ENVIRONMENTAL TESTING OF BITUMINOUS COATINGS

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INTRODUCTION

The topic of environmental testing of bituminous coatings covers a broad spectrum of test procedures and materials. As referred to in this paper, environmental testing includes utilization of both natural and artificial environments in testing programs other than in-service testing. The term "Bituminous Coatings" applies to coatings directly derived from petroleums, coal-tars and gilsonite. The environments of concern include atmosphere, water (both fresh and sea water) and soils.

PURPOSE OF ENVIRONMENTAL TESTINGS

Each environmental test program must first take into account the purpose for conducting the tests, so that reasonable objectives may be established. Let us first consider some of the purposes that could motivate a test program.

Production Quality Control: This is probably the least frequently encountered reason for an environmental test program, because there are very few environmental tests that yield data quickly enough to be of use in controlling production quality. One applicator of protective coating for underground piping does utilize such a test in his mill. A number of samples of coated pipe are cut out of each production run. This run is not released from the mill until the samples are tested in a salt water solution, under cathodic protection for a 24 - 48 hour period. Disbonding of the coating due to the cathodic protection must not exceed certain limits set up by the applicator. We are not completely convinced of the value of this procedure, due to its highly artificial environmental conditions, but it does represent an effort to control the end-quality of the product through an environmental testing procedure.

Aid in the Development of a New Or Modified Product: As in the previously described case, here we are faced with the pressure of attaining results susceptible to evaluation in as short a time as possible. Frequently, attempts to "accelerate" effects of environments leads to the development of extremely severe artificial environments. Unfortunately, the intensification of one or more environmental factors does not produce the same short-term effects as a less intense factor operating over a longer period of time. This necessitates that such tests be developed with care, and that their results be interpreted knowingly. Obviously, however, each new development can not be evaluated by full-term natural environmental testing. Intensified testing will be used, sometimes yielding data of great value, other times misleading the investigator. Such tests may, however, be utilized to determine weak points in a coating material and help determine if changes in formulation strengthen the weak points.

Substantiation of Sales Claims: This motivation leads frequently to somewhat less than objective testing, but must be mentioned because of the prevalence of test programs that originate from this need. There is no lack of ethics in providing the sales department with data from environmental tests that demonstrate the good qualities of a particular product. Unfortunately, on some occasions, researchers have destroyed their credibility by over-attention to a products strong points - or to a competing products weak points. This can lead to widespread indifference to what may be excellent data, because the investigator, no matter how highly placed, is suspected of lack of objectivity.

Compliance with Government or Other Purchaser's

Specifications: Environmental test programs of this nature effectively tie the hands of the investigator. The widespread dependence of some governmental agencies, in the past, upon specific environmental tests, that at times were not related to end-usage of the protective coating led to widespread hostility to certain environmental tests. This does not reflect on the validity of the tests, but emphasizes the importance of proper selection of tests and knowledgeable interpretation of their results.

Selection of a Coating for a Specific Need: The remainder of this paper will be devoted to exploring the use of environmental testing for this purpose.

ATMOSPHERIC ENVIRONMENT

Laboratory Testing: A number of procedures are available for laboratory evaluation of coatings. Most of these tests utilize artificial sources of radiation to simulate sunlight and fog chamber devices utilizing high humidity atmospheres and/or periodic spraying with water. ASTM Standard D529-62 "Accelerated Weathering Test of Bituminous Materials" covers the operation of light-and-water-exposure apparatus for the exposure of bituminous roofing and waterproofing materials having a minimum softening point of 200°F. These procedures utilize an enclosed carbon-arc lamp and a water spray apparatus.

While use of this apparatus will result in more rapid coating deterioration than found in natural environments, there is no reliable means of equating the time of artificial exposure to failure to the time of natural exposure to failure.

It is quite possible to arrive at substantial difference endurance rankings utilizing the "accelerated" test from that which would be arrived at under a natural environment.

Modification of this procedure by use of a xenon-arc type apparatus has yielded more meaningful data according to Martin[1] who compared carbon-arc, xenon-arc and natural radiation. Martin utilized microtechniques to increase the sensitivity of evaluation of asphalt degradation by use of the change in absorbence of the carbonyl functional groups. He found that his ranking of degradation as obtained by use of the xenon-arc compared directly with the ranking obtained by use of natural exposure to sunlight with radiation monitored by the Eppley pyroheliometer. He found discrepancies between these rankings and those obtained by the use of the carbon-arc. These observations, together with his demonstration of the use of microtechniques seem to be major contributions.

While the use of weatherometer-type apparatus has its limitations, careful evaluation of its results can be utilized to separate material with gross differences in weathering resistance qualities.

Others have used arbitrary laboratory tests to furnish at least data that will provide a basis for a rough weeding of obviously inferior materials. Flournoy[2] suggests a method for this purpose. He utilizes three tests, as follows:

1. Thickness: Minimum of 0.005-inch for corrosive atmospheres.

2. Flaws and Holidays: Electrical resistance of a coated 4-inch by 2-inch panel must be at least initially 1,000,000 ohms and must be at least 750,000 ohms after 24 hours of water immersion.

3. Flexibility: The test panel, after being bent 90° around a 1-inch diameter rod must have no obvious coating failures and must retain an electrical resistance of 750,000 ohms.

This presents rather arbitrary criteria that may be applicable to certain circumstances. The rather short term environmental test, immersing the test coupon in water could give a rough idea of initial coating condition, but a 24-hour test seems of little use other than to eliminate coatings that were very obviously inferior. Undoubtedly, arbitrary tests of this nature could be of significant value in specific instances, but would be of little general use.

Natural Environment Tests: Exposure to a natural environment seems to be the best method of environmental testing of coatings for atmospheric exposure conditions. The major disadvantage seems to be that the time required for a full evaluation can be excessive, and that there is some difficulty in knowing exactly to what factors the panels have been exposed. Caryl[3] suggests the use of mirrors to increase the intensity of solar radiation, together with a rotating mount that keeps the panels always facing the sun, both with and without periodic washing with distilled water. His studies indicated that this system could give failures in 14 weeks that would occur only after 3 years at a 45^o south exposure.

Others utilize special panels with angles, crevices, rivet heads, weld splatter and other severe surfaces to subject the coatings to the most difficult areas to coat, thereby providing a panel with conditions that lead to early failures in actual service. This type panel has considerable merit and can lead to early elimination of inferior coatings. The evaluation of coating conditions on panels such as this is more complicated and time-consuming than that of flat panels, but the data obtained can be of corresponding higher value.

Of course, natural environments vary substantially from location to location and indeed over a wide range of any given location. This necessitates methods of evaluating the environmental effects to which a coating has been subjected over its test period.

Solar radiation is one factor of major concern. The evaluation of this factor is relatively simple and can be accomplished by the use of the Eppley Pyroheliometer and appropriate recording and integrating devices. Generally, records are kept of total langleys, langleys over .823, and hours of radiation over .823. Martin demonstrated a close correlation between total langleys and asphalt oxidation. The pyroheliometer, together with further development of microtechniques, seems to offer a path for very meaningful, relatively short-term evaluations, utilizing natural sunlight.

Other factors of major importance are temperature ranges relative humidity, and any atmospheric pollutants that may be of major importance. Continuous recording of temperature and humidity are essential. Methods also have been devised to monitor sulfides in the atmosphere.

UNDERGROUND AND UNDERWATER EXPOSURE

The success of underground and underwater exposed bituminous coatings or any coating requires qualities not absolutely necessary for atmospheric exposed coatings. These environmental conditions impose the necessity for greater physical strength, higher electrical resistance, and usually require much longer useful life than atmospherically exposed coatings. These requirements vary according to the end-usage of the product and to economic considerations. Coatings for corrugated galvanized steel storm sewers do not have to meet the rigid requirements applied to coatings used on thin-wall high pressure gas and oil pipelines. Any environmental testing program must, therefore, fully consider the end-usage of the materials tested. For purposes of further discussion, we will consider only those applications where the minimum length of coating effectiveness is required to be in excess of 30 years.

Laboratory Tests: The U. S. Department of Reclamation[4] utilizes a series of tests to evaluate coatings for use in submerged service. These tests include: Fresh water immersion[5]; salt spray box[6]; outdoor weathering exposure[7]; and weatherometer[8]. In cases where specific problems exist, the Bureau designs specific tests.

While there have been a few standards developed for the evaluation of the quality of certain specific bituminous coatings, we lack standardization of tests for comparing performances of various coatings to each other. Generally, investigators develop their own tests and procedures in much the same manner that the Bureau of Reclamation has done. It appears that this situation will not change in the immediate future.

Let us consider, therefore, the development of a test program to evaluate the relative merits of protective coatings for underground structures, with the understanding that a good coating must perform well for at least 30 years. The structure to which the coatings is to be applied might possibly be cathodically protected.

Usually the investigator is faced with a vast array of proposed coatings. His first problem then is the development of a method for "weeding" the grossly inferior materials from those that may possess some merit. The judgment of an experienced investigator generally is the fastest "weeding" mechanism; however, he most frequently must substantiate his judgment with test data.

The "salt-crock" test, or a modification of it, most generally is applied to this stage of the investigations. While there are infinite variations of this test, it consists essentially of placing a coated specimen in a container of salt water. The electrical resistance between the sample and a fixed electrode is then measured; the measured resistance must exceed an arbitrary value to qualify the coating for further consideration, and must continue to maintain an arbitrary minimum resistance over an equally arbitrary period of time. The duration of this test is generally short, in the order of 3 - 6 months. In some cases the sample is maintained electrically negative to the solution in which it is immersed in order to get some evaluation of its performance on a cathodically protected structure.

As a preliminary "weeding" test, the salt-crock test has considerable merit. There are many factors that can lead to erroneous results, however. Certain coatings can not readily be applied to small coupons. For example, a heavy asphalt-mastic pipe coating applied by hand has substantially different qualities from the same material extruded on pipe by commercial machinery. A hand-applied sample could be expected to fail in a shorter period of time than a machine-applied coating. Conversely, some other coatings have much better qualities when hand-applied than they would when applied on a commercial scale. A short-term salt-crock test would reveal inherent porosity in a coating, but would not, in six months, necessarily show a tendency for moisture absorption that could lead to failure in 5 - 10 years. There is also the hazard that electrical connections to the sample might be difficult to coat and electrical leakage caused by the connection could lead to erroneous results.

One method of conducting a salt-crock test may be of some interest. A standard procedure adopted by the author's company uses a coated pipe sample, preferably with the coating applied by the same equipment used to apply the particular coating on a commercial scale. The sample is selected so that no accidental flaws exist. The coated sample, generally about 18 inches in length, is placed concentrically in a second uncoated pipe of a convenient larger size. Generally, a 3/4-inch diameter sample would be placed in a 2-inch pipe. A rubber stopper the size of the casing pipe is cut so that the sample will fit snugly in it. The assembly is then made as shown in Fig. 1, with the space between sample and pipe being filled with water made highly conductive by adding salt. The electrical resistance between sample and casing is used as a measure of the coating effectiveness. High quality coatings will maintain resistances of in excess of 1,000 megohms per square foot of surface area for long periods of time.

If a value of about 10 megohms per square foot is used as a criterion, most grossly inferior coatings will be detected in a few weeks time, and can be eliminated from further testing. Higher quality coatings can be maintained in this test for extended periods of time to give information regarding long-term moisture absorption and performance under cathodic protection conditions.

The decision to give further consideration to a given coating that performed well in the salt-crock test must also consider the physical requirements for the coating. Resistance to impact, shock and sustained loads must be determined by appropriate testing. Resistance to underfilm water migration should be evaluated by the use of samples with scratched or impacted areas. The salt-crock test can be used for this evaluation, although electrical resistance measurements, since the underfilm-migration path may itself be high in electrical resistance. Generally, this test is conducted both with and without cathodic protection. The cathodically protected samples would be affected by the physical pressure of liberated gasses and high pH conditions at the pipe surface. The use of unrealistically high voltages for this test could yield very misleading data.

Coatings surviving the above tests can next be considered for further testing in environments simulating, as close as possible, those conditions that will be encountered in service. Engineering judgment may also lead to the beginning of in-service testing at this time. Where service conditions are such that high soil stress conditions may be expected, tests specifically designed to evaluate soil stress conditions may be used. Samples can be placed in a bentonite environment that is alternately wetted and dried. The performance of the coating can be evaluated electrically by measurement of the resistance between the sample and a ground reference. This would be supplemented by physical examination at the end of the test period or when electrical measurements indicated that failure had occurred.

This method of testing seems to be worthwhile only if extreme soil stress conditions are expected. Otherwise coatings that might perform well, and economically, under actual service conditions may be unwisely eliminated.

Natural Environment Tests: Where possible, it seems best to test the coatings under conditions as close as possible to those encountered in service. One approach is to use an outdoor test box filled with soil typical of the area. Such an arrangement is shown in Fig. 2. A permanent box is constructed outdoors. An aluminum foil lining is placed in the bottom of the box. Test samples are inserted through sleeves in the box, with plastic casing spacers on the samples where they pass through the sleeves. Standard rubber casing end seals are then used to seal the space between sample and sleeve. The box is then carefully filled with the selected soil. The coating extends to the ends of the pipe samples. Stainless steel guard rings are clamped on the pipe about 6 inches outside the casing seals. If the coating includes glass fabric or felt, the guard ring must be in contact with the bottom layer of felt or glass fabric. These rings have to be installed very carefully, and are essential to reliable readings.

Use of a soil box such as this simplifies electrical measurements and keeps the samples relatively accessible for physical examination. The shallow nature of the box causes the samples to be exposed to more severe wetting-drying and temperature cycles than would be experienced under service conditions. If grass is allowed to grow in the soil, the effects of roots would also be more severe. These factors must be weighed in evaluating test results. In one 7-year test, the electrical resistance of high quality coatings was in excess of 50 megohms per square foot.

Another approach would be to bury samples directly in a typical environment. If the entire sample is buried in this manner, great care must be taken to assure that the pipe ends and electrical connections are coated at least as well as the sample coating. This can prove to be very difficult. One arrangement is shown on Fig. 3. The most critical factor is the encapsulation of the ends and connections. As long as this is effective, the resistance between the guard rings and earth will be extremely high - any leakage of water into the encapsulation will be detected by a change in the guard ring resistance to earth. Of course it is essential that the test wire insulation be perfect. While this test is probably the most difficult to set up properly, its long-term results are probably the most meaningful.

Perhaps the largest scale natural environmental test program for protection coatings was conducted by the American Petroleum Institute[9]. These tests utilized 16 test sites and involved different coating systems applied to 3-inch pipe at each of the test sites. There were also 19 coatings applied to 14 working pipelines. The test program was of about ten years' duration ending in 1940. Great progress has been made in underground protective coatings since the test started in 1930, but unfortunately the tests were not continued to include new coating developments. While the results of this program are not directly applicable to today's problems, the program itself is of interest and could be used as a starting point for the resumption of similar industry-wide tests.

SUMMARY

Environmental testing of bituminous coatings can provide valuable information. Extreme caution must be exercised in the use and interpretation of "accelerated" tests. Coordination of "Environmental Testing" and "In-Service" testing is essential to the intelligent solution to coating problems.

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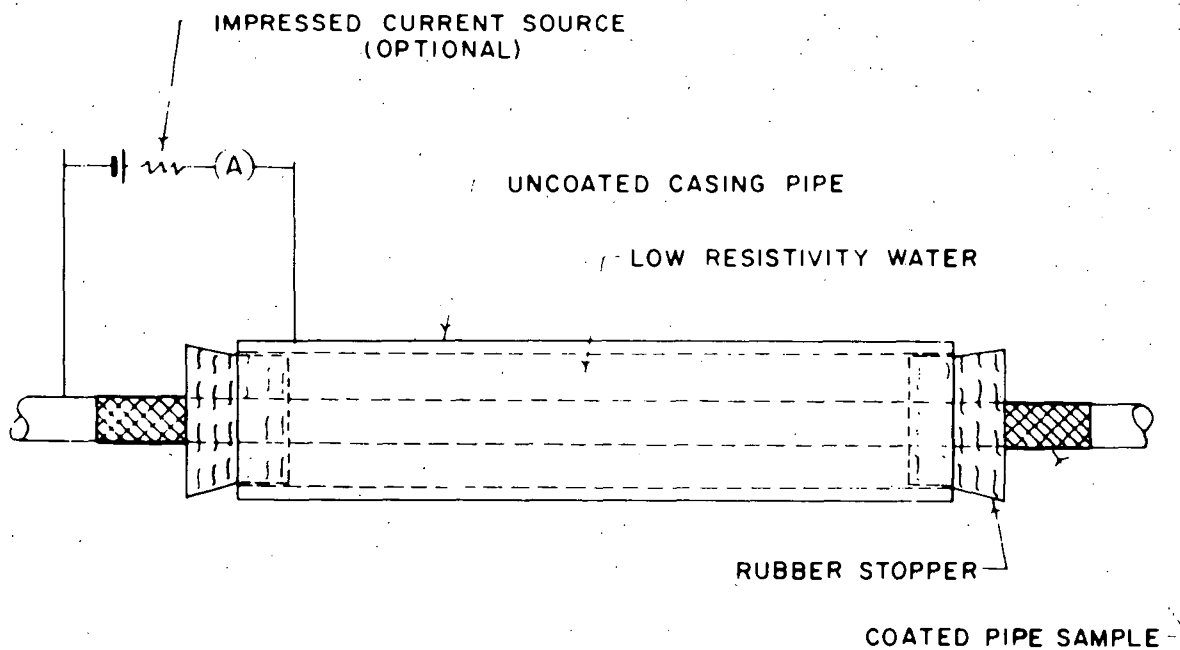
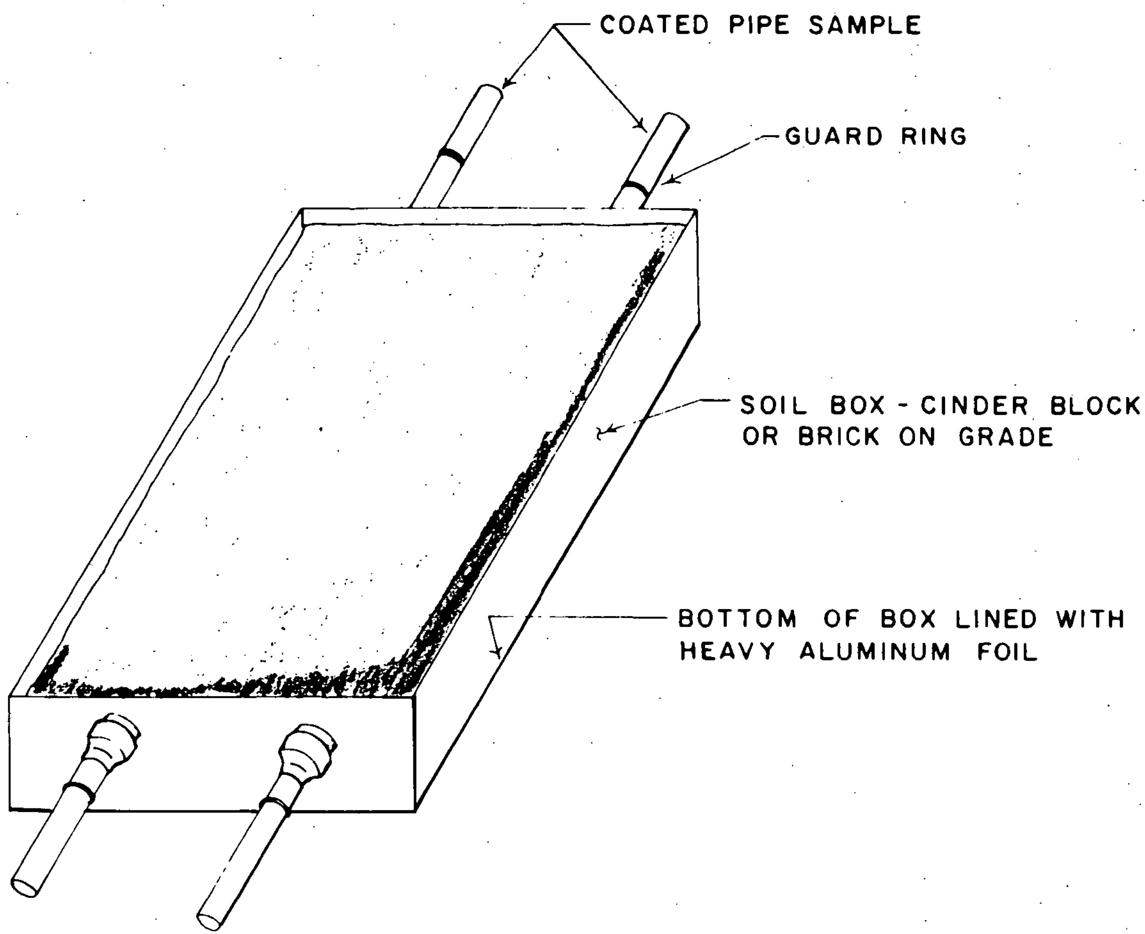


MODIFIED SALT-CROCK TEST

FIGURE 1



CONTROLLED SOIL ENVIRONMENT TEST

FIGURE 2

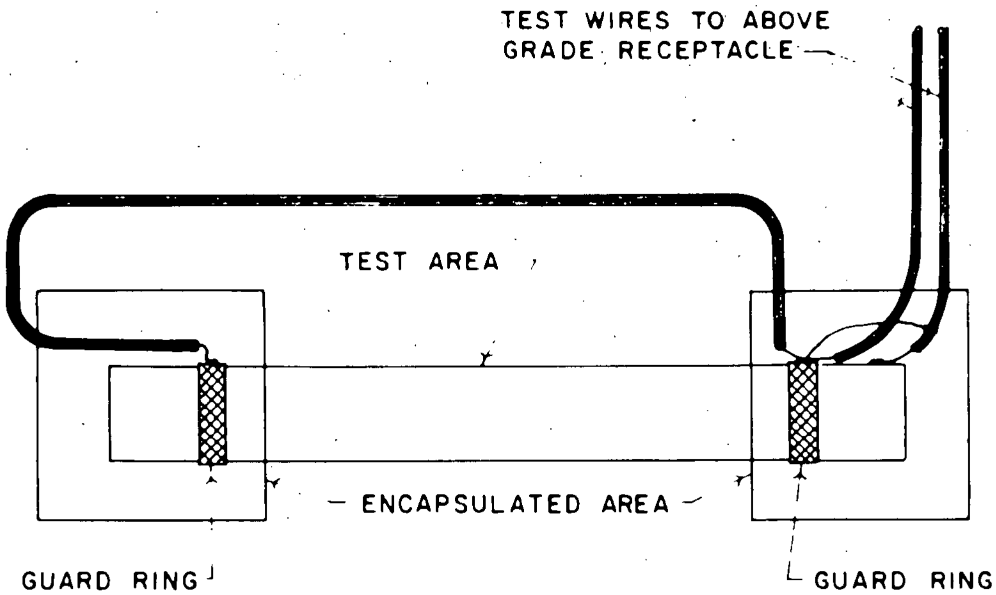

TEST WIRES TO ABOVE
GRADE RECEPTACLE
TEST AREA
ENCAPSULATED AREA
GUARD RING
GUARD RING

FIGURE 3