

Twenty Five Years of Paint Testing"

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The span of years marked by the twenty fifth anniversary of the Division of Paint, Varnish, and Plastics Chemistry of the American Chemical Society has witnessed a tremendous advance in the quality and general utility of organic finishes. This advance has been due in no small degree to a considerable elaboration and refinement of testing technique.

In many ways it is difficult to differentiate between cause and effect in the parallel improvement of both quality and test technique. The primary functions of paints, varnishes and lacquers are protection and/or decoration. As an increasing number of usages for such coatings were developed, it is a natural corollary that methods of test had to be engineered to ascertain the capacity of the finish to meet the requirements imposed. At the same time, the wider scope of application and performance characteristics which could be determined led to a greater appreciation of the versatility of the material. This in turn opened a broader vista of potential usages. Thus the contribution of the test chemist and the testing engineer, both in control of quality and in expansion of usefulness of protective and decorating coatings must be recognized.

Progress in Testing Technique

The term "testing" has three connotations: a review of things that have already occurred; an inspection of the current situation; and on the basis of knowledge of all available facts, a prediction of the future. Testing as it applies to paint technology has several aspects:

- (1) In formulation, the determination of the characteristics of a finish which fix its particular niche in the protective and decorative field.
- (2) In production, the determination of the level and uniformity of quality compared with a standard set up for that material.
- (3) In development, the analysis of service and durability records upon which may be predicated improvement of the product.
- (4) In research, the combination of findings from all such tests, giving an insight into the complex structure of the paint film, which is essential to all progress.

Testing in the paint industry certainly was not a new development within the past twenty five years. It is true that since 1922 there has been a large increase in the number of chemists, chemical engineers and technicians employed in the paint industry, and the importance of the test chemist and test engineer has increased proportionately. However, much still remains to be done, as was demonstrated in the 1946 report of the Dallas Paint and Varnish Production Club, covering a survey of methods used by paint manufacturers in testing paints and varnishes. (1)

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- (1) Official Digest, Federation Paint and Varnish Production Clubs
263, 653, 1946

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Out of approximately 290 laboratories reporting, some 13% still did not test their production for such fundamental properties as fineness of grind, weight per gallon, consistency, brushing and flow characteristics, opacity, color, gloss, hardness, etc., or did so only when such tests were specified by the consumer. This means, however, that 87% of the paint manufacturers run such tests as a matter of routine. Volume-wise, the proportion of paint which receives regular and careful inspection and testing is undoubtedly much higher.

Gardner's "Physical and Chemical Examination - Paints, Varnishes, Lacquers, Colors," now in its tenth edition, has for the past twenty five years been the outstanding compilation of test methods employed by the paint industry. The first edition was published in 1922, the second in 1925, with successive editions appearing at frequent intervals up to the tenth of 1946. Therefore, this outstanding work covers precisely the period under consideration. A review of the various editions as they appeared affords in itself a picture of the progress in technology and testing technique.

This division of the American Chemical Society has made numerous contributions to the present knowledge of paint testing. The American Society for Testing Materials, especially Committee D-1, has established methods of tests which are accepted standards for material suppliers, producers and consumers of coatings. The member clubs of the Federation of Paint and Varnish Production Clubs also have contributed much to the current knowledge of testing in the paint industry. Federal and other governmental or military purchasing agencies and industrial consumers through composition or performance specifications present a wide range of pertinent tests. The literature constantly adds new methods of test or refinement of current technique.

The testing of industrial finishes was discussed in detail in a Symposium on Paint and Paint Materials, sponsored by the American Society for Testing Materials in Philadelphia in February, 1947. Also, the Buxton Conference of the Oil and Colour Chemists' Association held in England in May, 1947 had as its general theme the assessment of paint performance. While some of the discussion applied to trade sales types of paint, considerable emphasis was placed on the testing of industrial finishes.

A complete review of all test procedures applying to the composition, development or formulation, and the performance of protective and decorative coatings is neither necessary nor feasible within the scope of this paper. However, since the field of exterior house paints is one of the most widely explored in all paint technology, it will serve as an example of the contribution of the test chemist and testing engineer.

Progress in Paint Formulation - The improvement in performance and service qualities of exterior house paints over the past 25 years has been extensive. See Figure 1. The four photographs illustrate the comparative appearance and durability of two-coat systems of representative white ready mixed house paints of 1927, 1935, 1940 and 1943, all after four years' simultaneous exposure. The paints were all on one continuous board of white pine siding, exposed south vertical in suburban Chicago, Illinois.

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The 1927 paint is representative of the 60% lead, 30% zinc oxide, 10% extenders paint of that era. Such paints tended to collect dirt which was then shed unevenly, and the film failed by checking and cracking. By 1930 titanium pigments were being used in conjunction with lead and zinc pigments and extenders, with the titanium usually introduced in the barium sulphate extended form. By 1935, titanium oxide plus selected extenders was replacing the titanium barium pigment to a considerable extent. Appearance qualities had improved, due to the brightness, opacity and chalking tendencies of the titanium oxide, and durability had been augmented. By 1940, specialized house paint undercoaters were widely used, and the pigmentation of the top coat had moved in the direction of higher titanium and extender content with somewhat reduced lead pigments.

Up to 1943, pigment-volume-concentration usually was held at about 28% to 30%. War time shortages necessitated conservation of drying oils and led to the use of increasing proportions of bodied oils, with less total binder. The pigment-volume-concentration increased to approximately 36%. The net result was a better house paint in such important qualities as flow, drying, and reduction in dirt collection, checking and cracking tendencies.

The steady progress in protective and decorative properties of house paints illustrated in Figure 1 was made possible to a considerable degree by the progress in methods of testing. The backbone of this testing was actual outdoor exposures - panels, house tests and other forms of field testing. There is probably no other type of paint coating in which durability over a span of years plays a more important role. Since house paints have a life expectancy of several years, tests which expose the paint to the weather in much the same fashion as in regular service are necessarily laborious and time consuming. The improvement that has been made in house paints in the past 25 years has extended the time required for exterior exposures to show significant failures. Hence paint durability testing continues to consume more and more time and progress becomes slower and slower. When the test chemist faces such a situation he tries to devise special laboratory tests which will give him in a short time the same results which he would get on longer normal exposure. So far there has been no such happy solution to the problem of testing exterior house paints.

Exterior house paints are exposed to air, sunlight, wind, various degrees of humidity, dew, fog, rain, sleet, ice, snow, various and varying temperatures, dirt, soot, and in some cases industrial gases, mildew, salty atmosphere, etc. The severity of these conditions changes from day to day and year to year. All of these states of weather influence both the initial drying and the service performance of the paint. Other factors exclusive of weather or composition include the wood or type of repaint surface on which the paint is applied, film thickness, direction and angle of exposure, to mention only a few.

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(2) Browne, F. L., U. S. Dept. Agr., Misc. Pub. No. 629, 1947

Laboratory Tests to Accelerate Weathering

Carbon Arc Type Exposure Apparatus - Devices of this general character represent developments of the first school of thought and have been in use for many years. (3) (4)

- (3) Nelson, H. A. and Schmuts, F. C., Am. Soc. Testing Materials, Proc., 22, Part II, 456, 1922
- (4) Nelson, H. A. and Schmuts, F. C., Am. Soc. Testing Materials, Proc., 24, Part II, 920, 1924

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On the types most widely used, the paint films are radiated with ultra-violet light from mercury vapor or carbon arcs, usually the latter. At intervals the films are subjected to thermal shock and moisture penetration from a water spray, plus additional thermal shock and the stresses set up by the expansion on freezing of the water retained in the film if refrigeration is used. Expansion and contraction due to the alternate soaking and drying out occur at various stages in the cycle. The intensity, amount, and rage of radiation, ambient temperature and humidity, panel temperatures, thermal shock and degree of moisture penetration, rest periods and the relative time distribution of test conditions during the cycle are determined by the design and operation of the machine.

Thus it appears that such devices introduce a large number of the test factors present in natural weathering and it might be anticipated that they should permit prediction of performance in actual service. Although they have been in use for over 25 years, there has been no unanimity in that conclusion, especially in the testing of long oil exterior paints. (5)
(6) There is still no agreement as

(5) Am. Soc. Testing Materials, Proc., I, 366, 1936

(6) Nat. Paint, Varnish Lacquer Assoc. Sci. Sect. Circ. 671, February, 1944.

to the best cycle to reproduce natural weathering. Failures to correlate accelerated test results with outdoor exposures, or tests run on identical materials by different cooperator, or to classify various classes of exterior finishes in their actual order of exterior durability or color retention, etc. in some instances have been ascribed to failures to duplicate test conditions precisely.

The Cleveland Paint and Varnish Production Club has been active for a number of years in an investigation of carbon arc machine operation. (7) (8) (9)

(7) Official Digest, Federation Paint and Varnish Production Clubs, 250, 454, 1945.

(8) Official Digest, Federation Paint and Varnish Production Clubs, 262, 518, 1946.

(9) Am. Paint J., 32, No. 7-B, 8, 1947

They found that a satisfactory correlation of such devices in the Cleveland area and duplication of Miami, Florida test fence exposures did not extend to arc cabinets run by cooperator more widely distributed geographically, who were not all confined to the Cleveland area, using the same water supply.

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they found an apparent necessity for adjustment of water supply, duration of water exposure, maximum temperature and duration of nearly maximum temperature, to achieve uniform machine operation in various parts of the country. They infer that after such adjustments are made further cooperative work must be done before any statements can be made as to whether there is a correlation with Florida results.

There are two principles of acceleration as far as laboratory weathering tests are concerned. First, it should be possible to intensify the weathering factors so that visible failure occurs more rapidly than under actual service conditions. This is the scheme used most widely. Second, it should be possible to diagnose the deterioration of the finish under test at an earlier stage when visible failure is not apparent. The Cleveland Club's efforts in the latter direction have been interesting and have met with some success. In addition to the observation of the usual loss of gloss, chalking, checking and color changes as criteria of the effects of weathering, they checked additional properties such as distensibility, adhesion, scrape hardness and rusting on the metal panels used in their tests.

The American Society for Testing Materials has Tentative Specification D 822 - 46 T, "Tentative Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer and Related Products." (10) It specifies the types of carbons, measurement and control of

(10) Am. Soc. Testing Materials, Standards, Part II, Nonmetallic Materials - Constructional, 1614, 1946.

current, voltage, temperature of panels, temperature and pressure of the water spray, rotation of the test specimens, and the operating cycle. The specifications for the water other than temperature and pressure are quite general.

Hollis (11) points out that it is desirable that a light source be

(11) Hollis, H. J. Oil Colour Chemists' Assoc. 30, 431 (1947).

developed which (1) is constant in output, (2) gives a fair proportion of light in the wave band from 2900 - 3300 Å but little in the extreme ultraviolet, and (3) does not vary in intensity during use due to fouling of the glass globes or filters by degradation products. Corax D glass globes show a relatively sharp cut-off of wave lengths below 2900 Å, reasonably close to the low limits of solar radiation after passing through the earth's atmosphere, at about 2950 Å. (12)

(12) Coblents, W. W. and Stair, R., B. of S. J. of Res. 3, No. 5, 629, 1929

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A.S.T.M. Specification D 822 - 46 T (10) calls for the replacement of the glass filters (globes or windows) after 2000 hours of use, or when pronounced discoloration or milkiness develops. Filters must be washed each day with detergent and water. Thus it would appear that the radiation factors are susceptible to reasonable control.

A rather general criticism of carbon arc machines is that undue emphasis is placed on chalking tendencies at the expense of checking and cracking, especially in long oil paints. (6) (11) There is a growing realization of the part moisture plays in causing destruction of the weathering film. (9) Wirshing (13) found that plotting the monthly mean temperature and the percentage relative

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(13) Wirshing, R. J., Ind. Eng. Chem., 33, 234, 1941.

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humidity at Miami, Florida more nearly approached the failure curve of automotive lacquers exposed at that point than did the radiant energy curve. There is a general trend to revise carbon arc machine cycles to prolong the water spray period so that the paints become thoroughly soaked with moisture. It is believed that the sequence of swelling during the wet stage of the cycle followed by contraction on drying out will accentuate rupture of the film, and in conjunction with some ultraviolet radiation will simulate more closely the film failures which result from normal weathering.

House Paints in Carbon Arc Units. - A definite question in conducting carbon arc tests to accelerate weathering of long oil air drying house paints is when to initiate the test. (11) Films which reach maximum film strength quickly, such as those which dry by simple loss of solvent, or baked finishes, presumably may be subjected to accelerated tests relatively quickly. However, slower air drying films such as house paints pose another problem. As soon as a paint is spread on a house two actions are initiated. One is the drying of the paint in which oxidation and polymerization and sol to gel transformation occur which give the film strength and resistance to the elements. It is known that these processes continue for a long period eventually leading to unfavorable film qualities, such as loss of elasticity. Also as soon as the paint is applied, the destructive forces of the elements start their attack. It is difficult to fix the point at which the balance changes from the positive to the negative side.

D 822 - 46 T Specification (10) calls for a drying period of seven days for "normal drying paints." This would appear inadequate for house paints, but it is appreciated that if too much time elapses before the film may be considered properly conditioned to be placed under test conditions, the main point - that of accelerating the development of normal failure - is lost to a degree. A revision of the early stages of the exposure to reduce the severity of ultraviolet radiation and operation at lower temperature to avoid distortion of polymerization processes is worthy of consideration. However, this is often impractical in apparatus which is used simultaneously for various types of materials and groups which are started at different times. Cost is a consideration in addition of sufficient instrumentation. Also, the proper proportionation of attacking factors over the entire range of the testing cycle has not been determined.

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So far there is no data available on the results obtained on apparatus in which all the means of control on the light source, water, and other factors are employed, and the optimum cycle is utilized. As reported in the Cleveland Club investigation, (9) when such instruments are available and properly handled it should be possible to correlate results between different machines. The questions which still would have to be answered are whether costs can be held within limits and whether there would then be a correlation with natural weathering results.

Outdoor Durability Tests

Until fully dependable methods of accelerated weathering tests are developed, the paint technologists and testers must rely on time consuming outdoor tests. The technique of outdoor paint testing has been improved considerably in the past twenty five years. Early tests were often applied without too much regard for spreading rates and film thickness, or type and uniformity of wood surface used. A rather common practice was to expose the paints on flat boards at an angle of 45° facing south, often at only one geographical location, with Florida a rather popular selection. It has been pointed out that exposures at 45° south at times do not simply accelerate the normal weathering of house paint exposed vertically and may actually distort the performance in regular service. (14) (15)

(14) "Photographic Supplement to 'Leaves From a Paint Research Notebook,'"
New Jersey Zinc Co., 1938.

(15) Troutman, R. E. and Vannoy, W. G., Ind. Eng. Chem., 32, 232, 1940.

Today, most house paints are exposed on lengths of beveled clapboard siding, mounted in the regular manner, which means that the paint surface is actually slightly off the vertical. Much greater differences in dirt collection and cleaning are shown on the slightly angular beveled siding than on strictly vertical boards. (16) As a consequence, strictly vertical exposures are avoided

(16) Vannoy, W. G. Official Digest Federation Paint and Varnish
Production Clubs, 235, 177, 1944.

except for special types such as trim paints, whose major usage would be on such surfaces. North vertical exposures on beveled siding are particularly useful in studying dirt collection.

Panel Exposures - There are several good methods of panel exposures. A description of all the methods and an analysis of the effects of the variations in technique are beyond the limitations of the paper. Therefore, without prejudice to other methods the discussion will be limited to those employed for the evaluation of exterior performance of house paints in our own laboratory.

Three-foot sections of 6-inch or 8-inch beveled siding are selected for uniformity of wood characteristics within the series. The species used

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most are edge grain Western Red Cedar for Group 1 wood, (2) White Pine for Group 2, especially in checking small differences in performance of high grade paints which might not be detected on woods of such good paintability as cedar or redwood, and Ponderosa Pine for Group 3 woods. Southern Yellow Pine, as a Group 4 wood, is now used only in special studies of painting systems for that wood, due to difficulties with warping when beveled siding of regular thickness was used.

The paints are applied by brushing at controlled spreading rates, preferably in an enclosed paint shop at the exposure farm during the outdoor painting season. Weather conditions, temperature and humidity at the time of painting are recorded. The wet panels are then moved to the adjacent drying room which is roofed but open to the weather on two sides except for cheesecloth to keep off insects. If the weather permits, the panels are then mounted temporarily on southern exposure for the time remaining before recoating. The next coat is usually applied two days after the previous coat in good weather and preferably within four days under any circumstances. The top coat is then allowed to dry until it has hardened sufficiently to accept stenciled identification. It is then ready to be placed on the fence if it is to be exposed locally, or a series of panels may be boxed and shipped for exposure elsewhere.

Such panels as must be painted indoors during winter months are given two hours of radiation in a special twin arc cabinet for each coat. This is done to simulate the beneficial drying effects of sunshine which a paint receives outdoors.

Range of Tests - Since the wood characteristics of any one panel will not duplicate precisely those of any other panel, conclusions based on the results obtained on any one panel must always be suspect. Elm (17) has pointed out that

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(17) Elm, A. C., Am. Soc. Testing Materials, ASTM Bull., 142, 9, 1946.

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problems of this nature can be solved by a multiplication of tests on a sufficiently extended scale and the application of statistical methods to the exposure results. There has been a considerable increase in the application of statistical methods to paint testing in recent years, and a further increase is to be anticipated.

As mentioned earlier, weather is not a fixed constant, - rather it varies from season to season and has wide fluctuations in temperatures, precipitation, sunshine, etc., from year to year. Tests run for relatively short periods of time will yield different results depending on the season of exposure, or they may not be representative of average behavior at that location because of unusual weather conditions. Since house paints are usually kept on exposure for several years, deviations from the mean in weather conditions tend to cancel out. However, the effects of wide deviations will

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very according to the point at which they occur in the test period. Therefore, it is necessary to run repeated exposures before the normal performance can be determined reliably.

Recognizing the possibility of erratic behavior of a paint on any single panel, whether due to wood, application or manner of exposure, a guide section is applied on every panel. A paint whose normal behavior is thoroughly understood by an adequate background of exposure experience is applied on 1/3 of each panel. The test paint is applied on the remaining 2/3 of the panel and its performance is rated with regard to the guide on the same panel. The broad experience with the guide also permits allowance to be made in case unusual weather conditions are encountered.

A multiplicity of panels is always desirable and duplicate or triplicate panels are used for critical tests. It is recognized that this does not give a broad base for statistical evaluation. However, a working farm must cover such a mass of problems that there is a physical limit to the number of panels that can be prepared, mounted, and examined. For most tests the problem is handled by the use of the guides and multiple exposures. Usually this involves exposures south and north vertical in suburban Chicago, Illinois, south vertical at Coffeyville, Kansas, and south vertical at Miami, Florida. The use of both edge grain cedar and Ponderosa Pine or other combinations further multiplies the units of tests, and in total cover a wide variety of panels, geographical distribution and direction of exposure. Operating in this manner, experience has shown that the average performance of a paint may be evaluated with sufficient reliability to allow national sale in large volume with safety.

The panels are mounted on a vertical fence as shown in Figure 2. They are held against conventional studding by a fitted clamp, with an enclosed air space of 4" depth between the panels and the shiplap backing. A double fence permits exposures both to the north and south. Readings are made and recorded at regular intervals and weather conditions are also recorded as an essential part of the exposure history.

Variations in Exposure Results - When judgment so dictates, the panels are called in from the several exposure locations and jointly examined to get the over-all picture of performance. It should be pointed out that Florida exposures do not simply accelerate the incidence and development of film failures which appear in the more northerly exposures, nor do the Kansas exposures simply lie intermediate. In general, the Florida panels tend to accentuate checking and cracking tendencies, with some surface flaking and erosion a minor failure. Suburban Chicago exposures show relatively little checking and cracking in better grade house paints, but erosion tendencies are aggravated.

Figure 3 shows companion exposures of five years' duration of the same paint, at the three locations, all south vertical. Magnified areas illustrate the type of failure at each location. 4532 C, the Chicago panel, shows the results of active chalking leading into erosion, especially visible at the brush marks. There is no characteristic checking and very little cracking,

can in the very thin spots. 4532 COF, the Coffeyville, Kansas panel, shows very little chalking and no erosion, but well developed cracking along the brush marks. The elliptical cracks are marks from hail-stones. 4532 F, the Miami, Florida panel, shows some surface pitting from chalking, and cracking almost equal to the Kansas exposure.

The fact that such differences exist has been proven by many exposures, using different paint systems, different species of wood, with panels painted and exposed at different times of the year. Why such differences occur is by no means as clear. They would appear to involve the total amount of ultraviolet absorbed by the paints, the temperatures of the films, the amount of moisture absorbed and how long such moisture is retained, the expansion and contraction due to temperature changes and alternate soaking and drying, and similar factors. However, the precise reasons why the weather conditions at one point accentuate checking and at another, erosion, remain obscure.

Chicago had approximately the same total hours of sunshine as Miami in both 1946 and 1947, for example. The angles of incidence of the sun's rays, of course, were different. The average temperature in 1946 was 51.8° F. in Chicago, 60° in Coffeyville, Kansas, and 75.4° in Miami; in 1947, 49.3° in Chicago, 57° in Coffeyville, 76.1° in Miami. The extremes in temperature in 1946 were -3° F and 100° in Chicago, 7° and 107° in Coffeyville, and 45° and 89° in Miami; in 1947, -2° and 101° in Chicago, -7° and 105° in Coffeyville, and 35° and 89° in the first eleven months in Miami. Total precipitation in 1946 was 31.88 inches in Chicago, 33.15 inches in Coffeyville, and 75.46 inches at the Miami exposure field; in 1947, 39.21 inches in Chicago, 31.20 inches in Coffeyville, and 79.30 inches at the Miami exposure field for the first eleven months. The comparatively heavy precipitation at the Miami exposure field and higher average temperatures would mean relatively higher average moisture content in the air than in Coffeyville or Chicago. However, information on how long the panels at the various locations remain wet after periods of precipitation is not available in this instance. It is apparent that the relation between weather and paint performance needs more investigation. The obvious question also arises whether accelerated weathering devices should be designed to simulate results of normal exposure in Florida or at other points having weather representative of areas with larger paint consumption.

Since repaint work is proportionately much larger than application on new wood, a relatively large number of panels are on exposure constantly, utilizing standard paint systems. This affords a backlog of surfaces for repaint studies, which otherwise are handled in the usual manner.

House tests - When specific paints have shown sufficient merit to deserve further evaluation they progress from the panel stage to the final step of field testing - practical house tests, in locations selected over the entire country. Houses are chosen on the basis of representative construction and condition, with a knowledge of previous paintings if repaint work. Relatively open exposure is preferred unless studies such as mildew, blistering or peeling, performance over knots and similar special conditions are the objective. The house surface is usually divided into sections using control or comparison paints. The painters' comments, weather conditions, spreading rates, drying, gloss and finally service performance are recorded.

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Conclusion

It is obvious that when house paints have survived such rigorous testing, consumer protection is much greater. The test chemist and testing engineer have not only contributed to paint technology but to the development of better paints which are made available to the general public. However, it should also be pointed out that the procedure is too laborious and time consuming, and improvements which should have been made in paints, to the benefit of the public, have been unduly delayed. Therefore, further study of methods of accelerated testing is not only desirable but mandatory.

It is entirely possible that the present instrumentation and technique will serve as the foundation upon which completely satisfactory methods will be built to evaluate all properties of the paint, including ultimate durability. There is still much to be learned about the internal structure of the film and the changes which occur during its life cycle. Long (18) has shown interesting

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- (18) Long, J. S. Official Digest, Federation Paint and Varnish Production Clubs, 255, 150, 1946.

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possibilities in the study of absorption of light by clear films, particularly in the ultraviolet range, as a measure of decomposition reactions and therefore a measure of durability. It was planned to examine both normally dried films and films which had been swollen with water after they were thoroughly dried. In this way the influence of water as a factor in influencing ultraviolet absorption, and consequently reaction and decomposition, would be measured.

Further studies of pigments, extenders, particle size distribution, surface areas, binder requirements, critical pigment volume concentrations, wetting, dispersion, composition and the chemistry of binders, distensibility, moisture permeability and many other factors are indicated. The expansion in research and testing will continue, and the responsibilities and the contribution of the test chemist and testing engineer will increase accordingly.

What is true of house paints is equally true of all types of protective and decorative coatings. The improvement in quality of finishes is always paralleled by a companion improvement in methods of evaluation and testing. There is no question but that the progress made during the past twenty five years will be dwarfed by the progress which will be made in the next quarter century.

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