New Accelerated Weathering Tests Including Acid Rain

Magnus Palm and Bo Carlsson—SP Swedish National Testing and Research Institute*

A procedure for the design of reliable accelerated weathering tests for service life prediction of automotive coatings was developed and used in designing an accelerated test based on SAE method J 1960. The influence of exposure conditions was investigated in several accelerated tests. Results from these tests and from Florida exposures were used to optimize test conditions. A test cycle that includes acid rain spraying was developed and evaluated by exposing 16 different coating systems using various methods. The results indicate that the new cycle is more reliable than a cycle commonly used today.

INTRODUCTION

When introducing new coating systems it is important to predict long-term performance of the coating in a fast and reliable way. Accelerated weathering tests carried out in the laboratory are often used for this purpose. However, accelerated weathering tests are so far considered to be unreliable and often fail to reproduce the degradation behavior of coatings exposed in the field.

To improve the reliability of accelerated test procedures for automotive coatings, a research program was initiated. One major objective was to find accelerated test methods that simulate coating degradation that occurs during outdoor exposure as closely as possible. Another overall objective was to develop methodologies for service life prediction of automotive coatings. Hence, the relationship between climatic parameters (irradiance, temperature, humidity, acid rain) and coating degradation has been investigated.

METHODOLOGY FOR TEST DESIGN

The procedure for the design of a test program is based on a general methodology for accelerated life testing.[1] Two commonly used basecoat/clearcoat systems were chosen for this work. One clearcoat was based on an acrylic melamine binder and the other on an acrylic urethane binder. For test purposes formulations were made both with and without light stabilizers. The first step was to define a critical performance criterion. Since visual appearance is the most important property from a consumer point of view, gloss was chosen as a performance criterion. The penalty level was set to a maximum of 25% loss of gloss after two years' exposure in Miami, Florida. In the next step, several accelerated tests were carried out using different levels of irradiance, temperature, and acid rain. To be able to compare quantitatively the loss of gloss in the accelerated tests with Florida exposure, an acceleration factor, the equivalent light dose (ELD) factor, was introduced in earlier work, as defined in equation (1).[2] If the ELD factor is lower than 1 some parameter is not simulated correctly. Also, the higher the ELD factor, the shorter the exposure time required during accelerated testing. In order to obtain useful results from the accelerated tests, it is important that the ELD factors are as similar as possible for different coating systems.[2]

To identify degradation mechanisms that cause loss of gloss, aged coatings can be analyzed with infrared spectroscopy. It has been shown that photoinduced breakage of the urethane crosslink in an acrylic urethane clearcoat is the main cause of loss of material from the coating, and that acid catalyzed hydrolysis which results in breakage of the ether crosslink is most likely to cause loss of material for an acrylic melamine clearcoat.[2] These reactions have also been described in detail elsewhere.[3-5] Furthermore, in an earlier work within the research program, it was found that the gloss loss of a polyurethane coating correlated well with changes in the FTIR spectra.[6]

$$ELD\ factor = \frac{average\ light\ dose\ in\ Florida\ at\ 75\%\ gloss\ retention}{light\ dose\ in\ accelerated\ cycle\ at\ 75\%\ gloss\ retention} \quad (1)$$

*P.O. Box 857, SE-50115 Borås, Sweden.

Table 1—Coatings Used for Test Design

Coating No.	Description of Coating
D1	Polyurethane (acrylic) clearcoat on silver metallic basecoat
D2	Melamine (acrylic) clearcoat on silver metallic basecoat
D1U	Polyurethane clearcoat on silver metallic basecoat without UVA and HALS[a]
D2U	Melamine (acrylic) clearcoat on silver metallic basecoat without UVA and HALS

(a) UVA = UV light absorbers, HALS = Hindered Amine Light Stabilizer.

Earlier work indicated that accelerated testing according to SAE J 1960, including manual spraying with an acidic solution, resulted in better agreement for the reduction of gloss between Florida exposures and accelerated tests than for SAE J 1960 without acid rain.[2] Since the infrared analysis also indicated that acid rain was an important parameter it was decided in this work to optimize the accelerated test cycle with respect to the load of acid rain spray, as well as to temperature and irradiance.

EXPERIMENTAL

Coatings

All coatings used were based on commercial formulations, which are commonly used systems in the automotive industry. All topcoats were applied on steel panels, which were pretreated with an electrocoat and primer. For test design two different clearcoats were used, an acrylic melamine and an acrylic urethane coating. The two clearcoats were formulated and tested both with and without UV light absorbers (UVA) and hindered amine light stabilizers (HALS), see *Table* 1. They were applied on a silver metallic basecoat.

Table 2—Coatings Used for Evaluation and Further Studies

Coating No.	Description of Coating
E1	1K acrylic melamine clearcoat on red basecoat
E2	2K polyester—polyurethane clearcoat on red basecoat
E3	Powder clearcoat on red basecoat
E4	2K polyurethane clearcoat with high scratch resistance on red basecoat
E5	2K waterborne polyurethane clearcoat on red basecoat
E6	2K polyurethane clearcoat, low bake, for repairs, on red basecoat
E7	2K polyurethane clearcoat, low bake, for bumpers, etc., on red basecoat. Applied on plastic panels.
E8	Same as E7 but applied on conventional steel panels.
E9	2K polyurethane clearcoat for door handles, on red basecoat. Applied on conventional steel panels.
E10	1K polyurethane clearcoat on red basecoat.
E11	Powder clearcoat on red basecoat
E12	1K solid alkyd melamine red coating
E13	2K solid polyurethane coating, dark red, low bake meant for plastic
E14	Powder slurry clearcoat on red basecoat
E15	Solid powder coating, white
E16	1K solid alkyd melamine coating, red

For evaluation of the optimized test method, and for further studies of the effects of acid rain spraying, 16 different coating systems were used, as listed in *Table* 2. Note the large variation in coating chemistry, ranging from basecoat/clearcoat systems for high-class cars to solid coatings for trucks, and also including a variety of new environmental coatings, e.g., powder coatings and waterborne polyurethane clearcoats.

Exposure Conditions

Sets of panels were exposed to direct weathering in a test site near Miami, FL, at 45°, 26°, and 5° according to the ASTM E782 Variable Angle Schedule. Panels were exposed for 24 months, starting in November 1991. For evaluation of the optimal test, panels were also exposed in Jacksonville, FL, and also according to the so-called Equatorial Mount with Mirrors for Acceleration plus Aqua (EMMAQUA®) test, where mirrors are used to intensify solar radiation onto the test object.[7] In the Jacksonville exposure a fixed angle of 5° was used instead of the Variable Angle Schedule. The Miami and Jacksonville exposures started in November 1998.

The test panels were exposed to different accelerated test programs, called CAM1, CAM2 and CAM3, all being based on the SAE J 1960 method.[8] The test programs include alternating dark and light, wet and dry periods, and have been described in detail elsewhere.[2] Data for modeling and optimization was obtained by varying irradiance, black standard temperature, and frequency of acid rain spraying.

The black standard temperature and irradiation level were varied according to *Table* 3.

Simulation of acid rain was achieved by 20 min front spraying in the designated test cycle using a solution consisting of sulphuric acid, nitric acid, and hydrochloric acid, 1:0.3:0.17 by weight, with pH = 3.2 in the Weather-ometer®. Schulz and Trubiroha used a solution of this composition in one study.[9] The frequency of acid rain spraying was defined as the inversion of the number of cycles between each spraying with acid rain, i.e., if the acid rain is sprayed once every fourth cycle the frequency is f = 0.25.

The accelerated test cycle ISO 4892-2 was used as reference to evaluate the new test cycle, since this method is the most commonly used cycle for accelerated weathering in Scandinavia. Exposure conditions included a 65°C black standard temperature, Xenon arc light-source with irradiance 0.50 W/m^2 at 340 nm and 102 min dry period (relative humidity 50%) followed by 18 min spraying with deionized water.

Accelerated Test Equipment

The equipment used for the accelerated tests with acid rain simulation was a modified Atlas XR35 Weather-ometer®, with a Xenon arc light source with inner and outer borosilicate filters. The original equipment was immensely modified in order to obtain fully automatic simulation of acid rain and better control of relative humidity.

Improved relative humidity control was accomplished by modifying both the hardware (fan, damper for moisture evacuation, etc.) as well as the controller software. The hardware for simulation of acid rain was obtained by constructing a system of pumps, valves, switches, containers for acid solution, etc. The measurement of acid rain spraying was performed by an in-line conductivity measurement. Deionized water with a conductivity of 1-2 μS/cm was used for spraying and for preparing the acid solution. No corrosion in the Weather-ometer was observed.

Chemical and Physical Analysis

Specular gloss was measured at 20° angle of incidence before and after exposure and reported as gloss retention, i.e., percentage of the original gloss. All panels were washed with water before measurements. The instrument used was a BYK-Gardner Micro-Tri-Gloss gloss meter. Color was measured using an instrument from Datacolor International, and recorded as Δl, Δa, and Δb, but for investigation of test performance only the ΔE value was used $(\Delta E = \sqrt{(\Delta a^2 + \Delta b^2 + \Delta l^2)})$. Scanning Electron Microscopy (SEM) was used to characterize damage caused by acid etch, using a J EOL JSM-5800.

MODELING AND OPTIMIZATION

The equivalent light dose factors (ELD factors), should exceed 1 for a good simulation and acceleration of conditions in Florida.[2] Furthermore, the difference (e.g., the standard deviation) among the ELD factors of the different coating systems should be as small as possible in order to obtain useful results from accelerated weathering tests. One objective with this study was to find the optimal conditions, i.e., the light intensity, temperature, and frequency of acid rain spraying that give the lowest difference between the ELD factors of the four different coating systems.

In order to optimize test conditions, the ELD factors have to be modeled. The models must not necessarily be derived from chemical and physical laws of nature, but the temperature dependence of coating degradation is expected to follow an Arrhenius expression. Assuming a constant rate of gloss loss, this rate, ΔG/Δt, could be described by the following general model:

$$\Delta G/_{\Delta t} = (a + b\gamma)(c + dI)e^{-E/RT} \quad (2)$$

where I is the irradiance in W/m^2 at 340 nm, T is the black standard temperature (K) during light exposure, γ is some other parameter which accelerates material loss (time of wetness, acid load, etc.), E is an Arrhenius energy, and R is the gas constant. Note that a, b, c, and d are specific coating parameters that have to be determined by experiments. Since the ELD factor is the ratio of light doses in Florida and Weather-ometer, the model for ELD factors will follow the same general expression.

The modeling and optimization procedure was divided into two steps in order to minimize the number of experiments. The first step covered temperature and irradiance and, based on the general expression [equation (2)], the model becomes:

Table 3—Black Standard Temperature and Irradiation Level for the Test Programs CAM1, CAM2, and CAM 3, All Being Variations of SAE Method J 1960

Test Program	Black Standard Temperature (°C)	Irradiance (W/m^2 at 340 nm)
CAM1	70±3	0.35
CAM2	70±3	0.5
CAM3	80±3	0.5

$$ELD = \left(\alpha_1/_I + \alpha_2\right)e^{E/RT} \quad (3)$$

where α_1 and α_2 are coating parameters that have to be determined by fitting the model to data obtained from different accelerated tests and from outdoor exposures in Miami, FL. This problem corresponds to a system of nonlinear equations.

The second step investigates the frequency of acid rain spraying. A so-called Weibull function was used (often used in service life prediction analysis):

$$ELD = \beta_1 + \beta_2 e^{-(\beta_3 f)^{\beta_4}} \quad (4)$$

where *f* is the frequency of acid rain spraying and β_1, β_2, β_3, and β_4 are coating parameters obtained by fitting the model to experimental data. The advantage with such a function is that it is very flexible. Using these models, test conditions can be optimized. First, solving a system of linear equations assessed the coating parameters. This was carried out with the aid of the software Matlab. Optimal test conditions were then calculated by minimizing the difference in ELD factors between the different coatings using a mathematical approach including the least square method. Various software was used for model fitting and optimization, e.g., Matlab and Sigmaplot.

RESULTS AND DISCUSSION

Varying the Frequency of Acid Rain

The spraying with acid rain proved to have a considerable effect on gloss retention. The rate of gloss loss at different acid rain spraying frequencies for three coating systems is shown in *Figure* 1. The graphs have a threshold-like appearance as shown in the figure, although the threshold is not that pronounced for the acrylic/urethane coating. The "threshold" appears at a lower frequency for the acrylic/melamine clearcoats than for the acrylic/urethane clearcoats, indicating that the melamine binder is more susceptible to degradation by hydrolysis than the urethane binder; acid-catalyzed hydrolysis has been confirmed by others.[3-4] Results from the acrylic/urethane coating without UVA and HALS are not included in the figure since that coating degrades and cracks very rapidly.

Modeling and Optimization

The optimization of the temperature and light intensity gave conditions similar to the CAM2 cycle, which includes an irradiance of 0.5W/m^2 and a black standard temperature of 70°C. It should be pointed out that the

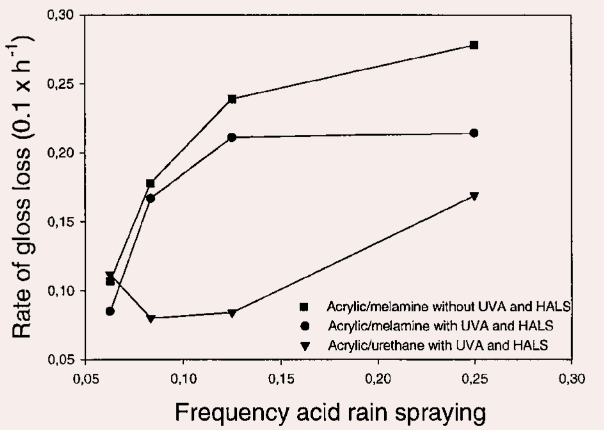

Figure 1—Rate of gloss loss at different frequencies with acid rain spraying. UVA = ultraviolet light absorber, HALS = hindered amine light stabilizer.

process of getting reliable models for the ELD factors is not straightforward since the difference in coating chemistry and behavior was large and the number of data was small. The Weibull function worked very well in modeling the effect of acid rain spraying, especially for the acrylic/melamine coatings. The model fit for the acrylic/melamine coating is shown in *Figure* 2.

The optimization gave a clear minimum at f = 0.07, which means acid rain spraying every 14th cycle.

The Test Cycle

The result of the test design was an accelerated weathering test program including acid rain spraying:

SPART 14 (SP Acid Rain Test, f_{acid}=14)

Irradiation level:	0.5 W/m^2 at 340 nm
Climate cycle:	40 min light only, 20 min light + water spraying on the front side of the panels, with or without acid solution, pH = 3.2. 60 min light, 60 min dark + spraying on the back side of the panels
Climate, light period:	black standard temperature: 70°C, dry temperature: 47°C, relative humidity: 75%
Climate, dark period:	dry temperature: 38°C, relative humidity: 95%
Acid rain:	spraying every 14th cycle, pH = 3.2, during the 20 min spraying

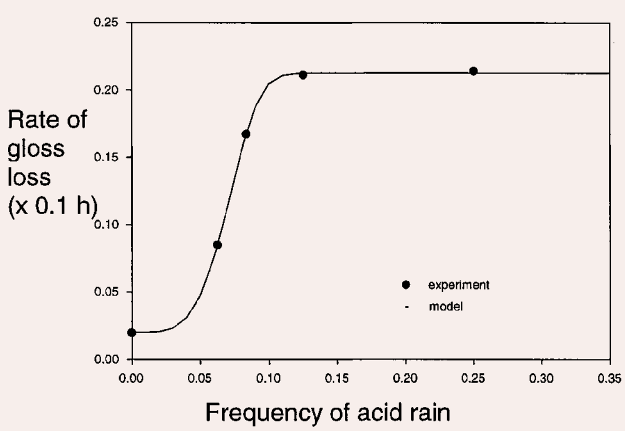

Figure 2—Rate of gloss loss as a function of acid rain spraying: model versus experiment.

The acid rain should be sprayed automatically and the pH monitored on-line to achieve reliable test conditions.

Evaluation and Effects of Acid Rain

As shown in *Tables* 1 and 2, the coatings for evaluation were different from the ones used in the optimization work. The coatings were exposed in Florida for one year. All coatings were exposed according to the two accelerated cycles SPART 14 and ISO 4892-2 for 5000 hr. The two cycles were compared using a procedure to predict gloss retention after one year of outdoor exposure:

(a) The required light dose to obtain 75% gloss retention was calculated by linear regression for the coatings systems E1—E16, for SPART 14 and ISO 4892-2, as well as for outdoor exposures in Florida. For coatings that lost gloss rapidly the last data was excluded in the regression since gloss retention flattens out at values lower than 20%, and since values lower than about 40% are too low to be of any (industrial) interest.

(b) ELD factors for all coatings and for both test methods were calculated using the results from (a).

(c) Predicted gloss retention after 12 months of Florida exposure was calculated for all coating systems using the median value of the ELD factors from each cycle resulting from accelerated weathering.

Predicted versus actual values from 12 months' exposure at the Miami site are shown in *Tables* 4 and 5. The penalty level was 75% gloss retention after two years of Florida exposure, which means that the gloss retention

Table 4—Predicted Gloss Retention (G_{Pred}) According to Results from SPART 14 vs Actual Gloss Retention (G_A) from 12 Months' Exposure in Miami, FL, and the Difference Between the Two, for Coating Systems E1—E16.

Coating System	G_{Pred}	G_A	G_{Pred}-G_A
E1	90	98	-8
E2	97	98	-1
E3	97	95	2
E4	97	97	0
E5	97	96	1
E6	97	98	-1
E7	95	92	3
E8	96	95	1
E9	97	96	1
E10	94	95	-1
E11	96	94	2
E12	67	57	10
E13	79	8	71
E14	97	98	-1
E15	94	97	-3
E16	75	67	8

Table 5—Predicted Gloss Retention (G_{Pred}) According to Results from ISO, 4892-2 vs Actual Gloss Retention (G_A) from 12 Months' Exposure in Miami, FL, and the Difference Between the Two, for Coating Systems E1—E16

Coating System	G_{Pred}	G_A	G_{Pred}-G_A
E1	98	98	0
E2	97	98	-1
E3	98	95	3
E4	97	97	0
E5	96	96	0
E6	97	98	-1
E7	98	92	6
E8	96	95	1
E9	97	96	1
E10	97	95	2
E11	97	94	3
E12	72	57	15
E13	90	8	82
E14	97	98	-1
E15	85	97	-12
E16	85	67	18

Table 7—Predicted Gloss Retention (G_{Pred}) According to Results from ISO, 4892-2 vs Actual Gloss Retention (G_A) from 12 Months' Exposure in Jacksonville, FL, and the Difference Between the Two, for Coating Systems E1—E16

Coating System	G_{Pred}	G_A	G_{Pred}-G_A
E1	96	93	3
E2	93	95	-2
E3	95	91	4
E4	94	91	3
E5	91	92	-1
E6	94	93	1
E7	96	93	3
E8	92	94	-2
E9	93	93	0
E10	93	89	4
E11	94	92	2
E12	43	59	-16
E13	79	39	40
E14	94	92	2
E15	68	96	-28
E16	68	70	-2

should exceed 87.5% after one year, assuming linear gloss loss. As shown in the tables, three coatings, E12, E13 and E16, do not fulfil the criterion. Predicted values, calculated from results from the SPART 14 exposure, comply with actual data according to the criterion, i.e., the accelerated test approves of all coatings except E12, E13, and E16, see *Table* 4. The ISO 4892-2 test, however, fails to give a correct judgement of coating E13 and E15 according to the same criteria (*Table* 5). Using the same pass/fail criterion for the Jacksonville exposure, the two accelerated tests succeed with all coatings except one, E1 for SPART 14 and E15 for ISO 4892-2, see *Tables* 6 and 7. Note, however, that the penalty level (75% gloss retention after two years) is set for Miami exposures; the pollution levels are higher at the Jacksonville site.

To get a numerical value of the performance of the cycle the parameter ∇ was calculated according to equation (5):

$$\nabla = \sqrt{\sum_i (G_{i,real} - G_{i,pred})^2} \tag{5}$$

where $G_{i,\,real}$ is the actual gloss retention after one year of outdoor exposure for coating *i*, and $G_{i,\,pred}$ is the predicted gloss retention for coating *i*, calculated from results from accelerated exposures. The lower the value of ∇, the better the prediction. In this analysis the coating E13 was excluded since it would totally dominate the sum for Miami exposures and, thus, give a misleading result. The parameters ∇ for the two different cycles are shown in *Table* 8, coating system E13 excluded. As shown in this table, the parameter ∇ is essentially lower for the SPART 14 cycle, for both exposure sites, suggesting that the SPART 14 cycle predicts coating weathering better than the ISO 4892-2 cycle. The ELD factors used were median values: 4.0 for the SPART 14 cycle and 2.5 for the ISO 4892-2 cycle. In terms of time, ELD = 4.0 for the SPART 14 cycle means that one year of Miami exposure corresponds to approximately 750 hr of accelerated exposure. As for the ISO 4892-2 cycle, one year of Miami exposure corresponds to approximately 800 hr of accelerated exposure.

For color changes, an ELD factor can be defined in the same way as for gloss and an arbitrary penalty level (in

Table 6—Predicted Gloss Retention (G_{Pred}) According to Results from SPART 14 vs Actual Gloss Retention (G_A) from 12 Months' Exposure in Jacksonville, FL, and the Difference Between the Two, for Coating Systems E1—E16

Coating System	G_{Pred}	G_A	G_{Pred}-G_A
E1	85	93	-8
E2	95	95	0
E3	95	91	4
E4	95	91	4
E5	95	92	3
E6	96	93	3
E7	92	93	-1
E8	93	94	-1
E9	96	93	3
E10	90	89	1
E11	94	92	2
E12	47	59	-12
E13	67	39	28
E14	95	92	3
E15	90	96	-6
E16	60	70	-10

Table 8—Parameter ∇ from Gloss Prediction, Coating System E13 Excluded, for the Cycles SPART 14 and ISO 4892-2 vs Outdoor Exposures

Test Cycle	∇
SPART 14—Miami	16
ISO 4892-2—Miami	27
SPART 14—Jacksonville	20
ISO 4892-2—Jacksonville	33

Table 9—Parameter ∇ for Color Change Prediction for the Accelerated Methods SPART 14, ISO 4892-2 and EMMAQUA vs Outdoor Exposures in Miami, FL

Test Cycle	∇
SPART 14—Miami	1.96
ISO 4892-2—Miami	3.17
EMMAQUA—Miami	3.14

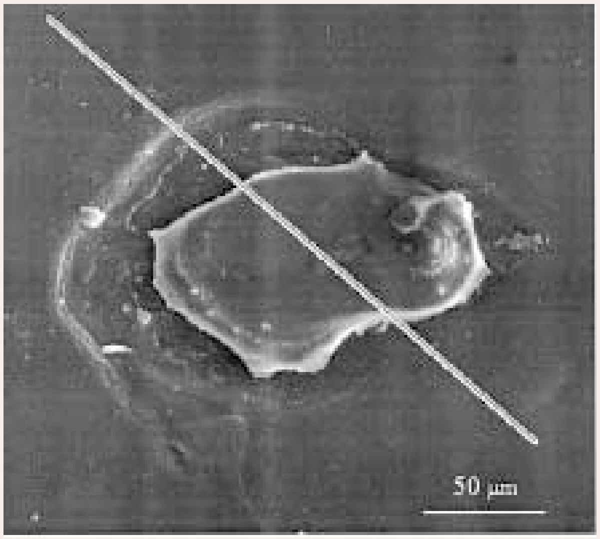

Figure 3—An SEM micrograph showing an acid etch damage in coating E8 after 5000 hr of exposure according to SPART 14. The line shows where a depth profile has been recorded.

this case ΔE=2) set. Using the same procedure as for gloss, it is then possible to predict color changes. The results for the Miami exposures are shown in *Table* 9, where the EMMAQUA accelerated "mirror" test is also included. These results indicate that the SPART 14 test is more reliable than the ISO 4892-2 cycle, just as in the case for gloss. For color changes, mechanisms other than loss of material are involved, which means that the SPART 14 test conditions give rise to degradation mechanisms closer to outdoor conditions in Florida than the ISO 4892-2 cycle. In terms of time acceleration for color change (ΔE), one year of Miami exposure corresponds to approximately 2000 hr of accelerated exposure. As for the ISO 4892-2 cycle, one year of Miami exposure corresponds to approximately 1050 hr of accelerated exposure.

The SPART 14 cycle resulted in acid etch damage on many clearcoats. The acid etch pits were studied with SEM. The etch pits were around 10 μm deep and up to 250 μm in diameter. An SEM micrograph is shown in *Figure* 3 and a depth profile in *Figure* 4. The depth profile shows that there is material in the middle of the pit that rises above the surface of the coating, indicating that this material adheres loosely to the underlying material, the top of a microscopic blister. The pits cover a small fraction of the surface, which means that the contribution to gloss reduction is negligible.

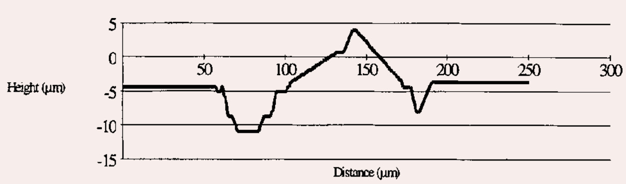

Figure 4—Depth profile along the line in Figure 3.

CONCLUSIONS

A new procedure to develop more reliable accelerated weathering tests was presented. A mathematical approach was adopted for optimization of test conditions, where variations in the ELD acceleration factors were minimized. Simulation of acid rain was introduced to obtain better correlation between accelerated test and Florida exposures. The frequency of acid rain spraying had a considerable influence on the rate of gloss loss. Modeling of acceleration factors and optimization of test conditions resulted in a test program based on SAE J 1960 with the addition of acid rain spraying every 14th cycle, a so-called SPART 14 test cycle.

A number of coatings were exposed to SPART 14, ISO 4892-2, and to several outdoor sites. The results indicate that the SPART 14 cycle correlates better with outdoor exposures than does the ISO 4892-2 cycle, both for gloss reduction and color changes. The SPART 14 cycle gave rise to acid etch pits on many clearcoats, but that has a negligible effect on gloss.

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