm^2}$ (Average)	σ
Carboxylated hydrocarbon (CC-11)	0.028	0.0049
Styrene-acrylate (CC-17)	0.058	0.0073
Phthalic alkyd (CC-13)	0.030	0.0096
Sodium silicate (CC-19 and -20)	0.182	0.0190

11. An examination of the data in paragraphs 7 and 10 shows that all of the colorless compounds except one, CC-17, met the requirements of CRD-C 302 method of test for sprayability and unit moisture loss through the membrane formed by a concrete curing compound. The sample designated as CC-17 is the only one of the group that was identified by infrared spectrophotometry as a copolymer resin of the styrene-acrylate type. Both of the water-based compounds, CC-19 and -20, failed the moisture loss test, and the losses were greater by a factor of 3 than the loss by the colorless compound CC-17. The data in paragraph 10 indicate that the test precision is not a function of the test average; the coefficient of variation is not constant, but varies from 10 to 50%. The moisture loss test precision appears to be a function of the chemical class of the materials being tested.

Flash Point, Sprayability, Specific Gravity,
Dry to Touch, and Loss of Volatiles

12. Tests, in addition to moisture retention, currently used to evaluate pigmented curing compounds and applicable to colorless or water-based compounds are flash point, sprayability, specific gravity, and dry to touch. The results of these tests plus the percent volatiles lost in the controlled temperature cabinet (100 F) are listed below:

Sample Symbol No. <u>CRD-CC-</u>	Flash Point °F	Sprayable at 40 F	Specific Gravity	Dry to Touch hr	Percent Volatiles Lost at 100 F
8	83	Yes	0.950	1	77
9	80	Yes	1.018	1	56
15	95	Yes	1.000	1-1/2	62
16	100	Yes	0.947	1-1/2	73
14	100	Yes	0.914	1-1/2	47
18	103	Yes	0.846	2-1/2	69
13	78	Yes	0.924	1	77
10	100	Yes	0.796	1-1/2	74
11	105	Yes	0.827	1-1/2	62
12	103	Yes	0.828	2	66
17	102	Yes	0.875	2	79
19	*	Yes	1.285	4	57
20	*	Yes	1.289	4	57

* Not applicable.

13. Each of the values reported in the tabulation above is the average of two results that in all instances were the same or very close. All of the compounds met applicable specification requirements. The percent volatiles gives an indication of the amount of solvent in the material.

14. Correlation coefficients were calculated for the moisture loss results and the physical data in paragraph 12. The coefficients indicated the absence of good correlation between results of any of these tests and moisture loss. Also, there were no clear distinctions of any of the physical tests according to chemical classes. However, the specific gravities of the chlorinated rubber materials (CC-8, -9, -15, and -16) were greater than those of the other materials designated as colorless, but the fluctuation within the chlorinated rubber group was wide, being 0.071. Physical test data for both water-based compounds (CC-19 and -20) were in good

agreement with each other, but the specific gravities and dry-to-touch results were significantly different from those of the colorless compounds.

Color and Weatherometer Tests

15. If colorless curing compounds are to be used when pigmented compounds would be unsuitable, it is necessary to evaluate the colorless materials for their lack of color. Therefore, several tests were made on the 13 materials to determine the color of the as-received materials and the change in color of the materials after exposure under conditions of natural daylight, controlled humidity and temperature, and ultraviolet light and rainfall in a weatherometer.

16. All of the as-received materials designated as colorless possessed some color ranging from very clear light yellows to cloudy shades of brown and red, whereas the two as-received water-based materials actually possessed less color than the colorless compounds, being colorless with a slight cloudiness. The colors of all these materials were noted visually and by using a color comparator upon receipt and after exposure to laboratory conditions plus some natural daylight for 28 days. The comparator selected for use with the curing compounds was one normally used for varnishes, with the range and steps of standards from 1 to 18. As the numbers on this comparator increase from 1 to 18, the colors become darker. The colors of the materials (from visual observations and using the comparator) are noted below:

Sample Symbol No.	Color Based on Visual Observations of As-Received Material	Color Comparator Readings for As-Received Material and at 7-, 14-, 21-, and 28-day Ages					Change from 0 to 28 Days
		As Received	7	14	21	28	
8	Cloudy, yellow	6	6	6	6	7	+1
9	Cloudy, yellow	5	5	6	6	7	+2
15	Clear, yellow	6	6	7	8	9	+3
16	Slightly cloudy, yellow	5	5	5	6	7	+2
14	Dark, reddish-brown	14	14	14	14	14	0

(Continued)

Sample Symbol No.	Color Based on Visual Observations of As-Received Material	Color Comparator Readings for As-Received Material and at 7-, 14-, 21-, and 28-day Ages					Change from 0 to 28 Days
		As Received	7	14	21	28	
CRD-CC-18	Slightly cloudy, golden brown	11	11	11	11	11	0
13	Slightly cloudy, brown	10	10	11	11	11	+1
10	Cloudy, tan	10	11	11	9	9	-1
11	Cloudy, yellow	6	5	4	4	4	-2
12	Cloudy, red	18	17	18	12	12	-6
17	Clear, light yellow	1	1	1	1	1	0
19	Slightly cloudy, neutral	1	1	1	1	1	0
20	Slightly cloudy, neutral	1	1	1	1	1	0

17. An examination of the data above shows that the colors of the materials include the entire range of the comparator, 1 through 18. Five of the materials, CC-14, -18, -17, -19, and -20, showed no change upon exposure to light. Two of these were modified styrene-butadienes; one was a styrene-acrylate; and two were sodium silicates. There were five materials, CC-8, -9, -15, -16, and -13, that slowly darkened with age. Four of these materials were chlorinated rubbers; the other material was a phthalic alkyd. The remaining three materials, CC-10, -11, and -12, lightened upon aging. These were the carboxylated hydrocarbons. CC-12 may contain a fugitive dye, although no tests were conducted to determine the presence of a dye.

18. Since one of the purposes for using colorless curing compounds is the need or desire for lack of color on some concrete surfaces, an investigation was conducted to determine the color and color change imparted to mortar specimens by coating them with the compounds included in the program. Two groups of tests involving coated mortar specimens were conducted and are described in the following paragraphs.

19. In the first series of tests, mortar specimens were prepared and sprayed with each of the 13 materials in accordance with the methods outlined in CRD-C 302 in the Handbook for Concrete and Cement¹ except

that the mortar surfaces were troweled rather than broomed before spraying. These specimens were stored for 28 days in the same controlled temperature-humidity cabinet as used for the moisture-loss specimens. At ages of 1, 7, 14, 21, and 28 days, the specimens were removed from the cabinet and reflectance readings were taken using the infrared, amber, blue, and green filters with a multipurpose reflectometer. The readings were taken as directed by the instrument manufacturer, standardizing the instrument with reference standards of MgO with the appropriate filter prior to taking readings on test specimens. Initially, two series of reflectance readings were taken for each specimen using two different reference standards. Since there was little difference in the two groups of readings, the standard that was selected for obtaining reflectance readings for all the specimens was one used for obtaining reflectance readings for white-pigmented curing compounds. The reflectometer was therefore standardized with MgO standard with the following settings: infrared, 68; amber, 63.6; blue, 64.8; and green, 63.9. Reflectance readings of the coated mortar specimens did not vary appreciably when standardizing the reflectometer with infrared, amber, or green filters. Readings obtained with the blue filter were appreciably lower for several of the specimens and too low (<9) on others. Therefore, only readings obtained using the infrared filter are reported herein. The duplicate tests of these coated mortar specimens varied in their reproducibility, dependent upon the variation in mortars, uniformity of the compound coverage, and uniformity of drying of the mortar as well as changes that occurred in the compound.

20. Initially the reflectance readings were converted into tristimulus color scale values, and the color differences were calculated from color scale values of the reference and test specimens according to ASTM Test Method D 2244-64T.² These calculations were discontinued because they provided no significant information in addition to that already revealed by the unconverted reflectance readings. Since the reflectance readings increased upon aging, the calculated color difference became smaller, which indicates only that the difference between the reference specimen and the test specimen decreased with age. The average reflectances and changes in reflectances of the 2 coated mortar specimens for

each of the 13 compounds after storage in the controlled temperature-humidity cabinet from 1 to 28 days are shown in the following tabulation.

Sample Symbol No. <u>CRD-CC-</u>	Average of Two Reflectance Readings at 1-, 7-, 14-, 21-, and 28-day Ages, %					Change from 1 to 28 Days, %
	<u>1</u>	<u>7</u>	<u>14</u>	<u>21</u>	<u>28</u>	
8	12.1	19.8	23.0	25.9	28.6	16.5
9	14.7	19.1	22.3	25.2	26.6	11.9
15	13.2	16.9	19.8	22.0	23.6	10.4
16	13.8	19.1	22.5	24.7	26.8	13.0
14	9.9	10.5	11.1	12.6	14.8	4.9
18	10.8	15.5	18.8	22.3	24.2	13.4
13	15.8	20.1	21.8	11.7	23.4	7.6
10	11.3	14.8	17.6	19.2	20.4	9.1
11	11.0	11.6	11.3	11.8	11.5	0.5
12	14.5	17.4	17.8	18.6	19.1	4.6
17	23.3	33.9	37.4	39.3	40.6	17.3
19	19.7	21.0	21.8	22.5	23.0	3.3
20	19.5	20.4	20.6	20.8	20.6	1.1

21. As can be seen in the tabulation above, all of the mortar specimens coated with the curing compounds lightened during the period of 1 to 28 days storage in the controlled temperature-humidity cabinet. Generally, the greatest color change occurred between 1 and 7 days, with a more gradual lightening thereafter. The specimen coated with sample CC-11 evinced very little change (0.5) for the entire 28-day period, and the two specimens coated with the water-based materials showed a small increase in readings (1.1 and 3.3). The reflectance changes of the remainder of the specimens ranged approximately from 5 to 17, with the average change of the CC-17 specimen being the greatest.

22. The reflectometer readings noted in paragraph 20 show only the changes that occurred when the coated mortar specimens were exposed to controlled temperature and humidity with no ultraviolet radiation. Therefore,

the second group of tests was made to detect color changes, degradation, and/or other chemical-physical phenomena of the curing compounds when subjected to artificial weathering.

23. In the first series of this group of tests, small, round, empty metal molds $4\text{-}\frac{3}{8}$ in. in diameter were sprayed at a coverage rate of 200 sq ft/gal with each of the 13 materials being investigated. In addition, for purposes of comparison, a blank mold and a mold sprayed with white-pigmented carboxylated hydrocarbon curing compound were prepared in a like manner. In the second series, similar metal molds were filled with mortar and sprayed with each of the colorless and water-based materials as described in paragraph 19. One mortar specimen with no compound and one sprayed with a white-pigmented curing compound were also prepared. After 24 hr curing in the controlled temperature-humidity cabinet, specimens from both test series were exposed in a weatherometer that accommodated fifteen specimens in circular horizontal disks that slowly rotated during the weathering process.

24. For tests involving the coated metal molds with no mortar, the weatherometer was operated for 112 hr with no simulated rainfall and a single carbon-arc lamp as the source of ultraviolet radiation. After 112 hr, the molds were subjected to an additional 24 hr of weathering consisting of 102-min cycles of ultraviolet radiation alternated with 18 min of rainfall. A maximum temperature of 165 F was attained during these tests. The specimens were removed after periods of 8 or 16 hr up to a total exposure of 112 hr, then after 24 hr of alternating ultraviolet radiation and rainfall, and observed for evidences of film deterioration and changes in color using the reflectometer. The reflectance readings showed a general increase for all the coated plain molds except for the two coated with the carboxylated hydrocarbons, CC-10 and -12, throughout 112 hr of ultraviolet radiation. After 24 hr of alternating ultraviolet radiation and rainfall, the reflectance readings varied somewhat--some showing no change, some an increase, and some a decrease. This wide variation is primarily due to different degrees of washing of curing compound materials and resulting splotched effects. After only 8 hr of ultraviolet exposure, compounds CC-19 and -20 showed signs of cracking, dryness, and flakiness.

The chlorinated rubbers, CC-8, -9, -15, and -16, began to show signs of splotching at 80 hr, and small hairline cracks appeared at 112 hr. The styrene-butadienes, CC-14 and -18, showed signs of crazing and wrinkling at 16 hr. The visible change of the remaining colorless materials was gradual and appeared to be less than that of the materials cited above. The white-pigmented material adhered well during all the tests.

25. Examination of the coated mortar specimens subjected to the weatherometer was of greater interest in this investigation since the same changes can be expected under field conditions. Therefore, these specimens were tested as described in paragraphs 23 and 24, and, in addition, the weatherometer tests with alternate cycling of simulated sunshine and rainfall were continued for a total of 304 hr. Reflectometer readings using the infrared filter are shown in table 1.

26. Table 1 shows that generally the reflectance values of the coated mortar specimens increased with exposure in the weatherometer, which indicates a lightening of the surface of the specimens. The same chemical class of compounds appeared to react or change in a similar manner. The mortar specimens coated with chlorinated rubber (CC-8, -9, -15, and -16) showed a continuous increase in reflectometer readings up to about 48 hr, then a leveling off, showing practically no further change until they were subjected to the alternate cycles of rainfall and simulated sunshine at 112 hr. At this time, there was a sharp decrease in reflectance readings and then irregular readings until the test was terminated at 304 hr. Reflectance readings of the mortar specimen coated with styrene-acrylate, CC-17, followed the same pattern as those of the chlorinated rubber specimens except that the readings stabilized somewhat earlier (at about 24 hr). The reflectance readings of the specimens coated with the remaining colorless materials (CC-14, -18, -13, -10, -11, and -12) stabilized at about 24 hr, then leveled off until the specimens were subjected to rainfall, at which time there was a decided increase in the readings. The specimens coated with the water-based materials (CC-19 and -20) evinced little change in reflectance readings until the specimens were subjected to rainfall, at which time there was an increase in readings. The uncoated and white-pigmented specimens showed an increase in

reflectance readings after subjection to rainfall. Plots of reflectance readings versus hours exposure in the weatherometer for three typical specimens of the various classes (CC-8, -14, and -20) are shown in plate 1.

27. The coated mortar specimens were examined each time reflectance readings were obtained. Again, generally, materials of the same chemical class responded to weathering in a similar manner. General comments on the weathered coated mortar specimens after continuous ultraviolet exposure for 112 hr and after an additional 192 hr of alternating ultraviolet and rainfall exposure are presented in table 2.

28. This table indicates that weathering definitely affected the appearance of mortar coated with these curing compounds. The coated mortar surfaces responded in diverse ways to the simulated weathering--some improving in appearance after ultraviolet radiation and then developing a mottled unattractive appearance after subjection to rainfall, some being mottled and splotched after ultraviolet radiation but greatly improving upon exposure to rainfall, and others displaying little change throughout the testing. At first, the simulated rainfall generally lightened the coated surfaces and tended to wash off the curing compound, thus causing an unevenness and mottling or spotting effect. With additional rainfall, the unevenness and discolorations gradually washed away for most of the coated surfaces. In attempting to evaluate each material for suitability for use as a curing compound based on general aesthetic qualities or appearance of concrete surfaces cured with the material, several factors such as adherence of the material to the mortar surface, discolorations imparted to the surface, scaling, curing conditions, etc., must be considered in deciding whether a material would be acceptable.

29. Photographs were taken of all coated mortar specimens before subjection to the weatherometer, after 112 hr exposure to ultraviolet radiation, and after a total of 304 hr exposure (including 112 hr of continuous ultraviolet exposure plus 192 hr of ultraviolet radiation and rainfall cycles). Some of the photographs typical of the various chemical classes are shown in photographs 1 through 4.

30. Mortar surfaces coated with the chlorinated rubbers (CC-8, -9,

-15, -16), styrene-acrylate (CC-17), two of the carboxylated hydrocarbons (CC-10 and -12), and both of the water-based materials (CC-19 and -20) were aesthetically pleasing to the eye after subjection to ultraviolet radiation. However, definite surface changes were noted on some of the specimens after subjection to rainfall alternated with ultraviolet radiation. The chlorinated rubber specimens appeared unattractive due to glossy, brownish, mottled effects. Surfaces coated with materials CC-8 and -9 were not as dark and showed a less mottled effect than did the surfaces coated with CC-15 and -16. The surfaces coated with CC-10, -12, -17, -19, and -20 showed little change upon being subjected to rainfall. The hydrocarbons, CC-10 and -12, appeared to impart less color to the weathered mortar specimens than the other colorless materials and resulted in a mortar with the original surface exposed and showing less deleterious effects or objectionable stains. Specimens coated with one of the styrene-butadienes, CC-14, and the phthalic alkyd, CC-13, were judged visually as possessing the most mottled and unattractive surfaces under both conditions of weathering. The white-pigmented coated mortar surface remained white and showed no mottling or unevenness throughout the weatherometer tests.

31. The chlorinated rubbers and the styrene-acrylate appeared to adhere much more tenaciously to the mortar than the other colorless materials investigated. Therefore, these materials would require more weathering and washing to be completely removed than the other colorless materials investigated. This adhesive property may be objectionable because of the long-lasting mottled appearance of the concrete or mortar surface. The tenacious adherence of the white-pigmented material was similar to that of the chlorinated rubbers and styrene-acrylate; however, the appearance of the surface upon weathering can be considered objectionable only because of the white pigment and not because of discolorations or mottling effects.

32. The two nonpigmented carboxylated hydrocarbons (C-10 and -12) and both water-based materials (C-19 and -20) were the only curing compounds in this investigation that imparted a dull finish to the mortar surfaces; the remaining surfaces were glossy initially. The dull finish may be desirable when considering the appearance of the finished mortar.

33. The colors as noted initially for the coated mortar specimens

subjected to both the controlled temperature-humidity cabinet and the weatherometer ranged through shades of clear amber, gray, and green. Readings on the reflectometer were influenced by mortar preparation, uniformity of curing compound coverage, amount of gloss or color of the material, and drying of the mortar upon aging and exposure as well as changes that occurred within the compounds themselves. None of the mortar surfaces were colored sufficiently to be objectionable prior to weathering; however, the dark, mottled appearance produced by weathering for some of the materials would be considered objectionable if the mottled appearance lasted over unduly long periods of time.

PART III: INFRARED TESTS

Identification

34. The 13 materials evaluated in this investigation were examined and identified using a double-beam infrared spectrophotometer. Infrared spectra were obtained in the 2.5-16 μ region for each of the materials. Samples of the colorless curing compounds were prepared for infrared analysis by gently pressing the well-mixed, as-received material between sodium chloride (NaCl) crystals separated by a 0.015-mm spacer and then placing this cell in a demountable cell holder. In addition, thin films of the materials were cast on NaCl crystals and allowed to dry at 40 C for 2 to 5 days before spectra were obtained. The type of evaporable solvent in each material could then be determined by comparing the spectra obtained from these two techniques. These spectra are shown in plates 2-12.

35. It was difficult to obtain suitable infrared spectra for the two water-based compounds. Films were cast on a sheet of teflon and on IRTRAN-2 crystals. Films sufficiently thin were unobtainable for infrared measurements by this technique. Finally, by diluting the samples with water and placing the material between IRTRAN-2 crystals separated by a 0.015-mm spacer and using an attenuator in the spectrophotometer, measurable spectra were obtained. These spectra are shown in plates 13 and 14.

36. Each of the materials in this investigation was composed of several constituents including mixtures of resins, oils, solvents, and modifying agents. No attempt was made to identify all the individual additives positively. However, the major chemical components of each of the compounds were identified, and it was possible to detect differences within the same chemical classes due to different additives. Identification was made by a study of the functional groups as revealed by the spectra and by a comparison of the spectra with reference spectra of known materials. A study of the infrared spectra of these materials revealed that they represent six distinct types. Information obtained by a study of the infrared spectra of the materials is discussed in the following paragraphs.

CC-8 and -9

37. Infrared spectra of the dried films of these two compounds are characteristic of chlorinated rubber (plates 2 and 3). The presence of strong aliphatic C-H absorptions is noted at 3.38, 3.42, 3.49, 6.9, 7.0, 7.25, and 13.5-13.7 μ . The strong bands around 3.4-3.5 μ are ascribed to both CH₃ and CH₂ groups involving both asymmetrical and symmetrical stretching modes and in-phase and out-of-phase vibrations of the hydrogen atom. The strong bands around 6.9-7 μ are ascribed to asymmetrical CH₃ stretching and deformation of CH₂ and vinyl groups. The moderately sharp absorption at 7.25 μ is due to symmetrical stretching of C-CH₃ groups, and the strong diffuse absorptions around 13.7 μ are characteristic for the presence of long-chain hydrocarbons of the type (CH₂)_n (when n is four or greater) plus C-Cl stretching. The strong broad band around 7.9 μ is characteristic of the CHCl group vibration in chlorinated rubber, and additional C-Cl stretching vibrations are noted around 13 and 15 μ . The remaining moderately strong diffuse band at 10.7 μ is ascribed to olefin C-H wags. Although the CC-8 and -9 compounds have identical distinct features, there are some differences in their dried films that should be noted. The presence of aromatic groups is evident at 3.32, 6.2, and 6.7 μ in both materials but is much more pronounced in CC-9 (plate 3), indicating that the aromatic solvent in CC-8 evaporates much more rapidly than that in CC-9.

38. The spectra of the as-received CC-8 and -9 materials are practically identical (plates 2 and 3), but are quite different from the dried film spectra. The spectra of the as-received material contain both aromatic (3.3, 6.2, and 6.7 μ) and aliphatic (3.4, 3.5, 6.9, and 7.3 μ) constituents and represent chiefly the volatile portions of the compound. The volatiles or solvents appear to be a mixture and include the ortho-, meta-, and para-xylenes and other high-solvency naphthas.

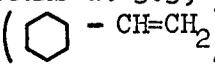
CC-15 and -16

39. The spectra of these two compounds (plates 4 and 5) are identical, and the compounds are identified as chlorinated rubber resins, but there are several features that distinguish them from the other two chlorinated rubbers, CC-8 and -9, discussed in paragraphs 37 and 38. The spectra of the dry films (plates 4 and 5) show less evidence of aromatic groups,

and the strongest absorptions are noted at 3.4, 3.5, 7.0, 7.25, and 13.7 μ , which are due to C-H deformations described in paragraph 37. The characteristic absorptions due to chlorinated rubber are evident at 8 and 15.2 μ . Also, the spectra of the dry films of these chlorinated rubbers show the presence of moderate bands at 5.8 μ , which are ascribed to carbonyl (C=O) groups. These absorptions may be due to tall oil or other fatty acid groups in the resinous compounds. Other differences noted between these spectra and the spectra of CC-8 and -9 in the region 10-16 μ are attributed to degree of chlorination and saturation of the rubber.

40. Spectra of as-received compounds CC-15 and -16 appear to be more representative of the solvents in the material than the nonvolatile constituents. The solvents appear to be a mixture containing both aromatic and aliphatic groups. However, based on the relative intensities of these groups, these materials contain more long-chain hydrocarbon groups and fewer aromatic groups than CC-8 and CC-9. Actually, all four chlorinated rubbers appear to contain essentially the same kinds of solvents, but the proportions are different.

CC-14 and -18

41. These two materials have been identified as modified styrene-butadiene resins. The spectra of the dried films (plates 6 and 7) are identical, and only minor differences are noted in the spectra of the as-received materials. Evidence of aromatic hydrocarbons is shown by the presence of moderate sharp peaks at 3.3, 6.25, and 6.7 μ , which are ascribed to the styrene group (). The strong band at 13.4 μ is ascribed to out-of-phase CH bonding of the monosubstituted aromatic, while the band at 14.35 μ is specific for the monosubstituted phenyl group. Strong aliphatic CH absorptions are noted at 3.4-3.5 μ . The presence of CH₃ and CH₂ groups is apparent by the strong absorption at 6.9 μ , and the absorption at 7.25 μ is ascribed to C-H stretching and bonding modes in methyl groups. The absorption band at 10.3 μ arises from the presence of trans 1:4 disubstituted ethylene addition in the butadiene. Very strong absorptions are evident at 3.0 and 5.9 μ , and are attributed to OH bonding and C=O bonding, respectively. These absorptions are attributed

primarily to oxidation products of the films, since it was noted that the 3.0 and 5.9 μ bands got progressively stronger as the films were allowed to cure in the laboratory, and since these bands were not detected in the as-received material.

42. The spectra of as-received materials CC-14 and -18 show the presence of olefinic, aromatic, and aliphatic hydrocarbons similar to those in the dry material; however, several differences are apparent that allow for obtaining additional information on the types of volatiles present. Comparison of the spectra shows that the as-received CC-14 apparently contains more aromatic constituents than the as-received CC-18 as evidenced by relative intensities at 3.3, 6.2, 6.7, 13.0, 13.5, and 14.3 μ . The absorption bands present in both spectra of these compounds are characteristic for petroleum fractions of naphtha or mineral spirits, xylene, or other coal-tar derivatives.

CC-13

43. This material has been identified by infrared spectra (plate 8) as an oil-modified O-phthalate alkyd resin. Strong absorptions of the dry film spectrum at 3.27, 3.3, 3.4, and 3.5 μ indicate a mixture of olefinic, aromatic, and aliphatic constituents. The very strong band at 5.8 μ is attributed to C=O and is due primarily to C=O bonding on the phenyl ring. The strong band in the 7.8-7.9 μ region is ascribed to the presence of C-O groups contiguous to the carbonyl group, while the bands at 8.9 and 9.3 μ are characteristic of C-O linking. The moderately strong band at 2.9 μ indicates OH bonding, which may be due to partially reacted polyhydric alcohol or fatty acid groups. Characteristic absorptions for the ring structure of phthalates are noted in the two fairly weak but sharp bands at 6.24 and 6.32 μ . The strong absorption at 13.5 μ is characteristic of the ring structure of phthalates; the strong absorption at 14.3 μ is due to either a monosubstituted or ortho-disubstituted aromatic ring. Additional evidence of aliphatic groups is noted at 6.9 μ , which includes CH vibrations of both CH₃ and CH₂ groups, and at 7.25 μ , which is characteristic of CH bonding and stretching modes in the CH₃ group. Comparison of the as-received spectrum with the dry film spectrum of CC-13 (plate 8) indicates that the volatile portion of the material is

primarily a mixture of ortho-, meta-, and para-xylenes.

CC-17

44. The spectra (plate 9) of this material are distinct for styrene-acrylic copolymers. The strong bands of both polymers are evident in the dried film. The bands at 3.24, 3.26, 3.30, 6.25, 6.70, 9.73, 13.3, and 14.4 μ are characteristic for phenyl groups found in styrene. The distinguishing feature of the acrylic portion of this copolymer is noted at 5.78 μ , which is quite strong and sharp, and is characteristic for C=O found in acrylic esters. Also, the strong band at 8.7 μ with a weaker absorption band at 8.0 μ is attributed to the C-O-C stretching mode characteristic of acrylics. The presence of long-chain molecules containing CH₃ and CH₂ groups is evident at 3.38, 3.43, 3.51, 6.9, and 7.27 μ .

45. The spectrum of the as-received material includes several bands due to solvent that are absent in the dried film. These bands are noted at 11.4, 12.0, 12.4, 13.0, 13.3, and 13.5 μ and are attributed primarily to a mixture of high-solvency naphthas of high aromatic hydrocarbon content including a mixture of the xylenes. However, the presence of aliphatic solvents is also quite evident by the very strong absorption in the 3.4-3.5 μ region, which partially obscures the aromatic absorptions around 3.3 μ . Additional aliphatic absorptions due to C-H vibrations of the methyl and methylene groups are present at 6.9 and 7.25 μ in the as-received material as well as in the dried film.

CC-11

46. The spectrum of this curing compound (plate 10) indicates that the material is primarily a mixture of carboxylated hydrocarbons of the butadiene type. The strong sharp absorptions noted at 3.42 and 3.50 μ are typical for C-H stretching modes in aliphatic materials. The positions of these bands and the presence of a strong band at 6.9 μ with a much weaker band at 7.25 μ (C-CH₃ linkage) indicate that the CH₂ group is more prevalent than the CH₃ groups. The medium absorption at 13.85 μ is typical for skeletal vibrations involving long-chain CH₂ groups and arises from a structural group rather than from a single unit. The other prominent features of the spectra of CC-11 arise from carboxylic groups. The strong band at 5.9 μ is characteristic of C=O vibrations found in

carboxylic acids or ester resins, while the medium-strong band at 8.0μ and weaker band at 8.5μ indicate C-O stretching vibrations within the carboxyl group. The strong absorptions at 6.5 and 7.1μ are typical of carboxylate absorptions found in resins. Since the presence of both free carbonyl groups (5.9μ) and carboxylate groups (6.5μ) is prominent in the spectra, it is deduced that some of the fatty oils used in the manufacture of the material have been partially reacted--probably with lime--and some are unreacted in the mixture. Hydroxyl bonding is strong as shown by a strong absorption around 3.0μ and may be due to OH stretching vibrations of the carboxylic group. Although the strong aliphatic absorptions in the region around 3.4 - 3.5μ may obscure any absorptions near this region due to aromatic or unsaturated constituents, a study of the dried film spectrum shows definite proof of unsaturated or alkene groups by the medium and weak bands at 10.3 and 11.0μ , respectively. The 10.3μ absorption is specific for trans structure ($-\text{CH}=\text{CH}-$) and is due to hydrogen atoms that are out of plane at the double bond.³

47. The spectrum (plate 10) of as-received material CC-11 is quite similar to that of the dried film, and the same strong bands are present. Vibrations due to the methyl group at 7.25μ are more evident in the as-received spectrum, while the region from 7.5 - 16μ is more diffuse. These comparisons indicate that the volatiles are long-chain simple hydrocarbons similar to mineral spirits or high-solvency naphthas.

CC-12

48. This material exhibits several prominent infrared bands (plate 11) similar to those of CC-11; however, there are distinct differences in the spectra that allow for definite differentiation between the materials. The common bands of CC-12 and -11 are found in the 3.4 - 3.5μ region and at 5.9 , 6.5 , 6.9 , 7.25 , 8.0 , 8.5 , 10.3 , 11.0 , and 13.9μ . The functional groups causing each of these absorptions are the same as those described for CC-11 in paragraph 46. The chief difference between the spectra of CC-12 and -11 is the presence of aromatic constituents in CC-12 as noted in the 3.3μ region and at 6.25 , 6.7 , 13.4 , and 14.3μ . The strong absorptions at 13.4 and 14.3μ are characteristic of monosubstituted phenyl groups, or more specifically of styrene. Doublet absorptions at 13.7

and 13.9μ occur for crystalline hydrocarbons of the ethylene type. The doubling of this bond in the crystalline material arises from interactions between neighboring molecules in the crystalline phase.³ This phenomenon is usually encountered if materials are crystalline in ethylene/ester copolymers and in esters of long-chain aliphatic acids. This material, CC-12, is classified in this study as being a carboxylated butadiene-styrene hydrocarbon.

49. There are no important differences noted between the spectra of the dried film and the as-received material for CC-12. The intensities of the bands changed due to volatiles, and the slight differences suggest that the chief solvent is naphtha.

CC-10

50. This material is identified as being a carboxylated ethylene material, and the spectra (plate 12) of the material are quite similar to those of the other carboxylated hydrocarbons, CC-11 and -12. Again, however, distinct differences in the spectra are noted among these materials. The strongest absorptions are noted in the $3.4-3.5\mu$ region, indicating aliphatic C-H stretching vibrations. The presence of both methyl and methylene groups is evident by the strong sharp absorption at 6.85μ , whereas the medium sharp band at 7.25μ indicates only methyl deformation vibrations that are more evident in CC-10 than in the other two carboxylated hydrocarbons. Bands at 5.9 , 6.5 , 7.8 , 7.9 , 10.3 , and 11.0μ are ascribed to the same functional groups as described in paragraphs 46 and 47 for CC-11. No indications of aromatic substances are present in the dried-film spectrum. The chief difference between CC-10 and CC-11 is the presence of the doublet at 13.7 and 13.9μ in CC-10, whereas only one absorption at 13.9μ is evident for CC-11. This clear doublet is similar to that of the CC-12 spectra and indicates the presence of ethylene groups in the crystalline state. Other minor differences between spectra of these materials are shown in relative intensities of the remaining bands, which indicate more of the ester-type material (probably in the form of oils) in the CC-11 and more ethylene (probably crystalline wax) in the CC-10 material.

51. The spectrum (plate 12) of the as-received material of CC-10 indicates that the material is primarily made of aliphatic hydrocarbons as

shown by absorptions at 3.4, 3.5, 6.85, 7.25, and 13.9 μ . Ester groups are evident, but in relatively small amounts. In addition, the presence of substituted aromatic groups is noted. A comparison of the dried-film spectrum with the as-received spectrum indicates the solvent to be primarily long-chain aliphatic hydrocarbons of the naphtha type, with fewer aromatic groups.

CC-19 and -20

52. These two materials have been identified by their infrared spectra (plates 13 and 14) primarily as water solutions of sodium silicate. The spectra of the materials are relatively simple. The strongest absorptions are in the 2.8-3.3 μ range and at 6.2 μ , are quite broad, and are attributed to unbonded hydroxyl stretching vibrations. It is possible that the materials contain other constituents that may absorb in these hydroxyl regions but would not be detectable due to the diffuseness of the bands. The medium-broad band in the 4.7-4.8 μ region is ascribed to Si-H stretching vibrations, while the less intense bands around 9 and 9.8 μ may be due to Si-O-Si linkage.

Exposure Tests

53. Physical and chemical changes occur in liquid resin or waxy materials when these materials are exposed to the atmosphere or light. These changes may be due to evaporation of the solvent and to actual chemical and/or physical changes occurring within the material such as cross-linking, degradation, or oxidation reactions. A comparison of the infrared spectra of the as-received material and the 1-day films gives an indication of the type of solvent, and these changes are described earlier in this report. Chemical changes produced by photooxidation are sometimes detected by infrared spectrophotometry in the regions around 3 and 5.9 μ . The change at 3 μ is characteristic of stretching of hydroxyl, and the change at 5.9 μ is characteristic of carbonyl vibrations. Therefore, in order to observe these phenomena, films of the colorless curing compounds were cast on NaCl crystals, and these crystals were stored at laboratory conditions with about 8 hr per day of natural sunlight exposure

for 28 days. At 1 and 28 days, infrared spectra were obtained between 2.5 and 16 μ . In addition, spectra of the regions around 3 and 6 μ were obtained at 4, 7, 14, and 21 days. The spectra of the materials at the various ages were studied to detect changes and rates of changes that occurred. The infrared spectra revealed that each material within a general type or class evinced similar changes upon exposure, but, as expected, the variation between classes was great.

54. The transmission values of the hydroxyl and carbonyl absorption bands were calculated for each colorless compound at 1, 4, 7, 14, 21, and 28 days. In an effort to determine quantitatively any changes that occurred in the hydroxyl and carbonyl regions during the 28-day exposure, the transmission values (which are not additive) were translated to absorbance values by means of the following equation:

$$A = -\log \frac{I}{I_0} = -\log T = \log \frac{1}{T}$$

where

A = absorbance

I = intensity of the transmitted radiation

I₀ = intensity of the incident radiation

T = transmission

The calculated absorbance values are given in table 3.

55. A study of the hydroxyl and carbonyl regions at various ages revealed the following facts: (a) very little or no significant changes were observed for the two chlorinated rubbers, CC-8 and -9, or the styrene-acrylate, CC-17; (b) the two chlorinated rubbers modified with additional aliphatic constituents, CC-15 and -16, showed slight irregular changes of the hydroxyl band and a slight increase of the carbonyl band; (c) only slight gradual increases at both wavelengths were detected for the phthalic alkyd, CC-13; (d) both styrene-butadiene resins, CC-14 and -18, evinced the greatest increase of hydroxyl and carbonyl bands of any of the materials investigated; and (e) the remaining materials, CC-10, -11, and -12, designated as carboxylated hydrocarbons, showed definite increases of both bands over a 28-day period, but the rates of changes were rather erratic.

56. A study of the absorbance values with reference to age or exposure reveals some irregularities. These irregularities may be due to the less-than-ideal transmission ranges obtained from some of the films (ideally transmission should be within the range from 25 to 65%),⁴ variation of film thickness among the various materials and within the same film, and exposure of the films to uncontrolled humidity in the laboratory. The humidity in the laboratory varied widely while the films were being exposed, and certainly this factor may have greatly influenced the intensities of the hydroxyl and carbonyl absorptions.

57. The cast films of the materials that displayed definite changes as evidenced by the infrared spectra and that showed transmission values within an acceptable range for making semiquantitative estimations were noted for only the two styrene-butadiene resins and the three carboxylated hydrocarbons. Changes in absorbances of the hydroxyl band at 3μ and the carbonyl band at 5.9μ for these materials from 1 to 28 days are plotted in plates 15 and 16, respectively. Some of the plotted curves are smoothed out to represent what would probably take place if the laboratory humidity were controlled. These graphs show that upon exposure to the atmosphere, there is an increase of both hydroxyl and carbonyl groups from 1 to 28 days for the two styrene-butadiene resins. This increase is quite rapid for the first few days and slows down considerably after 14 days.

58. Irregularities are more evident for the hydroxyl and carbonyl absorbances of the carboxylated hydrocarbons. However, the curves for the two materials designated as carboxylated butadiene-styrene, CC-12, and carboxylated ethylene wax, CC-10, show generally increased hydroxyl and carbonyl groups with increased exposure to the air, but the CC-11 material shows inconsistent intensities of hydroxyl groups upon exposure while the carbonyl group shows a smooth steady increase for 14 days, after which the increase tends to level off.

59. The noted changes at 3 and 5.9μ with respect to time are believed to be due to chemical changes as a result of exposure. There appear to be two competing reactions taking place with the formation of hydroperoxide groups.⁵ These reactions are probably due to the drying oils in the compounds. These reactions and observations may prove

valuable for some materials in correlation of reactions with actual performance of a material. It is difficult to find any correlation between moisture loss results or any of the other physical tests performed in this study and the infrared spectra obtained on the exposed films. However, the moisture loss results were found to be dependent upon the chemical class of the curing material. With the exception of the carboxylated hydrocarbons, no differences between compounds were found in any chemical class for moisture loss, color change, or chemical change as indicated by infrared spectra study. The following differences were noted between the carboxylated hydrocarbons: CC-11 moisture loss is significantly higher than that of CC-10 or -12 (the moisture loss of these two is statistically the same); the color comparator reading (see paragraph 16) of CC-11 is significantly lower than that of CC-10 or -12; the color change of CC-11 on mortar is significantly less than that of CC-10 or -12; mortar coated with CC-10 or CC-12 shows less mottling effects upon weathering than that coated with CC-11; and finally, the chemical changes on exposure were found to be considerably greater for mortar coated with CC-11 than for mortar coated with CC-10 or -12.

Conclusions

60. Ten of the eleven colorless curing compounds evaluated during this investigation met requirements of CRD-C 300 in the Handbook for Concrete and Cement¹ for moisture loss, sprayability, flash point, and dry to touch. The colorless styrene-acrylate and both water-based materials, which were also colorless, failed the moisture loss test but met other applicable requirements.

61. The colors of the as-received materials varied from clear light yellow to cloudy dark reddish-brown; however, it did not appear to make any appreciable difference what the initial color values were for the materials in this investigation since there was no significant color difference among the mortar specimens sprayed with the lighter and darker materials prior to exposure or weathering of the specimens. All the surfaces of the coated mortar specimens except that coated with CC-11, a carboxylated hydrocarbon, lightened upon exposure to ultraviolet radiation.

62. Based on general appearance after the coated mortar specimens had been subjected to conditions of weathering (ultraviolet radiation and rainfall) in this program, the two surfaces coated with the carboxylated hydrocarbons, CC-10 and -12, were judged as possessing a more aesthetic, natural appearing mortar than the other surfaces. These two materials were the only compounds of the colorless type that imparted a dull finish to the mortar, and this characteristic of the coated mortar may be an indication that the curing compound may be more easily washed off than the materials that impart a glossy finish to the mortar. In the exposure tests, it was difficult to determine exactly when the material was washed off these specimens because of the small gradual change and the natural appearance of the mortar. The other colorless materials adhered to the mortar surfaces more strongly and would require additional rainfall or washing to leave an unaffected surface appearance.

63. The chlorinated rubbers (CC-8, -9, -15, and -16) and the styrene-acrylate (CC-17) adhered more tenaciously to the mortar surfaces

than the other materials investigated. This tenacious adherence coupled with an unattractive appearance after weathering would be objectionable where original concrete or mortar surfaces were desired in a relatively short time. In instances where aesthetic appearance is not as important but prolonged curing is desirable or where a prime coat for paint is desired, the chlorinated rubbers may be preferable for curing.

64. No mottling effects were evident in the surface coated with the white-pigmented curing compound. This lack of mottling is attributed to even application of the film and the tenacity of the film in adhering to the mortar. If the weathering had been continued, the material undoubtedly would have washed off, but probably at a uniform rate. Since mottling effects as a result of weathering have been reported in field applications using white-pigmented materials, it is believed that these effects are due primarily to improper or uneven application. The presence of white pigment would augment any surface effects noted. Uneven application would also affect surfaces covered with colorless compounds, causing a more mottled effect during the initial stages of weathering.

65. The materials investigated ranged widely in price. In evaluating performance of the materials no differentiation could be made between the less expensive and the more expensive materials.

66. The use of infrared spectroscopy proved quite successful during this investigation in identifying each material and in detecting differences among the various classes of material from different manufacturers.

Recommendations

67. The use of infrared spectroscopy in identifying materials is recommended for future tests. Correlations between types of material, laboratory performance, and field performance can then be made.

68. For concrete or mortar requiring a colorless curing compound, it is recommended that the materials be tested by applicable CRD-C 300 methods. In addition, smooth mortar specimens should be coated with the colorless curing compounds at the same coverage rate as the moisture loss specimens and should be subjected to weathering tests consisting of

cycles of alternate ultraviolet radiation and rainfall for 48 hr. The specimens should be examined critically for undesirable effects before weathering tests, after 24 hr of weathering, and after 48 hr of weathering. During this period, the coated mortar surface should (a) be aesthetically pleasing to the eye; (b) lighten upon weathering; (c) exhibit no or only slight cracking, crazing, or mottled appearance; and (d) slowly wash off, leaving the natural look of mortar exposed.

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Table 1

Infrared Reflectometer Readings of Coated Mortar Specimens Subjected to Weatherometer

Sample Symbol No. CRD-CC-	Reflectometer Readings, %														
	Ultraviolet Radiation, hr										Alternate Ultraviolet and Rainfall, hr				
	0	8	16	24	32	48	64	80	96	112	136	160	208	256	304
8	20.6	28.3	31.4	33.6	38.2	40.6	40.5	40.5	40.3	40.0	15.2	21.0	14.3	17.0	21.3
9	28.6	34.6	37.4	37.2	38.6	40.6	42.6	43.5	43.5	42.7	14.9	22.8	13.8	14.8	19.4
15	13.3	20.6	24.0	28.0	30.8	34.0	34.5	34.4	34.4	34.2	12.6	13.3	15.8	19.8	23.6
16	11.0	20.4	28.3	33.8	35.0	35.0	34.6	34.5	35.6	34.4	11.3	11.8	12.8	13.5	13.8
14	14.0	21.2	32.2	34.8	35.4	35.8	35.8	35.7	35.8	36.2	41.8	43.8	44.0	43.7	42.2
18	22.2	31.9	31.9	31.7	32.0	31.7	32.2	32.0	32.2	32.2	38.8	40.0	39.4	37.0	34.8
13	17.1	26.8	31.4	31.4	31.6	31.7	31.6	31.6	31.8	31.6	36.0	40.7	41.2	41.2	40.4
10	18.3	27.4	20.0	28.0	28.2	28.4	28.8	28.9	29.0	29.2	32.8	35.0	34.4	28.2	24.5
11	10.6	9.0	9.4	9.5	9.7	9.8	9.8	9.8	10.0	10.2	29.4	32.3	28.8	25.3	24.1
12	18.2	19.1	21.1	25.2	25.7	25.8	26.3	26.3	26.5	26.7	37.1	40.0	42.3	43.1	43.3
17	26.8	34.8	39.8	38.8	38.9	38.8	38.6	38.4	38.4	38.2	14.5	30.0	23.2	28.5	33.4
19	16.4	17.2	17.5	17.6	17.6	18.4	19.1	19.1	19.4	19.3	23.5	27.7	19.8	35.9	38.3
20	20.3	20.0	19.8	20.4	20.4	20.5	20.7	20.8	20.9	20.8	26.8	29.8	26.7	34.0	37.0
White-pigmented	64.3	63.0	66.9	67.4	68.4	69.6	72.0	72.7	74.2	74.9	85.2	84.4	84.4	82.6	83.6
Plain mortar	29.0	29.2	29.4	29.1	29.2	29.4	29.4	29.5	29.7	29.6	31.8	33.6	30.6	34.0	33.2

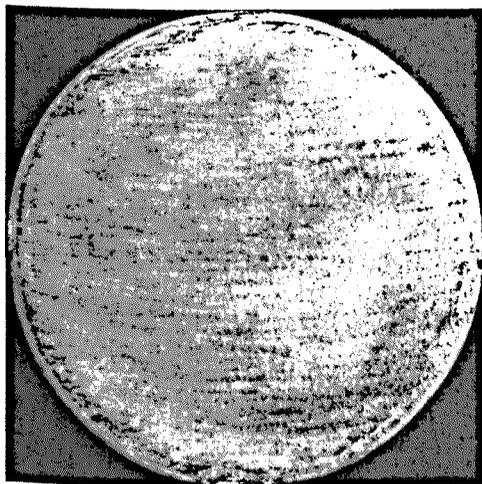
Table 2

General Appearance of Coated Mortar Specimens After Weathering

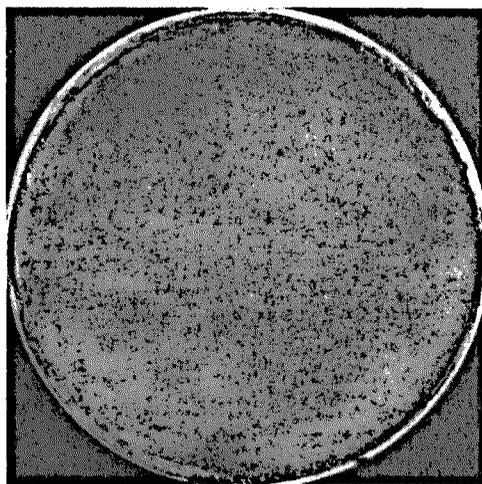
Sample Symbol No. CRD-CC-	Specimens After 112 hr of Ultraviolet Exposure		Specimens After 112 hr of Ultraviolet Exposure Plus 192 hr of Alternating Ultraviolet Exposure and Rainfall	
	General Appearance	Remarks	General Appearance	Remarks
8	Good	Light gray, glossy	Fair	Darker, yellowish, glossy, slightly spotted, most coating material still present
9	Good	Light amber, glossy	Fair	Darker, yellowish, glossy, slightly spotted, most coating material still present
15	Good	Light gray, glossy, few burst bubbles	Poor	Darker, glossy, mottled, most coating material still present
16	Good	Gray, glossy, slightly streaked	Poor	Dark, glossy, mottled, most coating material still present
14	Poor	Greenish-brown, glossy, uneven color, mottled, ugly	Poor	Lighter, dull, mottled effect fading
18	Poor	Light amber, glossy, mottled	Fair to good	Lighter, most of coating material washed off
13	Poor	Gray, glossy, mottled	Poor	Lighter, dull, mottled
10	Good	Gray, dull, smooth	Fair to good	Dull, most coating material washed off, little scaling of mortar
11	Poor	Greenish-brownish, glossy, dark, mottled	Fair	Dull, whitish residue, most coating material washed off
12	Fair to good	Gray, dull, slightly mottled	Fair to good	Dull, lighter, little scaling of mortar
17	Good	Light gray, glossy, smooth	Good	Glossy, slightly spotted, most coating material still present
19	Fair	Dull, whitish residue	Fair	Dull, little change, lighter
20	Fair	Dull, slightly mottled	Fair	Dull, lighter, more washed effect
White-pigmented	Good	Dull, white	Fair to good	Dull, slightly washed
Plain mortar	Fair	Dull, slightly mottled	Poor	Dull, few holes in surface

Table 3
Absorbance Values

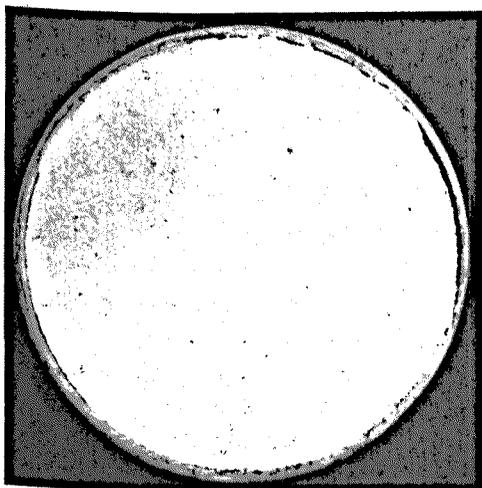
Sample Symbol No. CRD-CC-	Hydroxyl, at $\approx 3\mu$ at Indicated Age in Days						Carbonyl, at $\approx 5.9\mu$ at Indicated Age in Days					
	1	4	7	14	21	28	1	4	7	14	21	28
8 Chlorinated rubber	0.022	0.016	0.016	0.017	0.017	0.017	0.016	0.016	0.016	0.017	0.017	0.021
9	0	0	0	0	0	0	0	0	0	0	0	0
15	0.021	0.021	0.007	0.007	0.007	0.008	0.036	0.036	0.037	0.059	0.059	0.082
16	0.005	0.005	0.006	0.019	0.013	0.007	0.033	0.033	0.031	0.060	0.043	0.043
14 Styrene-butadiene	0.069	0.194	0.199	0.281	0.301	0.314	0.032	0.218	0.284	0.608	0.584	0.717
18	0.091	0.182	0.182	0.279	0.279	0.312	0.089	0.204	0.226	0.572	0.489	0.613
13 Phthalic alkyd	0.094	0.094	0.099	0.126	0.126	0.126	0.941	0.941	0.941	0.941+	0.941+	0.941+
17 Styrene-acrylate	0.016	0.008	0.011	0.011	0.011	0.011	1.105	1.105	1.105	1.105	1.105	1.105
11 Carboxylated hydrocarbon	0.138	0.477	0.470	0.423	0.572	0.434	0.658	0.864	1.058	1.336	1.336	1.432
10	0.035	0.069	0.049	0.070	0.050	0.078	0.126	0.154	0.167	0.272	0.166	0.272
12	0.077	0.091	0.099	0.125	0.135	0.134	0.244	0.295	0.322	0.506	0.416	0.517



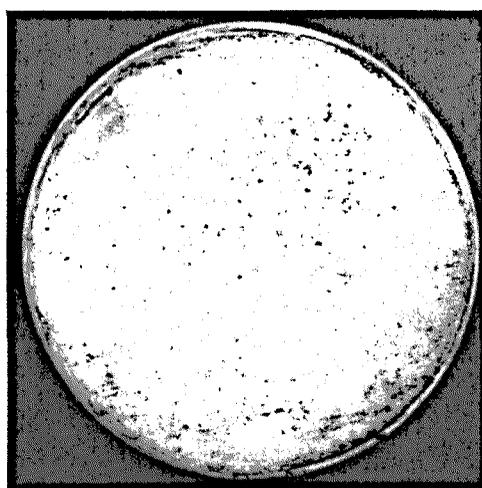
a. CC-8 prior to exposure



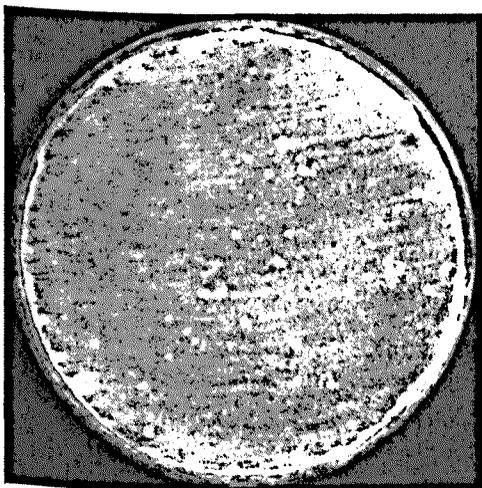
d. CC-15 prior to exposure



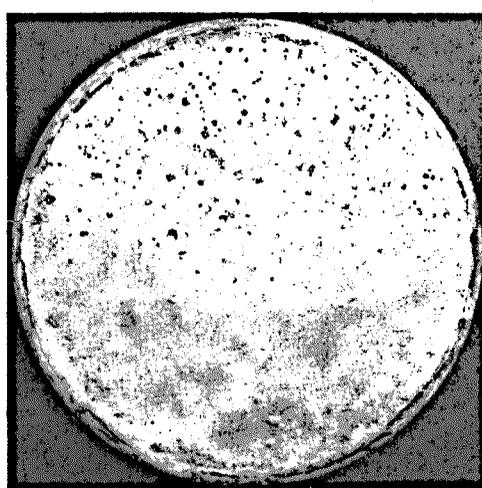
b. CC-8 after 112 hr exposure



e. CC-15 after 112 hr exposure

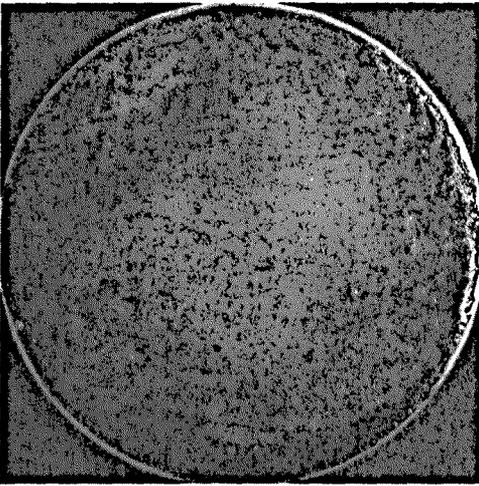


c. CC-8 after 304 hr exposure

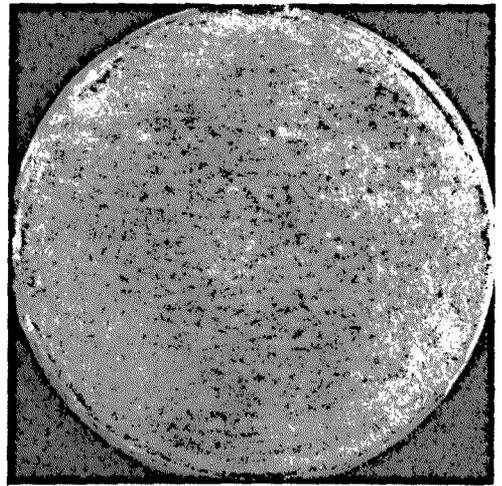


f. CC-15 after 304 hr exposure

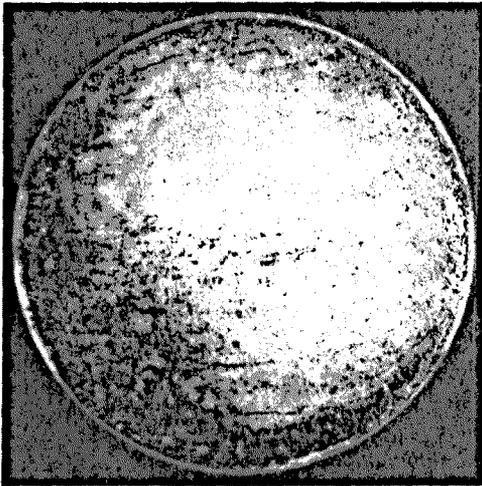
Photograph 1. Chlorinated rubber specimens (CC-8 and -15) prior to exposure in weatherometer, after 112 hr of ultraviolet exposure, and after 304 hr of total exposure



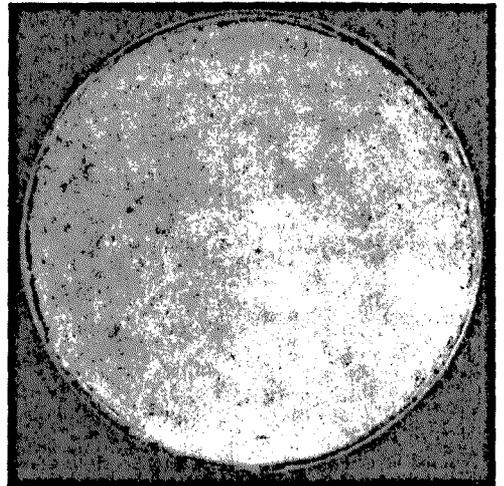
a. CC-14 prior to exposure



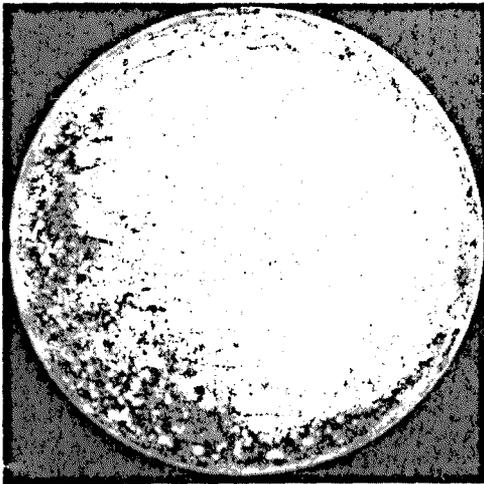
d. CC-13 prior to exposure



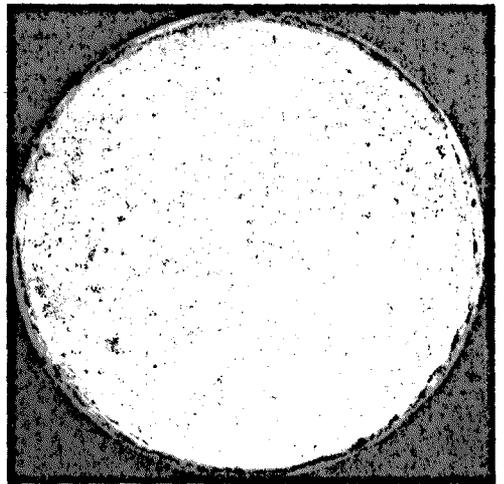
b. CC-14 after 112 hr exposure



e. CC-13 after 112 hr exposure

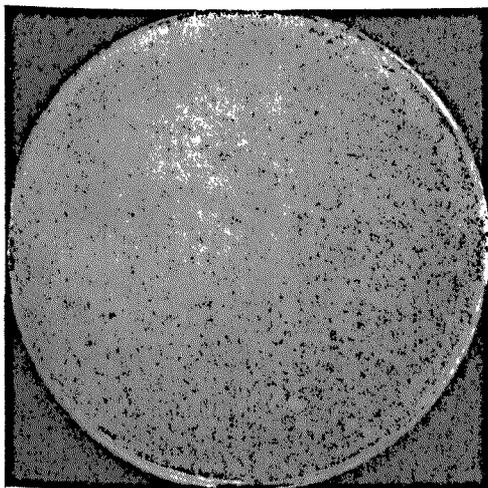


c. CC-14 after 304 hr exposure

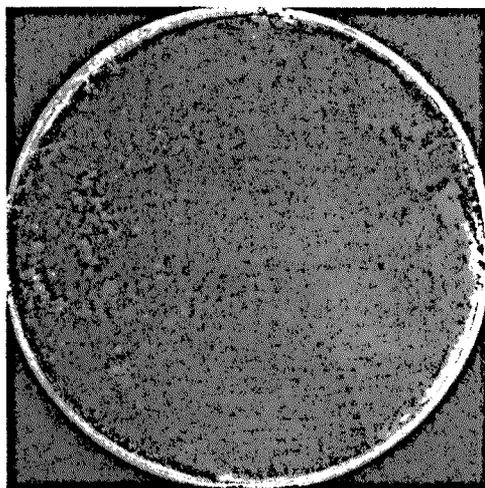


f. CC-13 after 304 hr exposure

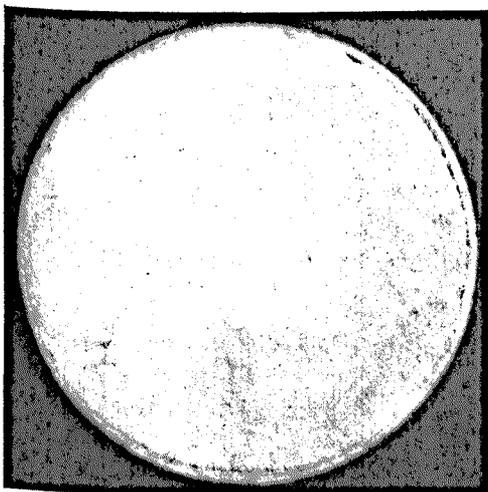
Photograph 2. Styrene-butadiene (CC-14) and phthalic alkyd (CC-13) specimens prior to exposure in weatherometer, after 112 hr of ultraviolet exposure, and after 304 hr of total exposure



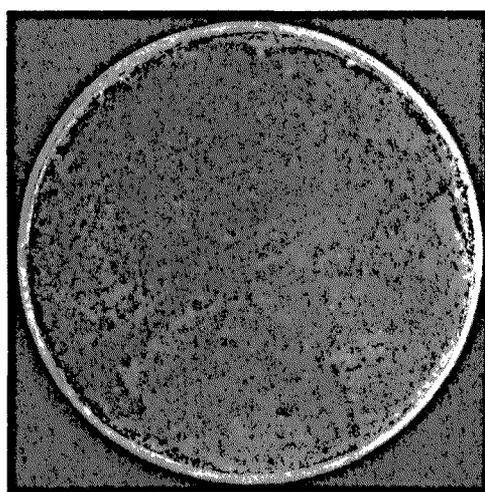
a. CC-10 prior to exposure



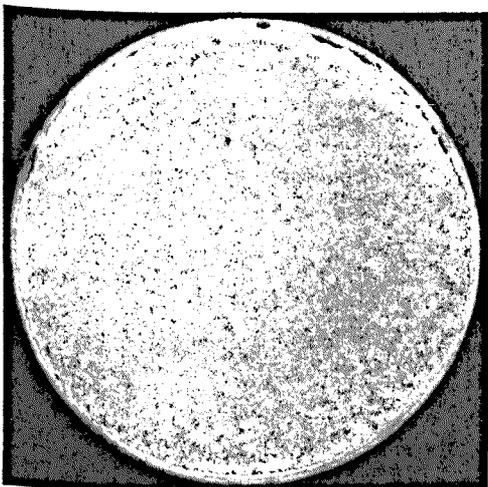
d. CC-11 prior to exposure



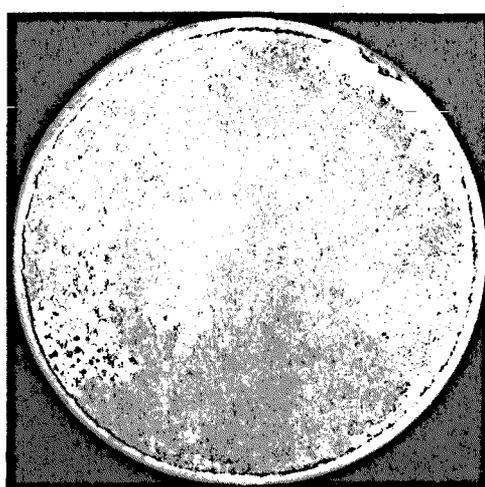
b. CC-10 after 112 hr exposure



e. CC-11 after 112 hr exposure

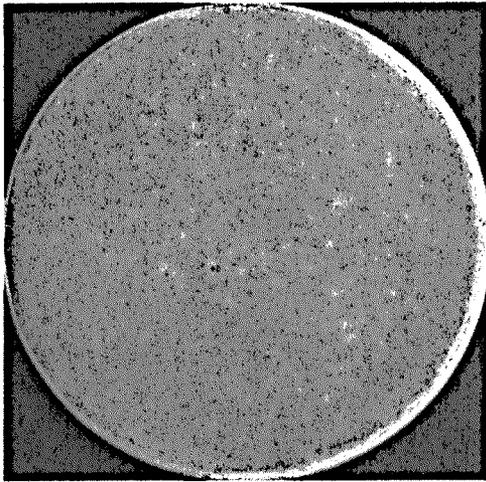


c. CC-10 after 304 hr exposure

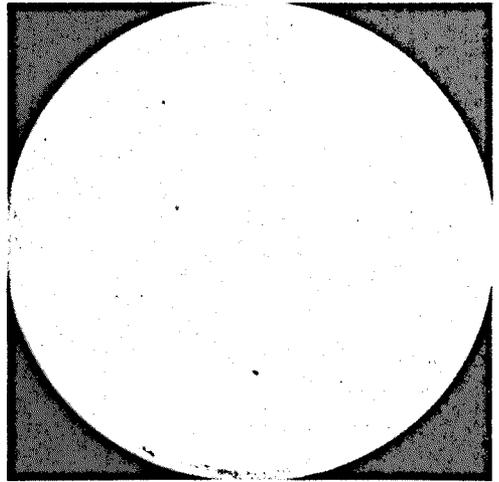


f. CC-11 after 304 hr exposure

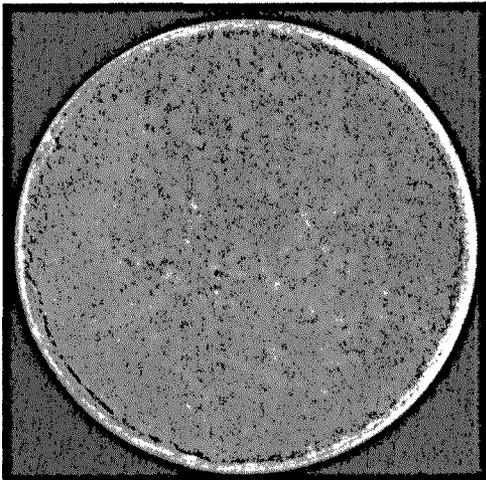
Photograph 3. Carboxylated hydrocarbon specimens (CC-10 and -11) prior to exposure in weatherometer, after 112 hr of ultraviolet exposure, and after 304 hr of total exposure



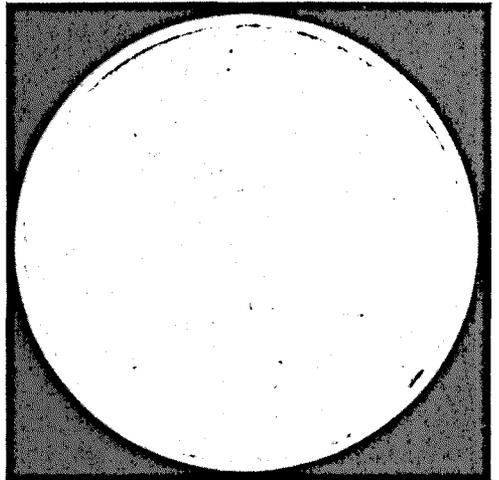
a. CC-20 prior to exposure



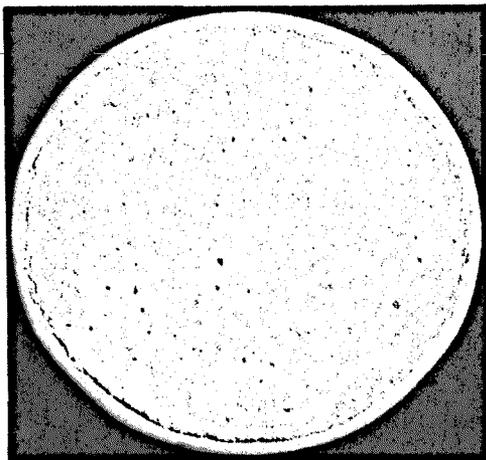
d. White-pigmented specimen prior to exposure



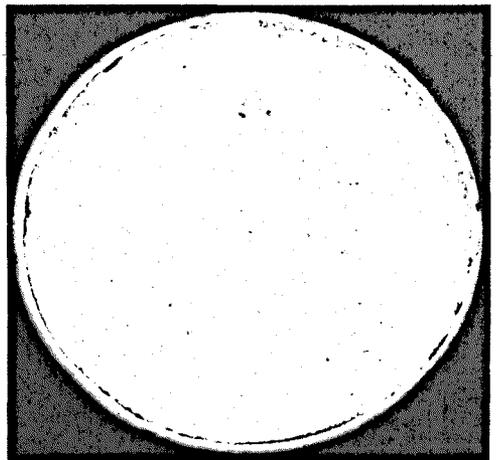
b. CC-20 after 112 hr exposure



e. White-pigmented specimen after 112 hr exposure

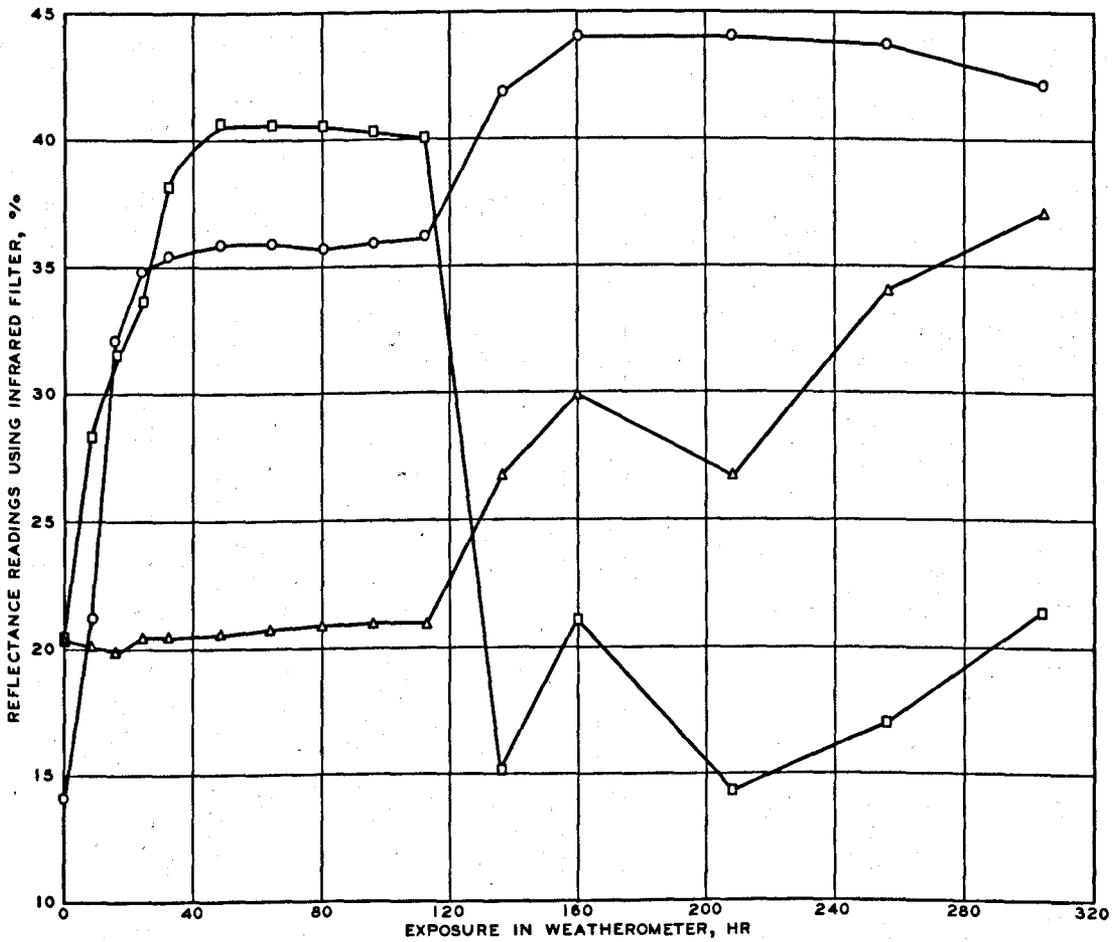


c. CC-20 after 304 hr exposure



f. White-pigmented specimen after 304 hr exposure

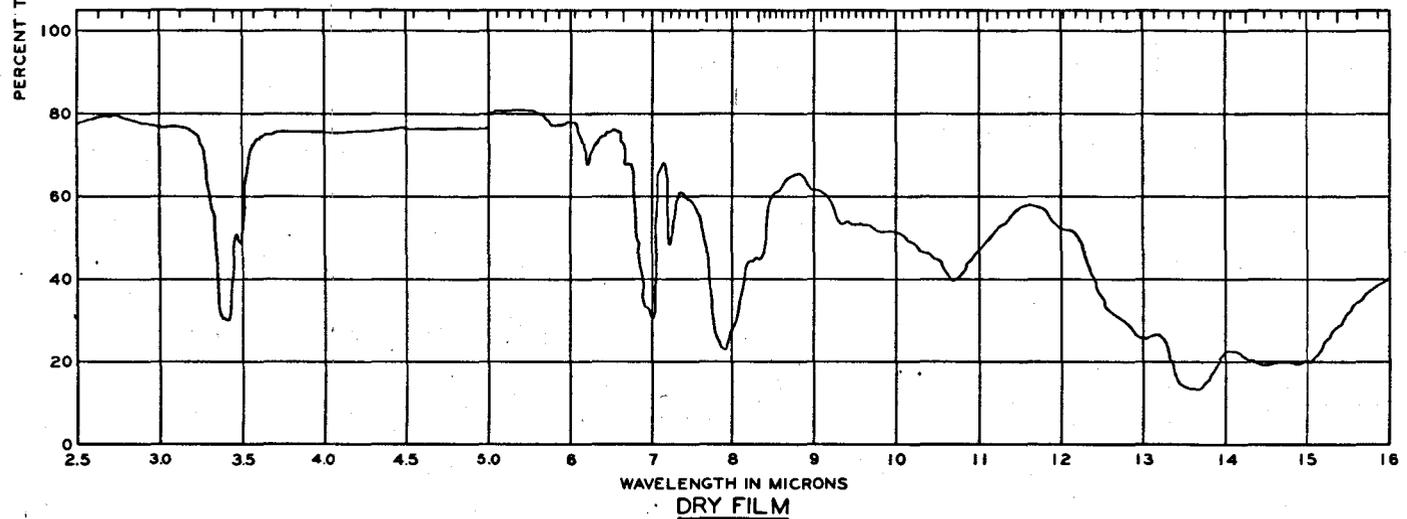
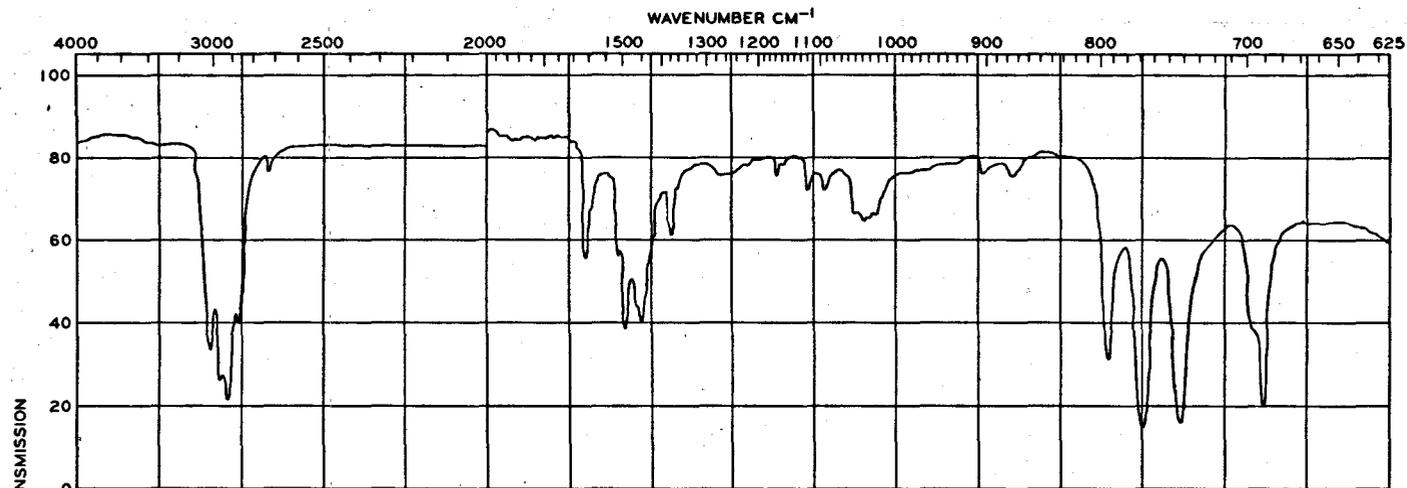
Photograph 4. Sodium silicate (CC-20) and white-pigmented specimens prior to exposure in weatherometer, after 112 hr of ultraviolet exposure, and after 304 hr of total exposure



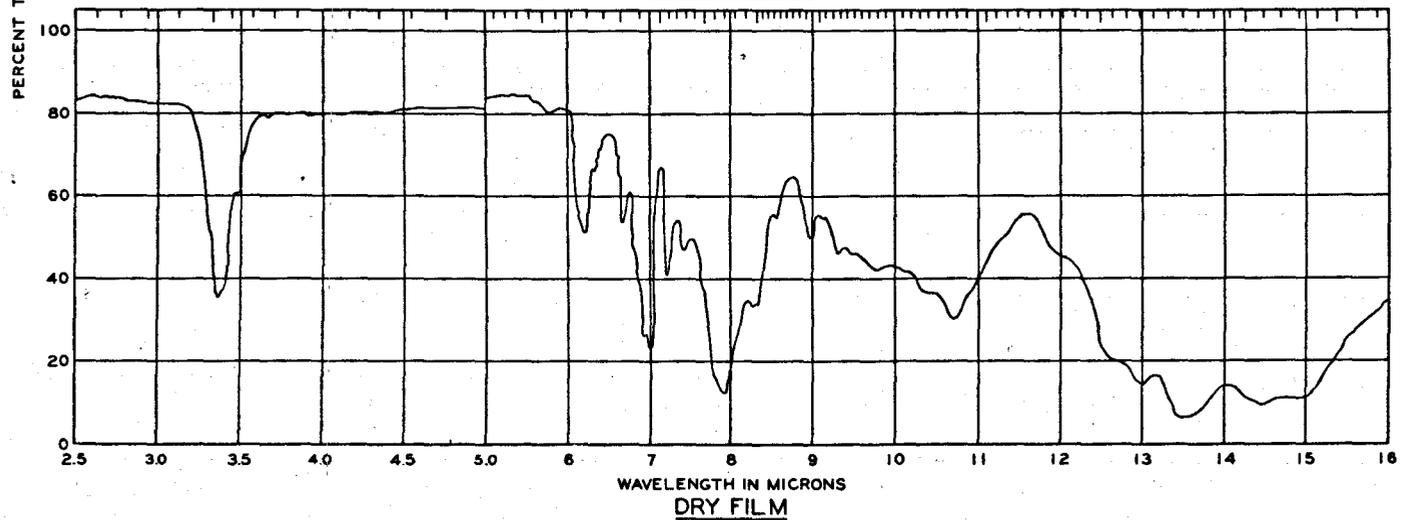
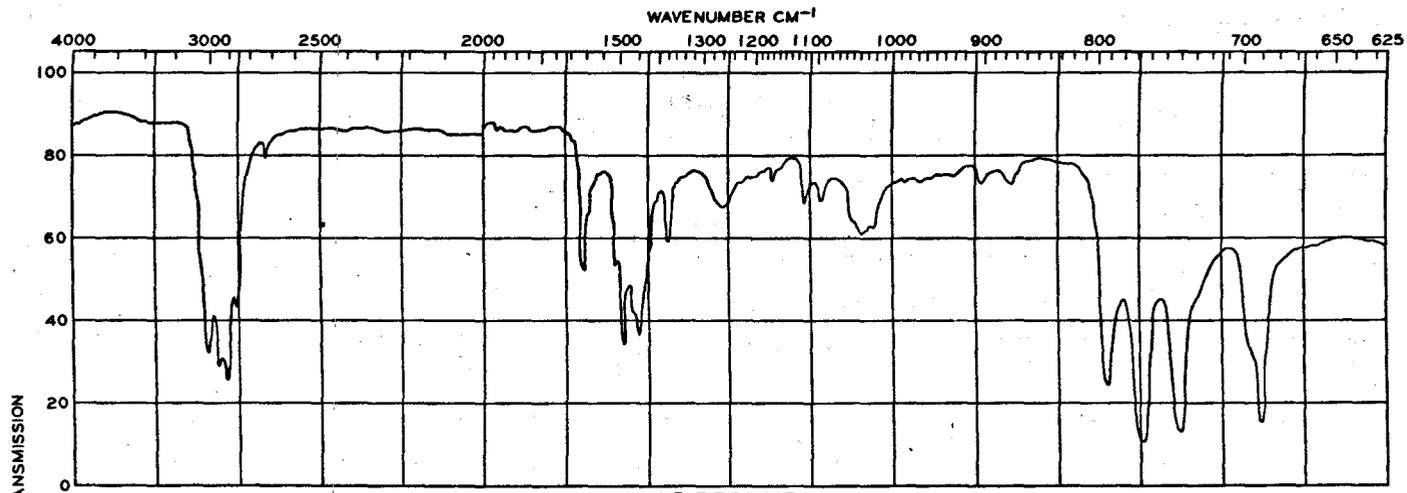
LEGEND

- CC-8
- CC-14
- △ CC-20

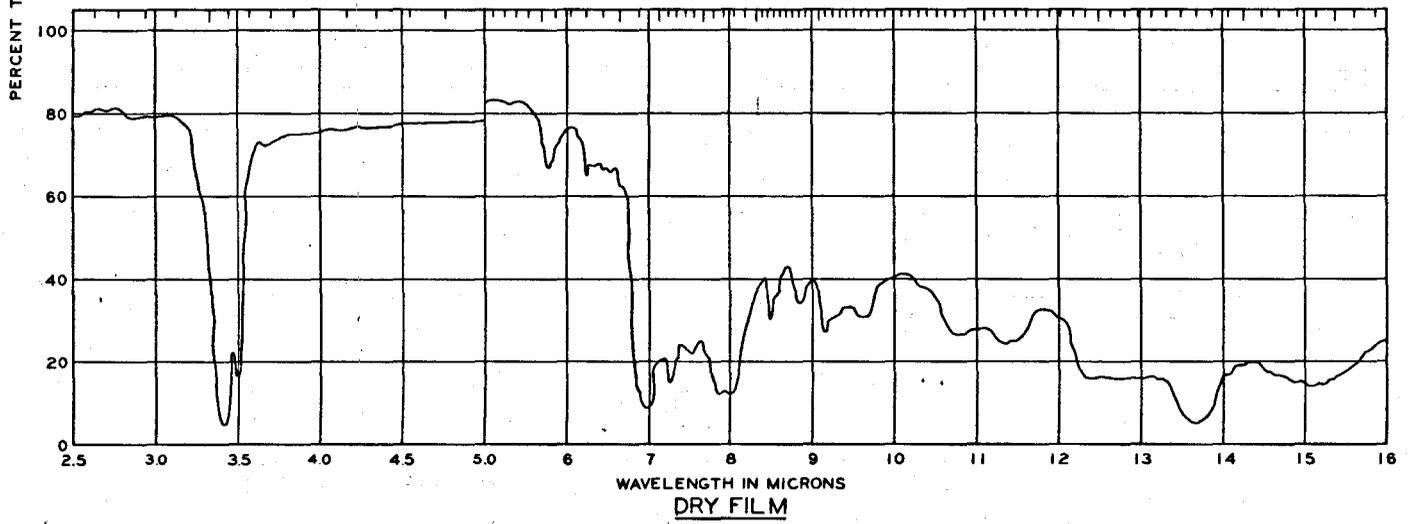
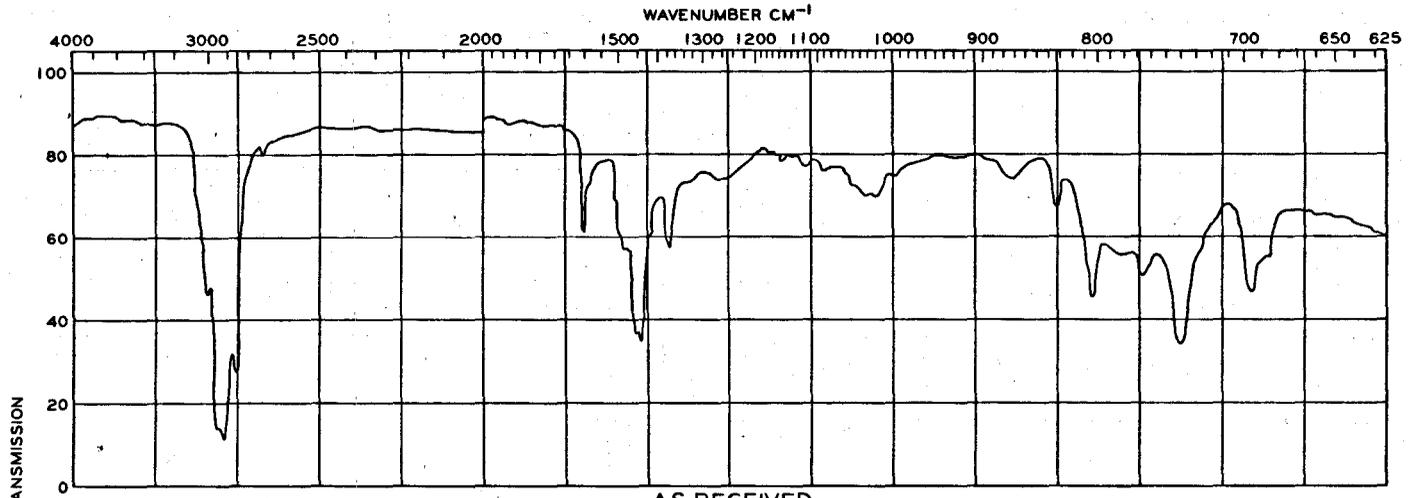
REFLECTANCE READINGS
 VERSUS EXPOSURE
 IN WEATHEROMETER
 MORTAR SPECIMENS COATED
 WITH CC-8, CC-14, AND CC-20



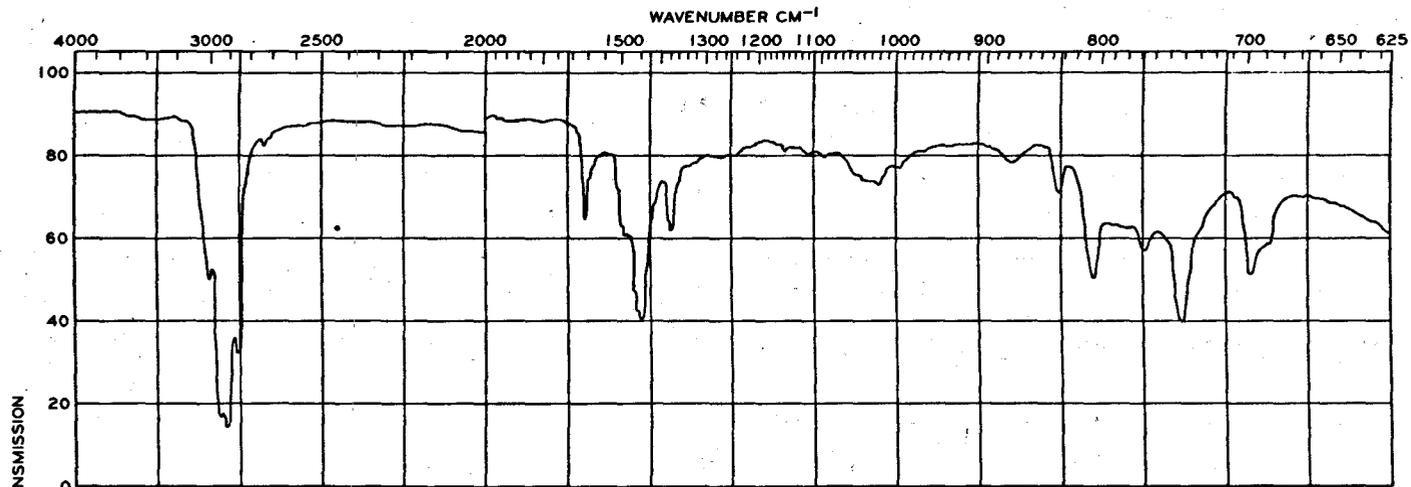
INFRARED SPECTRA
CRD-CC-8
AS RECEIVED AND DRY FILM



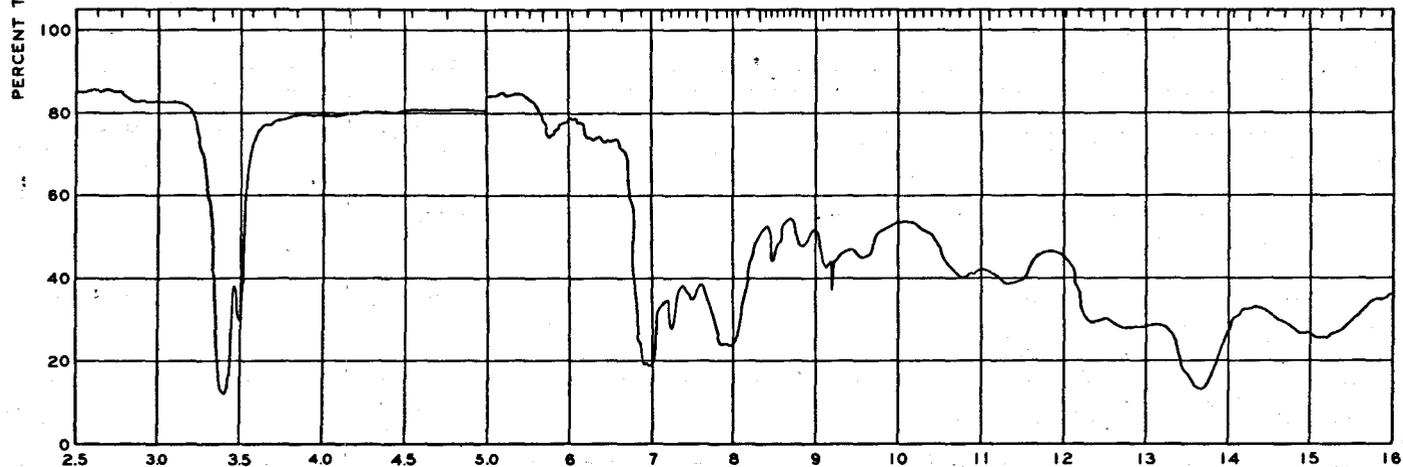
INFRARED SPECTRA
CRD-CC-9
AS RECEIVED AND DRY FILM



INFRARED SPECTRA
CRD-CC-15
AS RECEIVED AND DRY FILM

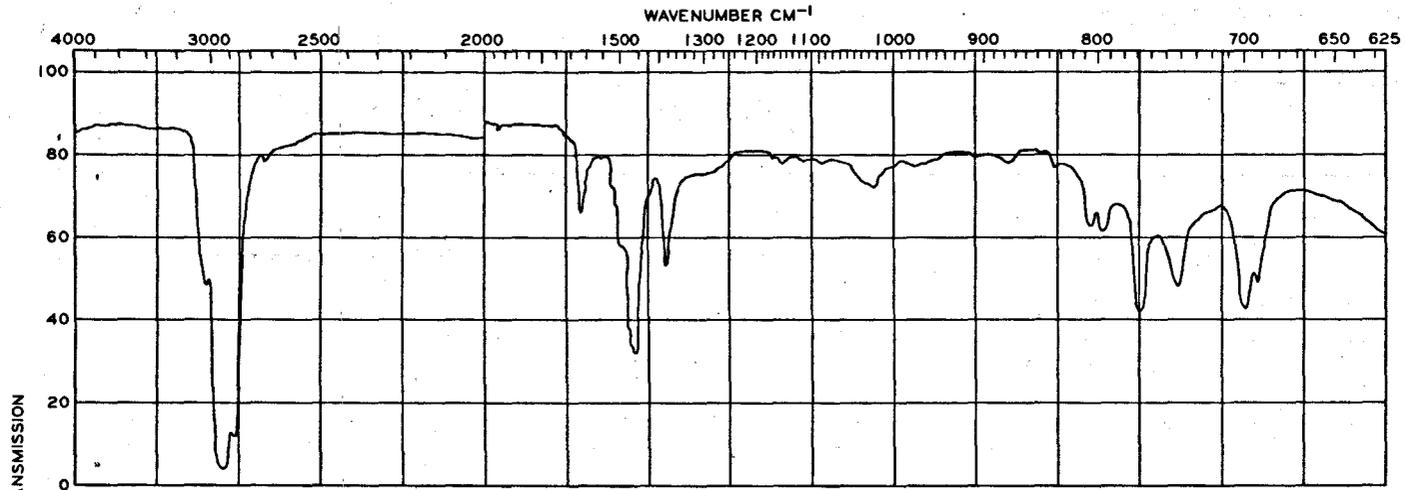


AS RECEIVED

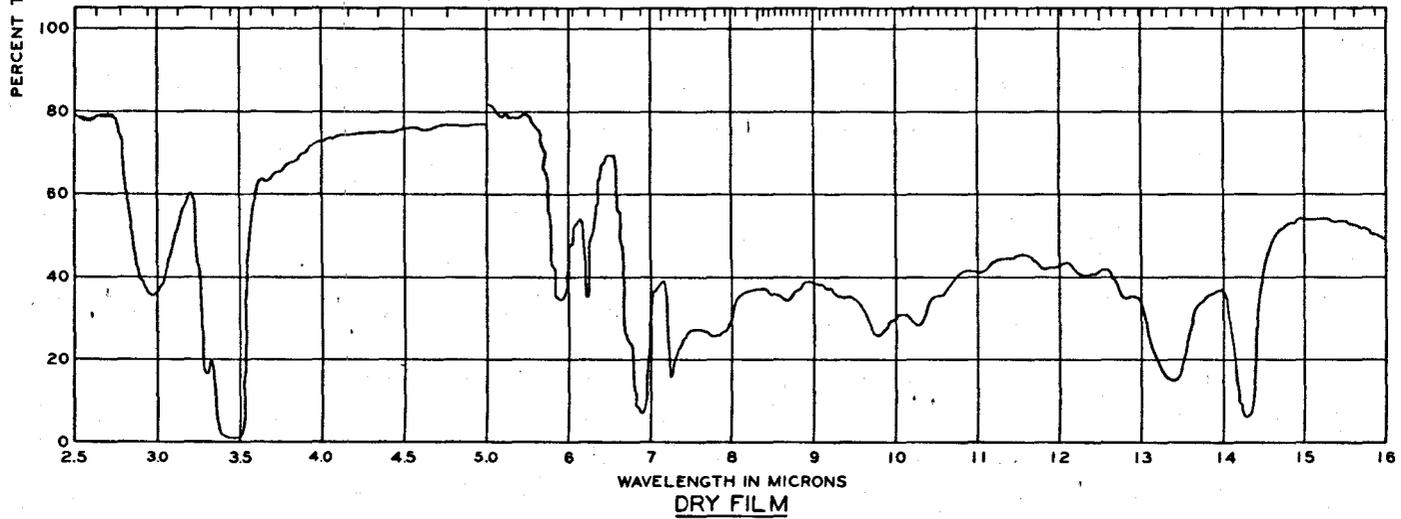


WAVELENGTH IN MICRONS
DRY FILM

INFRARED SPECTRA
CRD-CC-16
AS RECEIVED AND DRY FILM

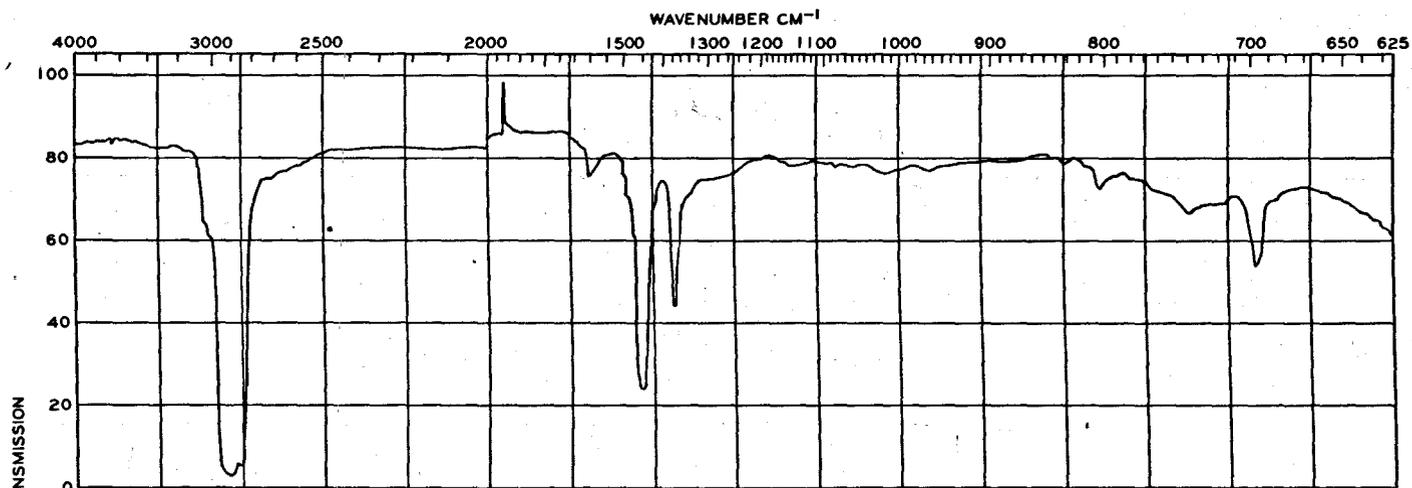


AS RECEIVED

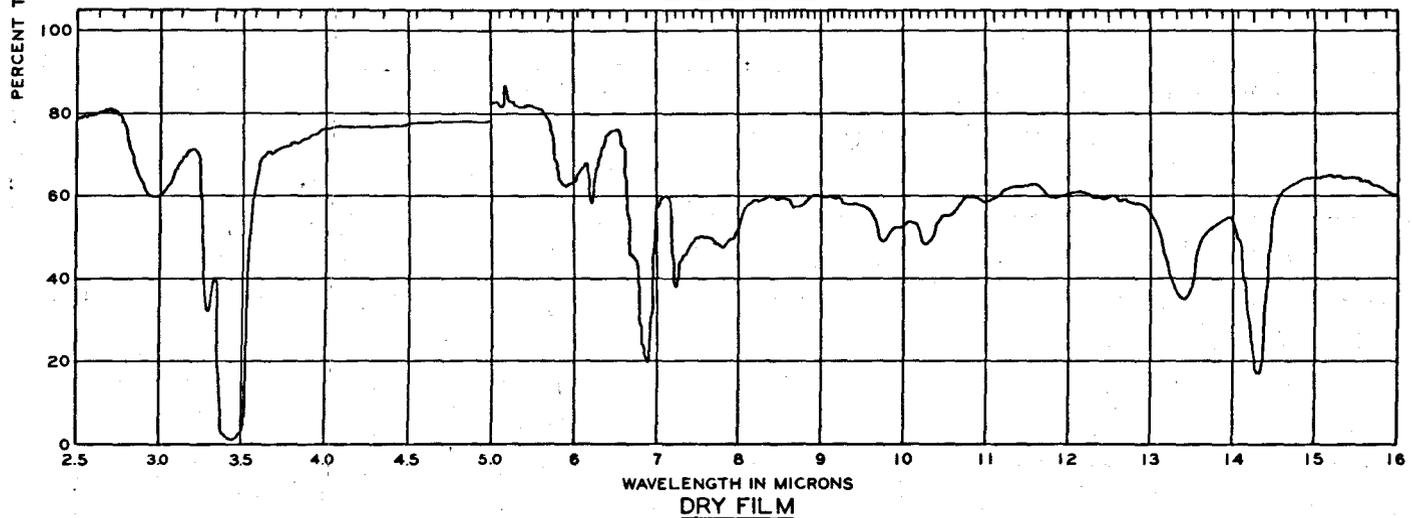


DRY FILM

INFRARED SPECTRA
CRD-CC-14
AS RECEIVED AND DRY FILM

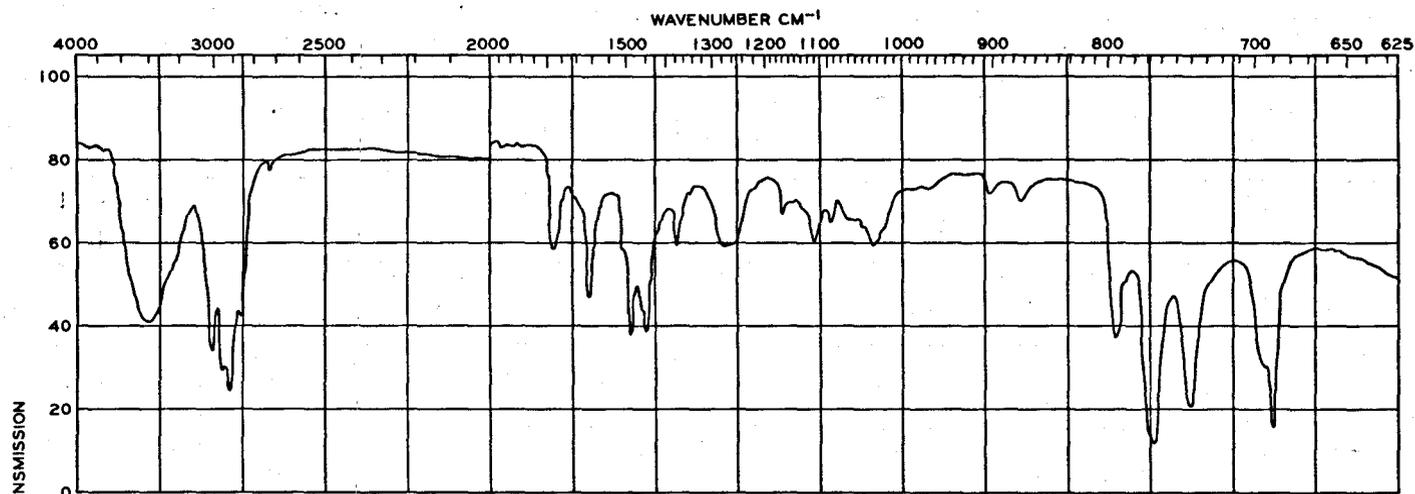


AS RECEIVED

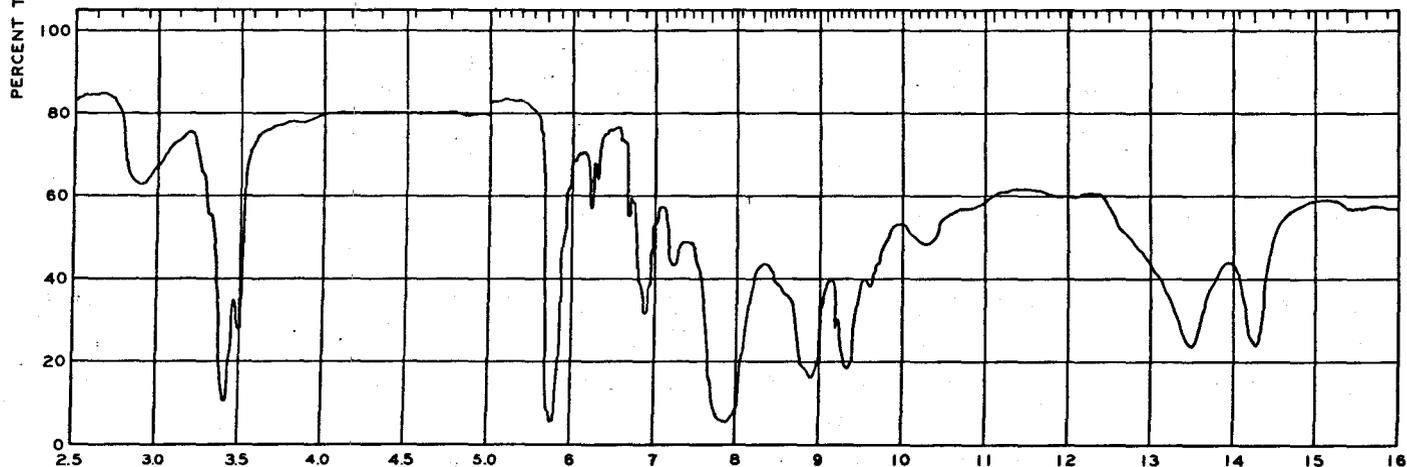


DRY FILM

INFRARED SPECTRA
CRD-CC-18
AS RECEIVED AND DRY FILM

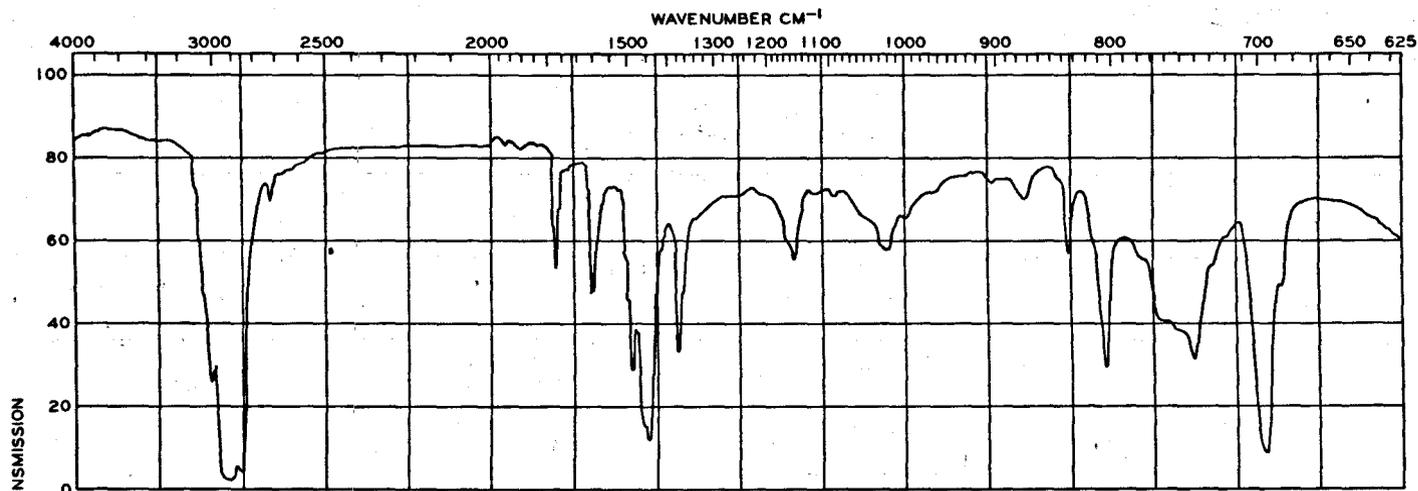


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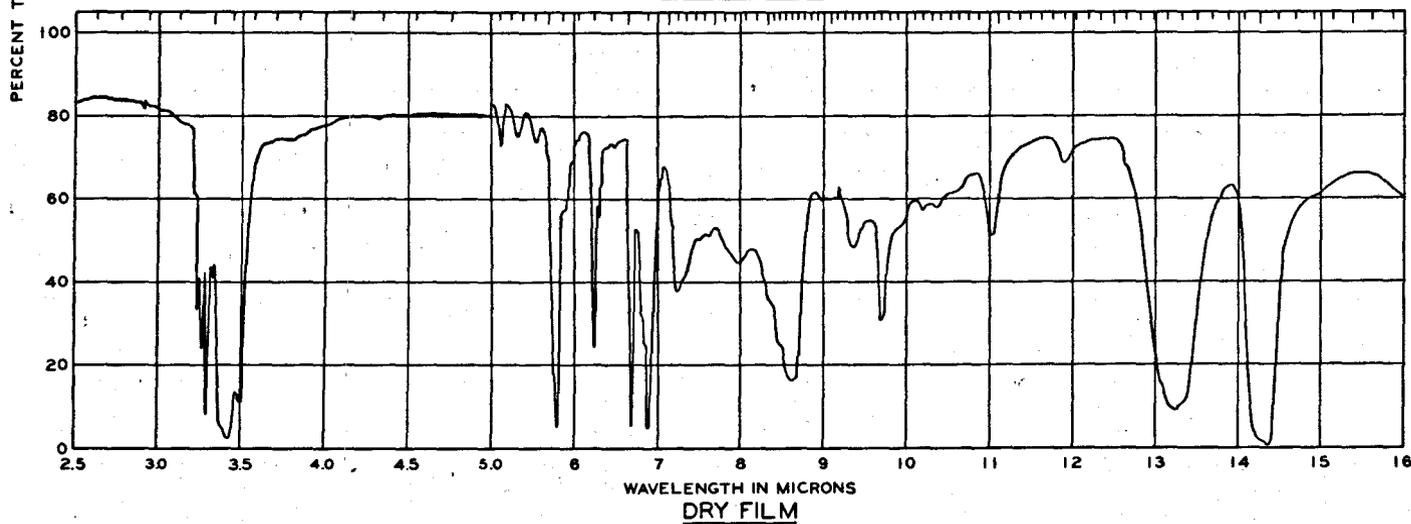


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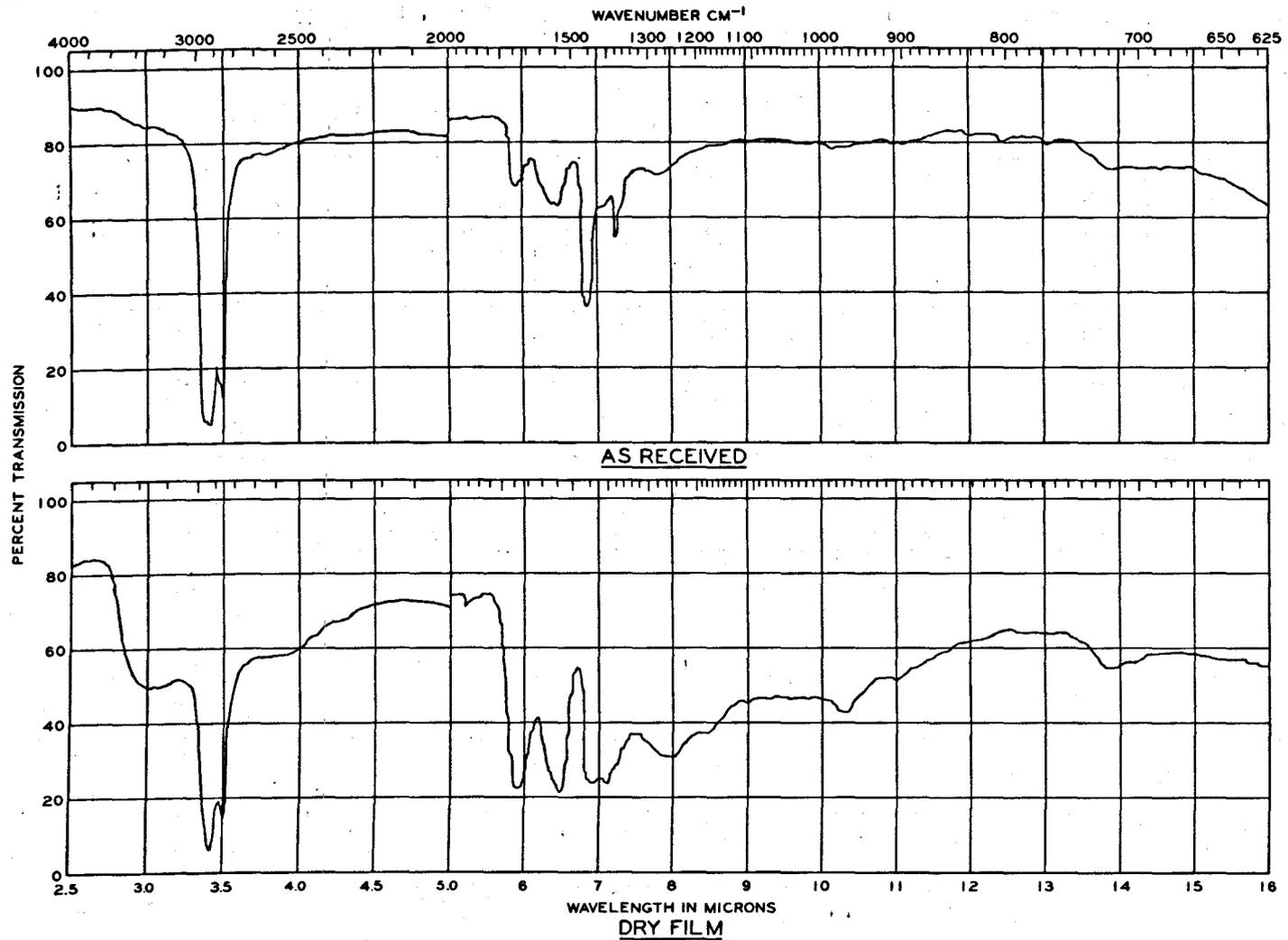
INFRARED SPECTRA
 CRD-CC-13
 AS RECEIVED AND DRY FILM



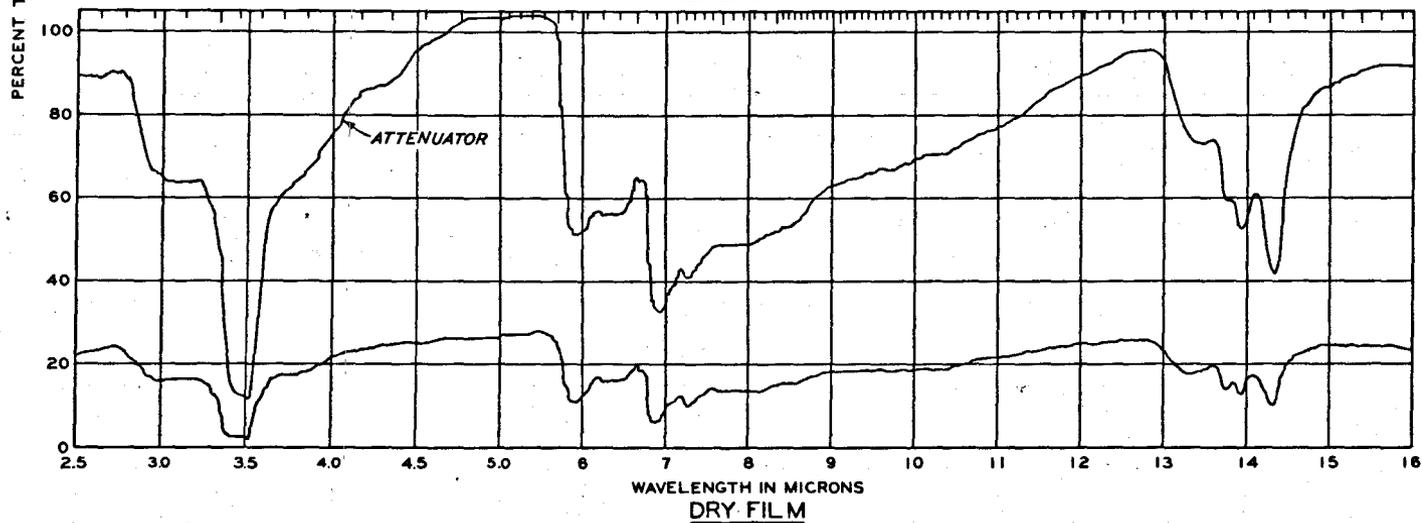
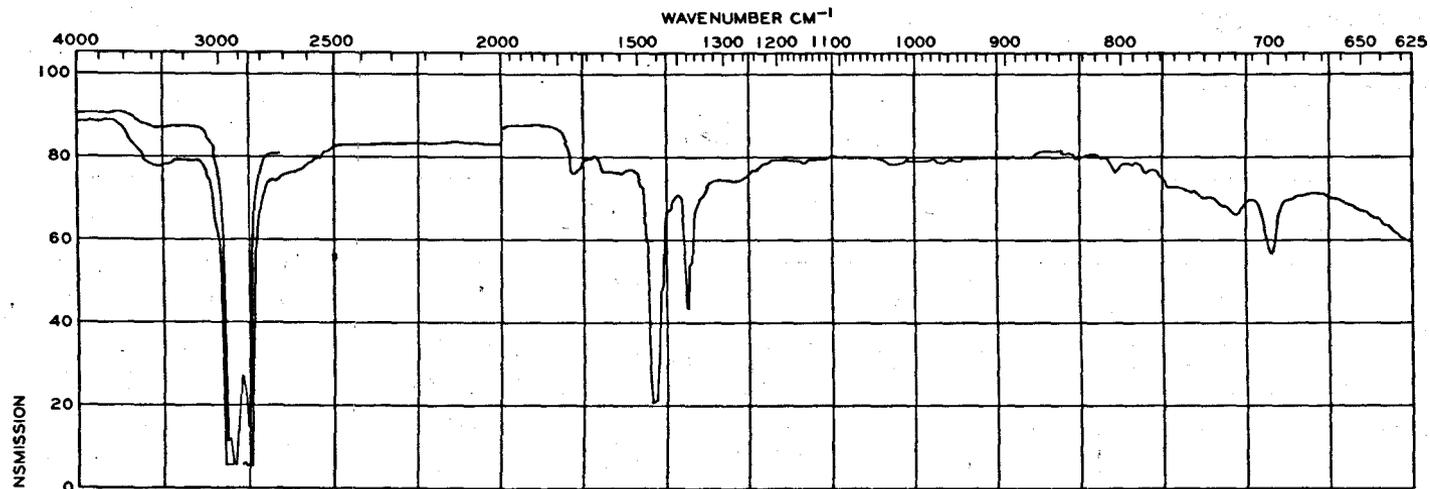
AS RECEIVED



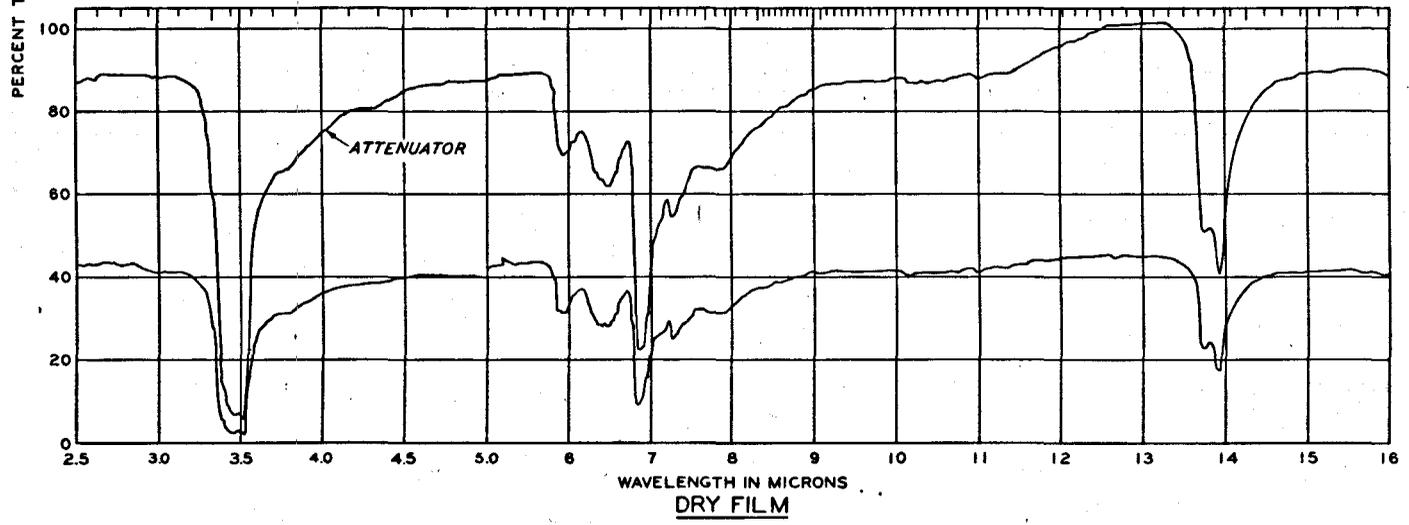
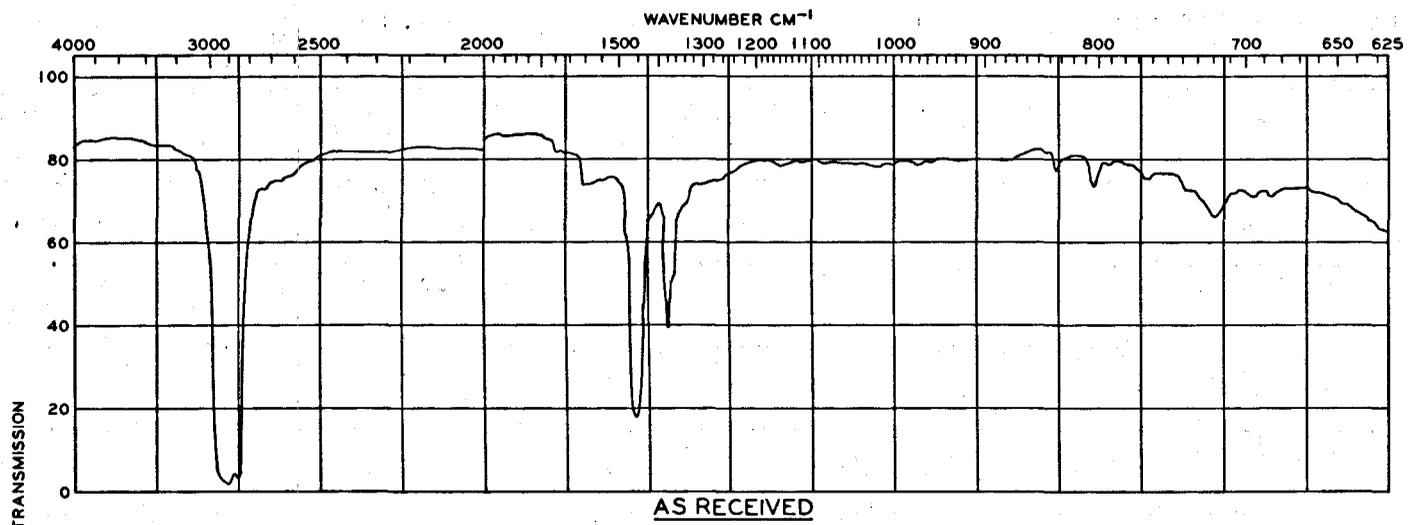
INFRARED SPECTRA
CRD-CC-17
AS RECEIVED AND DRY FILM



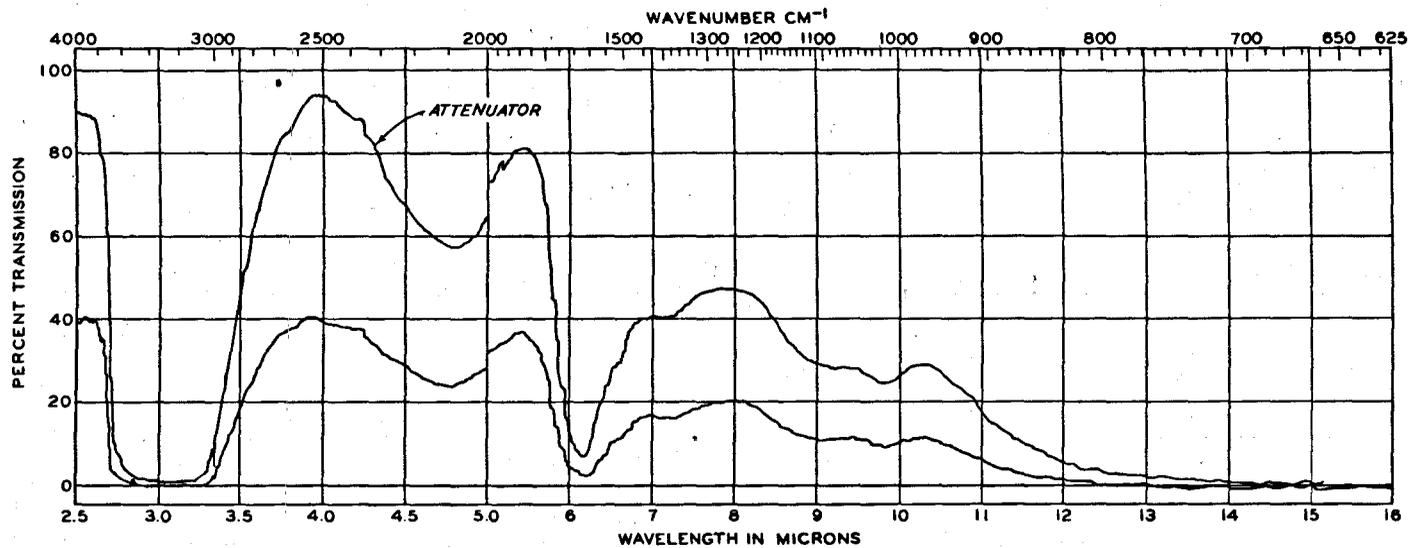
INFRARED SPECTRA
CRD-CC-11
AS RECEIVED AND DRY FILM



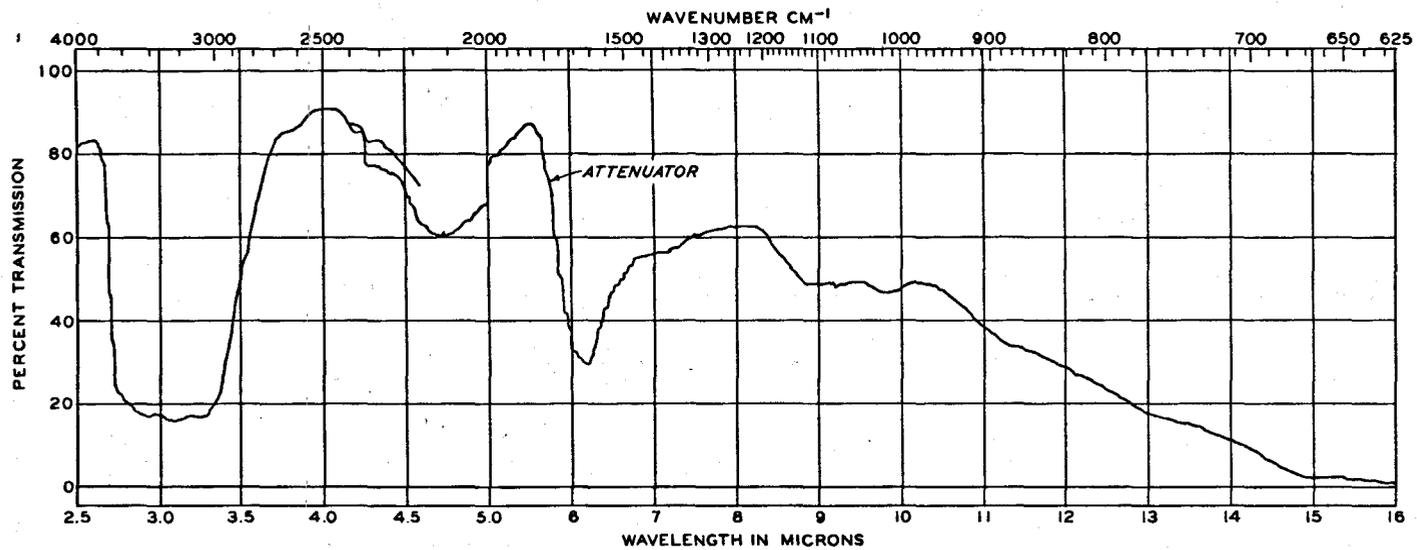
INFRARED SPECTRA
 CRD-CC-12
 AS RECEIVED AND DRY FILM



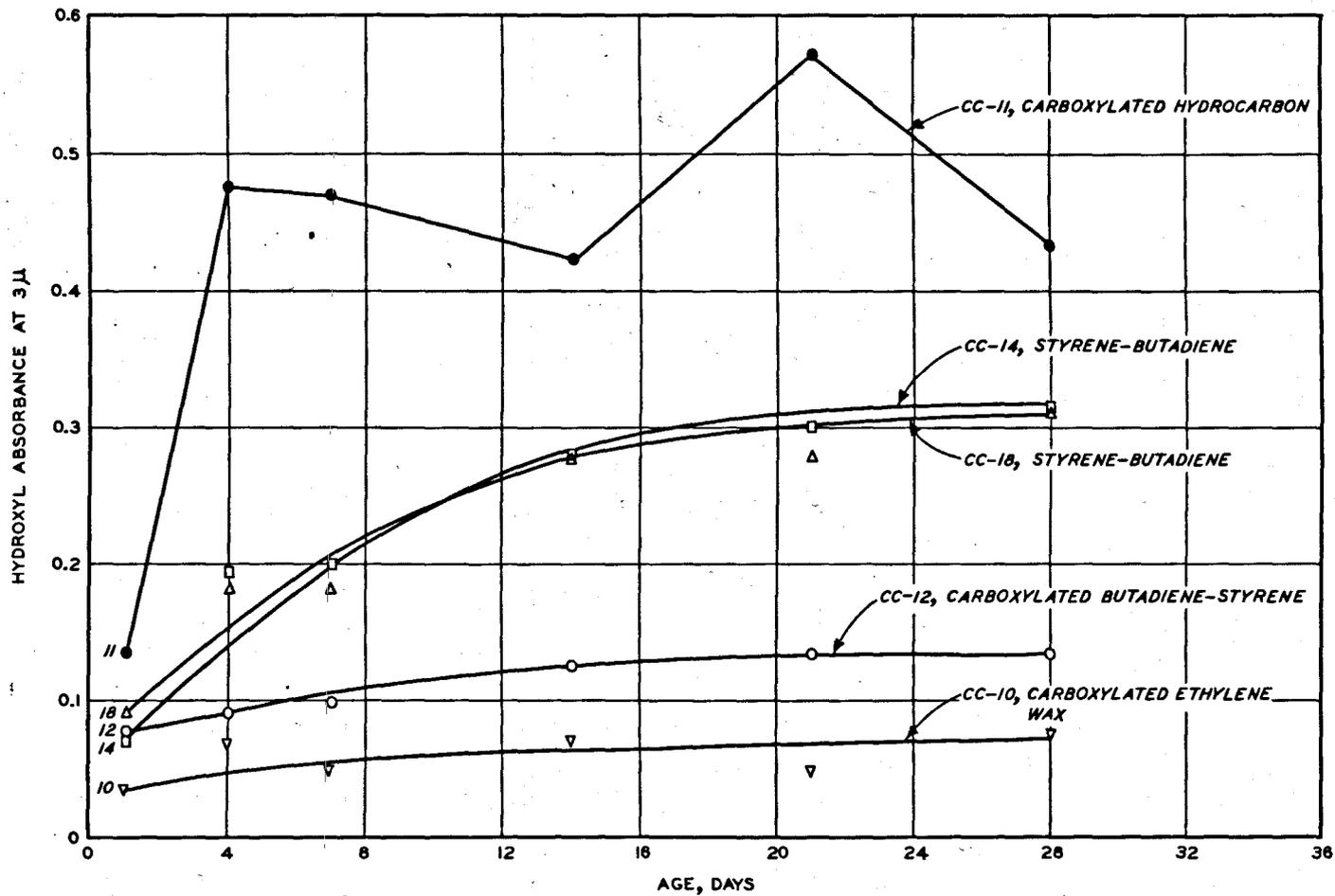
INFRARED SPECTRA
CRD-CC-10
AS RECEIVED AND DRY FILM



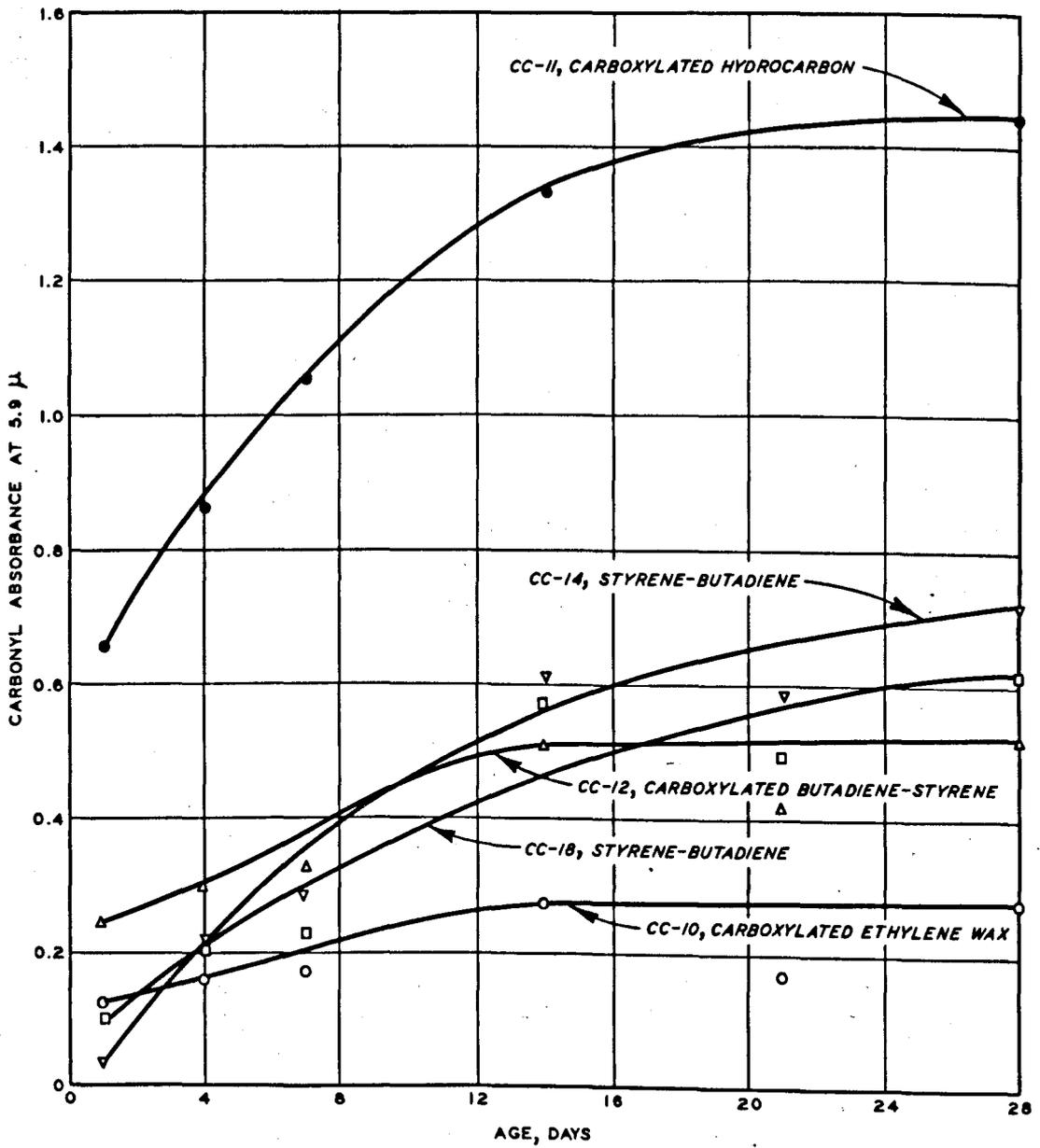
INFRARED SPECTRA
CRD-CC-19



INFRARED SPECTRA
CRD-CC-20



HYDROXYL ABSORBANCES
AT VARIOUS AGES
CRD-CC-10, -11, -12, -14, AND -18



CARBONYL ABSORBANCES
 AT VARIOUS AGES
 CRD-CC-10, -11, -12, -14, AND -18

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13. ABSTRACT Eleven compounds designated as colorless and two designated as water-based were evaluated for their suitability for curing of concrete and for their lack of color. All met present requirements for sprayability, drying time, and flash point. Only ten met the moisture-retention requirements. The three that failed the moisture test were one colorless type (styrene-acrylate resin) and both water-based compounds. An infrared spectrophotometer was used to identify and characterize each of the materials. Infrared analysis revealed that the 11 colorless materials represent 5 different chemical classes: 4 chlorinated rubbers, 2 modified styrene-butadienes, 1 phthalic alkyd, 3 carboxylated hydrocarbons, and 1 styrene-acrylate. Both water-based materials were identified as sodium silicate liquids. Infrared measurements were used to detect chemical and physical changes of the colorless materials when exposed to laboratory conditions and natural sunlight. The materials ranged from light yellow to dark reddish-brown and were rated from 1 to 18 on a standard color comparator. Generally, mortar specimens coated with the curing compounds lightened in color when subjected to both a controlled temperature-humidity cabinet and ultraviolet radiation in a weatherometer. However, upon being subjected to alternate cycles of rainfall and ultraviolet radiation in the weatherometer, some of the coated surfaces continued to lighten and the curing compounds washed off, while some became mottled and unattractive. There was no correlation between original color of the curing compound and color of the coated mortar surfaces either before or after exposure. Coating materials in the same chemical classes responded to weathering in a similar manner. Mortar surfaces coated with two of the carboxylated hydrocarbons possessed a dull finish and appeared more natural looking with fewer signs of mottling than any of the other coated surfaces.			

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