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Characterization and Quantitative Istimation of Waxy Materials in Membrane Curing Compounds.

Research Project 57 G-83, Spectrophotometric Identification of Membrane Curing Compounds. Research Lab. Report No. 285

Reported by: M. H. Janson

From our experience in Michigan, wax is considered an undesirable adulterant in membrane curing compounds and is prohibited by current Department specifications. Therefore, a study was undertaken to detect waxy materials in concrete curing compounds by means of infrared spectro-photometric techniques. Samples of a typical curing compound resin, a wax which might be added, and various mixtures of the resin and wax were obtained from Artco, Incorporated and Truscon Laboratories. Examination of the infrared spectra of these materials indicated that the presence of these waxes could be detected by absorptions at 13.7 and 13.9 microns and that quantitative estimation of the amount of wax present in the samples was possible.

In order to apply the above findings to other resins, and possibly waxes, samples of concrete curing compound were obtained from the Testing Laboratory at Ann Arbor. Samples of material from each company furnishing curing compounds up to the end of July, 1957 were selected on the basis of extremes in drying time. Test data revealed a considerable range in drying time and one sample showed "tracking off" after 96 hours. Table 1 contains the sample identifications and selected test results obtained at Ann Arbor. Infrared spectra of the vehicle solids of each sample listed in Table 1 were recorded and examined for the presence of wax. Two spectra indicated the possible presence of wax.

Chemical separation procedures were also studied and used to verify the presence or absence of wax in the curing compound samples. Results of the spectrophotometric procedure together with a chemical separation confirmed the presence of wax in two of the samples, 57 MR-96 and 57 MR-101, and the absence of wax in the remaining five samples. Gravimetric analysis by chemical separation indicated a wax content of 48.4 percent in Sample 57 MR-96 and 38.2 percent in Sample 57 MR-101. Infrared spectra indicated the presence of wax in approximately the same amounts.

The Permite curing compound represented by the two samples containing wax was rejected by the Testing Laboratory on the basis of drying time, which is adversely affected by the presence of excessive quantities of wax.

Spectrophotometric and chemical tests on two additional samples of Permite received later in the season, Research Laboratory Sample Nos. 57 MR-116 and 57 MR-117, indicated the absence of wax in both samples.

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While it was possible to detect and determine waxes in the particular samples examined, the possibility still exists that resins used in other commercial curing compounds might interfere with the spectrophotometric determination of waxes because the waxes and resins generally used are petroleum derivatives and may possess in common the particular organic groups which absorb at 13,7 and 13.9 microns. For unknown compounds. therefore, it would be advisable to supplement the infrared examination with chemical tests for the determination of wax.

In this connection, we feel that all new compounds submitted for qualification or acceptance under our current specifications should be tested for wax before approval for use. Moreover, it would be desirable to perform such tests once a year at the beginning of the season on all brands of membrane curing compounds used in State construction to verify the absence of wax in these materials.

> E. A. Finney, Director Research Laboratory

HAF:la

cc: C. J. Olsen

Table 1
Concrete Curing Compound Samples

Company or Brand Name	Batch No.	East Lansing Lab. No.	Ann Arbor Lab. No.	Drying Time, Minutes	Moist. Retentic Percent
Artco	12	57 MR-95	57 CH-267	78	98.5
Permite	542	57 MR-96	57 CH-271	tracked off,	98 .c
Servicised	706 M-03	57 MR-97	57 CH-284	after 96 hours 77	98.3
Phenix-Murphy	41	57 MR-98	57 CH-325	150	97.3
Truscon	2345J	57 MR-99	57 CH-195	240	97.1
Phenix-Murphy	25	57 MR-100	57 CH-87	50	97.1
Permite	547	57 MR-101	57 CH-68	33	99.3