criteria, then it should be assumed that all were containers filled from the same well-mixed vat of paint and, for the reasons discussed above in pouring-tank sampling, one chemical analysis on one sample should suffice. Also, it is worthwhile to note that, for sample sizes of 10 and 12 in Figure 2, the greater protection afforded by these sample sizes over that chosen (eight) does not justify the increased testing efforts that would be involved. Furthermore, because of methods of determining probabilities (which we will not discuss here) for small lot sizes, the OC curves shown in Figure 2 would be somewhat conservative and probably represent the worst case.

CONCLUSIONS

1. It was determined from historical data that the limits for viscosity of paint could be broadened.
2. The practice of accepting paints on the basis of "substantial compliance" with the specifications was eliminated, and this was believed to aid in improving the overall quality of paints accepted by causing producers to pay closer attention to their manufacturing processes.
3. No advantage was found for replacing NYSMM6 by FTMS 141a (method 1021).
4. It was determined that, if the uniformity criteria are met, then it is practical to assume that a paint lot can be considered as one bulk unit for further chemical analysis.
5. A new sampling scheme is suggested for container sampling.

ACKNOWLEDGMENTS

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# Accelerated Performance Testing of Bridge Paints for Seacoast Environments

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**The design and operation are described of an accelerated-corrosion-environment chamber for evaluation of metal protective paints. The findings are discussed of experiments designed to test the reproducibility of the results obtained in the chamber and are correlated with the limited available data from an exterior weathering test fence at a tidal estuary in Brunswick, Georgia. The fundamental premise underlying the design of the chamber is that the primary stresses that account for paint-system failures on structural steel in seacoast environments are caused by continuing cycles of wetting and drying and heating and cooling in the presence of the corrosion-stimulating chloride ion. The major conclusions are that the chamber exhibits high precision of test results within runs and an exceptionally close similarity in a greatly accelerated test to the modes of panel failure observed in the field. The prospects for close laboratory-field correlation appear very good but, for general use, this correlation will require control system techniques that have been proposed but not yet validated by comprehensive experimental studies.**

Research on accelerated-weathering devices spans a period of one-half century. During the 1920s, Nelson and co-workers (1, 2, 3, 4) developed artificial weathering machines and investigated various exposure cycles. An interesting illustrated review of much of this early work has been given by Gardner (5). Standard methods for operating weathering equipment (6) and preparing and evaluating test panels (7) were developed and published by the American Society for Testing and Materials (ASTM) during the 1930s.

Notwithstanding the intensive research over an extended period of time, difficulties in obtaining similar results from different machines and in correlating these results with field experience have continued to create problems (8). The multiplicity of factors involved in accelerated weathering and corrosion tests was discussed at length in 1963 by Valentine (9) and by Talen (10) (particularly, the aging process). Clearly, the problem of defining and measuring some important fundamental properties and variables had not been specifically addressed, and no evidence was seen of any effort to formulate a comprehensive physical theory of the performance of anticorrosive paints. Thus, in 1967, Burns and Bradley (11) referred to laboratory-field correlation research as follows: "This correlation has never been achieved despite the efforts of many laboratories over a period of many years." Later, however, in referring to the work of Gay (12), they observe that "The significant

variables in accelerated weathering of paints have been studied, and information has been obtained that should lead to improvements in durability testing procedures for paints."

This is almost certainly the most important problem in the range of paint technical problems.

The above observations are mostly relevant to the problem of accelerated weathering in its broad aspects. When interest is more specifically directed to the evaluation of coatings for use in a humid salt environment (i.e., a seacoast), then much more encouraging results were reported nearly 40 years ago. In 1932, Gardner described a test that involved overnight immersion of painted panels in seawater and daytime exposure at 45° south (13). Six weeks of exposure on this test was found to simulate about 1 year of normal exposure on structures. Later, Wray automated the test to provide cycles of 5-min immersion and 25-min sunlight in air and reported that results were obtained in a few weeks that were equivalent to 1 or more years at the seacoast (14). In 1961, in an extensively documented and comprehensive study involving 16 primers, Rischbieth and Bussell (15) found that a salt droplet test (British Standards 1391-1952) was superior to humidity or salt fog tests in rating the primers in agreement with exterior weathering tests. This study also showed the large magnitude of variations in exposure results among exterior weathering sites that would necessarily confound simple direct-correlation efforts.

**Figure 1. Chamber and controls.**

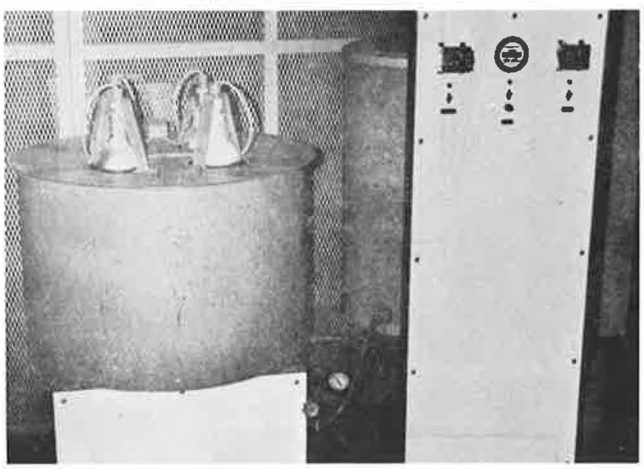
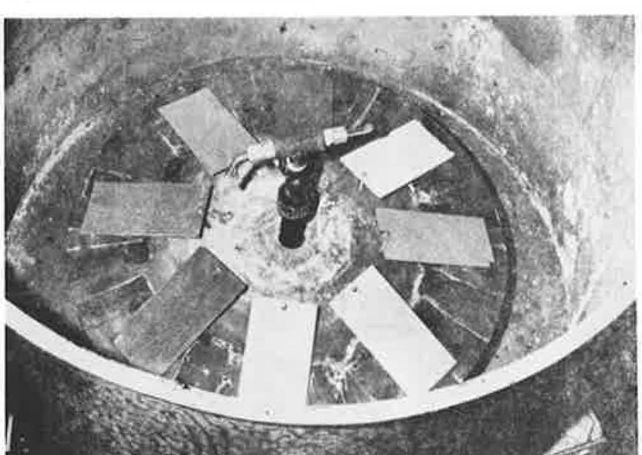


**Figure 2. Interior view of chamber.**



The literature on accelerated weathering is very large indeed. In a survey that covered the period 1955-1967, 228 papers were listed under the subject heading "coating exposure tests" (16). Thus, if unifying concepts can be developed, a huge store of data is available for experimental determination of parameters in the relevant equations.

## ENVIRONMENTAL SIMULATION CONSIDERATIONS

Conventional weatherometer studies were included as standard test procedures within the project in which this environmental test chamber study was pursued. Consideration was not given to the possibility of modifying the weatherometer into an environmental test chamber primarily because it was committed to conventional operation. Moreover, it was suspected that the elaborate carbon arc of the weatherometer was not necessary for the accelerated testing of moderately aggressive environments. Presumably, the chemical and physical stresses to be imparted to the test films in the chamber should exert their destructive effects before the radiation could cause significant damage. In addition, a flexible design that could readily accommodate various liquid or gaseous corrodents and provide rapid heating-cooling and wetting-drying cycles dictated a custom design for the apparatus. Although the primary immediate use for the apparatus was in simulating the humid salt seacoast environment, a capability of simulating various special chemical and cyclical environments was considered to be a desirable secondary design objective.

## CHAMBER DESIGN

### General Description

The environmental chamber apparatus has two main sections—the chamber enclosure and the control and servicing equipment (see Figure 1). The chamber is equipped with a rotating specimen table, four overhead ultraviolet (UV) lamps, two wetting-solution nozzles, and a drain (see Figure 2). The table rotates at a rate of 4 revolutions/min. Eight panels are mounted in a circular pattern on the table, sloping at a 30° angle toward the outer edge (see Figure 3). The rotation of the table provides a completely uniform positioning of the panels with respect to both light and wetting solutions.

The four 275-W UV lamps are mounted in the chamber cover. When in operating position, they are 36 cm (14 in) above the panels.

The two wetting-solution nozzles are located on a central pipe and are aimed so that one stream of solution hits the panels 6.4 mm (0.25 in) from the top and the other hits halfway down the panels. Both of the streams strike the panels at compound angles. This provides a uniform wetting over the entire surface of the panels. The nozzles are made of 6-mm (0.23-in) flint glass tubes with the end drawn and fire polished until only a small hole [approximately 0.3-mm (0.012-in) diameter] remains (see Figure 3) (because this equipment was designed and built to U.S. customary units, SI units are not shown on Figure 3). This nozzle design was adopted after metal and plastic nozzles were found to develop problems of corrosion and plugging. The glass nozzles operate for long periods of time without plugging and are easily cleaned if plugging does occur. Another advantage of these nozzles is that, because they direct a thin stream of solution directly onto the panels rather than produce a general spray of solution, there is less incrustation of

the lamps with salt. The expended wetting solution leaves the chamber through the drain.

Service and Control Systems

The ancillary equipment consists of a 114-L (30-gal) solution holding tank, the electrical system, the piping system, the solution pump, and the table rotation motor. A pump bypass line equipped with a valve and pressure gauge is used to control the pressure at the nozzles. A solenoid valve, which is connected in the electrical circuit of the pump, was installed in the line from the holding tank to prevent leakage at the nozzles (caused by the pressure head in the tank) when the pump is not operating (see Figure 4).

The wetting cycle and the UV light cycle are controlled by independent timers. This provides two unsynchronized cycles: (a) a wet-dry cycle in which a solution [normally synthetic seawater conforming to ASTM D1144-52 (revised 1965) (section 4)] is periodically sprayed on the panels and (b) a light-dark (heating-cooling) cycle in which the panels are periodically subject to UV radiation and heat gradients. An elapsed-time meter is connected

Figure 3. Rotating specimen table and nozzle assembly.

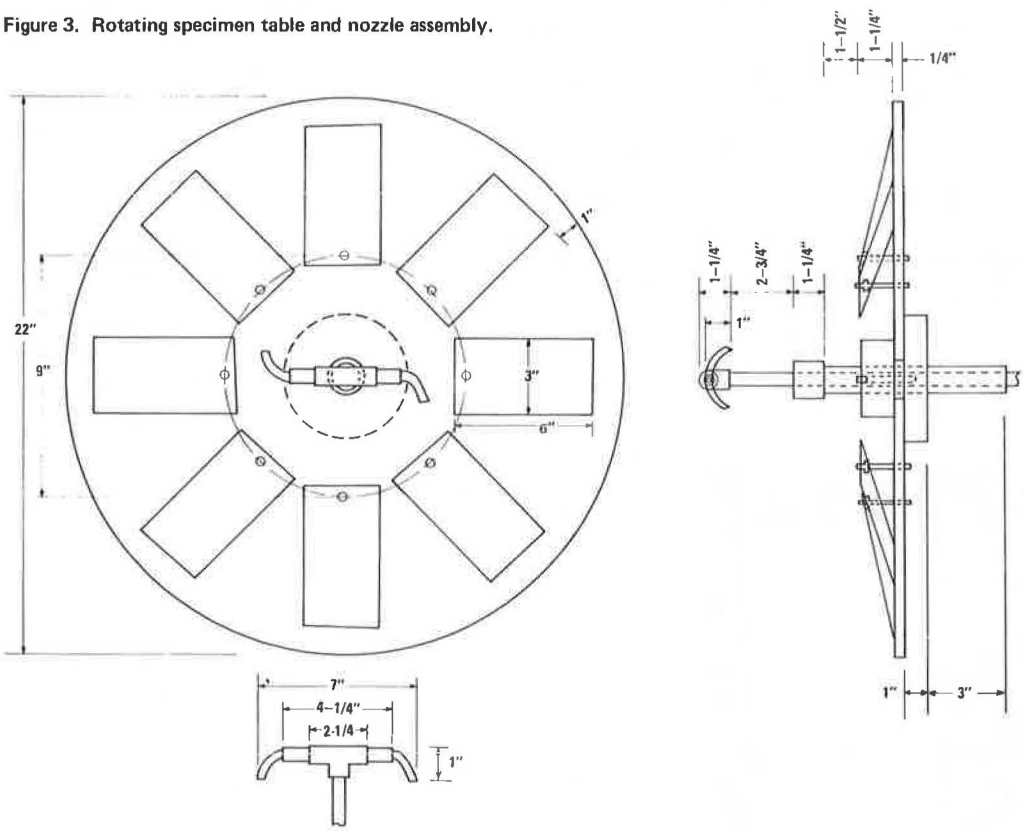



Figure 4. Piping system.

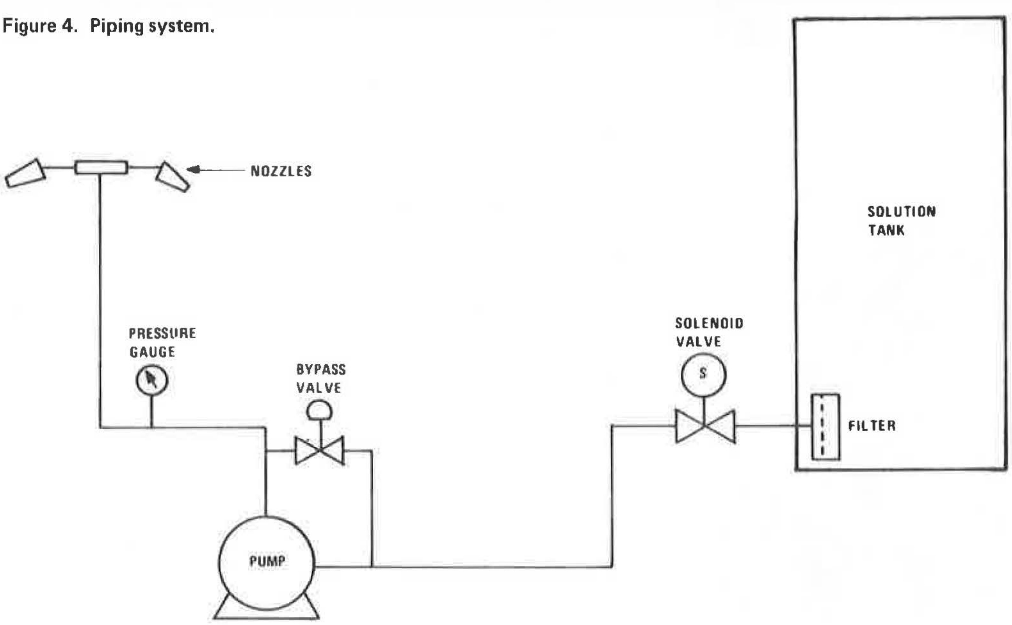
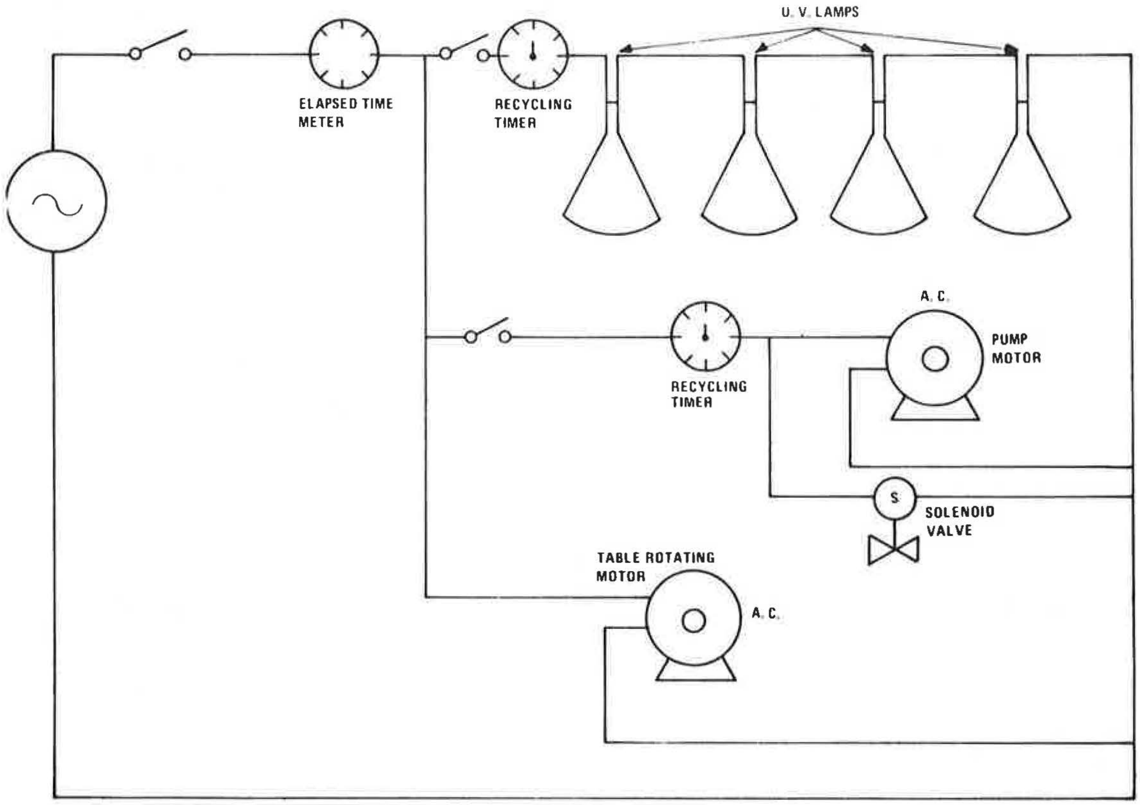


Figure 5. Electrical system.




to the main power line to indicate the number of hours of operation (see Figure 5).

## OPERATING CONDITIONS AND TEST PROCEDURES

The conditions and procedures used were selected with the particular objective of attaining an accelerated simulation of a seacoast environment.

### Operating Conditions

The standard operating conditions for the environmental chamber are as follows:

| Condition | Procedure |
|---|---|
| UV light (heat) | 40 min on and 40 min off |
| Wetting solution | Synthetic seawater conforming to ASTM 1141-52 (1965) section 4 |
| Wetting cycle | 30 s on every 15 min |
| Wetting rate | 65-75 ml of solution/cycle |

### Panels and Preparation

The standard panels for use in the environmental chamber are 7.6x15.2-cm (3x6-in) cold-rolled, 0.812-mm (20-gauge) thick, SAE 1020 steel panels meeting the specifications of ASTM D609-61. The test panels are prepared as specified in ASTM D609-61 method A, procedure 2 and sandblasted to white metal as specified in Steel Structure Painting Council standard procedure 5-63. The panel number is stamped in the upper left-hand corner, and the panels are painted both front and back with the paint system and by the application method specified (usually brushing). The coated panels are then conditioned by a 24-h oven heat-aging period at 80°C (176°F). After conditioning, the panels are scribed with a special scribing tool. The scribe is made from the upper corner to the lower center on both left and right. Film thickness is measured with a paint thickness gauge. The panels are then ready to be placed in the environmental chamber.

### Inspection and Grading

The panels are inspected and graded at intervals of approximately 100 h or as appropriate to the specific study.

The period of time required for testing depends on the resistance of the paint system to degradation and the objectives of the specific test. For structural steel paint systems, failure is considered to have occurred when repainting is required (as judged by an ASTM grade of rusting-5 in the scribe or integrity-9 on the planes). This condition will be reached by most structural steel systems within 600 h in the environmental chamber.

### Data Reduction

A computer program was developed that abstracts an integrity (integrity corresponds to the lowest ASTM type rating among the attributes rusting, blistering, cracking, flaking, and erosion) grade from both the scribe and the planes observations and performs a least-squares fit of the data to give separate degradation equations. The program computes a planes service life [$SL_p$ (integrity-9)] and a scribe service life [$SL_s$ (integrity-5)]. These are the main characterizing performance parameters from the environmental test. Examples of the data plots are shown in Figure 6. Note that $SL_s$ is computed from a simple log-decay equation, appropriate for panels predamaged by scribing, whereas $SL_p$ is computed from a growth-decay equation that provides for a necessary induction period before undamaged surfaces begin to deteriorate.

## PRECISION STUDIES

### Systems

The paint systems selected for this study included the Georgia specification green bridge-paint system (Pb), a standard system of chromate oil-alkyd primer with a gray alkyd finish (Cr), a zinc-rich system of organic zinc primer and aluminum phenolic finish (Zn), and three one-coat primer-only systems.

### Experimental Plan

The complete study involved three experimental runs for 750 h each of 8 panels/run—a total of 24 panels. Each run included duplicates of the three full systems (6 panels) and two single-replicate primer panels. The primer panels were scheduled so that a different paint was exposed together for each run, and each primer received two runs.

Figure 6. Computation of service life from experimental data.

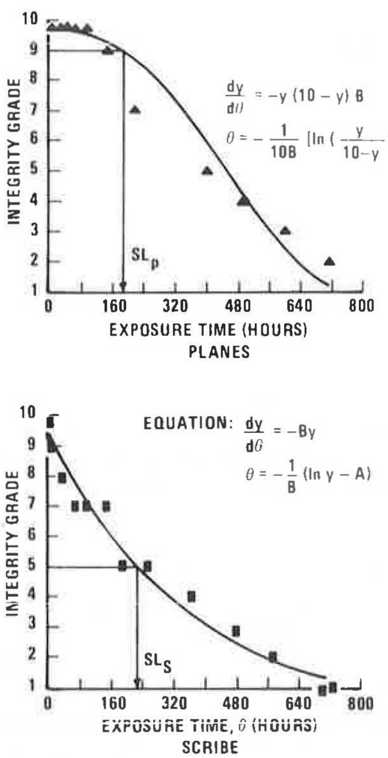



Figure 7. Service-life plots.

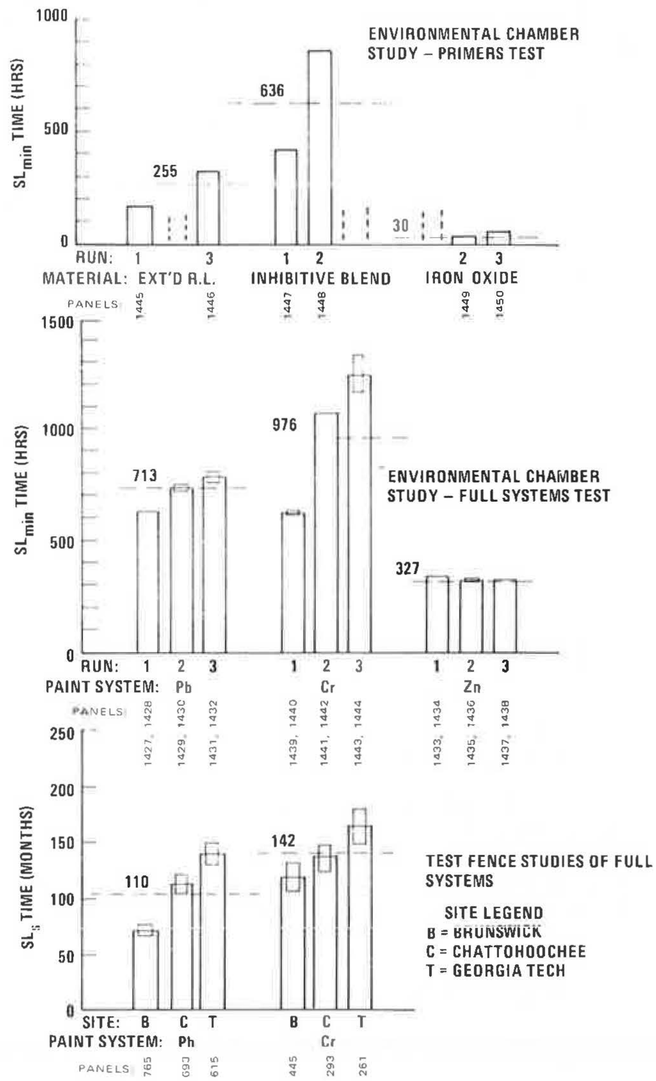



Table 1. Confidence limits for test results.

| Type of System | Statistical Index | Pb | Cr | Zn | Extended Red Lead | Inhibitive Blend | Iron Oxide |
|---|---|---|---|---|---|---|---|
| | | Computed Service-Life Difference[a] (h) | | | | | |
| Full | Detectability of system differences[b] [if, in common runs of a pair of joint (simultaneous) experiments[c], the results[d] of two paint systems are compared and the mean difference exceeds the tabulated value, then the systems are significantly different in performance] | 20 | 125 | 1.5 | | | |
| | Repeatability of experiments[e] (if, in separate runs of a pair of experiments, the results of two paint systems are compared and the mean difference exceeds the tabulated value, then the systems are significantly different in performance) | 191 | 833 | 34 | | | |
| Primers only | Detectability of system differences[b] [if the coefficient of variation of each of these primers is assumed to be similar to that of the chromate full system (Cr) and the experiment consists of two runs in single replicate (one panel), when the mean difference in results between two systems in common runs of joint experiments exceeds the tabulated value, then the systems are significantly different in performance] | | | | 56 | 141 | 8 |
| | Repeatability of experiments[e] (if, when the coefficient-of-variation assumption and the separate-runs procedure are used, the mean difference in results between the system exceeds the tabulated value, then the systems are significantly different in performance) | | | | 266 | 664 | 41 |

[a]Based on 95 percent confidence limits.
[b]From Equation 3a.
[c]An experiment here refers to three runs of one system.
[d]Results are the average service life of duplicate panels.
[e]From Equation 3b.

Figure 8. Experimental sensitivity as function of runs and replications: red lead paint systems.

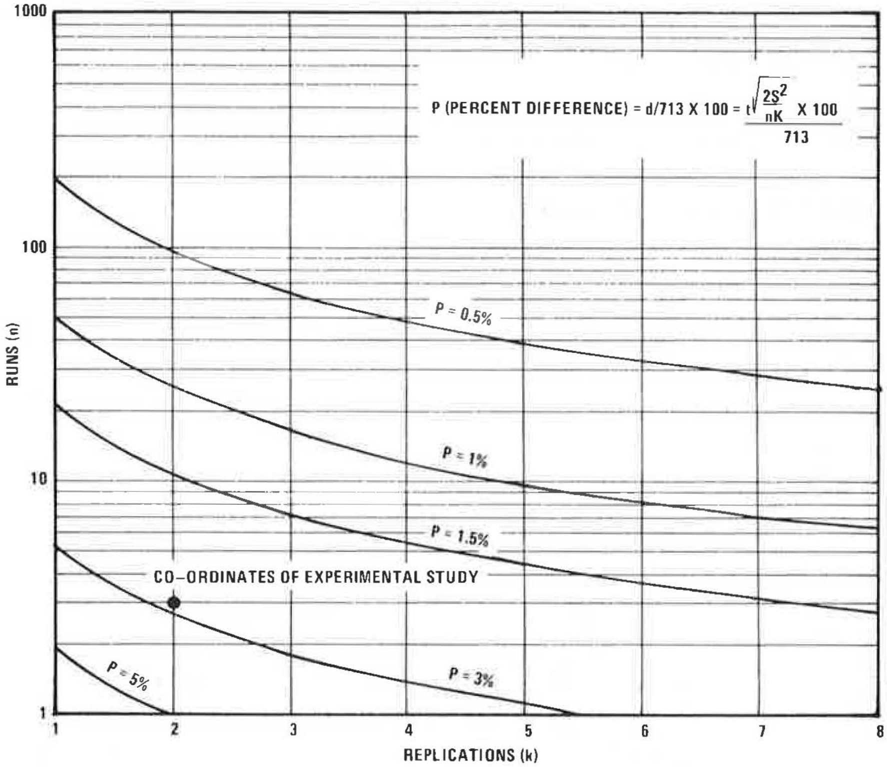



Table 2. Confidence limits for synthesized test-fence data.

| Statistical Index | Computed Service-Life Difference[a] (months) Pb | Cr | Zn |
|---|---|---|---|
| Detectability of system differences[b] [if, in common runs of a pair of joint (simultaneous) experiments[c], the results[d] of two paint systems are compared and the mean difference exceeds the tabulated value, then the systems are significantly different in performance] | 19 | 33 | - |
| Repeatability of experiments[e] (if, in separate runs of a pair of experiments, the results of two paint systems are compared and the mean difference exceeds the tabulated value, then the systems are significantly different in performance) | 89 | 63 | - |

[a] Based on 95 percent confidence limits.
[b] From Equation 3a.
[c] An experiment here refers to three runs of one system.
[d] Results are the average service life of duplicate runs.
[e] From Equation 3b.

## Test Results

Reduced service-life data ($SL_p$ and $SL_s$) were computed and are shown in Figure 7. A statistical analysis of the data from the full-systems tests leads to some useful quantitative generalizations. The variances for replicates ($S_R^2$) and the variances for runs ($S_E^2$) were computed by using Equations 1 and 2 and are shown below:

$$S_R^2 = nk\,\Sigma\,[(X_{rR} - \bar{X}_R)^2/(k-1)n] \qquad (1)$$

$$S_E^2 = n\,\Sigma\,[(\bar{X}_R - \bar{\bar{X}})^2/(n-1)] \qquad (2)$$

where

r = replication,
R = run,
k = replications per run, and
n = number of runs per system.

| Source of Variation | df | Full System, $S^2$ Pb | Cr | Zn |
|---|---|---|---|---|
| Duplicates within runs | 3 | 124 | 4 654 | 0.67 |
| Among run means | 2 | 5405 | 103 040 | 171 |

Values for the detectability of system differences were computed from the statistic

$$d = t \times (2S_R^2/nk)^{1/2} \qquad (3a)$$

and values for the repeatability of experiments were computed from the statistic

$$d = t \times (2S_E^2/n)^{1/2} \qquad (3b)$$

Some derived 95 percent confidence limits for these are given in Table 1.

## Discussion of Precision Studies

The data given in Figure 7 and the confidence limits given in Table 1 clearly show that the environmental chamber is capable of discriminating significant performance differences among the primers and full systems tested. For example, for two lead-type paints run simultaneously in the environmental chamber in duplicate, a difference of only 20 h in their observed service lives justifies a conclusion that the paints are really different.

The relationship between runs and replications for tests of the lead-type paint system (Pb) is plotted in Figure 8, in which the significant difference is expressed as a percentage of the mean service life. The sensitivity of the discrimination exhibited by the equipment used in this study was judged to be well advanced into the area of practical use. One notes, however, from Table 1, that the repeatability of experiments shows large values for significant differences. This means that the run-to-run variability is large and thus the machine-to-machine and machine-to-field correlation must be subject to the same large variation. Additional research to uncover

the source of this variability would be of value.

CORRELATION STUDIES

The purpose of these experiments was to compare the exposure results obtained in the environmental chamber with test-fence data from a marine atmospheric environment (Brunswick, Georgia) that the chamber is intended to simulate.

Figure 9. Single-coat primers: laboratory versus field.

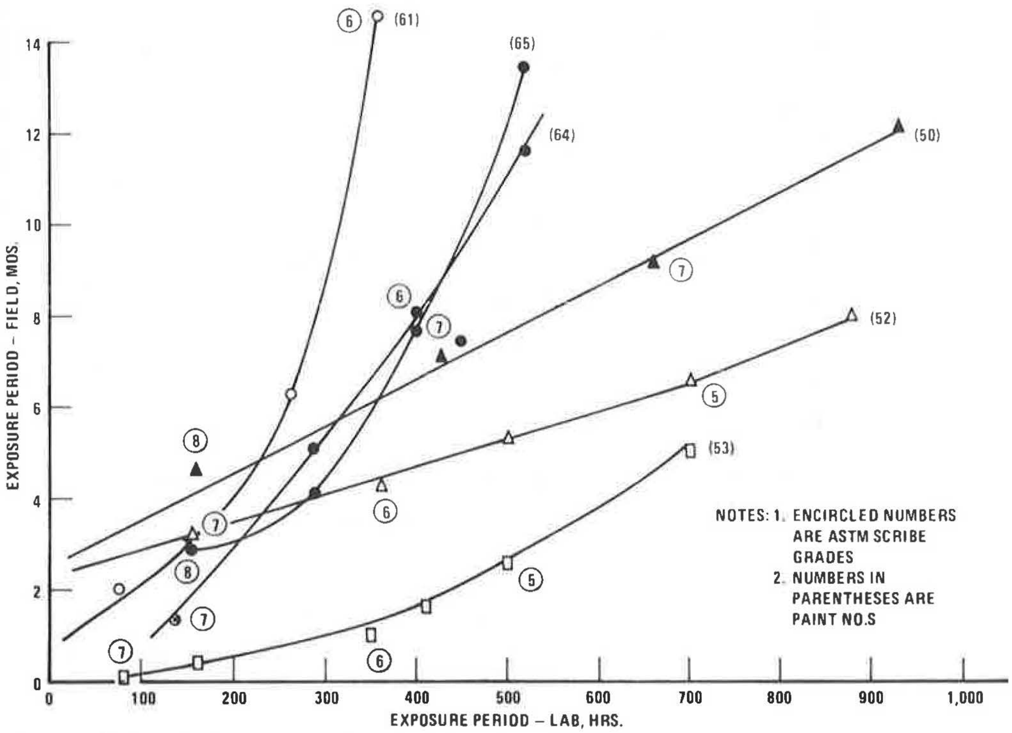



Figure 10. Full systems: laboratory versus field.

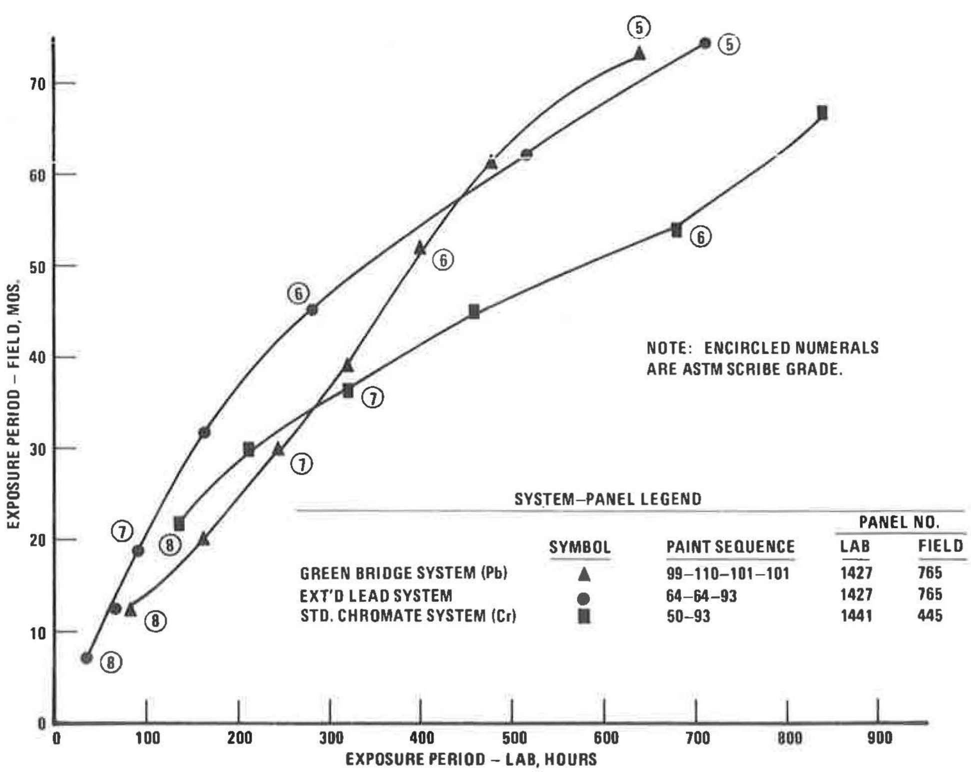
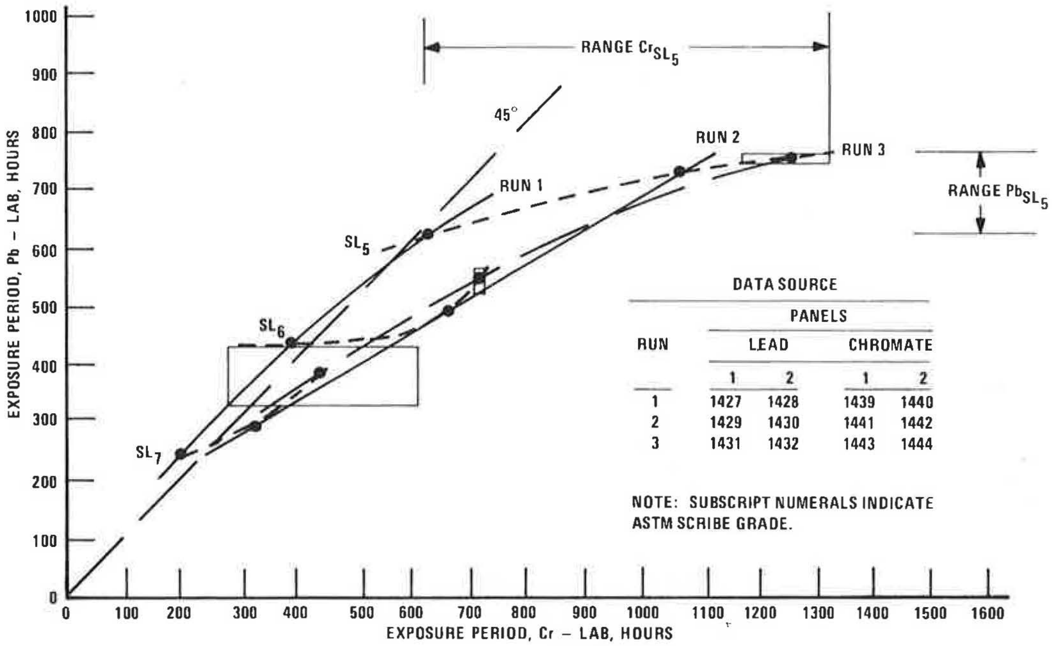


Figure 11. Correlation of two paint systems in environmental chamber.




Systems

This study includes all experiments of the precision study and a number of other runs for which corresponding laboratory-field data were available.

Experimental Plan

The correlation experiments were not designed to have the analytical rigor of the precision study but were expected to reveal much about the character of both the laboratory and the field exposure. The general procedure was to plot equal-integrity observations of corresponding single panels (laboratory and field) on time coordinates (hours and months) throughout the exposure periods. To abstract equal-integrity times from the exposure data, it was least-squares fitted to exponential decay curves. Plots were prepared for scribe and planes observations from panel pairs.

Test Results

All of the correlation plots are taken from single-panel data. The laboratory-field lines were traced on a continuous basis to reflect faithfully the correlation with two continuous curves that had been previously fitted to observed performance data. Further smoothing of a resultant curve would be a questionable procedure, despite the suggestion of unusual complexity in some of the correlations.

Finally, the matter of accuracy and precision of correlation was addressed. Available relevant basic data are given in Figure 7 and derived data in the form of confidence limits for laboratory and field data are given in Tables 1 and 2 respectively.

The number of test systems that had exact laboratory-field correspondence was not large; therefore, field tests involving several types of panels and test conditions were assembled to supplement the findings.

Figure 9 shows the laboratory-field correlation of single coat primers (blasted panels in the laboratory and mill-scaled panels in the field).

Figure 10 shows the correlation of laboratory-field systems of closest correspondence—full systems on blasted panels in the laboratory and similar systems on blasted panels exposed 45° south in the field.

Figures 11 and 12 show the Pb versus Cr systems in laboratory and field respectively.

Discussion of Correlation Studies

It would have been unrealistic to have expected that distinctively different paint systems and test conditions (surface preparation and vertical exposure) would all exhibit identical correlation curves. Families of curves as shown in Figure 9 are a reasonable result of diverse conditions. The observed diversity is augmented by the fact that all of the plots presented are for individual pairs of panels rather than for averages. The data shown in Figure 10 are the best available basis for any generalization that is to be drawn. As an approximation, based on a linear least-squares correlation, the mean slope is about 32.5 months/275 h = 0.118 months/h. This corresponds to a rate acceleration of 0.118 months/h × 24 h/d × 30 d/month = 85 field (Brunswick) h/laboratory h.

An effort to read particular interpretations into the individual curves of Figure 10 is probably not justified because of the limited statistical basis. Undoubtedly, however, some systems are nonlinear for the test conditions selected. Conceivably, these results could be linearized by rendering the laboratory test less aggressive (thereby extending the test period). But this may not be the procedure of choice, because an important feature of laboratory tests is the reduction of testing time to a minimum. A better procedure might be to develop standard performance curves for known reference (control) systems in both laboratory and field and use these controls to convert raw observations to standard laboratory results. Standard field results would also embody the advantage of substantially reducing run-to-run variability (which is a major source of experimental error). The efficiency of this general idea has been demonstrated by Mitton and Church (18) in their concept of an "average year of Florida weather."

A detailed development of this subject is beyond the scope of this report, but attention is directed to a dem-

Figure 12. Correlation of two paint systems on test fence.

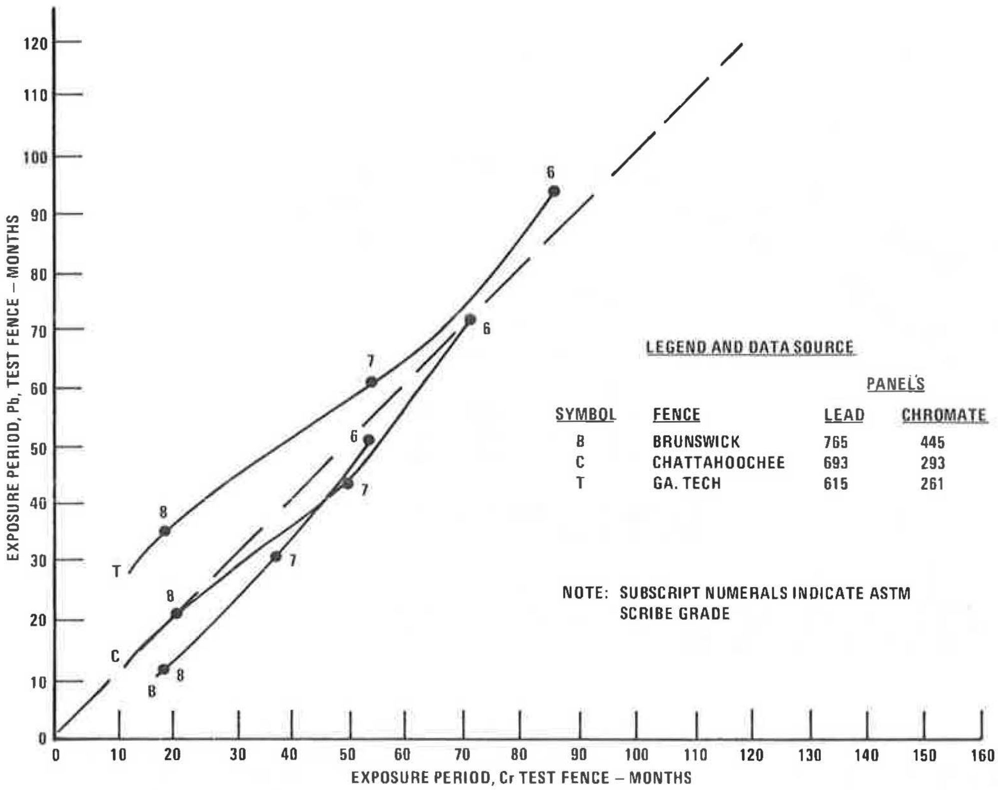
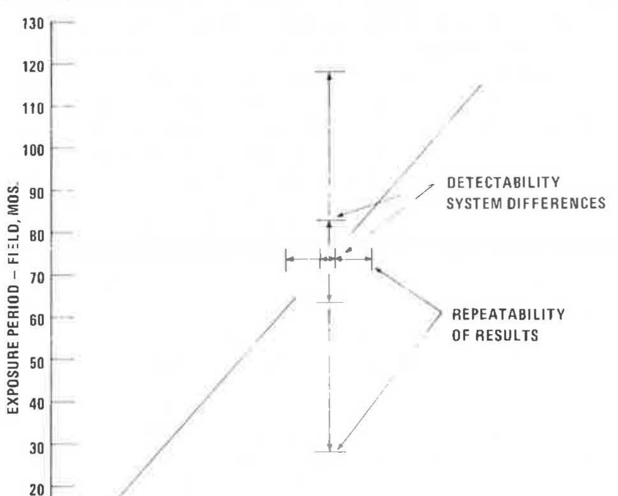



Figure 13. Joint (laboratory-field) 95 percent confidence limits for maximum differences expected under stated conditions: green lead paint system.




onstrated fact that weather variability (year to year as well as day to day) is a significant variable in exterior paint system tests, just as run-to-run variability is significant in the environmental chamber. A reduction in the variability of the latter by improved mechanical controls could be helpful, but this would not alter the variability of the former (exterior) in any way.

It is possible to derive 95 percent confidence limits at the service-life value (as illustrated in Figure 13 for the green-leaded paint system) for both laboratory and field data. The confidence limits for the field results represent only a conservative estimate, as discussed above. The limits are based on the definitions stated in Tables 1 and 2 wherein the ranges shown represent the maximum difference between two experiments. The outer limits may be regarded as accuracy (between-run) limits, which are applicable in the absence of standardization. The inner or precision (within-run) limits are those that would obtain if standardization eliminated run-to-run variation. Clearly, standardization procedures must become routine if a satisfactory basis for paint-system design is to be achieved.

## CONCLUSIONS

An environmental chamber of fairly simple construction, as assembled for this project, displayed a useful capability for accelerated simulation of the effects of corrosion on painted panels of a seacoast test-fence environment as observed at Brunswick, Georgia. More specific conclusions follow:

1. The observed average panel-degradation rates of several representative test systems were approximately 85 times the test-fence rate. (This is an order-of-magnitude figure and not intended for performance estimation.)

2. The reproducibility or precision within runs of the environmental chamber experiment was quite good; the procedure was capable of distinguishing, at 95 percent confidence limits, within-run differences as small as 7.3 percent of the mean service life [average of Pb and Cr system means = $100 \times [(20/713) + (125/976)]/2 = 7.3$ percent]. Thus, operating conditions for all specimen

positions within the chamber may be regarded as essentially identical.

3. The repeatability or accuracy of results between runs appeared to require the use of control systems to reduce the variability. With an effective control technique, the run-to-run variation could approach the within-runs limits.

4. The correlation with the Brunswick 45° south seacoast test-fence results was generally good, but sufficient distinct differences in correlation curves were observed among different paint systems to make the use of control systems advantageous for the detection and computation of nonlinear correlations.

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My associate, David R. Hurst, was actively involved in all phases of the work reported in this paper and contributed significantly to both conception and implementation. Lewis Young patiently led us through the intricacies of the design and analysis of experiments that use statistical methods. At the time the work was conducted (1971-1973), we were employed at the Georgia Institute of Technology Engineering Experiment Station. The work here reported was sponsored by the Georgia Department of Transportation in cooperation with the Federal Highway Administration. The contents of this paper reflect my views only and do not necessarily reflect the views or policies of the Georgia Department of Transportation or the Federal Highway Administration.

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# Measurement of Polarized Potentials in Concrete Bridge Decks

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**An investigation was conducted to determine the best method of measuring the polarized potential of the reinforcing steel in a cathodically protected bridge deck. The use of carbon rods and copper-copper sulfate and zinc-zinc sulfate half-cells as probes was studied in laboratory slabs and bridge decks. The carbon probes were found to be more accurate and reliable; the half-cells produced variable results. The coke layer was found to act as a half-cell and its voltage had to be taken into account when measuring the polarized potentials in the deck.**