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ACCELERATED TESTING AND ORGANIC COATINGS

by

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ESSAIS ACCELERES ET REVETEMENTS ORGANIQUES

SOMMAIRE

Les résultats dans quatre sphères d'essais sont analysés — les essais de pliage au mandrin, les essais au moyen de l'appareil à bille pour mesurer l'abrasion, les essais de corrosion et les essais de vieillissement accéléré. Dans les deux dernières catégories, l'efficacité de différents essais à reproduire les détériorations naturelles, est analysée. L'exactitude des essais pour prédire le degré d'acceptabilité des revêtements à une fin donnée est établie sur une base de degré de corrélation avec l'exposition naturelle. Nes exigences préalables pour l'usage satisfaisant des essais accélérés sont étudiées.

ACCELERATED TESTING *has stirred debate over correlation of results to normal use and exposure. Here are the findings of an investigation into the adequacy of these tests for predicting the performance of coatings under actual exposure conditions.*

BY H. E. ASHTON

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THIS article discusses the experiences of the Materials Section of the Division of Building Research in its attempts to predict, by means of accelerated tests, the suitability or durability of organic coatings, particularly with reference to their use on or in buildings. It is not intended to be a catalog of all the failures encountered while trying to develop meaningful accelerated tests, but because of the present state of the art, much of it, unfortunately, will be negative in character. The term "art" is used because it has been said that a science with more than seven variables is an art, and when it comes to accelerated testing, especially accelerated weathering, more than that number are present.

The Section's interest in accelerated tests stems from the approach taken in much of the work on organic materials. Generally, the scheme followed is: (1) find out which types of materials have the durability required for a particular use; (2) determine the important properties that confer this durability, and (3) develop accelerated tests that will accurately predict the required durability. If these tests can be developed, it should be unnecessary to return to the first step each time a new material is introduced.

The approach outlined was adopted because the Section, as part of an applied research division of the National Research Council, is called upon to give advice on the use (and sometimes after the misuse) of organic materials in buildings. So far, the work on organic coatings has concentrated mostly on the first two steps of the program.

Because of numerous difficulties, the validity of many accelerated tests has not been established to everyone's satisfaction, and consequently, there are wide differences in opinion concerning their usefulness and the manner in which they can be used. Some researchers maintain that accelerated tests, particularly those involving artificial weathering, are good only for comparing materials of the same basic type, and that as soon as a different kind of material is included in the test, the results will be doubtful. This idea suggests that an accelerated test may separate black sheep from white sheep, but cannot separate sheep from goats.

At the other extreme are those who claim an exact correlation between accelerated tests and normal usage. For example, some paint chemists state that one month in a Weatherometer is equal to x months plus y days exterior exposure in their locality. The Building Materials Section has found a great disparity in the value of the various accelerated tests; this article relates the Section's experiences with some tests that were applied to organic coatings.

Flexibility Tests

The first test to be discussed involves work carried out several years ago on clear finishes for exterior wood. Because the results were not favorable, they were reported to a Canadian Government Specifications Board (CGSB) subcommittee meeting, but were not published at the time. They are presented now to illustrate that an accelerated test may not be useful in predicting suitability of a coating if the wrong factor is accelerated. Publication may prevent others from unnecessarily repeating this work.

An important part of the research on exterior clears was to determine the properties important to durability and to find accelerated means for estimating this durability. One property that has always been considered of vital importance for a wood finish is flexibility. For many years, initial flexibility has been

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This article was originally presented as a paper before the Symposium on Accelerated Testing held November 8, 1968, by the NRC Associate Committee on Paint Research at Montreal, Quebec.

Table I — Laboratory Formula Numbers — Paraphenyl-Phenolic Varnishes

Type of Oil	Per Cent Oil on Solids 50.0	66.7	75.0	80.0	83.3
Tung	892	893	894 896*, 900**	901	—
Linseed	—	902	903	905	—
Soya	403	—	—	—	—
Dehydrated castor	—	—	—	—	404

Other phenolic varnishes: 906, U.S. Federal Spec. TT-V-119: 72% tung-linseed; 907, Reactive alkyl phenolic: 73.8% tung; 908, Non-reactive alkyl phenolic: 73.8% tung; 909, Non-reactive alkyl phenolic: 73.9% tung-linseed; 910, Cold mix t-butyl phenolic: 66.3% tung-linseed.
*Undercooked; **Overcooked.

measured by bending films on tinplate or other thin metal panels over a mandrel. This bend test was used partly because it was thought that if the film is not flexible to begin with it will not last long in service, and partly because the test is familiar and easy to run.

Since most varnishes are flexible enough to pass a one-eighth-inch mandrel, specifications usually require the Kauri reduction test, in which the loss of flexibility can be said to be "accelerated" by the addition of the hard, brittle resin. The Kauri test was consequently included as one of the accelerated tests, but it was soon found that the results were not related to the durability of the phenolic varnishes used in this work.

Next it was thought that performing the bend test at a low temperature would accelerate the test more meaningfully and would quickly determine which varnishes would be most likely to lose flexibility when exposed to natural weathering. A series of the phenolic varnishes of various oil lengths and natural durabilities was subjected to bend tests at four temperatures: 23, 0, —18 and —30° C. In addition to the test panels, a one-eighth-inch mandrel and a pair of cotton gloves were kept at the test temperatures in accordance with Method 119.4 of CGSB Specification 1-GP-71.

The composition of the varnishes is given in Table I, and the results of the bend tests are shown in Figure 1. The durability rating shown is the assessment by two observers of the coatings after exposure on western red cedar for two years at Ottawa, Ontario. The rating is on the conventional 10 to 0 scale, where 10 is perfect condition and 0 is complete failure.

When the exterior durability of these varnishes is compared with their flexibility at low temperatures, there is no correlation. As might be expected, there is correlation between low temperature flexibility and oil content of the varnishes. Ten-gallon (Imperial gallon) varnishes fail at around 20° C., 20-gallon varnishes at around 0, and 30-gallon p-phenylphenolic varnishes at around —15° C. The 30-gallon alkyl phenolics passed at —18, and one passed at —30° C., but they were not as durable.

The only two varnishes to pass at —30 had poor durability, failing after two summers. Only one of the six varnishes that passed the bend test at —18 had good durability, but this result may have been anomalous, since under- and over-cooked homologues failed at the same temperature. Lowering the temperature at which the bend test was carried out did not, therefore, enable prediction of the durability of these materials, even though they would experience temperatures as low as —30° C. during natural exposure at Ottawa.

Accelerated Weathering

An associated part of this work was to follow the changes in flexibility when the varnishes were subjected to accelerated weathering. This method would seem more likely to be related to durability than that of simply lowering the temperature. Coated panels were exposed in an Atlas Single Arc Weatherometer operating on the CGSB cycle of eight hours light only, ten hours light and water, and five and one-half hours water only. Bend tests were carried out at 23, 0 and —18° C. after two, four and eight weeks of accelerated weathering. The results are given in Figure 2.

Again it can be seen that there was no correlation between exterior durability and flexibility after accelerated weathering. After two weeks all varnishes failed at —18° C., and of the five that passed at 0, only one performed well on wood. After four weeks all failed at 0. With room temperature bend tests, durable varnishes failed after four and eight weeks and non-durable varnishes passed in accordance with their oil content.

Similar inconclusive results were later obtained from tests intended to

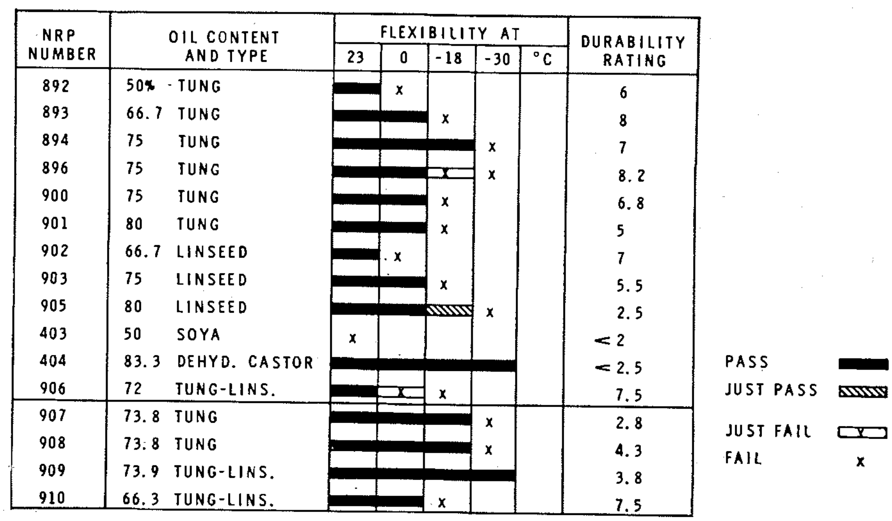


Figure 1—Low temperature flexibility.

determine the effect of drier content on flexibility and its retention using four varnishes used in the previous tests. It was concluded that the flexibility test, whether accelerated by low temperature, artificial weathering, or both, was of little assistance in predicting the durability of clear varnishes for exterior wood. Because a more discriminating test than the simple mandrel method was obviously required, the approach was abandoned.

Abrasion Tests

Another property of organic coatings that has not been amenable to accelerated testing is abrasion resistance. The difficulty probably arises because abrasion or wear resistance is not a single, unique property, but a combination of more fundamental properties such as hardness, tensile strength, elasticity and adhesion. It is, therefore, not surprising that simple tests have given results that frequently do not correlate with practice and usually are not reproducible between laboratories.

Many coatings chemists have been interested in predicting the service lives of coatings used where abrasion is considered to be an important factor. Such locations are floors, walls of public buildings and leading edges of aircraft surfaces.

The first and most rudimentary approach to accelerating the natural abrasion of coatings was to allow an abrasive to fall upon the film. Emery powder was used initially as the abrasive, but was replaced with sand, possibly because of the limited abrasion resistance of coatings in use at that time. To control and concentrate the flow of abrasive, a tube was used with a funnel attached at the top to act as a reservoir.

As more workers adopted this simple test, it became obvious that standardization was required if results from different laboratories were to be compared. The length and the diameter of the tube were fixed and a standard abradent incorporated in the test. In the United States, where much of the work on the method was conducted at the Bell Telephone Laboratories (1), Ottawa sand was adopted.

Because sand particles are round and not too hard, abrasion tests using sand take a long time, even with coatings of only moderate abrasion resistance. Consequently, testing authorities in Canada selected silicon carbide which is synthetic and, therefore, should be more dependably uniform in composition and particle size. Unfortunately, the carbide is in the form of needles that are easily shattered, thus changing the particle-size distribution.

With either of the standard abrasives, tough materials require excessive amounts before a breakthrough occurs, so that the test is very laborious and time consuming. The Paint Laboratory at the Division of Building Research, NRC, made a few studies to improve this situation, but had no significant results. In recent years the laboratory's chief connection with the test has been through the Canadian Co-operative Paint Exchange.

The falling abrasive test has been run on the Exchange five times. In spite of efforts to improve the procedure by specifying test conditions more closely, the reproducibility between and the repeatability within laboratories have not changed much over the years. Table II compares the different exchanges by means of the coefficients of variation based on the grams of abrasive per mil of coating.

As with many other exchanges, part of the variation between laboratories is due to errors in preparing films of a stated dry film thickness. On one exchange, however, when all the test panels were returned to one laboratory for measurement of film thickness, the precision was not greatly improved.

Review of the abrasion exchanges indicates that part of the difficulty is caused by the silicon carbide. Although the material is a recognizable chemical compound, it exists in two or three grain structures: cubic, referred to as "blocky," a more needle-like form (slivery), and a combination of the two. Each manufacturer has fairly good control over the proportions of the two grains in his product, but when different paint testing laboratories purchase from different suppliers there can be a wide variation in the abrasives. In addition, the sieve designation of commercial abrasives is not too rigorously followed.

In spite of these conditions, not all the discrepancies in abrasion test results can be attributed to silicon carbide. On one exchange where sand was included for comparison, the precision was equally poor and it took much longer to obtain the results.

On a special exchange conducted by two government testing laboratories and one paint company laboratory, there was no improvement in precision with aluminum oxide. This work illustrates what generally seems to happen when attempts are made to instrumentate a physical test that is apparently simple but which combines several basic properties. Even when numerical values can be assigned and the apparatus is sufficiently precise to differentiate between

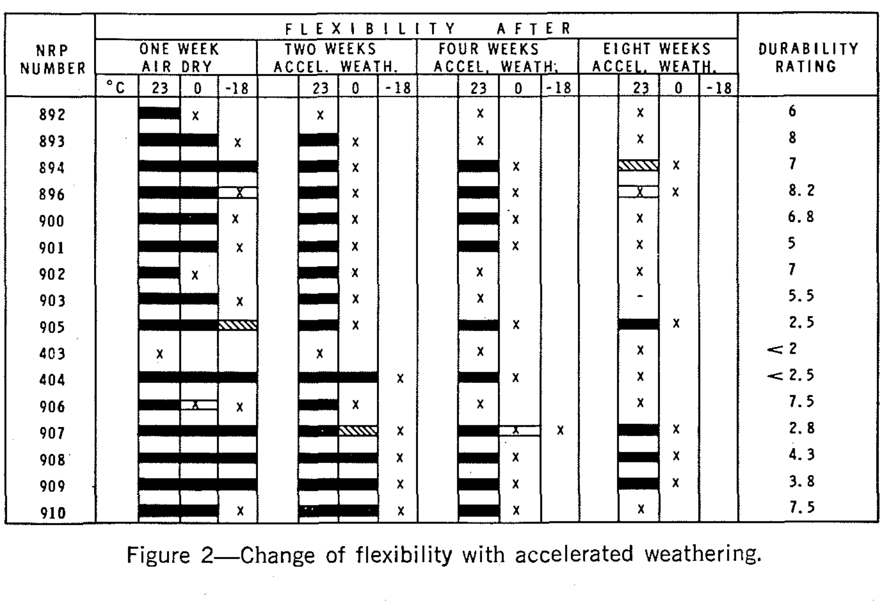


Figure 2—Change of flexibility with accelerated weathering.

Table II — Coefficient of Variation in Exchanges on Abrasion Resistance

Exchange Number	Material Tested	Substrate	Coefficient of Variation, % Between Laboratories	Within Laboratories
20A	Clear alkyd	Glass and steel	127.7	20.70
20B	Clear alkyd	Glass and steel	69.9	67.50
45	Floor enamel	Glass	92.4	16.90
62	Floor enamel	Glass	102.3	12.35
102	Floor enamel	Phosphated steel	91.6	18.00

somewhat similar materials, the test, although it may have fairly good repeatability, is unreproducible between laboratories and defies all efforts toward improvement. Other coatings tests of a similar nature are surface hardness, whether measured by Sward rocker, needle scratcher or pencil, and drying time, where attempts are made to duplicate mechanically the action of the finger.

Corrosion Resistance

The standard accelerated test for measuring corrosion resistance of coatings on metal is the familiar salt spray test, ASTM designation B116. Although it is called for in many specifications, it is another test with which various organizations have been struggling for years to obtain some reproducibility.

As a result of work carried out in the laboratories of the Materials Section (much of it by Mr. John Harris), it has been concluded that the salt spray test is good for what it was originally developed—the testing of metallic coatings applied to metals. Here, the degree and type of failure, particularly with the copper-acetic acid modification B368, are reported to correlate well with actual exposures. For organic coatings, however, it is believed that the test results have poor reproducibility, and bear little relation to exterior performance. Consequently, the Section has not operated its salt fog cabinet for at least four years.

Tests, some of which have been reported (2), indicated that there are two reasons why salt fog exposure does not correlate with actual exposure. First, the test is operated continuously at 100 per cent relative humidity, whereas in atmospheric weathering, wetting is intermittent and insufficient water to produce blisters is absorbed. Second, the cabinet is maintained at 35° C.

It was shown that practically all normal types of primers immersed in water at this temperature blister; most blister at 23° and very few near 0° C. The Division operates eight natural exposure sites located across Canada; in tests conducted at these sites blistering without rusting was not an important factor in failure of steel primers, yet it is one of the main causes of failure in the salt spray test.

For example, an iron oxide-zinc chromate primer to 1-GP-40a exhibited 20 to 60 per cent medium blistering after five days of salt spray, and 50 to 100 per cent medium-dense blistering after seven days. This same material was applied to steel prepared in various ways, including weathering and wire brushing, and topcoated with alkyd enamel. It performed well for eight years at all DBR sites except an industrial marine location. A limited amount of blistering occurred only on the undersides of panels, except for the wire brushed specimens where some also appeared on the face.

The first series of coatings exposed at the sites for the Associate Committee on Corrosion Research and Prevention included a similar type of primer. After six years of exposure there was little development of rusting and blistering on any primer, whether topcoated or not, except at the previously-mentioned site (3). Most of these primers would be expected to fail the salt fog test in seven to 14 days.

Because of results like these the salt spray test was abandoned and work concentrated on a cyclic condensation test in which water blistering would be reduced but corrosion would still be accelerated. After many tests on a small Marr apparatus designed to comply with British Standard 1391:1952, an Aminco Climate Lab was modified. The Climate Lab was originally designed to supply air of a given temperature and relative humidity by saturating air at the required temperature and then heating the air to get the desired conditions. Timers and an external heater were added to the machine to get a more rigorous condensation cycle.

The cycle is produced by first running the refrigerator for two hours, which cools the cabinet and test panels to 40° F. Warm, moist air is then blown into the cabinet for 30 minutes when the heater and fan shut off. Moisture begins to condense on the panels almost immediately and continues for several hours.

After equilibrium is reached, there is a period during which the condensate remains on the panels before the additional heater is turned on and blows warm air directly on the panels to dry them. Then the panels are allowed to cool, during which time they may be examined before the 24-hour cycle starts again. To duplicate service in industrial atmospheres, sulfur dioxide can be added to the system.

Salt Fog Testing

Since the salt fog test was condemned for poor correlation as well as poor reproducibility, there must be some basis for recommending this cycling test. A specific example from several years of work is one series of tests in which 88 organic coatings, mostly primers, were compared for performance in the Climate Lab and at the DBR Ottawa exposure site. The primers were commercial, specification or experimental. In the last category pigment volume concentration was systematically changed so that some primers were well above the critical PVC. About half the coatings were exposed (45° facing south) for three years; the remainder were exposed for two years. The exposure period in the cycling test was three months.

The simplest way to compare the two exposures is to see whether one is more severe than the other, or whether they give approximately the same result. On this basis, 60 of 88 samples, or about two-thirds, gave similar results. In three cases exterior exposure caused greater failure, and in 25 the reverse was true.

In a more detailed analysis of the results, the panels were rated on the 0 to 10 scale. The correlation coefficient between the exposures was calculated to be 0.47 where 1 is perfect correlation and 0 is no correlation at all. Although the observed value is not particularly high, it was found to be statistically significant at the 99 per cent confidence level. When the coefficient was recalculated using only the ratings of the materials exposed for three years, it increased to 0.55. Hence it would appear that a slightly shorter exposure time in the Climate Lab would have more nearly paralleled the severity of the natural exposure and would have increased the correlation coefficient.

It is probably safe to say that even these coefficients are much higher than would be obtained in a comparison of the salt spray test to natural exposure. In addition, the type of failure is much more representative of actual exposure results. Since only one laboratory is equipped with the modified apparatus, there is no information available on the reproducibility of the test.

Speeding Breakdowns

Recent work has shown that the rate of corrosion acceleration when primers are topcoated is less than many people would desire. Consideration is being given to changing the timers to get two cycles per day to increase the speed of breakdown. The correlation, of course, should then be checked again. A similar cyclic condensation test using a modified humidity cabinet with a four-hour cycle has recently been described (4).

Because many other laboratories have done considerable work with electrical conductivity, resistance and related properties, the Materials Section has not been very active in that field. To summarize the Section's position on accelerated corrosion tests for organic coatings on metals: (1) the salt spray test is not considered relevant; (2) a condensation cycle predicts corrosion resistance more reliably but more slowly; (3) immersion tests are related only to immersion exposures.

Artificial Weathering

The final section of this report considers those tests that most paint chemists think of when the term "accelerated testing" is used, i.e., accelerated or artificial weathering. It has been said that tests that set out to assess coating durability have given rise to the greatest discussion and least agreement between users of the tests. There are several methods of accelerating the effect of weather on coatings and the factors that are accelerated usually depend upon the interests of the investigator or the capabilities of the technique used. They can vary from the scheme once used in India, where the panels were exposed to natural light, which at that latitude is quite intense throughout the year, with the acceleration of a water spray from a hose at intervals, to a complicated apparatus that exposes panels to a concentrated light source, sprays them with water either on the front or back, and adds sulfur dioxide, oxides of nitrogen and/or ozone to the enclosed atmosphere.

It appears to the author that the more gadgets are added to a machine the more things can go wrong with it. Perhaps there is an optimum number of factors that should be included in any one test, and if others are considered important they should be incorporated in a separate test. Moroney (5) has stated that multiple correlation analysis with more than four variables is usually profitless; perhaps this is a clue as to how many factors should be included in one test.

One accelerated test that is reputedly simple because it is concerned with only one performance characteristic is the blister box. It is supposed to predict whether coatings will blister on wood substrates if moisture migrates through the wood. Some researchers have claimed that the box test is too severe and that blister *houses* correlate better with coating performance on wooden homes.

In spite of the considerable work (6) that has been carried out on blistering and the extensive discussion about it, field examinations by this Division, which usually are made because of complaints, have not shown it to be the most important paint problem. Cracking and peeling of oil paints is a much more common type of failure, at least in the Ottawa area. Consequently, some work in this field has been done by Dr. R. S. Yamasaki of the Materials Section.

In Dr. Yamasaki's project it was first necessary to be able to reproduce the phenomenon in the laboratory so that it could be studied systematically. The chief factors causing cracking and peeling were considered to be solar radiation, which decreases the elasticity of coatings, and water, which not only can affect adhesion but also, through continuous changes in water content, can bring about swelling and shrinkage of films. Despite the presence of both degradative elements in the customary accelerated weathering tests, cracking and peeling are not normally noted. Experiments were, therefore, carried out to see whether different wetting and radiation-drying cycles would cause cracking and peeling.

Two oil paints were used in the study: a paint made in the laboratory exactly conforming to the composition given in 1-GP-28b Type B, and a paint commercially supplied to the same specification. The latter had failed in the field by cracking and peeling. The materials were applied to white pine panels which had the sides and backs sealed so that moisture changes would take place through the test face.

Three Exposures

The panels were first subjected to one of three exposures that provided a range of wetting and drying conditions. Cycle I consisted of three days of immersion in distilled water, followed by three days of drying by radiation in a twin carbon arc Weatherometer. The second exposure was in a twin arc machine operating on the CGSB cycle of eight hours of light only, ten hours of light and water, and five and one-half hours of water only. The third exposure was in a single arc Weatherometer using the DBR cycle of 12 hours of light without water, 12 hours of water without light.

These exposures continued for about 30 weeks, and showed that all three cycles caused cracking and peeling if pursued long enough. Cycle I was most rapid, and the type of failure most closely resembled those in the field. Cracking and peeling were associated with the wood grain in the first cycle (Figure 3), with blisters in the third cycle (Figure 4) and with both in the second cycle. The Figures also show that the commercial paint underwent greater breakdown than the laboratory paint.

One disadvantage of the procedure, even with the first cycle, is that the acceleration is not great. It might be expected that periods shorter than three days for each part of the cycle would decrease the over-all exposure time by increasing the number of cycles per time interval. In fact, they were less effective. Apparently, the panels required three days of immersion to absorb as much water as those subjected to spraying for 12 to 15½ hours. They also required three days of radiation to remove almost all of this water.

The effectiveness of the first cycle shows that complete wetting and drying plays an important part in causing the failure. At the end of their drying periods, the panels exposed to Cycle II, which was intermediate in severity, had some water, and those in least effective Cycle III had most water. The same paints applied on glass panels did not exhibit the deep cracking and peeling that was observed on wood panels; this shows that substrate movement is an important factor in the breakdown.

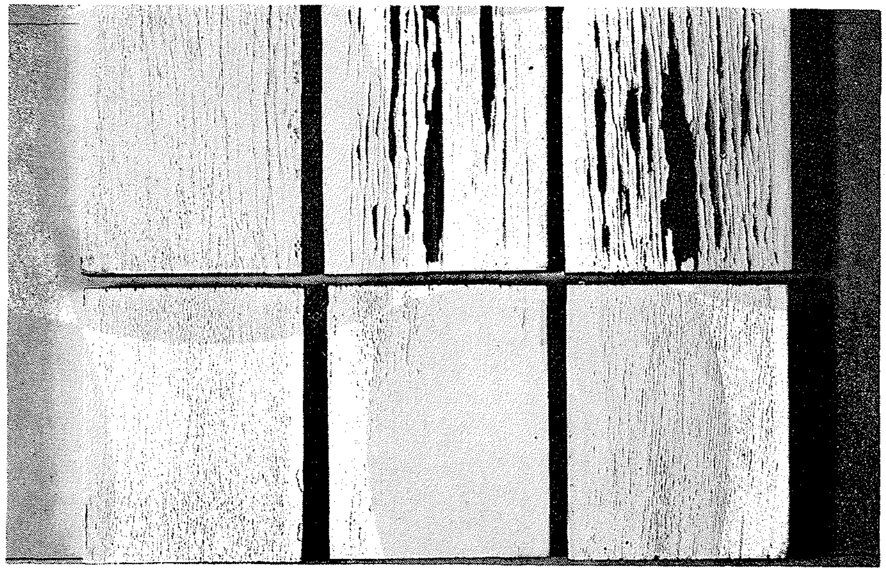


Figure 3—Cracking and peeling produced by cycle of three days of water immersion, three days of drying in twin carbon arc Weatherometer. Specification paint: top — commercial, bottom — laboratory.

Alkyds Hard to Crack

When alkyds were used in this test, they could not be made to crack within a reasonable time; this finding correlates with previously reported work which showed that alkyds have higher original extensibility and lose it more slowly on exposure than oil paints. Studies designed to determine the mechanism of cracking and peeling of oil paints are continuing.

Another aspect of accelerated weathering which was studied is the effects of different cycles that can be incorporated in artificial weathering procedures. The possible permutations and combinations of just the two factors of radiation and moisture are almost endless. Fortunately, only a few have come into common use.

Several years ago, the Test Methods Subcommittee of the CGSB Paint Committee surveyed a number of countries regarding the type of apparatus and the cycles used in their accelerated weathering tests. From the replies it appeared that the two main machines were the Weatherometer of Atlas Electric Devices and the Marr of Great Britain. Since that time, the Xenotest, manufactured at Hanau in Germany, has been introduced, although several laboratories use this machine more for color stability than for weathering tests.

An interesting finding of the survey was that laboratories in many countries were not very satisfied with the usual cycles specified for the two machines. It appeared that if cycles were used unaltered it was chiefly because they were standard cycles. In several countries, particularly those familiar with both machines, comments were made that the ASTM cycle did not have enough moisture in it or that the British procedure had too much.

In Australia, the Defence Standards Laboratories modified their Marr machine by adding a second arc, and their Atlas by adding a second spray. The British test, which was originally a Defence method (No. 26 of DEF/1053), has been changed to overcome the blistering caused by excessive moisture. In British Standard 3900, Part F3, a fan has been added and the arc wattage increased. The test is now claimed to be quite reliable, although not universally applicable (7).

The ASTM cycle has been altered by the Norwegians to include one and two-hour periods of water spraying instead of the usual nine minutes in one hour or 18 minutes in two hours, all periods with continuous irradiation. In Denmark, greater wetness was obtained by shutting off the arc during the 18-minute spray interval. The Swedish Paint Industry Laboratory did the same, except that it used the three and 17-minute variation of the cycle. Several cycles are used in the USSR; three to 17 appeared to be the one in most common use, but other cycles with more water are used to accelerate chalking or corrosion.

A somewhat embarrassing result of the survey was the question asked by one country concerning the reasons for adopting the CGSB cycle, i.e., the 8-10-5½ cycle already described. The only basis seems to have been laboratory convenience before the use of timing cams obviated manual switching. In the only large-scale comparison of the CGSB and ASTM cycles, conducted by the Canadian Department of Public Works, it was found that both gave similar results for color change, but the CGSB

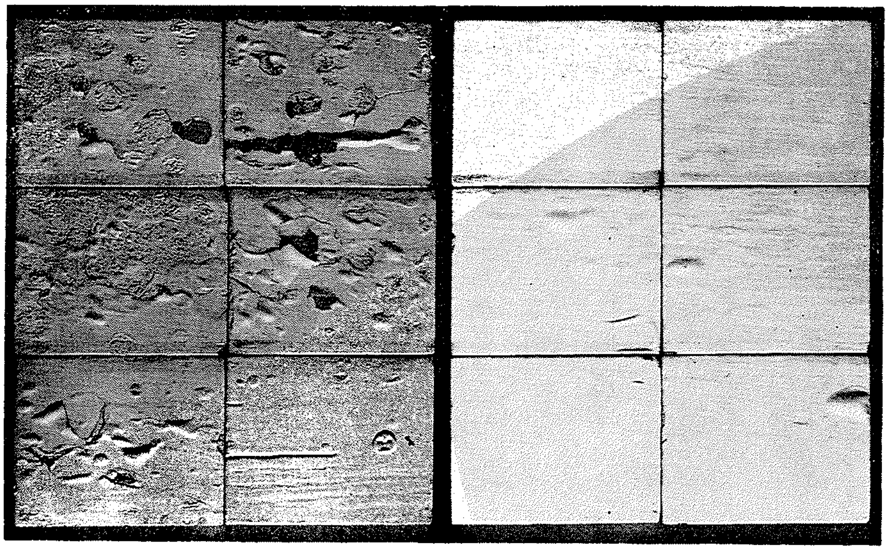


Figure 4—Blistering and cracking produced by cycle of 12 hours of light without water, 12 hours of water without light in single carbon arc Weatherometer. Specification paint: left — commercial, right — laboratory.

cycle caused a more rapid loss of gloss.

Because the Paint Laboratory of NRC, along with many others, was not satisfied with results from either cycle, investigations on other modifications have been carried out from time to time. In tests on clear finishes for exterior wood, neither the ASTM nor the CGSB cycle produced failures that resembled those occurring naturally. In the Weatherometer, too much failure occurred at the coating surface, while there was very little at the wood-coating interface where most failures took place in natural exposures at DBR. Hence, the usefulness of the tests for predicting exterior durability of the finishes appeared questionable. The type of failure suggested that the light, which operates 100 per cent of the time in the ASTM and 75 per cent of the time in the CGSB cycle, had too great an effect. The radiation period was, therefore, decreased to 50 per cent with the hope that failures would be more representative although probably slower to occur.

Simultaneous water spray was considered an unnecessary complication; similarly, the remainder of the cycle consisted of water spray without radiation. Because wood panels seemed to warp more in twin arc machines on the other cycles, it was decided to operate the 12-12 cycle with a single arc. This is the DBR cycle referred to in the cracking and peeling studies. Although it was least effective in that test, with clear finishes it gave the types of failures observed on exterior exposures, i.e., alkyds failed by delamination, while p-phenylphenolic-tung varnishes lasted longer but failed by checking and cracking. These types of materials have now been tested often enough to show that in this cycle alkyds start to fail in 16 weeks, while the phenylphenolic should last 24 weeks before much failure is observed.

Because the 12-12 cycle gave satisfactory results with clear finishes on wood, its use was continued, although with the assumption that it would be slower than the other cycles due to the reduced number of arcs and the lower percentage of time in which the arc operated. In connection with other work, occasionally a material has been exposed to two or three different cycles. Surprisingly, in some cases with pigmented materials, the DBR cycle gave results that were not too different.

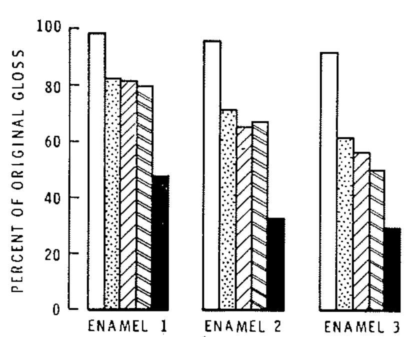
Gloss Retention

Figure 5 compares the gloss retention of three alkyd enamels exposed for 21 days in three carbon arc and one xenon arc Weatherometers. The last was operating on the dew cycle: one hour of light only, and one hour of no light with water spray on the back of the metal panels to cause condensation. As expected, the greatest loss of gloss occurred in the xenon machine.

The second most effective cycle was the 12-12. For example, enamel three, the control panel of which lost gloss most rapidly and threfore probably has the poorest gloss retention, retained 50 per cent of its original gloss in the DBR cycle, 57 per cent in CGSB and 61 per cent in ASTM.

The results with the other two enamels were similar for the DBR and CGSB cycles. These cycles tended to be more effective than ASTM; the DBR, or 12-12 cycle, was therefore at least as effective with only one carbon arc as the others were with two. With the small number of samples, the 12-12 cycle is not shown to be significantly faster statistically, but the test does indicate that with pigmented finishes the cycle can yield comparable results with a very great saving in light operation over the ASTM cycle and of both light and water over the CGSB cycle.

With several Weatherometers the saving of deionized water in the latter case is significant. Figure 5 shows that the ASTM cycle was



CYCLE	CONTROL	ASTM	CGSB	DBR	DEW
ARCS	-	2	2	1	1 XENON
LIGHT ONLY	-	51 MIN	8 HR	12 HR	1 HR
LIGHT & WATER	-	9 MIN	10 HR	-	-
WATER ONLY	-	-	5½HR	12 HR	1 HR
HOURS LIGHT EXPOSURE	-	456	354.5	234.3	220

Figure 5—Gloss retention.

least efficient in the use of radiation. It appears that when both elements of degradation are operating at the same time they tend to counteract each other; consequently, during those cycles when the light remains on while water is being sprayed, much of the water is wasted. It can readily be observed that the surface of a sprayed panel is dried by the radiation before the panel makes a complete revolution, with the result that the radiation does not have a chance to impinge upon a film with a high moisture content, as can occur during natural weathering. Whatever the reason, if 234 hours of exposure to a single arc are as effective as 456 hours to a twin arc, the latter procedure is lacking in acceleration.

It is, perhaps, a coincidence that the three cycles that have proved to be effective for different purposes—i.e., the dew cycle, the 12-12 and the three-day-three-day for cracking of oil paints—have 50 per cent radiation without water and 50 per cent water without radiation.

Xenon Arc

In addition to possible variations in the number or wattage of the light sources, there is another variable in the type of source. The source that has received the most attention in recent years is the xenon arc, which is available in accelerated weathering machines from at least two manufacturers. It has three advantages: (1) when properly filtered the radiation closely resembles terrestrial sunlight; (2) its intensity is stable over relatively long periods after initial breaking in; (3) the acceleration factor is high.

The disadvantages of the xenon source are its inefficiency, high cost and relatively short life. A doubtful advantage is the ease of replacing the Pyrex filters with quartz filters, which transmit well down into the UV. This results in very rapid degradation of coatings, but is contrary to the very reason for adopting the xenon light, i.e., that it closely reproduces sunlight.

The working group on light and water exposure apparatus of ASTM Committee D-1 has adopted this course of accentuating speed rather than reproducing natural durability. Consequently, interlaboratory tests using xenon arcs with quartz filters have shown marked reversal in results with rapid failure of acrylics known to be very durable in Florida.

Experience with the xenon Weatherometer at DBR has not been extensive. To date, the dew cycle has been most used in the machine. The work already mentioned on cycles for use with carbon arcs indicated that the one-hour high-humidity periods in the usual dew cycle might not be long enough for coatings to absorb much moisture. Consequently, the effect of longer times for both sections of the dew cycle is currently being investigated.

Three air-drying and two baking enamels which have been exposed for one year in Ottawa are being subjected to dew cycles of 1-1, 2-2 and 3-3 hourly periods. In an attempt to reduce the time variable as much as possible after an initial exposure of one day, each set is being exposed to its cycle for one week at a time. Because only one machine was available and because the intensity of the xenon source gradually decreases, this procedure seemed best, even though it allows a recovery period of two weeks.

At present there are no significant differences between cycles, although the baking enamels which lost most gloss under natural conditions are just starting to lose gloss rapidly in the two cycles with longer periods. If significant results are obtained, further tests will be run to determine whether there is an optimum dew cycle period.

Conclusions

This report has related the experiences of the Division of Building Research with tests for flexibility, abrasion, corrosion and artificial weathering to predict the serviceability of coatings. Because of these and other studies a certain approach to accelerated tests has developed.

(1) Wherever possible, the normal or average result of actual service should be established. In spite of the difficulties that can arise, such controls are necessary yardsticks against which the suitability of accelerated tests can be measured. Granted, a long time may be required to obtain results if durability in the normal environment is involved, but the author maintains that it should be attempted.

In addition to the problem of time, problems arise due to the great variability possible in results concerned with natural phenomena. It was no accident that the first scientists to make much use of statistical treatment of results were agriculturists. With such variability, either the tests must be statistically well designed or a fair number of replicates must be run to get a reliable estimate of the performance of the material. Mitton and Church (8) have shown how much of the variability due to differences in weather during differing exposure periods can be eliminated if controls are exposed a sufficient number of times to obtain a reliable average durability.

(2) It is possible to use accelerated tests for prediction purposes provided a test appropriate for the properties of interest is selected. For example, when cracking and peeling are being studied, a test must be used in which those failures play a prominent part. As previously proposed, the number of factors in any one test should be limited or the test becomes unreproducible. It is not, for instance, considered necessary to include ultra violet radiation in corrosion tests, although one might expect it would cause cracking of exposed films, thus allowing water to reach the substrate. In actual practice, corrosion is frequently more severe on the underside of panels, indicating that wetting and drying are more important factors than UV light.

(3) The preceding does not mean that one factor should be emphasized to the virtual exclusion of all others. In many tests, factors or elements are accelerated merely because they can be easily concentrated or accentuated, or because they cause the duration of the test to be markedly reduced. One example is use of the xenon arc with quartz filters. According to Brand et al (9), disproportionate acceleration is at the root of the troubles with present methods. They suggest that truly reliable methods will be developed only when each deteriorative factor is accelerated a proportional amount, but

conclude that this is extremely difficult, if not impossible.

(4) In accelerated tests, especially artificial weathering, it is good practice to include materials with poor as well as good durability in both the accelerated test and normal use. With such controls it is believed that accelerated weathering provides a good indication of the durability that can be expected from a coating.

(5) More attention should be given to methods of accelerated detection. Instead of waiting for the appearance of visually observable defects, the effects of weathering should be measured by tests that are sensitive to changes in basic properties. In his survey of accelerated testing of paints, Hearst (10) concluded that methods of accelerated detection of normal deterioration will probably give better correlation with performance than methods using normal observation under conditions giving accelerated deterioration. Such detection methods can, however, be used with accelerated tests. For example, one can use changes in the infra red curves and in tensile properties of coatings subjected to accelerated weathering.

(6) Accelerated tests have probably provided as much as one can rightfully expect until basic studies provide a fuller understanding of the processes involved in deterioration. So many factors are interrelated that a universal test or cycle will never be developed, but further study should indicate ways to improve present accelerated methods.

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