SOME LABORATORY EVALUATIONS OF BITUMINOUS COATINGS

Harold C. O'Brien, Jr.
Royston Laboratories, Inc., Pittsburgh, Pa.

Bituminous Substances have been used as preservatives and protective coatings for thousands of years. Their effectiveness is based largely upon their resistance to moisture and chemical attack. The term "Bituminous Substances" includes a wide variety of chemical compounds. These are the native asphalts, oils and resins, of both animal and vegetable origin, the products derived from them by further treatment and chemical means, the refined and chemically treated petroleum asphalts and coal tar products, and the pitches and residues from the treatment and processing of resins and oils.

The current major volume of bituminous materials, by-products of the coal and petroleum industries, have been developed and used as protective coatings over a long period. This has been due to their ready availability, low cost per unit volume and to their performance characteristics.

Many bituminous compounds have performed as underground protective coatings exposed to soil and water for a half century. With such long service performance, shorter-term laboratory performance tests are a requisite in order to evaluate the newer bituminous compounds, other coatings, and particularly those applied at lesser film thicknesses than in past practices.

In corrosion control the most effective property of a coating is electrical insulation. The proper insulative values limit galvanic and cell type corrosion. This fact applies generally, for coatings used both above and below ground or

immersed in electrolyte. Water may destroy mechanically, by water sorption and desorption, coatings in transient water exposures, flexing the coatings to physical failure. Water may destroy the electrical insulation property of coatings by penetrating and carrying in electrolytes and acting as an ionizing vehicle to solubilize coating constituents. Water-saturated coatings are, in effect, a broth of electrolyte and have very limited protective values for metal. Therefore, to preserve the utmost coating integrity and performance, water must be excluded.

In order to accomplish this, the compounder must produce a coating which will resist water absorption at least to a degree sufficient to prevent its being penetrated by water to become water-saturated and thereby electrically conductive. The compounder must develop means of evaluating the materials already known to have excellent performance in this property and means of studying these materials in relation to new compounds and systems.

The criteria which must be determined in order to produce new coatings equal to past excellent ones are: (1) water absorption; (2) water penetration; (3) water saturation; (4) electrical insulation values; and (5) performance in relation to the flow of galvanic currents.

WATER ABSORPTION

Much work has been done in determining the water absorption properties of coal tar and asphalt by gravimetric methods[1, 2]. Earlier workers applied coatings to glass or metal and determined the sample weight increases after immersing in

water for various periods of time[3]. The increase in sample weight generally was expressed as an increase percent by weight and was assumed to be entirely due to the absorption of water. In most cases the possibility of solution of the sample or sample loss to the water was not considered. A.S.T.M. Method D-95 uses flat free films of the coating material as samples and in this method the actual water absorbed is removed from the weighed sample, collected and measured[4]. The results may be expressed either as weight percent increase, or as weight per unit area exposed to the water. Most workers agree that for coatings which may vary widely in specific gravity, it is preferable to express the figure as weight gain per unit of area exposed.

The samples of this paper were prepared as free films by coating on Mylar sheet and stripping after cooling or drying. Either hot- or cold-applied materials may be stripped from Mylar. The removal of most tacky compounds can be effected by chilling the sample. The free films are cut to measure approximately 5.0 x 10.0 cm. per side. The thickness is approximately 100 mils. The exposed area is calculated to be roughly 100 sq. cm. These samples are (after sponging dry the surfaces) immersed in tap water of 70,000 ohms cm^3 resistivity and are weighed at intervals for a period of 1,000 hours. This period was chosen after it was discovered that no trend of early equilibrium could be established in short-term gravimetric tests. This is also evidenced by the work of Henry Lee[5]. In most cases, after attaining the gravimetric-time curve for at least 1,000 hours, the samples are removed and a desorption or weight loss-time curve is obtained. It was found that such a procedure depicts any loss in weight

by leaching from the sample.

Figure 1 shows gravimetric curves for hot-applied plasticized coal tar enamel and for room-temperature cured coal tar-epoxy. The exposure was at 70°F and demonstrated that at this temperature there is some leaching loss with the plasticized coal tar enamel and none with the epoxy compound. The leaching effect can be so pronounced as to become the major effect with some bituminous substances such as Gilsonite (Figure 2) which continuously dissolves in water at room temperature.

A number of gravimetric absorption tests were made at 160°F, and it was found that practically no bituminous material, either coal tar or asphalt, was suitable for exposures at these temperatures. As shown in Figure 3, coal tar enamel at 160°F absorbs more than twice as much water as it does at 70°F. The leaching effect in this example is not noticeable at 70°F. It is, however, considerably more pronounced than the absorption effect when carried out at 160°F. For a total of thirty bituminous samples tested at 70°F and 160°F, the average absorption at 160°F was approximately fifteen times that at 70°F. A series of bituminous materials representing those which are most resistant to water absorption is shown in Figure 4. From this chart, one may determine that both asphalts and coal tar materials can be highly resistant to water absorption at room temperature. It is also shown that certain solvent solution cold-applied materials are as resistant as the hot-applied resins. The asphalts, other than asphalt enamel, are all blown asphalts of similar physical properties but of different origin. They vary more in absorption properties at 160°F than at 70°F. It would appear characteristic

of coal tar types to leach somewhat at 70°F and considerably at 160°F. With asphalt types this is not evidenced.

Aside from the inherent water susceptibility of a particular bituminous resin itself, it is characteristic that the presence of loading compounds, fillers, talcs, etc., in such a bituminous coating compound can seriously affect the properties of water resistance. While pigments and loading compounds are frequently added to improve the physical properties of tensile strength, flow and deformation resistance, hardness, etc., pigmentation always results in increased water absorption. It is essential, therefore, that only pigments low in water susceptibility and ion content be used, in order to keep water absorption low and to prevent water saturation and electrical conductivity upon prolonged exposures to water.

Figure 5 demonstrates the effect of variations in weight percent additions of such a carefully chosen pigment to a cold-applied insulative asphalt coating. The exposure was at 70°F. While 25% additions do not markedly increase water absorption above the value for the resin alone (30 milligrams) percentages higher than this are certainly clearly detrimental to water absorption resistance. Typical values for coal tar and asphalt enamels are found to be in the range of 20 to 35%.

It should be pointed out that the degree of water absorption in coating materials is determined to a large extent by the effects of osmotic pressure. Elm[6] showed that the higher the osmotic pressure of a solution the lower the rate of water absorption. Using distilled water, he found no equilibrium

established after 800 hours of immersion. Elm also pointed out the increased water absorption in films subjected to potential gradients, the electroendosmotic effect. It can be understood, therefore, that solution gradients produced by chemical electrolytes are quite apt to be different than those produced by distilled or tap water. For this reason, as well as due to specific chemical reactivities, electrolyte weight absorptions may vary from those obtained with water with the specific electrolyte and with the particular bitumen or coating compound. (Figure 6).

WATER PENETRATION BY ELECTRICAL METHODS

A number of workers have explored methods to relate electrical measurements to water absorption and to water saturation of coating materials. Arthur[7] points out the value of electrical resistance measurements as a means of determining "failure to protect." His test is not an accelerated test.

While the volume resistivity values of most bituminous substances fall in the range of 1×10^{12} ohms cm.^{3} to 1×10^{15} ohms cm.^{3}, there occurs a wide variation in their resistance values measured during immersion in water. From time to time, resistance values may vary up or down 10, 100 or even 1,000 times the prior reading. Adventitious pinholes or conductive leakage paths are formed giving little indication as to whether the entire coating volume is conductive or merely a single tiny area.

Conway and Smith[8] suggest the use of magnetic resonance to determine the moisture content in hygroscopic materials. While

the method is non-destructive and fast, it may not prove suitable for use at low percentage values of water in coatings such as are found with bituminous materials. McKinley and Seaberg[9] used gravimetric data, specific inductive measurements, and power factor values to set up minimum specification performance criteria for cable insulation. They showed early failures with voltage and current stresses, but they could not demonstrate a suitable short-term test to predict insulation breakdown.

Brasher and Kingsbury[10] in 1954 set forth a comparison of gravimetric methods and capacitance methods for measuring water absorption. They pointed out that it is possible to estimate water up-take from the capacitance changes that occur during the immersion of the painted panel. They suggested methods of water distribution within the coating and that possibly water is distributed in layers parallel to the coating surface.

At the time of such early workers, we developed a method particularly suitable for the water-capacitance evaluation of bituminous compounds. We found that the distribution of water in these insulative type coatings with limited concentrations of pigments could be considered to be essentially in layers parallel to the sample surfaces. For most bituminous type coatings we have been able to corroborate this, and we have also found it particularly valuable to relate the changes in capacitance to a calculated value of the actual depth of penetration of the water front into the sample.

Whereas, volume resistivity values indicate either pinhole penetration or absorption or a combination of both in a

qualitative fashion, the dielectric constant values more nearly indicate the water absorption, quantitatively. While an adventitious pinhole may affect the volume resistivity reading adversely, and change the value by its presence several thousand percent, it will produce only a small effect in the dielectric constant value of the sample.

Since the dielectric constant value of immersion water is from eighty to several hundred times that of air and since the dielectric constant values of electrically insulative bituminous coatings are much nearer to those of air than to the values of water or electrolytes, the inclusion of water within an insulating coating material so markedly changes its dielectric constant value that a sensitive index to quantitative absorption and depth of penetration is provided.

A series of dielectric constant values readily obtained from water immersed samples furnish indices to the quantity of water absorption or depth of penetration of water saturation at any instant. When these values are taken over a period of time and the capacitance values indicate that the value of dielectric constant for the sample is 81 (distilled water), water saturation and penetration are complete and the coating will have little, if any, protective value.

The rate of water absorption with time may be plotted and the theoretical t_1, or penetration depth, may be used as an index of coating performance.

After only a few weeks exposure, curves of t_1 values versus time show a tendency with good coating material to establish t_1 at a very low rate of change. Study of these

early curves furnishes an index of ultimate performance and aids in establishing a predicted time of saturation failure.

A condenser specimen is set up using a 4" x 10" panel on which is placed a uniform coating. The thickness is measured accurately. A one pint can with bottom removed is placed against the dried coating and sealed around the edge with caulking compound. The can is then filled with water, thus forming a parallel circular plate condenser with water as one plate and the steel panel as the other (Figure 7). The diameter of the can is measured to calculate the area of one of the plates.

Immediately, upon filling, the initial capacitance and resistance are measured. The specific inductive capacity (K_o) for the whole coating insulation is calculated by using the following equations for parallel circular plate capacitors:

$$C_o = \frac{.0885\ K_o \cdot S}{t} \qquad\qquad K_o = \frac{C_o\ t}{.0885 \cdot S}$$

Where in the above; (initial capacitance reading) C_o is in micro micro farads (MMfd.), K_o is the dielectric constant, S is the area of one plate in square centimeters, and t is the thickness in centimeters.

By considering the coating as made up of two series circular plate capacitors (Figure 8) composed of (1) C_1 a thin capacitor of water-saturated dielectric insulation, and (2) C_2 a thicker capacitor of water-free dielectric insulation, calculation of the approximate water penetration depth, t_1, can be made.

The total capacitance $C = \frac{C_1 \quad C_2}{C_2 + C_1}$

The capacitance for the water-saturated dielectric insulation layer is: $C_1 = \frac{.0885 \cdot 81 \cdot S}{t_1}$ Note: $K_1 = 81$

The capacitance for the water-free dielectric insulation layer is: $C_2 = \frac{.0885 \cdot K_2 \cdot S}{t_2}$ K_2 = the initial K_o value calculated

Equations:

$$\frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2}$$

$$\frac{1}{C} = \frac{1}{\frac{.0885 \cdot 81 \cdot S}{t_1}} + \frac{1}{\frac{.0885 \cdot K_2 \cdot S}{t_2}}$$

$$\frac{1}{C} = \frac{t_1}{.0885 \cdot 81 \cdot S} + \frac{t_2}{.0885 \cdot K_2 \cdot S}$$

Also:

$$t = t_1 + t_2$$

$$t_2 = t - t_1$$

$$\frac{1}{C} = \frac{t_1}{.0885 \cdot 81 \cdot S} + \frac{t - t_1}{.0885 \cdot K_2 \cdot S}$$

$$\frac{1}{C} = \frac{t_1}{7.1685\ S} \quad \frac{t - t_1}{.0885 \cdot K_2\ S}$$

The value for K_2 is the same as for K_o at the start of the test when $t_1 = 0$ i. e.

$$C_o = \frac{.0885 \cdot K_2 \cdot S}{t} \quad \text{and } K_2 = \frac{C_o\ t}{.0885 \cdot S}$$

Then, substituting for K_2:

$$\frac{1}{C} = \frac{t_1}{7.1685 \cdot S} + \frac{t - t_1}{\frac{C_o\, t}{.0885 \cdot S}\,(.0885 \cdot S)}$$

$$\frac{1}{C} = \frac{t_1}{7.1685 \cdot S} + \frac{t - t_1}{C_o \cdot t}$$

$$7.1685\, C_o \cdot S \cdot t + C \cdot C_o \cdot t \cdot t_1 + 7.1685\, C \cdot S\, (t - t_1)$$

$$7.1685\, C_o \cdot S \cdot t = C \cdot C_o \cdot t \cdot t_1 + 7.1685\, C \cdot S \cdot t - 7.1685\, C \cdot S \cdot t_1$$

$$t_1\, (C \cdot C_o t - 7.1685\, C \cdot S) = 7.1685\, C_o \cdot S \cdot t - 7.1685\, C \cdot S \cdot t$$

$$t_1 = \frac{7.1685 \cdot S \cdot t\, (C_o - C)}{C \cdot C_o t - 7.1685\, C \cdot S}$$

t_1 is the depth of water penetration in centimeters.

t_2 is the thickness of water-free dielectric in centimeters.

S is the area in centimeters of one plate of the capacitor.

t is the thickness of the coating in centimeters.

C_o is the initial measured capacity in micro micro farads.

C is the present capacity of the capacitor.

C_1 is the series capacitor of water-saturated dielectric ($K_1 = 81$).

C_2 is the water-free series capacitor in micro micro farads.

Using the above equation, and several substituted values of capacitance between the initial capacitance C_o and a final value when the depth of penetration t_1 is equal to the specimen thickness t, a graph of capacitance versus depth of penetration may be plotted, example, Figure 9. From this graph, for any measured value of capacitance, t_1, the depth of penetration of water through this particular coating can be found.

Figure 10 shows a sample curve which in two weeks levels off at about 70 percent penetration. Such a curve is valuable in predicting life, ascertaining minimum film thickness for an application, and in evaluating the relative absorption characteristics of the coating system.

In addition to the use of water as an environmental contacting agent, various other electrolytes, varying in values of dielectric constant, conductivity, pH, osmotic pressure and other specific effects upon the coating may be used. The sample as shown, Figure 7, is particularly adaptable to the above investigations as well as for the study of the effects of galvanic phenomena such as polarity and cathodic currents. Anodes, cathodes and potential carrying electrodes are insertible within the electrolyte contained in the can.

Using this method for bituminous materials, it was found that in some cases several months are required to reach equilibrium. As shown in Figure 11, one sample required ten months. In most cases, much earlier predictions of coating life can be made. As shown in Figure 12, a group of the better bituminous materials was evaluated by this method for 400 days. At the end of this period, all samples were over 10^{14} ohms cm.^{3} in resistivity. The original thickness, t, the original dielectric constant, K, and the depth of penetration, t_1, are shown. As with the gravimetric absorption method, t_1 varies in this group of samples. From this it can be concluded that coal tar and asphalt coatings, both hot- and cold-applied, are similarly satisfactory in this test.

Typical water penetration curves for hot-applied coal tar enamels are shown in Figure 13. Generally the higher plasticizer and pigment content of plasticized enamels cause greater water penetration values than for the standard enamels. Standard enamels or hard coal tar pitches may demonstrate cracking and thus show more erratic results.

Shown in Figure 14 are three water penetration curves for hot-applied asphalt coatings. It may be seen from such a curve as that for enamel G that a sufficient minimum mil film thickness specification must be considered in view of the depth of penetration and the rate of penetration increase with time. At 100 days, the water is saturated 25 mils into the sample and is increasing penetration at approximately three mils per year.

In Figure 15 three water penetration curves are shown for cold-applied asphalt and coal tar coatings demonstrating again that no great differences need exist between coal tar and asphalt coatings. At applied thicknesses double their penetration depth, the projected life of each, to a state of water-saturation, is a considerable period.

Where it is desired to combine bituminous materials such as a bitumen resin with epoxy resin and attain the necessary physical properties to permit thin coating applications, it becomes necessary to evaluate water penetration values to determine a minimum safe film thickness for satisfactory projected life. In Figure 16 is a bitumen-epoxy showing a projected life of approximately fifty years at 20 mils film thickness.

In order to study the smaller compounding effects produced by variations in types of pigment in the same base

formula of a bituminous coating, it is advantageous to combine the methods of gravimetric absorption and water penetration as in Figure 17. While all samples are over 10^{14} ohms cm.^{3} in resistivity, after 400 days some show increased leaching over others, and the capacitance penetration values can vary over 100%, a sensitive index to such compounding changes.

It was pointed out in the studies of gravimetric water absorption that higher temperatures (160°F) produce more rapid water absorption than is attained at room temperature. This same effect is noted as in Figure 18 when the capacitance method is employed. All samples failed within five weeks at 190°F.

In order to determine the electro-osmotic effect of applied potentials and their relation to water penetration by the capacitance method, several bituminous samples were subjected to water anodic potentials of 90 volts and 360 volts. The resultant curves are shown in Figure 19. Within three days the curves are well established. Early failures are predicted where resistivity values are relatively low, i.e., under 1×10^{12} ohms cm.^{3}. Where resistivity values are high, i.e., over 1×10^{14} ohms cm.^{3}, attenuation and equilibrium occur but at a faster rate than with no applied potential.

In review of the gravimetric water absorption and water penetration data obtained for bituminous materials, it appears that each method is a valuable tool for relatively short-term evaluation. By combining data, there is a definite relationship for a particular type of bituminous coating. Once this is established, either test may supplant or implement the

other. Figure 20 depicts the relationship for a bitumen-epoxy coating giving values for penetration and for 1,000-hour gravimetric absorption.

As in Figure 21, it is sometimes convenient to plot extended life in years using both the data obtained for weight absorption and capacitance penetration. Thus, one is able to determine a useful life based upon a specified initial film thickness application.

CATHODIC CURRENT DISBONDING TESTS

In addition to insulative coating evaluation by the methods described, it has become current practice to test insulative coatings immersed in electrolytes and subjected to cathodic potentials varying from less than one volt to several hundred volts. It is interesting to note that most workers have attempted to find a short-term cathodic current disbonding test for evaluating performance life of insulative coatings.

There are presently many variations of this type test being carried out. Higher voltages and currents and electrolytes of higher conductivities than are encountered in service are employed to produce fast breakdown. In general, it is difficult to recognize or control all of the factors contributing to coating breakdown. Some of the factors which we have found to be of importance are: (1) the pH values produced at the cathodes; (2) the applied potential; (3) the nature of the anode (Weast)[11]; (4) the temperature; (5) the solution concentration and conductivity; (6) the type of surface preparation; (7) the type of primer; (8) the area exposed; (9) the electrical

resistivity value of the applied coating; and (10) the thickness of the sample.

Our cathodic current disbonding tests are generally carried out using entirely coated steel panels with waxed edges. A given area of panel is immersed in distilled water, tap water, or other more conductive electrolytes. While different anodes produce different pH values, we have found magnesium to be quite effective in producing high cathode pH values. Since magnesium is most commonly used in practice, we apply 1.5 volts D.C.$^{+}$ to magnesium as the anode and 1.5 volts D.C.$^{-}$ to the samples as cathodes. Small holes are drilled through the coatings to initiate areas of current flow. Initial current readings are taken and successive readings are made at daily intervals. When the coatings display a disrupted appearance, they are removed and the disbonded coatings are cut away to expose the entire bared area. This area is measured and can be related to current, potential and time.

In our work with cathodic current tests of bituminous materials, we have observed that thin films are readily disbonded. This is in accord with the fact that thin films are readily and quickly penetrated by water and electrolytes especially when migrating under potential stress. Tomashov et al.[12] have found that the coating ifself can become cathodic,acting as a conductive film.

Cathodic disbonding susceptibility due to thinness is possibly due also to the factor of increased current flow at short-length pinholes in comparison to pinholes which are axially

long, even though the diameters of each are alike. The effect is explained by Sunde [13].

It is interesting to note that Koenecke[14], who places coating specimens under anodic and cathodic stress and measures capacitance index (Shaw and Twiss)[15], resistance, and disbonding, concludes that a basic film thickness must provide an insulating barrier under either test or service conditions to maintain electrical insulation and adhesion.

When cathode deposits occur at the coating, a thin film can be assumed to be highly saturated with alkalies which can react chemically to disintegrate many bituminous coatings. We have found the asphalt coatings generally to be more susceptible to high pH values than are coal tar materials (see Figure 6). In addition, both animal and vegetable residual pitches which are saponifiable by virtue of unsaturation or other reactive grouping are readily disintegrated in alkali at cathodes.

We have found that the proper metal surface preparation can effect up to several hundred percent increased cathodic current resistance to disbonding. Bituminous coatings over solvent-degreased steel panels disbond approximately five times the area of the same coatings over phosphated or grit-blasted steel. Almost as important as surface preparation is the use of proper primers.

Figure 22 was prepared to demonstrate a number of hot-applied bituminous coatings. Number 1 demonstrates the effect of a particularly good primer applied at 1 mil film thickness. The remaining samples shown without primers demonstrate the

relative resistance of each bituminous type. It can be noted that asphalts are generally more susceptible than coal tar types. Figure 23 shows a similar trend for the cold-applied bituminous coatings.

We have noted that a number of cold-applied type materials having very high values of volume resistivity, i.e., over 1×10^{14} ohms cm.^{3}, tended to disbond more than certain cold-applied materials having much lesser values of resistivity. We have been able to establish that in some cases disbonding is accelerated in materials having very high volume resistivities. Note samples 9 and 10 in Figure 23. By adding 20% coal dust to bring the volume resistivity value from 1×10^{14} to 1×10^{11} ohms cm.^{3}, the disbonded area of sample 10 was reduced to one-eighth that of sample 9. This accelerating effect is possibly due to insulation retention of higher potential differentials in local cathode cells formed at interfaces between bare and coated metal.

When compared to other conventional coating systems, the bituminous materials are highly resistant to cathodic current disbonding. All of the drying oils and oil-modified alkyds, epoxy esters, urethane esters and many vinyl type coatings are highly susceptible to cathode environments.

In Figure 24 the right panel is the same base bituminous asphalt as the left plus the addition of 10% fish oil and 10% zinc chromate. With these additions the asphalt is highly degraded and is readily disbonded.

In Figure 25 the left panel is 20% alkyd and 80% asphalt. The right panel is 100% asphalt.

In Figure 26 the left panel has no primer and the right panel is primed with an iron oxide alkyd primer. The coating is refined cold-applied asphalt mastic. Increased susceptibility to disbonding is noted in the primed panel.

In Figure 27 disbonding due to pigment reactivity is demonstrated. The same base asphalt cold-applied coating is pigmented with glass fibers on the left and with asbestos fibers on the right. The glass is subject to attack by the alkali present.

CONCLUSIONS

The evaluation of long-term performance types of bituminous coatings is possible in the laboratory, based upon laboratory test procedures being considered and established in terms of normal-range exposures.

Water, the single most important factor in the excellent performance life of bituminous coatings, is absorbed by osmotic and electro-osmotic effects. The absorption may be measured gravimetrically and related to quantity per unit area. By this method, also, the solubility or leaching characteristics of certain bitumens may be detected by means of drying and weighing samples after water immersion exposures. Coal tar types were found to be more susceptible than asphalts in this regard. In gravimetric absorption tests carried out at 160°F, this fact was accentuated. At 160°F all of the bituminous materials tested showed multifold absorption increases.

Electrical properties have been studied in relation to water absorption characteristics and to the performance of

bituminous materials as insulative coatings. The bituminous coatings have volume resistivities in the range of 1×10^{12} to 1×10^{15}. It was found that volume resistivity measurements alone do not lend themselves to short-term evaluation methods.

Using bituminous coatings, a new method was developed to derive measurements of depth of water penetration by electrical capacitance. This method permits evaluation of bituminous compounds of known performance against newer compounds. Water penetration values can be co-related to gravimetric absorption. This method permits projected life studies of coatings and permits safe film thickness specifications for new compounds. The method is fast, non-destructive and sufficiently sensitive to detect small changes in exposure conditions or compounding ingredients. It is an effective short-term test giving projected values for the long-life bituminous coatings, well within a 400-day test period. The philosophy of a short-term rather than an accelerated test is preserved.

In applying accelerated test methods such as the cathodic current disbonding test to bituminous materials, it was found that there can be many factors which affect the results obtained. Using a moderate version of the cathodic current disbonding procedure, a number of bituminous materials were evaluated and were found to be, in general, superior to many other coating types.

REFERENCES

1. Fair, W. F., Jr., "Properties Specifications, Tests and Recommendations, Part 1.," Corrosion, November, 1956, p. 519.

   Fair, W. F., Jr., "Properties Specifications, Tests and Recommendations, Part 2.," Corrosion, December, 1956, p. 605.

2. Hayes, H., Journal American Water Works Association, Vol. 32, pp. 1705-1722.

3. McComb, G., "Electrical Insulation of Underground Pipelines, Part I," The Pipeline Engineer, July, 1957, p. D-38.

4. Shideler, N. T., "Testing and Evaluation of New Coating Materials," Paper to American Gas Association, Dallas, Texas, May 18-19, 1959.

5. Lee, Henry, "Attaining Equilibrium in Water Absorption Tests of Epoxy Coating Resins," Materials Research and Standards, November, 1963, p. 910.

6. Elm, A. C., Official Digest, 1949, Vol. 267, p. 197.

7. Arthur, Marion A., "Electrical Method for Evaluation of Protective Coatings as Moisture Barriers," ASTM Bulletin, September, 1949.

8. Conway, T. F., and R. J. Smith, "Magnetic Resonance Determines Moisture," Electronics (Engineering Edition), 1958.

9. McKinley and Seaberg, "Moisture Vs. Rubber Insulation," General Electric Review, July, 1952.

10. Brasher, D. M., and A. H. Kingsbury, "Electrical Measurements in the Study of Immersed Paint Coatings on Metal. 1. Comparison Between Capacitance and Gravimetric Methods of Estimating Water Uptake," Journal of Applied Chemistry, February, 1954.

11. Weast, Robert C., and Donald C. Melrose, "Influence of Anodes on Solution pH in Salt Crock Test," Materials Protection, March, 1965.

12. Tomashov, N. D., Yu N. Mikhailovskii and V. V. Leonov, "Mechanism of Electrochemical Corrosion of Metals Under Insulating Coatings. I. Kinetics of Deterioration of Insulating Coatings on Electrolytes," Corrosion, Vol. 20, No. 4, April, 1964, pp. 125-128.

Tomashov, N. D., Yu N. Mikhailovskii and V. V. Leonov, "Mechanism of Electrochemical Corrosion of Metals Under Insulating Coatings. II. Kinetics of Cathodic Processes on Insulated Metals in Electrolytes," Corrosion, Vol. 20, No. 7, July, 1964, pp. 218-221.

13. Sunde, E., Earth Conduction Effects in Transmission Systems, Princeton, New Jersey: D. Van Nostand Company, 1949, p. 200.

14. Koenecke, D. F., "Organic Coatings and Impressed Currents," Enjay Laboratories.

15. Shaw, Manuel, and Sumner B. Twiss, "The Capacitive Index as a Measure of Organic Coating Deterioration," Pre-Prints of Papers, American Chemical Society, Dallas Meeting, April, 1956, pp. 47-58.

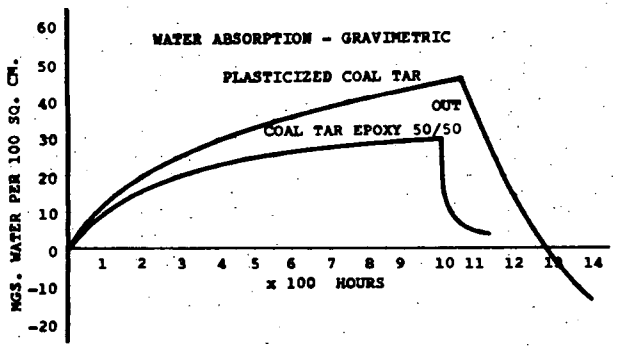

Fig. 1

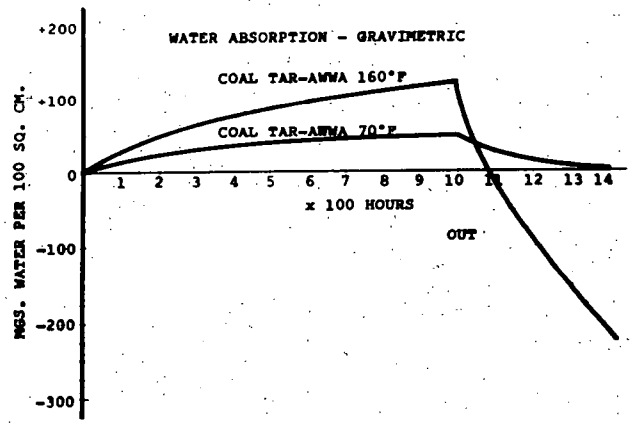

Fig. 3

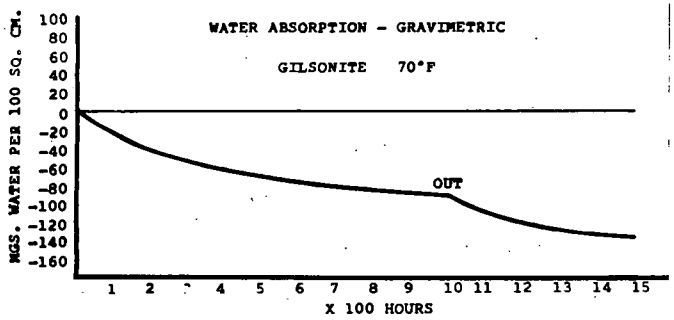

Fig. 2

WATER ABSORPTION - GRAVIMETRIC

MGS. PER 100 SQ. CM. PER 1000 HOURS IMMERSION - TAP WATER
VOLUME RESISTIVITY = 70000 (OHMS)

TYPE	MG. GAIN AT 70°F	LOSE ON DRYING	MG. GAIN AT 160°F	LOSE ON DRYING
ASPHALT M 220°F (HOT-APPLIED)	30	0.0	—	—
ASPHALT SOLUTION 240°F (COLD-APPLIED)	33	0.0	—	—
ASPHALT OXIDIZED ALIPHATIC 200°F (HOT-APPLIED)	36	0.0	156.00	0.00
ASPHALT OXIDIZED ALIPHATIC 230°F (HOT-APPLIED)	38	0.0	184.00	0.00
ASPHALT H 220°F (HOT-APPLIED)	39	0.0	270.00	0.00
COAL TAR PLASTICIZED ENAMEL AWWA (HOT-APPLIED)	40	-5.0	120.00	-346.00
COAL TAR STANDARD ENAMEL PITCH (HOT-APPLIED)	40	-7.0	—	—
ASPHALT-STYRENE (COLD-APPLIED)	40	-42.0	438.00	-187.00
ASPHALT PIPELINE ENAMEL 4	40	0.0	—	—
MYLAR (POLYESTER) PRE-FORMED FILM	41	0.0	—	—
COAL TAR PLASTICIZED ENAMEL (HOT-APPLIED)	42	-13.0	930.00	-774.00
ASPHALT S 220°F (HOT-APPLIED)	44	0.0	1053.00	0.00
ASPHALT K 220°F (HOT-APPLIED)	47	0.0	556.00	0.00
ASPHALT H2 220°F (HOT-APPLIED)	49	0.0	533.00	0.00
ASPHALT E 220°F (HOT-APPLIED)	55	0.0	270.00	0.00
ASPHALT (REFINED) COATING 240°F (COLD-APPLIED)	64	0.0	702.00	0.00
COAL TAR EPOXY (AMINE CURE) 70°F CURE	76	-19.0	-31.00	-200.00
COAL TAR-NITRILE COATING (COLD-APPLIED)	83	-7.0	3800.00	-423.00
COAL TAR MASTIC CA-50 (COLD-APPLIED)	107	-208.0	1400.00	-730.00

Fig. 4

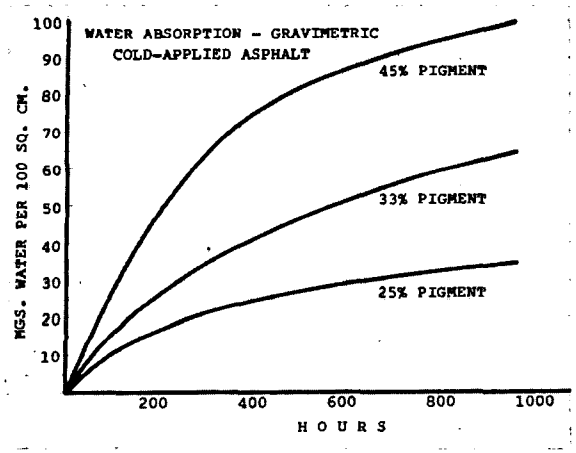

Fig. 5

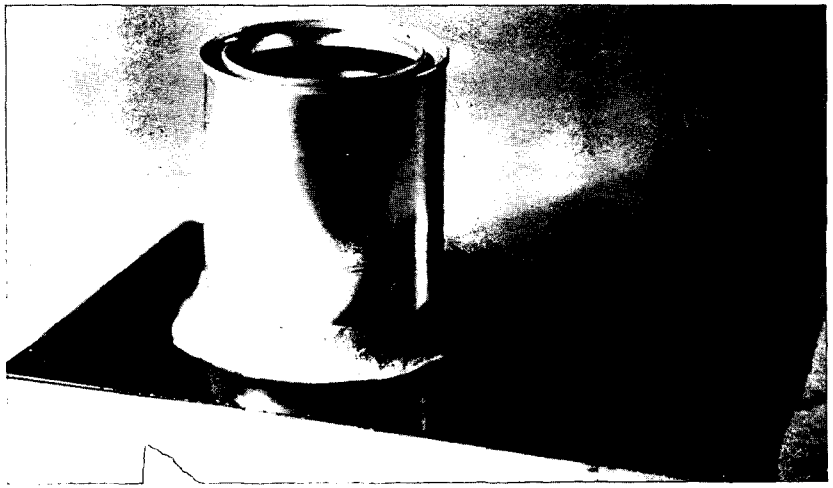

Fig. 7

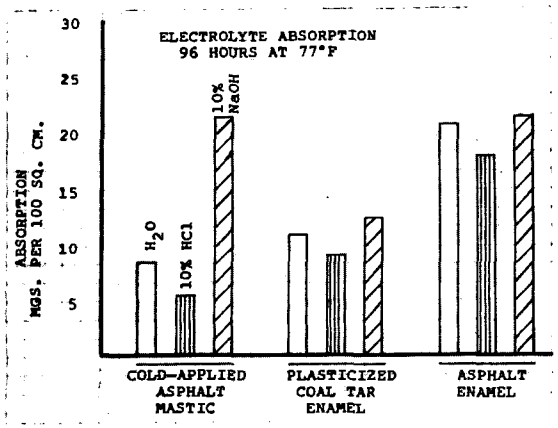

Fig. 6

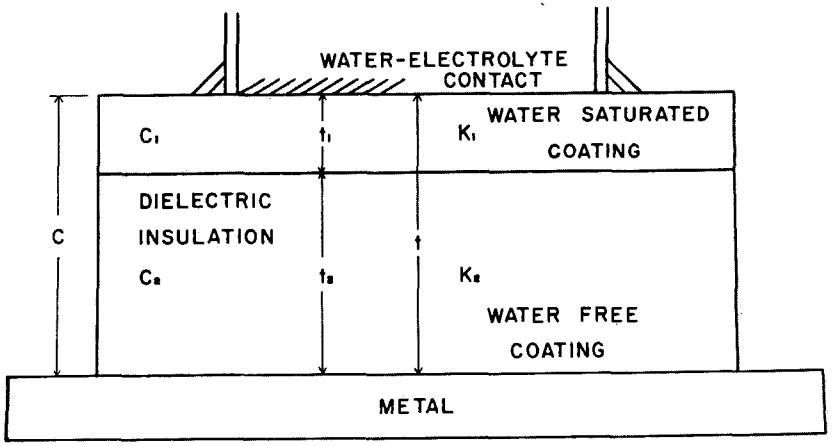

Fig. 8

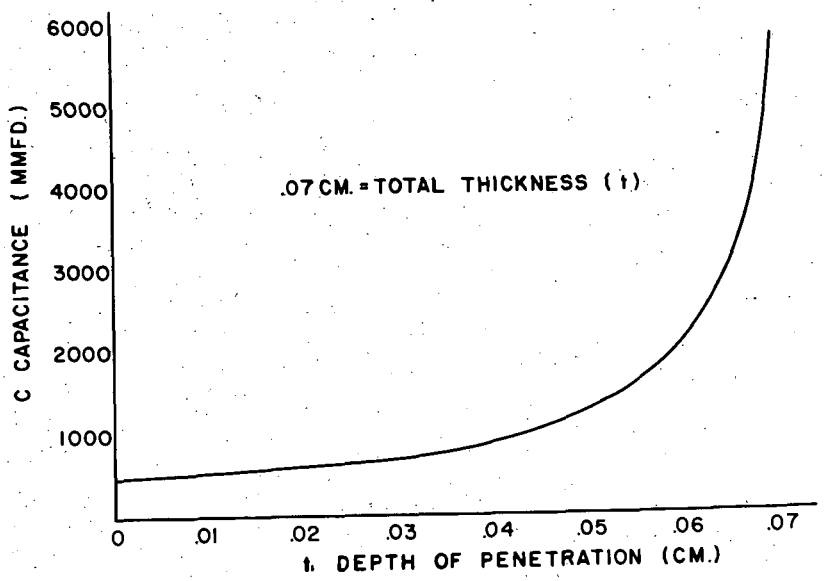
6000
5000
4000
3000
2000
1000
C CAPACITANCE (MMFD.)
.07 CM. = TOTAL THICKNESS (t)
0
.01
.02
.03
.04
.05
.06
.07
t, DEPTH OF PENETRATION (CM.)

Fig. 9

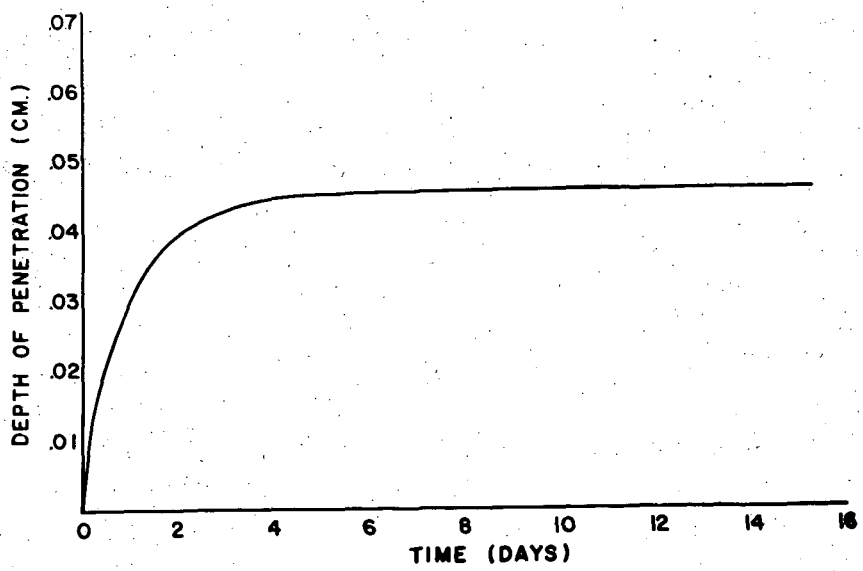
.07
.06
.05
.04
.03
.02
.01
DEPTH OF PENETRATION (CM.)
0
2
4
6
8
10
12
14
16
TIME (DAYS)

Fig. 10

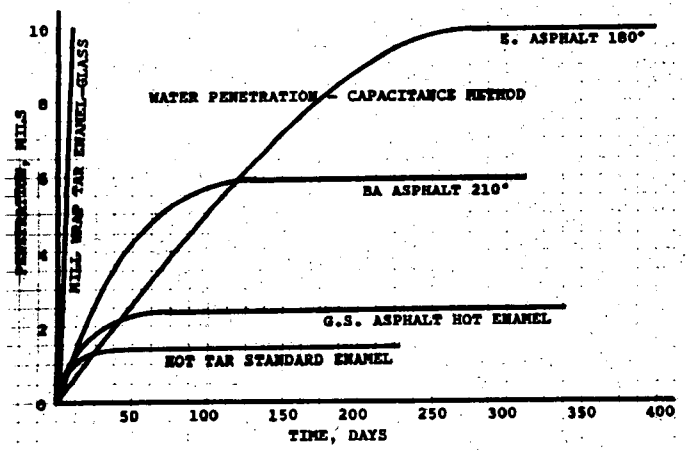

Fig. 11

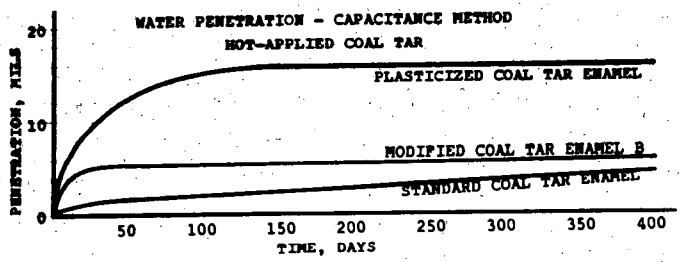

Fig. 13

WATER PENETRATION - CAPACITANCE METHOD

400 DAYS - 70°F WATER - VOLUME RESISTIVITY 70000 OHMS

TYPE	t (MILS)	(K)	t_1 (MILS)	R (OHMS)
ASPHALT M 220°F (HOT-APPLIED)	18	3.89	2.00	over 10^{14}
ASPHALT SOLUTION 240°F (COLD-APPLIED)	24	4.92	4.92	over 10^{14}
ASPHALT OXIDIZED ALIPHATIC 200°F (HOT-APPLIED)	51	6.34	2.00	over 10^{14}
ASPHALT OXIDIZED ALIPHATIC 230°F (HOT-APPLIED)	36	5.55	1.50	over 10^{14}
ASPHALT H 220°F (HOT-APPLIED)	95	6.68	1.50	over 10^{14}
COAL TAR PLASTICIZED ENAMEL AWWA (HOT-APPLIED)	73	14.23	6.00	over 10^{14}
COAL TAR STANDARD ENAMEL PITCH	38	3.90	8.00	over 10^{14}
ASPHALT-STYRENE (COLD-APPLIED)	26	7.50	6.00	over 10^{14}
ASPHALT PIPE ENAMEL 4 (HOT-APPLIED)	39	10.95	1.00	over 10^{14}
COAL TAR PLASTICIZED ENAMEL (HOT-APPLIED)	70	9.40	16.00	over 10^{14}
ASPHALT S 220°F (HOT-APPLIED)	106	7.40	2.00	over 10^{14}
ASPHALT K 220°F (HOT-APPLIED)	86	7.25	2.30	over 10^{14}
ASPHALT H2 220°F (HOT-APPLIED)	113	8.70	2.60	over 10^{14}
ASPHALT E 220°F (HOT-APPLIED)	65	5.49	3.20	over 10^{14}
ASPHALT (REFINED) COATING (COLD-APPLIED)	12	17.30	4.80	over 10^{14}
COAL TAR-EPOXY AMINE CURE (COLD-APPLIED)	20	6.40	4.00	over 10^{14}
COAL TAR-NITRILE COATING (COLD-APPLIED)	25	8.26	6.75	over 10^{14}
COAL TAR MASTIC CA-50 (COLD-APPLIED)	31	9.00	17.00	over 10^{14}

Fig. 12

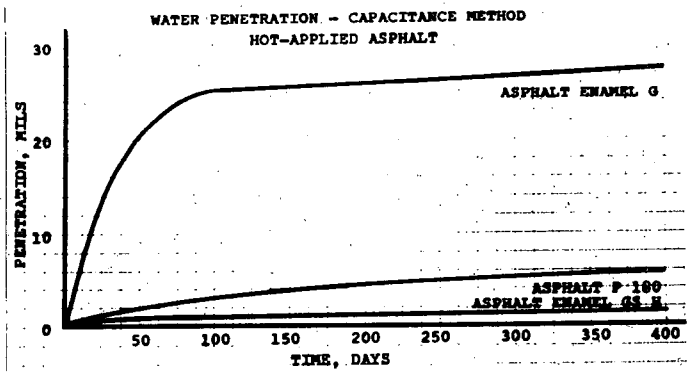

Fig. 14

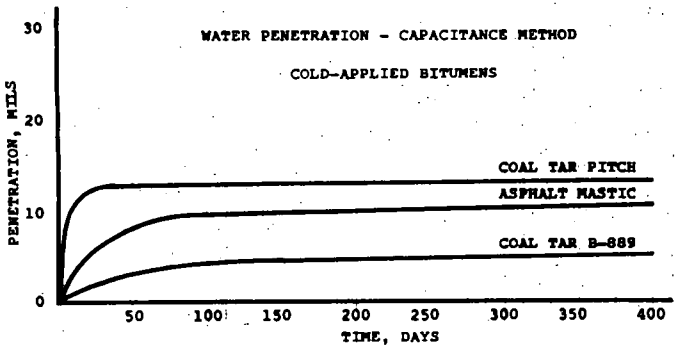

Fig. 15

WATER ABSORPTION - GRAVIMETRIC
AND
WATER PENETRATION - CAPACITANCE METHOD

USING A COMMON BASE FORMULA - REFINED ASPHALT
(COLD-APPLIED) AND VARYING PIGMENT TYPES
(35% PIGMENT)

FORMULA	MGS. GAIN PER 100 SQ. CM. PER 1000 HOURS	MGS. LOSS ON DRYING	t' (MILS)	(K)	VOLUME R.	t_1 (MILS) PENETRATION
B-909	22.0	-17	22	9.18	over 10^{14}	5.50
B-910	23.0	-20	27	10.40	over 10^{14}	7.60
B-911	19.0	-42	18	8.24	over 10^{14}	5.50
B-912	15.7	-39	23	7.92	over 10^{14}	10.70
B-913	15.6	-50	22	9.87	over 10^{14}	7.10
B-914	9.5	-54	25	8.05	over 10^{14}	11.70
B-915	47.0	-53	24	8.48	over 10^{14}	12.20
B-916	37.0	-51	20	9.97	over 10^{14}	6.70
B-917	-12.0	-82	24	10.56	over 10^{14}	8.05
B-918	11.0	-53	23	10.63	over 10^{14}	6.80

Fig. 17

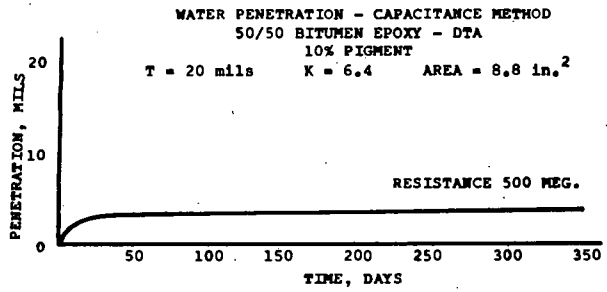

Fig. 16

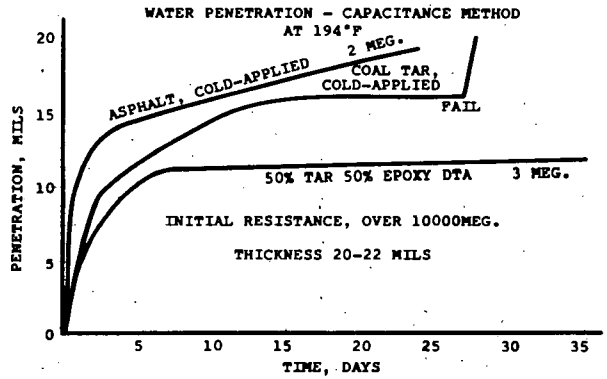

Fig. 18

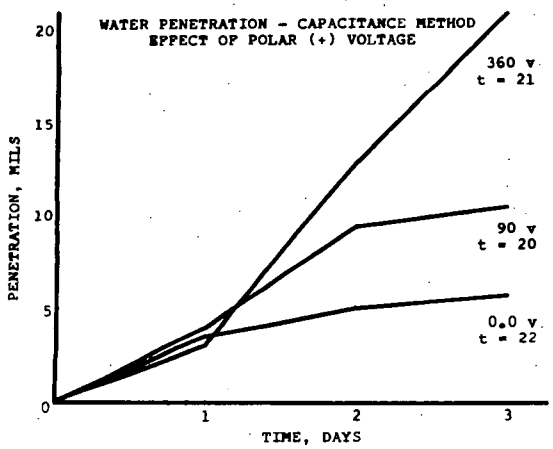

Fig. 19

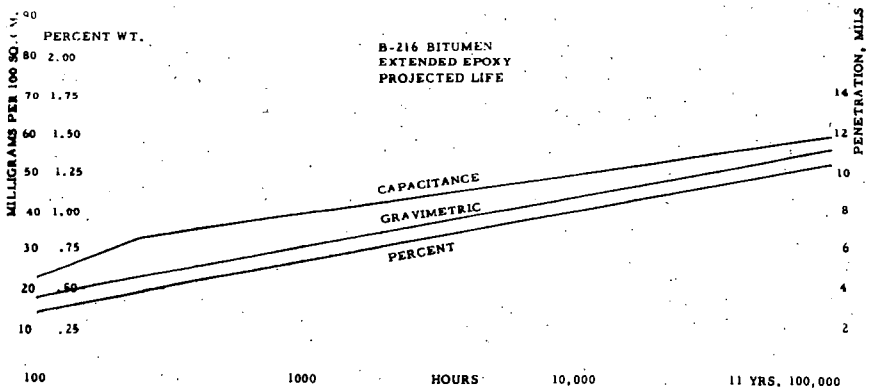

Fig. 21

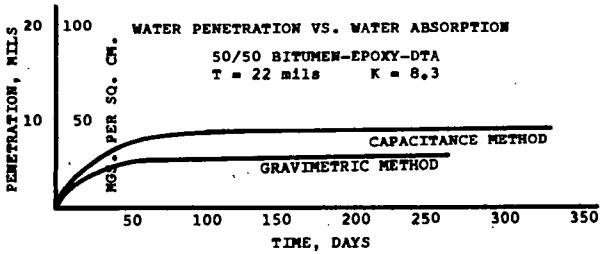

Fig. 20

CATHODIC CURRENT DISBONDING TESTS

COATED 2 X 4 INCH PANELS IMMERSED 2½" IN 70000 OHMS, R, WATER, 12 CM. FROM MG. ANODE, 1.5 VOLT CLOSED CIRCUIT POTENTIAL, 1/16" PINHOLE THROUGH PANEL IN SOLUTION.

	COATINGS (HOT APPLIED)		DISBONDED AREA SQ. M.M.
1.	ACRYLIC PRIMER + PLASTICIZED COAL TAR ENAMEL		1.0
2.	MODIFIED COAL TAR ENAMEL	(NO PRIMER)	56.0
3.	COAL TAR STD. ENAMEL PITCH	" "	144.0
4.	ASPHALT (OXIDIZED ALIPHATIC)	" "	380.0
5.	COAL TAR STANDARD ENAMEL	" "	500.0
6.	WURTZELITE ASPHALT	" "	621.0
7.	COAL TAR PLASTICIZED ENAMEL A.W.W.A.	" "	800.0
8.	COTTONSEED PITCH BITUMEN	" "	1,254.0
9.	ASPHALT (230°F. ALIPHATIC)	" "	2,250.0
10.	ASPHALT (BLOWN) 220°F.	" "	3,500.0
11.	ASPHALT PIPE ENAMEL	" "	3,500.0
12.	ASPHALT (PALM OIL PITCH)	" "	3,500.0

Fig. 22

CATHODIC CURRENT DISBONDING TESTS

COATED 2 X 4 INCH PANELS IMMERSED 2½" IN 70000 OHMS, R, WATER, 12 CM. FROM MG. ANODE, 1.5 VOLT CLOSED CIRCUIT POTENTIAL, 1/16" PINHOLE THROUGH PANEL IN SOLUTION.

THICKNESS RANGE, 17 - 35 MILS

	COATINGS (COLD APPLIED) (NO PRIMERS)	DISBONDED AREA SQ. M.M.
1.	COAL TAR MASTIC CA-50 TYPE	64.0
2.	COAL TAR MASTIC " " "	100.0
3.	COAL TAR EPOXY (D.T.A.)	100.0
4.	COAL TAR NITRILE COATING	440.0
5.	COAL TAR-EPOXY (POLYAMINE CURE)	1,575.0
6.	ASPHALT - STYRENE	3,000.0
7.	ASPHALT - REFINED	3,500.0
8.	ASPHALTIC RESIN PRIMER + ASPHALTIC BITUMINOUS TAPE	3,500.0
9.	COAL TAR NITRILE (C-37)	700.0
10.	C-37 + 20% COAL DUST	56.0

Fig. 23

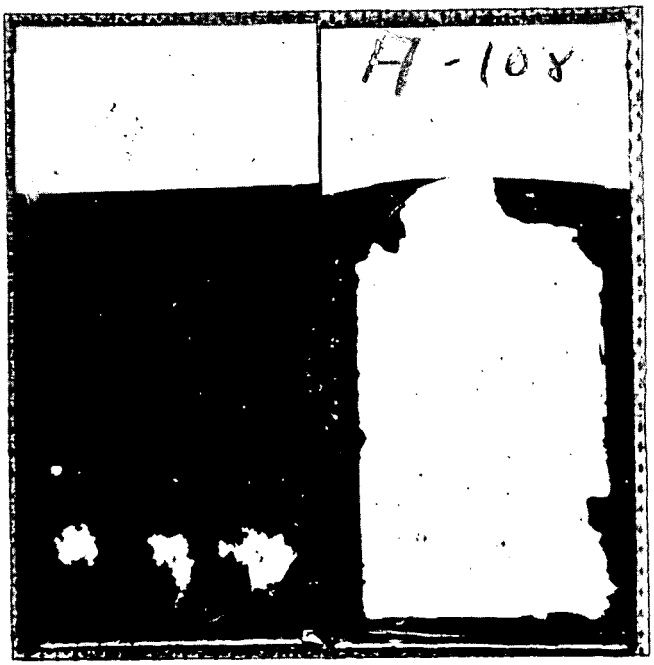

Fig. 24

Fig. 25

Fig. 26

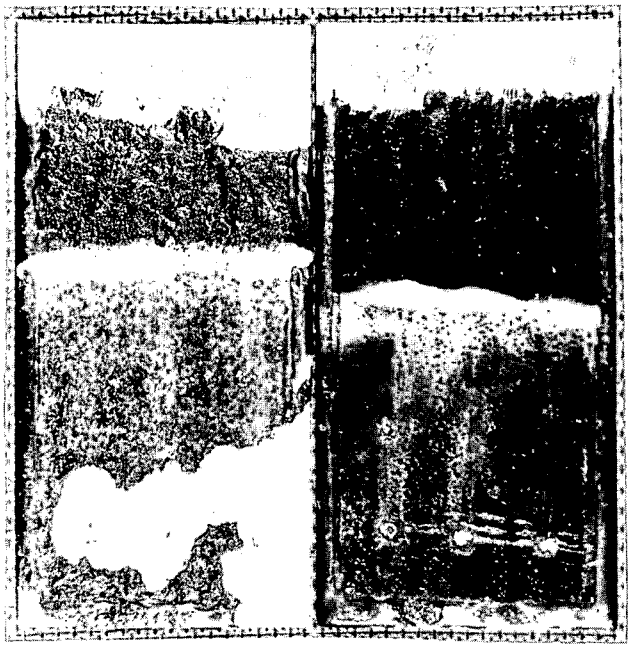

Fig. 27