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Fourier-Transform Infrared (FTIR) Characterization of Chemical Agent Resistant Coatings (CARC) Durability Using Infrared (IR) Cards and Silicon Wafers

by William Lum
and Philip Patterson

ARL-TR-1494

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Fourier-Transform Infrared (FTIR) Characterization of Chemical Agent Resistant Coatings (CARC) Durability Using Infrared (IR) Cards and Silicon Wafers

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Weapons and Materials Research Directorate, ARL

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Abstract

Rapid, cost-effective sampling accessories for Fourier-Transform Infrared (FTIR) analysis are quite suitable with minimum sample preparation in coating materials studies. The U.S. Army's camouflage, polyurethane topcoat MIL-C-46168, "Chemical Agent Resistant Coating" (CARC) was applied to infrared (IR) cards and silicon wafers. After curing, the coating's durability was tested using two separate accelerated weathering techniques (QUV-CON and Xenon-Arc), and as well as, two outdoor exposure test methods (South Florida and EMMA(QUA)*-NTW). Characterization of the binder's chemical changes during both natural and accelerated weathering processes was monitored using the FTIR. This final report summarizes the experimental results of the coating's resistance to the accelerated-weathering and outdoor-exposure conditions. Additionally, a brief description of the author's method is described for the preparation of silicon wafers and subsequent FTIR analysis.

*Trade name.

TABLE OF CONTENTS

	<u>Page</u>
LIST OF FIGURES	v
1. BACKGROUND	1
2. APPROACH	2
3. EXPERIMENTAL	4
4. RESULTS	6
5. CONCLUSIONS	11
6. PLANS	11
APPENDIX A: EMMA(QUA) LOG FILE	13
APPENDIX B: XENON-ARC LOG FILE	17
APPENDIX C: QUV-CON LOG FILE	23
APPENDIX D: FORT BELVOIR LOG FILE	27
APPENDIX E: SOUTH FLORIDA EXPOSURE	31
DISTRIBUTION LIST	35
REPORT DOCUMENTATION PAGE	39

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LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1. Coated IR cards with metal apertures after 3-mo exposure at Fort Belvoir site . . .	8
2. Coated aluminum holders with silicon wafers after 800 hr of xenon-arc exposure	8
3. Coated IR cards after 600 hr of QUV-CON exposure and a 3-M type 62 background	9
4. Coated aluminum holders with silicon wafers after 300 MJ/m ² EMMA(QUA) exposure	9
5. "Spreadsheet" IR spectra of resins after 0, 400, 800 hr xenon-arc exposure	10
A-1. Before EMMA(QUA) exposure	15
A-2. After 150 MJ/m ² of EMMA(QUA) exposure	15
A-3. After 300 MJ/m ² of EMMA(QUA) exposure	16
B-1. Before xenon-arc exposure	19
B-2. After 200 hr of xenon-arc exposure	19
B-3. After 400 hr of xenon-arc exposure	20
B-4. After 600 hr of xenon-arc exposure	20
B-5. After 800 hr of xenon-arc exposure	21
C-1. Before QUV-CON exposure	25
C-2. After 200 hr of QUV-CON exposure	25
C-3. After 400 hr of QUV-CON exposure	26
D-1. Before Belvoir-site exposure	29
D-2. After 1 mo of Belvoir-site exposure	29

<u>Figure</u>	<u>Page</u>
D-3. After 3 mo of Belvoir-site exposure	30
E-1. Before South Florida exposure	33
E-2. After 6 mo of South Florida exposure	33

1. BACKGROUND

This comprehensive paper will cover three main objectives, as well as, initial and final results of the 2-yr investigation. The first aim is to utilize and explore capabilities of advanced instrumentation and sampling accessories available in the commercial market. The second purpose is to attempt a better understanding and relationship between accelerated-weathering and outdoor-exposure testing. The material is the U.S. Army's Chemical Agent Resistance Coatings (CARC) polyurethane topcoat, comparing test results to its weathering durability. Finally, the third intention is to establish a rapid, easy-to-do, cost-effective technique to make quick determinations of the coating's infrared (IR) characteristics and its chemical changes during the weatherability testing.

The 2-yr Department of the Army (DA) work package (#3261) project utilized the technique of Fourier transform IR (FTIR) spectroscopy with different sampling accessories. The search for better tools for rapid analysis has been on-going as demands increase for test information of newly formulated coating materials. This unique opportunity has been addressed entirely by determining the most efficient technique. It was identified and selected through testing and evolution of several coating materials.

The Army's basic camouflage CARC topcoat is the two-component military specification MIL-C-46168D polyurethane. The CARC system is required on all Army combat, combat-support, and ground-support equipment, and tactical aircraft. Because of its significant importance, this coating was used in our study. The applied coating on different sampling accessories consisted of a clear, polyester polyurethane. The resins, Reichhold Chemicals/ Sherwin-Williams polyester polyol and Bayer Desmodur 851 aliphatic polyisocyanate,* were obtained from Sherwin-Williams Co.

* Reichhold Chemicals, polyester polyol and Bayer Industrial Chemicals Desmodur 851 aliphatic polyisocyanate, Sherwin-Williams Co., Chicago Il.

2. APPROACH

The project began by reviewing application literature on FTIR accessories and sampling methods. Technical training on weathering techniques and IR interpretation was obtained, followed by procurement of supplies, accessories, and related equipment.

The FTIR spectrometer* was used to investigate the various sampling techniques. The accessories and techniques identified and selected for this study were as follows:

- (1) magnetic sample holders with electronic-grade silicon wafers;[†]
- (2) 3-M disposable IR cards, both polyethylene (PE) and polytetrafluoroethylene (PTFE) microporous film substrates;^{**}
- (3) polyvinyl chloride ECRAN Screen Cell card;^{††}
- (4) vertically-oriented attenuated total reflectance (ATR) spectroscopy and;
- (5) free films via transmittance.

Preliminary coating samples were run on a trial basis using different spectroscopic techniques and accessories. A spectrum of each sample, before and after outdoor-exposure and accelerated-weathering techniques, was scanned and saved in the Nicolet's Omnic FTIR software spectrum file or the .SPA extension as a standard saving procedure. Omnic was employed to acquire background and sample spectra, peak positions, and tables. Initially, version 1.1 was installed in the computer. However, an updated 2.0 version became available and was implemented for use in all parts of this project.

A new background was used for each sample. Twenty-four actual scans of the sample were time-averaged with the Fourier transform technique, and the final spectrum was used with

* Model 5DX-B, Nicolet Instrument Corp., Madison, WI.

† Harrick Scientific Corp., Ossining, NY.

** 3-M New Products Development, Inc., St. Paul, MN.

†† Janos Technology, Inc., Townshend, VT.

enhancements for comparison with other spectra. Transmittance was converted into absorbance in order to do a baseline correction. The appearances of the spectra were adjusted using baseline corrections and data-smoothing enhancement.

Thallium bromo iodide (KRS-5) crystal was used with a vertical ATR.* The spectral range of the crystal is from 2200 cm^{-1} to 250 cm^{-1} , which allows an extended view of the lower region of the spectrum. The KRS-5 crystal is relatively insoluble and reasonably durable and will produce the best spectra for the widest variety of samples. The resolution was set at 4 cm^{-1} . Spectra were measured in the range of $4,000\text{ cm}^{-1}$ to 400 cm^{-1} primarily.

All the initial findings are covered in a U.S. Army Research Laboratory (ARL)[†] progress report. This interim publication mentioned the selection and elimination of most accessories and techniques and then focused on two accessories and techniques.

In the second phase, as was in the conclusive phase of the first year, specific IR absorption bands were monitored to follow the chemical degradation process. The traditional outdoor Florida exposure** plus EMMA(QUA)^{††} accelerated outdoor Arizona testing and accelerated-weathering techniques and devices*** were used for all samples. Techniques were fine-tuned as problems arose from the trial runs. Changes were made to optimize the FTIR instrument and sample film preparation for the best reproducibility.

The American Society for Testing and Material (ASTM) method designation G 53 was selected in using the QUV-CON device with UVA-340 fluorescent sunlamps. This method is also cited in our coatings specifications, which require 8 hr of ultraviolet (UV) light exposure and 4 hr in a condensation environment in a 12-hr cycle. Temperature settings during UV cycle were

* Spectra-Tech, Inc., Shelton, CT.

† Lum, W., and P. Patterson. ARL-MR-319, U.S. Army Research Laboratory, Aberdeen Proving Ground (APG), Aberdeen, MD, July 1996.

** South Florida Test Division (SFTD), Atlas Electric Devices Co., Miami, FL.

†† DSET Laboratories Division, Atlas Electric Devices Co., Phoenix, AZ.

*** QUV-CON UV/condensation and xenon-arc weather-ometer, Atlas electric Devices Co., Chicago, IL.

at 70° C and condensation cycle at 50° C. The specified practice in using the Weather-Ometer device refers to the ASTM method designation G 26, which describes operating conditions of 102 min of UV light and 18 min of both UV light and deionized water spray and 0.35 W/m² of irradiance. The conditions are also used in CARC coatings testing program. The EMMA(QUA) testing followed the ASTM-D-4141 procedure, in which we also included night time wetting (NTW). NTW is a 3-min water spray done five times during the night to simulate rainfall and temperature-cooling effect and, therefore, to accelerate degradation.

All test samples were sprayed to a dry film thickness (DFT) of 0.3–0.4 mils of clear resins and air-dried for at least 1 wk. A tight requirement of extremely thin films was required in order to get sufficient signal throughputs and reasonable spectra. The main concern was the loss of adhesion after a period of any exposures. In fact, this had happened on one EMMA(QUA) sample after 300 MJ/m² of UV radiance.

The final objective was to determine the most efficient technique for evaluating coating materials based on their weathering properties. Evaluation encompassed quick IR characterization, tracking of chemical changes, and rapid service life determination. This technical report concludes the entire investigation by encompassing all the results of the analysis.

3. EXPERIMENTAL

Coated and uncoated silicon wafer magnetic holders, screen cells, and disposable IR cards underwent both natural and accelerated weathering. The applied coating consisted of a clear, two-component polyester polyurethane, typical of a CARC coating. The resins used were Desmophen 650A-65 polyester polyol and Desmodur N-75 polyisocyanate, obtained from Bayer Corp.* The resins also used were Reichhold/Sherwin-Williams polyester polyol and Bayer Desmodur 851U polyisocyanate.

* Desmophen 650A-65 polyester polyol and Desmodur N-75 polyisocyanate, Bayer Corp., Industrial Chemicals Division, Coatings, Pittsburgh, PA.

The natural-weathering phase was performed at the Fort Belvoir, VA site with the racks facing in a southerly direction at a 45° angle. Open backing holders were used for direct-weathering (DWI) exposure. This procedure follows the requirements as specified by the ARL CARC military specifications.

A concept that was initially attempted involved the preparation and analysis of free-standing strips of cured films. The free-film concept was somewhat effective; however, it proved to be extremely sensitive to film thickness and very difficult to reproduce quality spectra. Therefore, it was dropped as a further consideration in the first year.

Thirteen-millimeter-diameter silicon wafers with lab-improvised aluminum holders were used as the only technique whenever a high temperature (above 60° C) was present as a key accelerating element of a test. The disposable IR cards as a stand-alone did not fare well under high temperature. CARC-coated IR cards did better than the uncoated ones, but still, the limitation is the microporous film construction. Magnetic holders did not withstand direct sunlight (natural or simulated), rain, and humidity. We found that the silicon wafer was the best way in tracking particular spectral bands relating to the chemical degradation process. However, this approach had a higher start-up cost than the disposable IR and screen cards. Most projects have to be within budget and schedule; therefore, cost is an important consideration.

Once the equipment was ready to run, both the silicon wafer and disposable IR card methods took only a minute when analyzing a sample. This approach allowed the authors to turn around the samples quickly and test again for another cycle of weathering exposure. It was proven to be less time-consuming, more effective, and easily done, even by a spectroscopist with a limited coatings' background.

However, both the Janos Screen Cell and 3-M IR cards are quite limited in their high temperature and moisture tolerance. They turned brown in color and warped in shape after accelerated testing. We therefore improvised our own aluminum holder design for the silicon wafer as our first-choice method.

4. RESULTS

Particular bands were tracked for each analysis. In the case of CARC coatings, one of the absorbance bands of interest is at approximately $2,265\text{ cm}^{-1}$, which signifies a presence of unreacted isocyanate. Based on the curing time, the relative intensity shows the completeness of the polyurethane reaction (polyisocyanate with polyol). This band was used by the authors to monitor the chemical change that was occurring, such as the completeness of the reaction or the cure time of the coating.

The carbonyl band is around $1,740\text{ cm}^{-1}$, signifying the presence of anhydrides, ketones, or esters used in a particular coating. Examples of usage are various polyesters, medium-oil maleic anhydride and o-phthalic anhydride alkyds, and polyketone polymers. Quick IR runs had provided a lot of spectral information about a coating, making IR characterization a proven process.

The polyurethane cross-linking band, also known as the amide II band, is around $1,540\text{ cm}^{-1}$. This band indicates the integrity of its urethane cross-links. A loss of intensity of this band is a result of urethane cross-linking breakage. This significant change was monitored to assess product performance, relating to 3-mo outdoor Belvoir-site exposure and to 24-mo outdoor Florida exposure. The 6-mo samples from South Florida Test Division (SFTD) were received and are shown in Figure E-2.

The other important bands are hydrocarbon stretch (around $2,950\text{ cm}^{-1}$), C-H deformation band (around $1,450\text{ cm}^{-1}$), and out-of-plane bending of hydrogens on an aromatic ring C-H band (around 750 cm^{-1}), along with the aromatic ring bending band (around 710 cm^{-1}).

The clear resin films were completely cured in all exposure methods by the end of the first exposure cycle. We noticed the diminished band for unreacted isocyanate between the before-exposure samples and after-exposure samples (Figures A-1–D-3, Appendices A–D).

By examining at the amide II band region, it was apparent that as exposure time increased, the urethane cross-links were breaking down. Given that fact, something could be said about the service-life performance of a coating at a drastically reduced testing time. The Reichhold/Sherwin-Williams resins were able to endure comprehensive simulation of end-use conditions. This same resin system also passed the CARC test, which is done at the U.S. Army Dugway Proving Ground, Dugway, UT.

Figures A-1–D-3 (Appendices A–D) represent visual comparisons of some spectra obtained from different exposure methods and durations. We used the log-file feature in the Omnic software to generate the spectra series, along with their text describing data collection, processing operations, and results of the operations. However, application “bugs” were found when we attempted to make changes to them after we were done with the initial layout.

Figures 1–4 illustrate appearances of samples after 3 mo of natural DWI at the Fort Belvoir site, after 800 hr of xenon-arc exposure, after 600 hr of QUV-CON exposure along with a designated background card for comparison purposes, and after 300 MJ/m² of UV radiance of EMMA(QUA) exposure. SFTD test samples were all back at the time of writing this report.

Figure 5 shows an overview of IR spectra of polyester polyol and isocyanate resins at different exposure durations (0, 400, and 800 hr) in the xenon-arc device. The “fingerprint” region between 2,000 and 500 cm⁻¹ was expanded to distinguish three bands in the same IR region. Increased loss of intensity in the amide II band at 1,540 cm⁻¹ clearly demonstrates that there was a breakdown in the film’s integrity. We saw similar occurrences in other weathering results with different exposure techniques. This information can be further tied to the nature and composition of the material. Certain blends of resins give unique characteristics that may withstand end-use conditions under a more extreme environment.

A brief description of our method is as follows. First, we cut the aluminum panels to a size that would fit in our sample compartment. We used one panel as the front piece and the other as

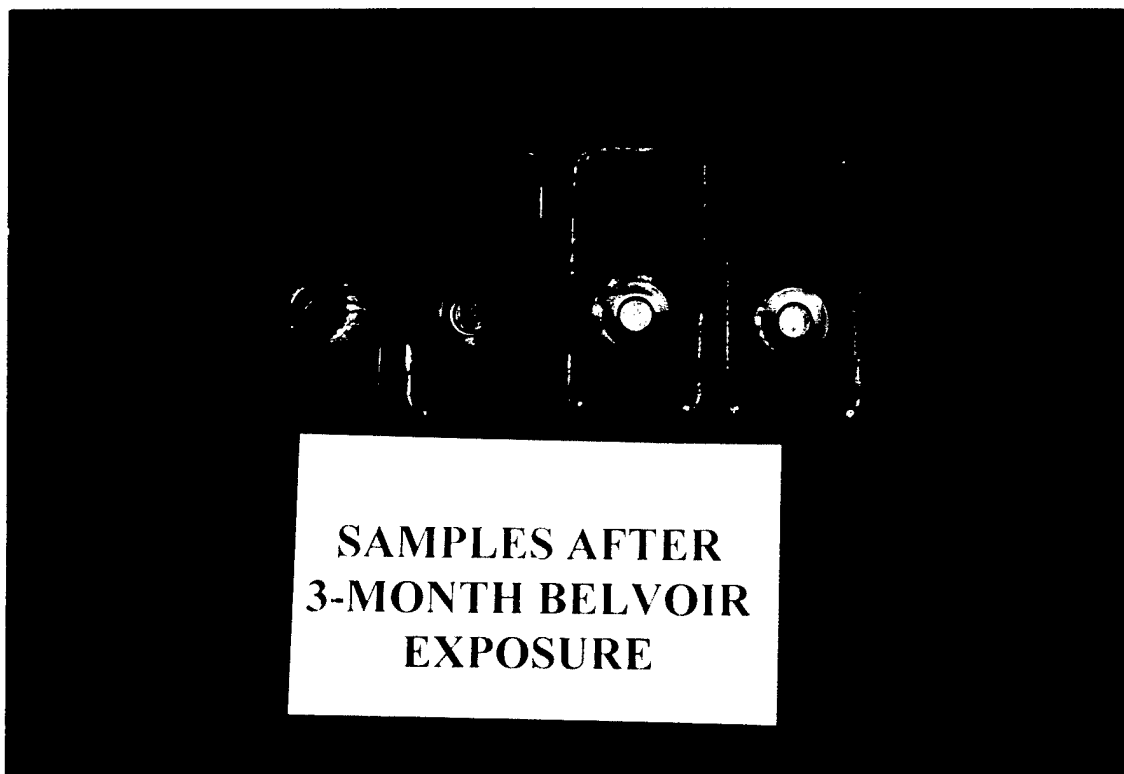


Figure 1. Coated IR cards with metal apertures after 3-mo exposure at Fort Belvoir site.

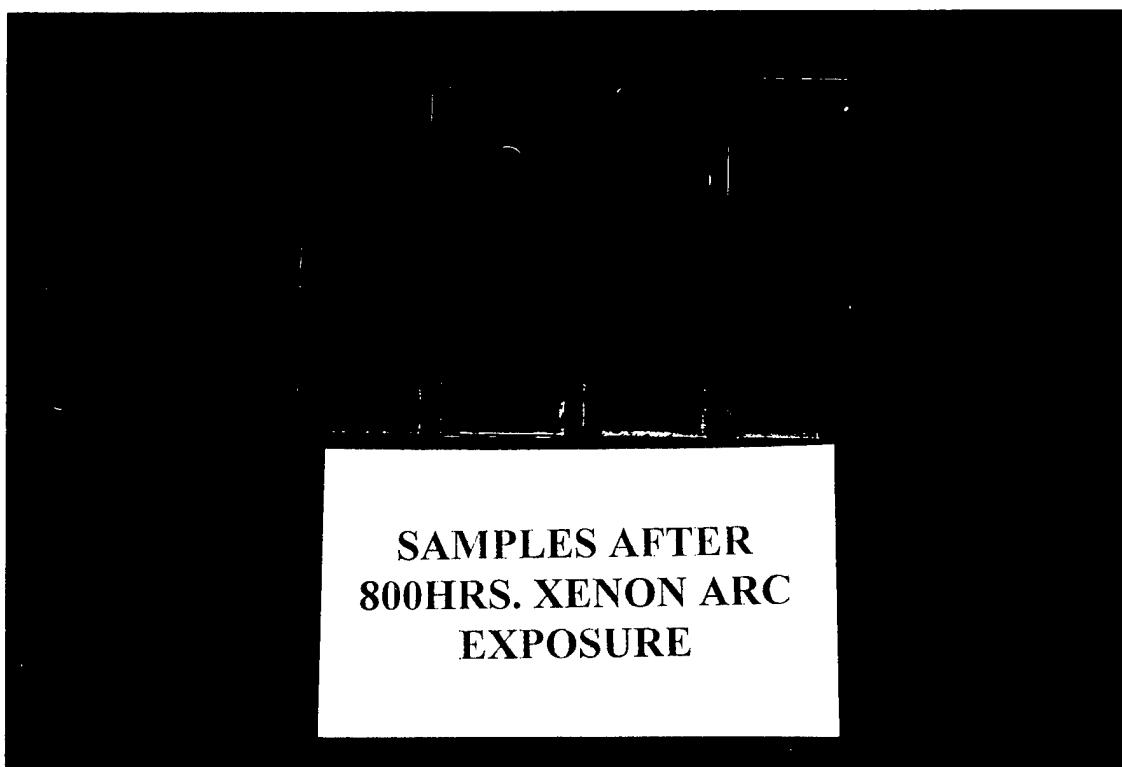


Figure 2. Coated aluminum holders with silicon wafers after 800 hr of xenon-arc exposure.

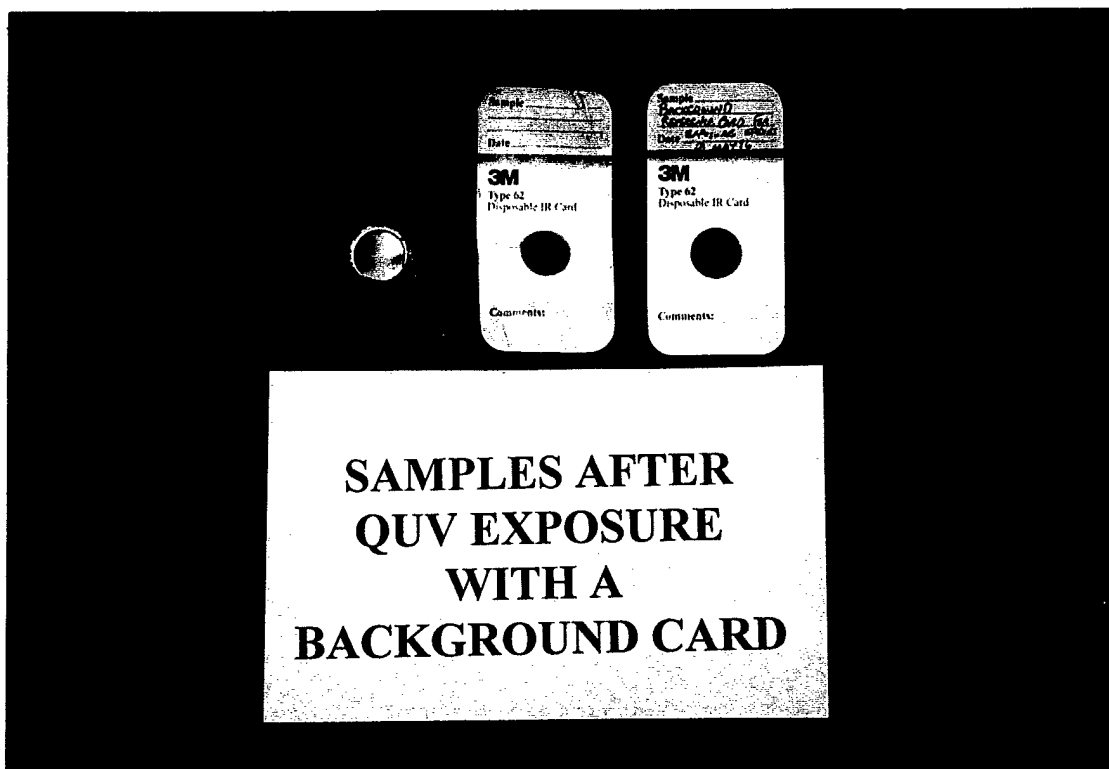


Figure 3. Coated IR cards after 600 hr of QUV-CON exposure and a 3-M type 62 background.

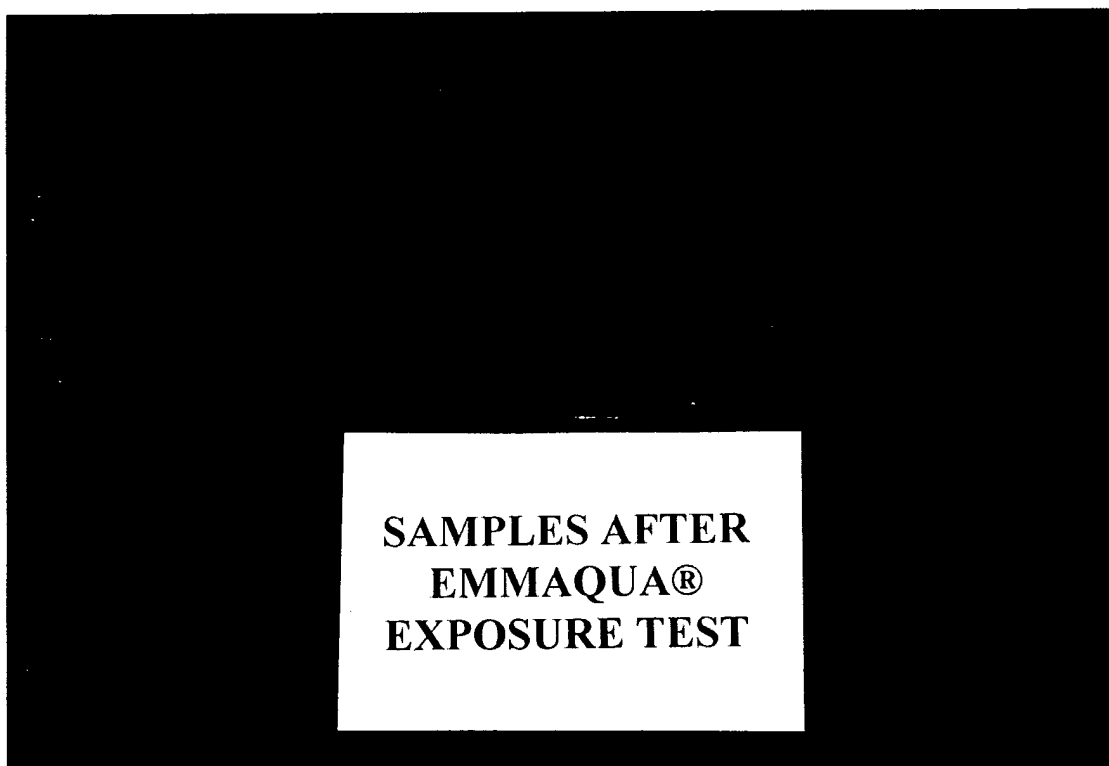


Figure 4. Coated aluminum holders with silicon wafers after 300 MJ/m² EMMA(QUA) exposure.

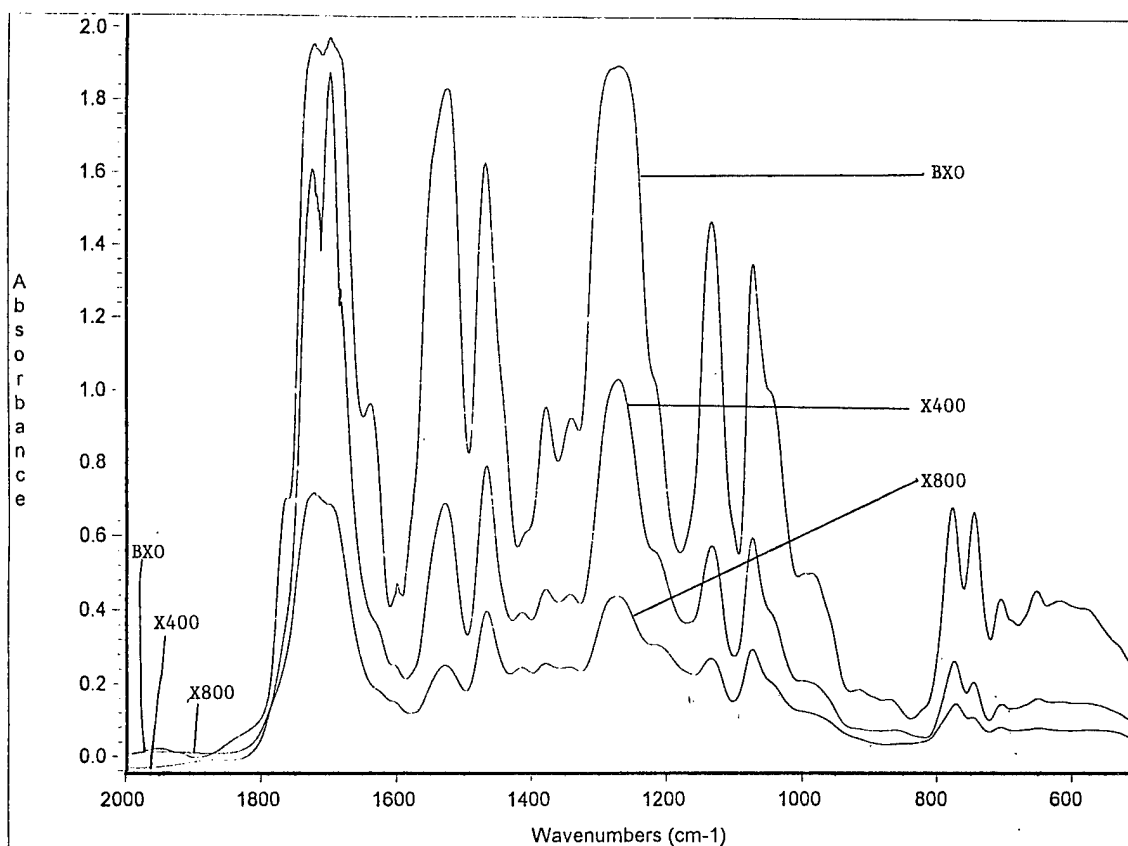


Figure 5. "Spreadsheet" IR spectra of resins after 0, 400, 800 hr xenon-arc exposure.

the back piece. Second, we punched holes at the right size and location for maximum signal throughput to keep the silicon wafers in place. Then, we sprayed CARC Green 383 on the front and back of the panels and sprayed clear resins on the silicon wafers. Pieces were assembled together into holders with the wafers in the application sample areas. Epoxy adhesive was used to keep the whole assembly together. We applied a rubber flexible coating along the edges to keep the moisture out as much as possible. This method worked out well for us. Although we thought about many other ways, they were discarded because of their time consumption and tedious sample preparation. In Appendices A–D, a log file is shown for each technique with its corresponding hours of exposure (Figures A-1–D-3).

5. CONCLUSIONS

IR band tracking of chemical changes is a highly feasible task. It has been proven that a wealth of spectral information can be extracted by using the FTIR technique with easy-to-use accessories. The chemical changes can explain the reasons for the loss of gloss, color, or both; adhesion; the subject failure in CARC testing; and the gradual inability to resist weathering effects.

The use of silicon wafers and IR cards can help to provide rapid, reliable answers to coatings' weatherability questions. Now, studies and results about a certain products' durability can be realized much sooner. Accelerated weathering can be done with new commercial products of FTIR accessories in making material prediction more accurate.

The best possible method is the usage of silicon wafers, followed closely by IR cards. Coatings' testing with natural and accelerated exposure systems is the only area we explored. However, this paves the way of analyzing a wide variety of samples with minimum sample preparation.

FTIR spectroscopy is definitely a valuable tool in the evaluation of coating materials relating to their durability characteristics. Advancements in commercially available accessory products has made spectral analysis simple and enhanced productivity and capabilities.

6. PLANS

Two-year exposure test results from SFTD will conclude this investigation. Also, authors plan to obtain the last test result from EMMA(QUA) Arizona testing. Correlation of all weathering data will be prepared.

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APPENDIX A:
EMMA(QUA) LOG FILE

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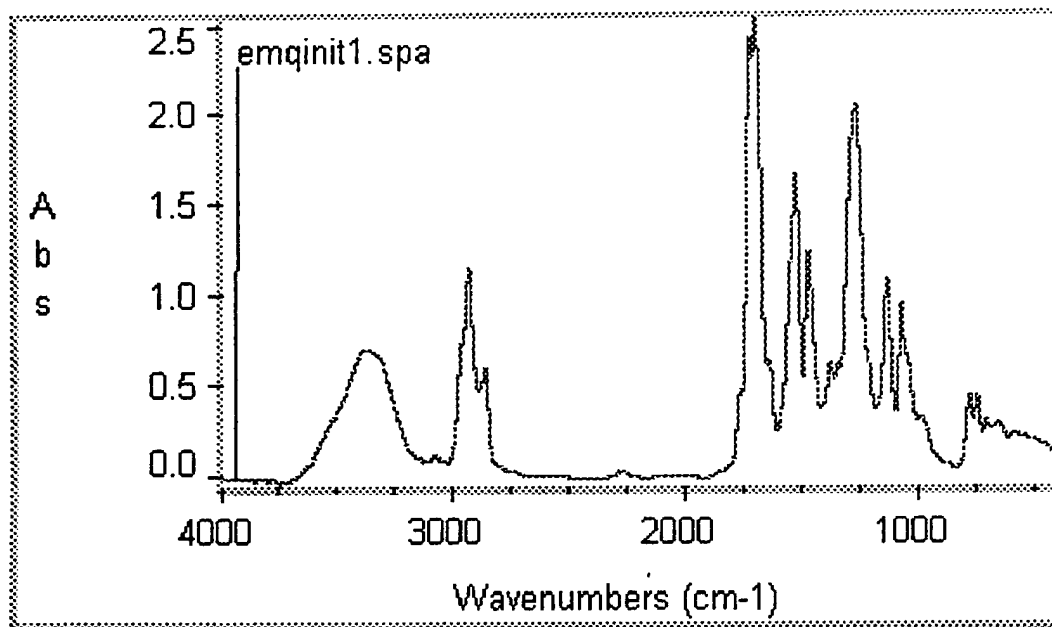


Figure A-1. Before EMMA(QUA) exposure.

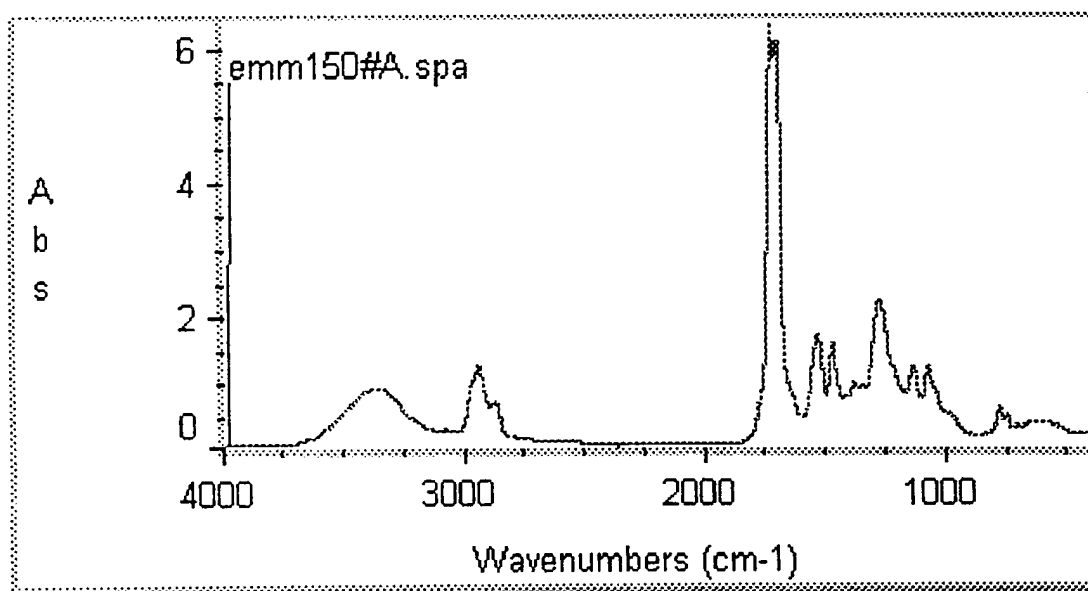


Figure A-2. After 150 MH/m² of EMMA(QUA) exposure.

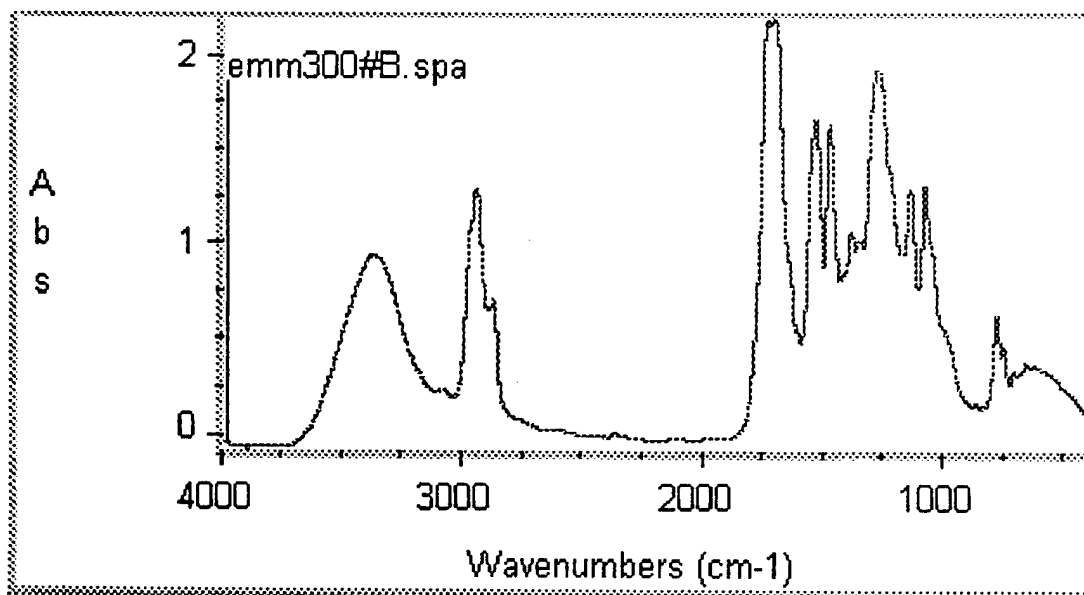


Figure A-3. After 300 MJ/m² of EMMA(QUA) exposure.

APPENDIX B:
XENON-ARC LOG FILE

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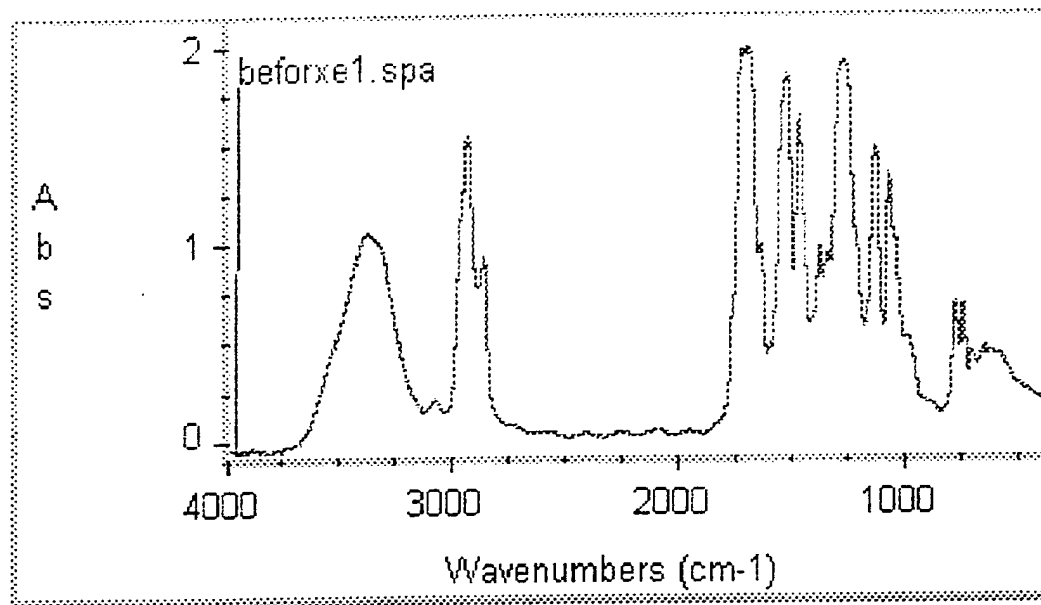


Figure B-1. Before xenon-arc exposure.

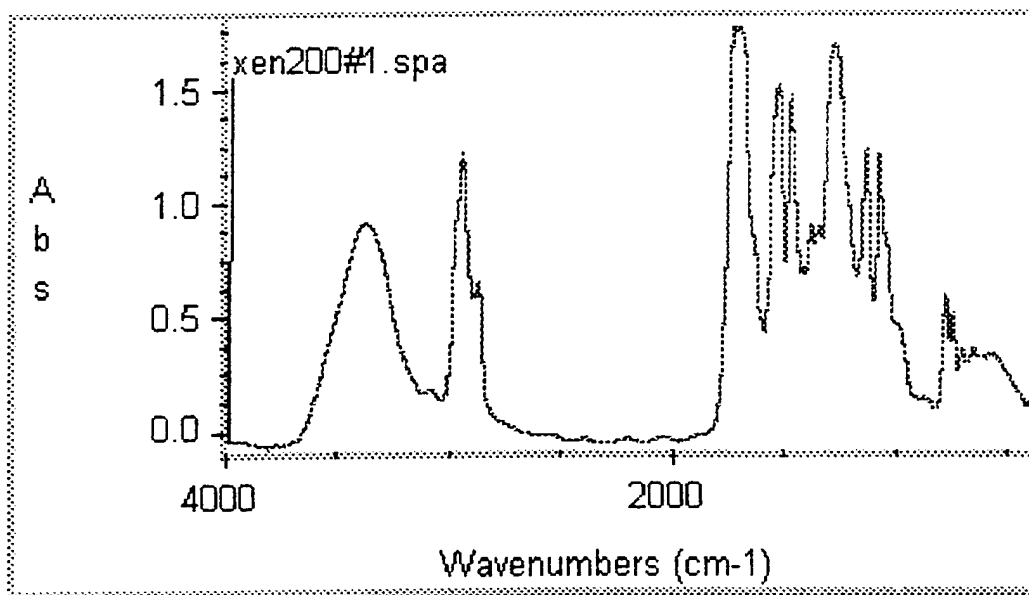


Figure B-2. After 200 hr of xenon-arc exposure.

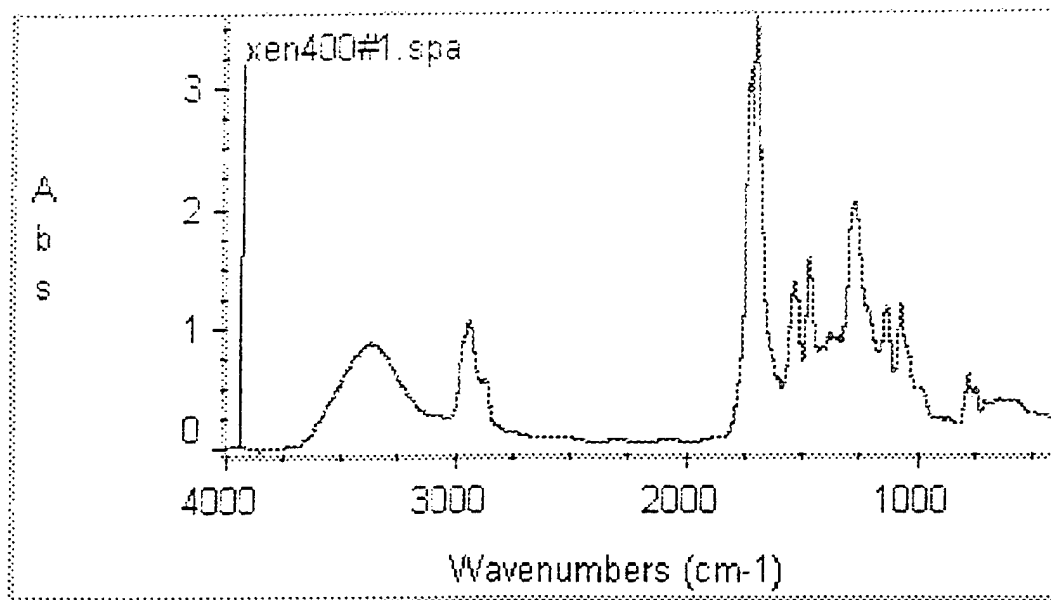


Figure B-3. After 400 hr of xenon-arc exposure.

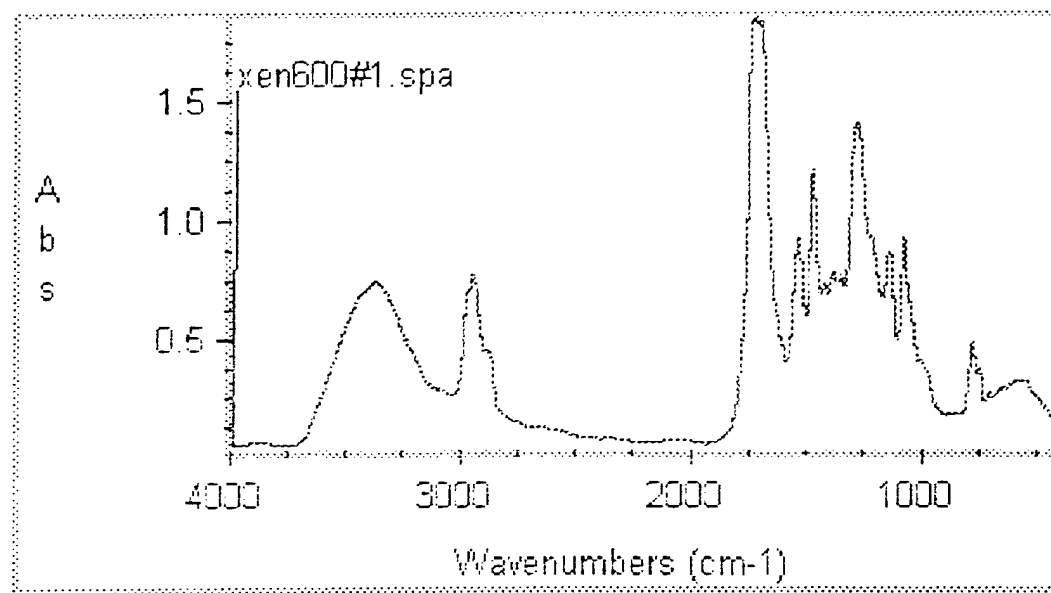


Figure B-4. After 600 hr of xenon-arc exposure.

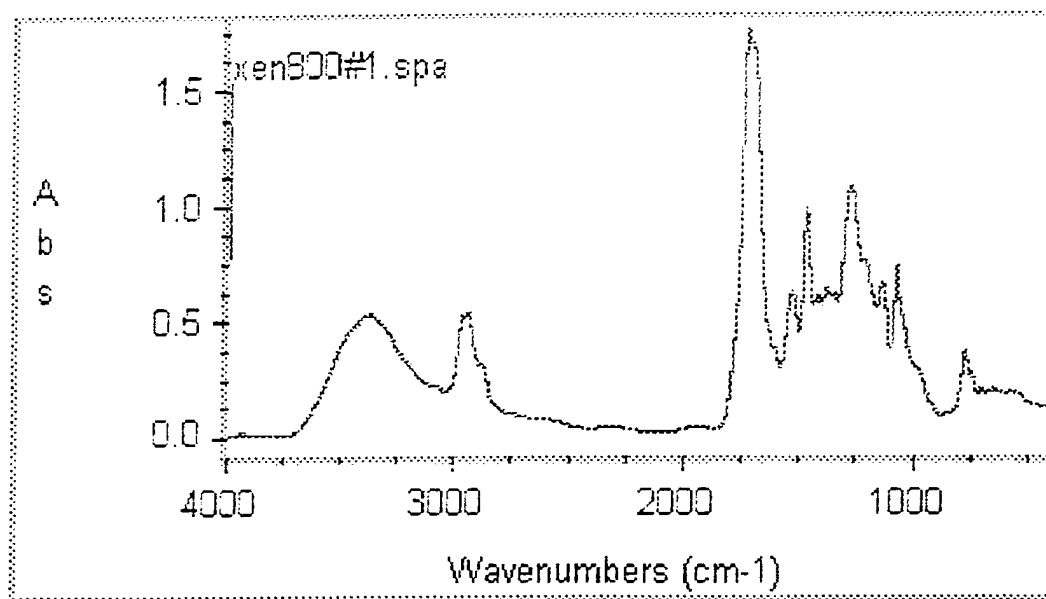


Figure B-5. After 800 hr of xenon-arc exposure.

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APPENDIX C:
QUV-CON LOG FILE

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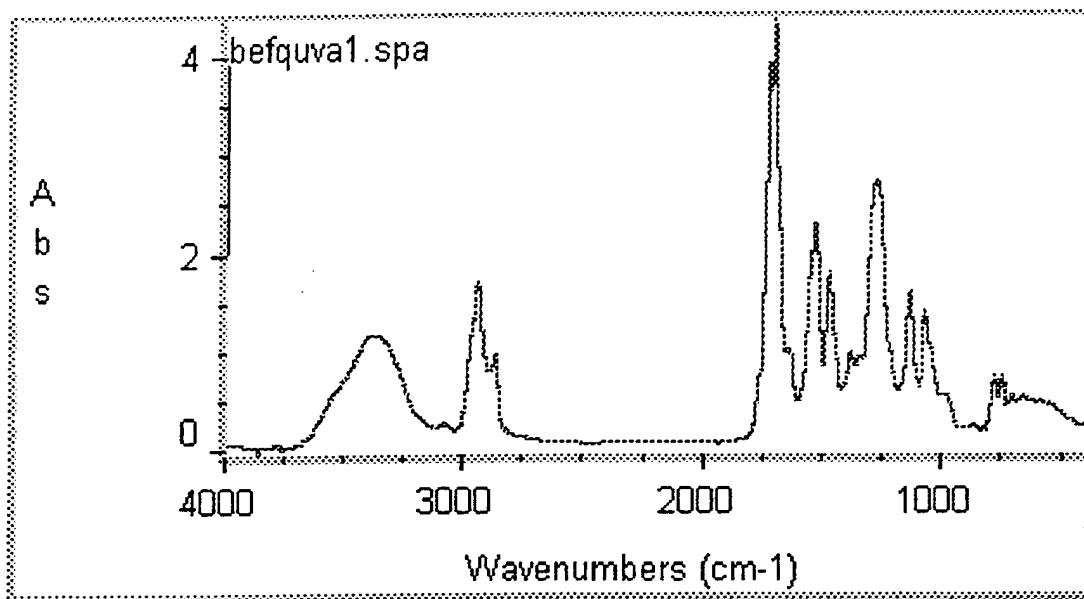


Figure C-1. Before QUV-CON exposure.

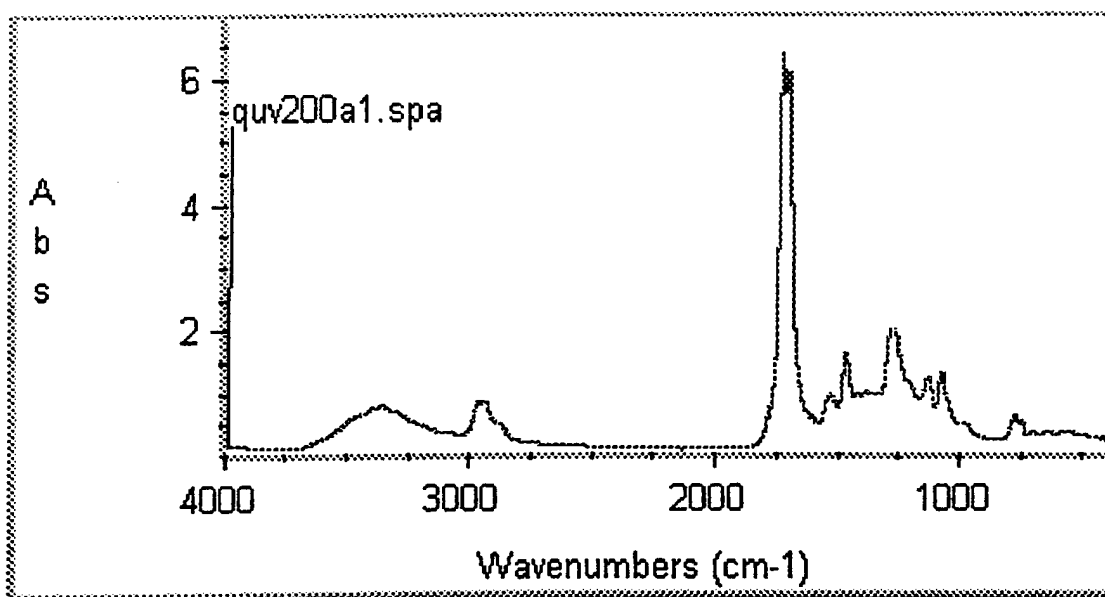


Figure C-2. After 200 hr of QUV-CON exposure.

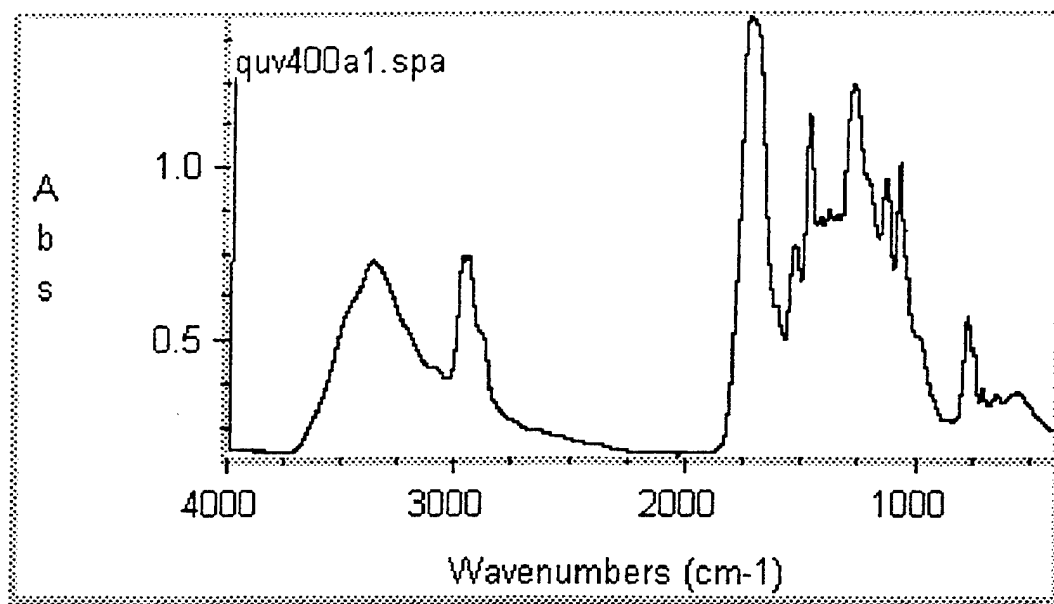


Figure C-3. After 400 hr of QUV-CON exposure.

APPENDIX D:
FORT BELVOIR LOG FILE

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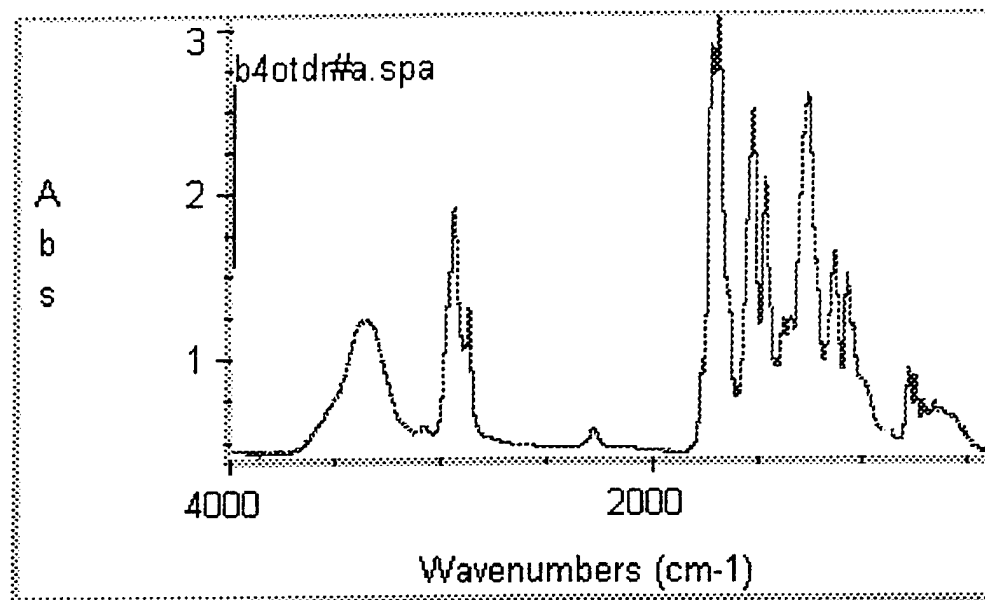


Figure D-1. Before Belvoir-site exposure.

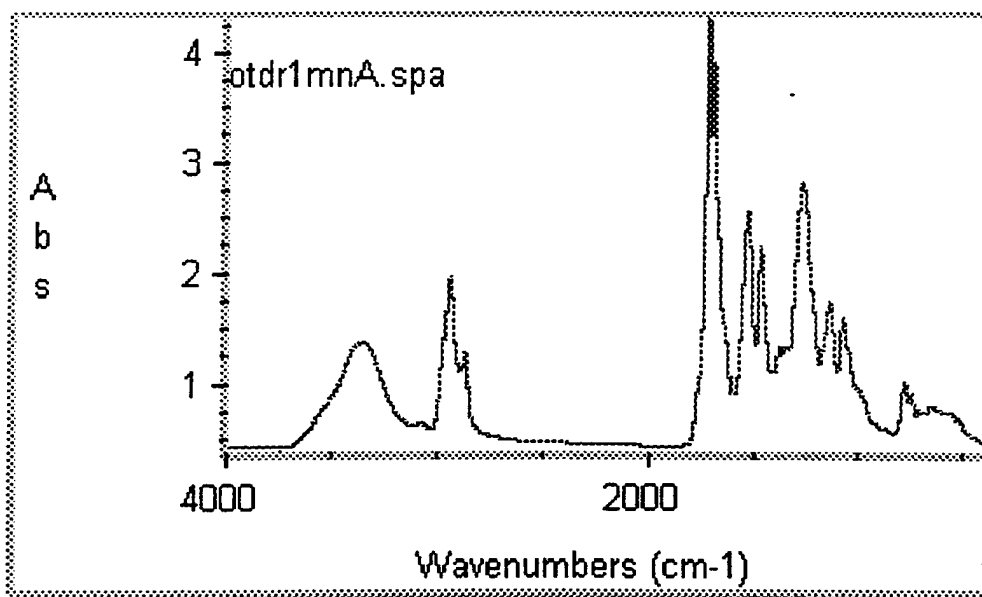


Figure D-2. After 1 mo of Belvoir-site exposure.

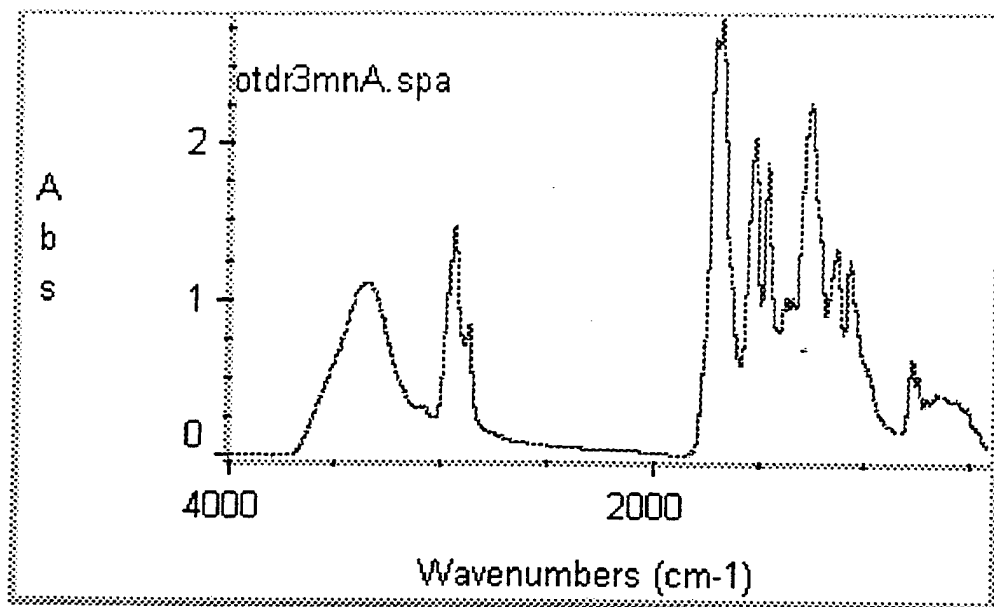


Figure D-3. After 3 mo of Belvoir-site exposure.

APPENDIX E:
SOUTH FLORIDA EXPOSURE

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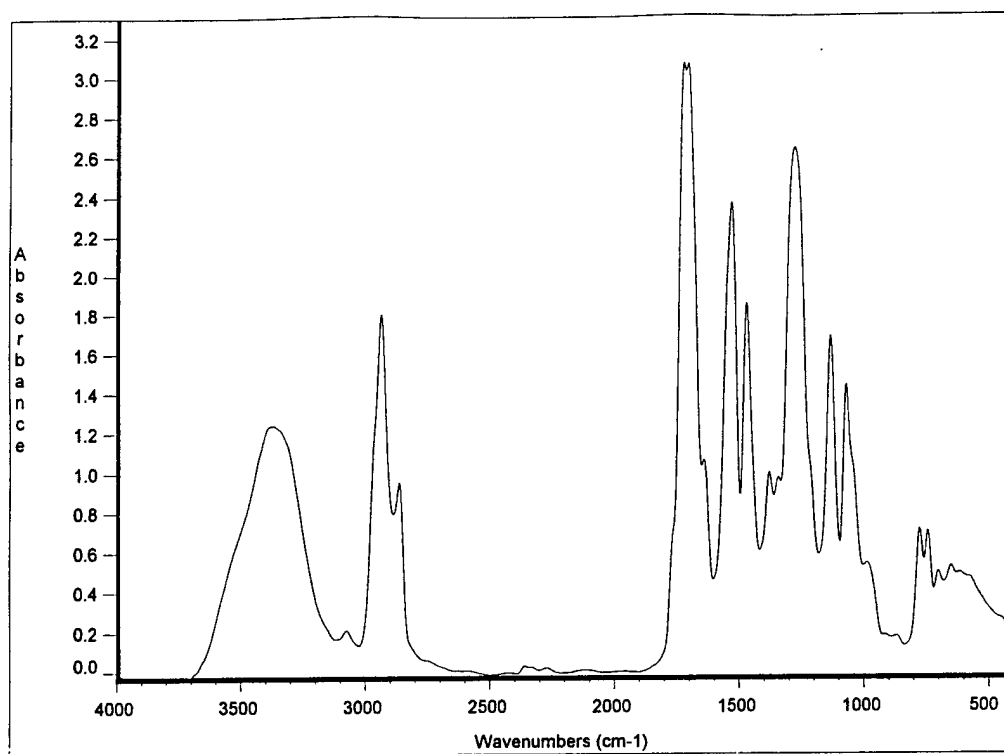


Figure E-1. Before South Florida exposure.

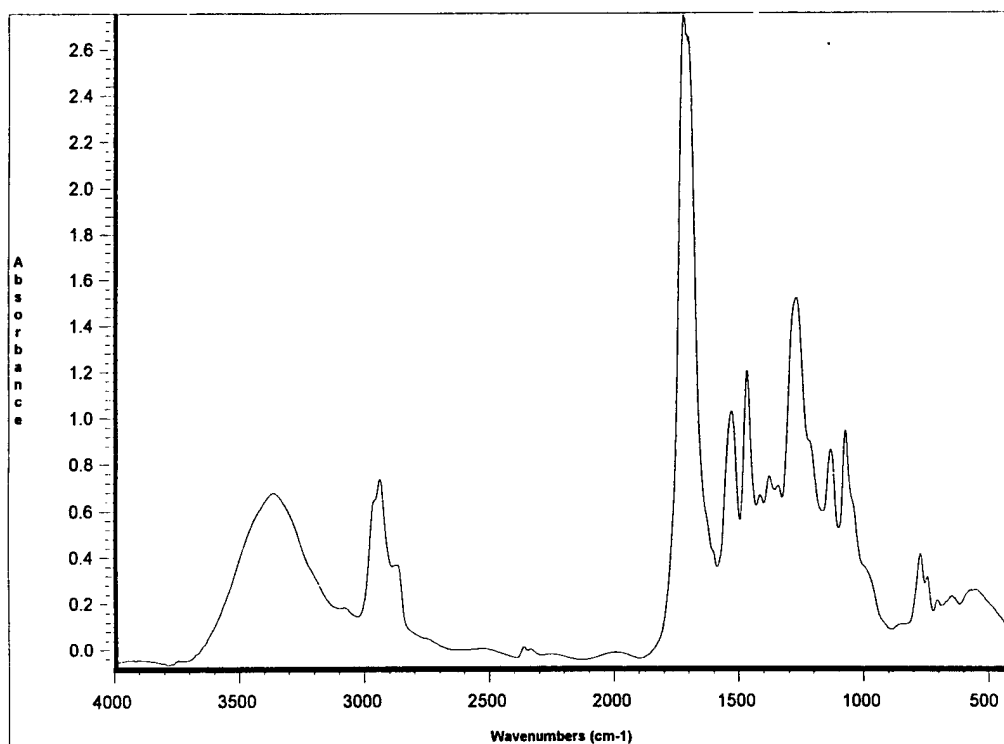


Figure E-2. After 6 mo of South Florida exposure.

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13. ABSTRACT (Maximum 200 words) Rapid, cost-effective sampling accessories for Fourier-Transform Infrared (FTIR) analysis are quite suitable with minimum sample preparation in coating materials studies. The U.S. Army's camouflage, polyurethane topcoat MIL-C-46168, "Chemical Agent Resistant Coating" (CARC) was applied to infrared (IR) cards and silicon wafers. After curing, the coating's durability was tested using two separate accelerated weathering techniques (QUV-CON and Xenon-Arc), and as well as, two outdoor exposure test methods (South Florida and EMMA(QUA)*-NTW). Characterization of the binder's chemical changes during both natural and accelerated weathering processes was monitored using the FTIR. This final report summarizes the experimental results of the coating's resistance to the accelerated-weathering and outdoor-exposure conditions. Additionally, a brief description of the author's method is described for the preparation of silicon wafers and subsequent FTIR analysis. *Trade name.				
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