

Photovoltaic Manufacturing Technology (PVMaT)

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EXECUTIVE SUMMARY

As outlined in our original proposal of January 1992 for this encapsulant development program, the primary goals are to:

- * Better define the problem of yellowing/browning of EVA-based encapsulants through examination of published papers, internal reports, case histories, and interviews with industry representatives.
- * Through laboratory investigation, determine probable mechanisms and the role of various parameters, such as heat, UV exposure, module construction, EVA interfaces, and EVA thickness, in the browning of EVA-based encapsulants.
- * Develop stabilization strategies, or a family of strategies for various module constructions, for protecting the encapsulant, based on EVA or other low-cost material, from degradative failure.
- * Conduct laboratory, accelerated outdoor, and field testing of encapsulant, laminated test coupons, and full modules in order to demonstrate the functional adequacy of the stabilization strategies.
- * Implement these strategies by: 1) wide dissemination of information developed under this program and 2) by making the improved or modified encapsulant formulation(s) available to the photovoltaic industry.
- * It is also a primary goal that the improved or modified encapsulant be low in cost (ie. competitively priced with present EVA [resin based] formulations), amenable to lamination with existing production lamination equipment with only minor modifications to processing conditions, and when combined with cover material will allow a minimum of 90% light transmission over the range of 350 to 1100 nm."

Key findings, as they relate to each of these goals, are summarized as follows:

- 1) Results of the review of published information and case histories, which was conducted during year one of this subcontract, are detailed in the first annual subcontract report (NREL/TP-411-7352, November 1994).

2) A detailed study of compositional and aging variables and degradation mechanisms, has revealed several important insights:

* While "standard cure" A9918P EVA resin-based encapsulant, laminated between low iron glass, showed significant yellowing after 17 weeks in an Atlas Ci35A xenon-arc Weather-Ometer, "neat" Elvax™ 3185, EVA resin with no additives, showed little or no yellowing after the same exposure.

* When similar laminates were prepared and exposed in the Weather-Ometer, using A9918P with no Lupersol™ 101, color development after 10 weeks was reduced by approximately 2/3. This result strongly implicated Lupersol 101 in the discoloration process. And when Lupersol TBEC was substituted for Lupersol 101 in the encapsulant (i.e. "fast cure" 15295P formulation rather than "standard cure" A9918P), the rate of yellowing was reduced by a factor of approximately 2.5 based on 17 weeks in the Weather-Ometer.

* Subsequent controlled Weather-Ometer aging studies of other laminates, prepared using A9918P with various combinations of additives, provided strong correlational evidence suggesting that A9918P discoloration arises primarily from stabilizing additives and their interaction with Lupersol 101. It appears that transformation products of these additives are giving rise to yellowing, rather than Elvax 3185, the base resin in A9918P.

* While it was not a goal of this program to fully explore all facets of the yellowing mechanism, instrumental analysis suggests that additive reactions and their interactions are a key step in the overall discoloration mechanism. Therefore, development work has focused on eliminating or interrupting this step.

3) Development work has yielded three strategies which limit photothermal browning:

* **Use of cerium-oxide-containing glass superstrates, especially with "fast-cure" 15295P encapsulant:** 30 weeks exposure in the Weather-Ometer (0.55 watts/m² /nm @ 340 nm, 100C) of glass/EVA/glass samples with cerium-containing glass gave a Yellowness Index of 5.2, barely detectable by eye, and one year exposure produced a 13 Index. By comparison, after the same 30 weeks exposure a 15295P control with Starphire™ low-iron glass had an Index of 87. Also, 36 weeks EMMA exposure in Phoenix resulted in almost no color for samples with cerium glass, versus a -0.7 Index with Solite™ glass and 13.8 with Solite glass when combined with "standard-cure" A9918P encapsulant.

* **Use of a Tefzel™ cover film in place of glass:** After 60 weeks exposure in the Ci35A, a Tefzel/A9918P/glass laminate had a Yellowness Index of 2.0, which is undetectable by eye, while 40 weeks of EMMA gave no measurable yellowing.

* **Use of one of four new EVA-based encapsulants, three "standard-cure" (X9903P, X9923P, and X9933P) and one that processes under "fast-cure" conditions (X15303P):**

After 14 weeks in the Ci35A, laminates of two of these encapsulants with Starphire, a glass highly transparent in the UV-B region of the spectrum, developed Yellowness Indexes of 1.6.

For added protection of these EVAs, a cerium-oxide-containing glass, either Solatex II™ or Solarphire™ should be used as the superstrate. These glasses should contain a sufficient quantity of cerium oxide to provide a lower end light transmission cut-off of approximately 325 nm.

Finally, the new encapsulants have the following features: - Employ the same Elvax 3185 used in A9918P and 15295P; - Handle, laminate, and cure under the same conditions as A9918P or 15295P; - Are comparable in cost.

1.0 INTRODUCTION

The goals of the NREL PVMaT program are, among others, to reduce module manufacturing costs and improve the quality, and we might add here the reliability, of manufactured PV products. But lower production costs will require economies of scale, and this means that the large potential markets of central power generating stations and distributed power applications need to be opened up. However, due at least in part to reliability issues, "Electric utilities seem to be in a "wait-and-see" mode, and show no interest at the present time in making investments of the magnitude that would make PV more competitive with other power sources.¹

One component critical to the service life of PV modules is the useful life of the EVA resin-based encapsulant which is employed extensively by module manufacturers on a worldwide basis. This pottant has been in commercial use since 1982², and over that time has proven to be a dependable material from the standpoint of production, module fabrication, and end-use. But despite the widespread acceptance of the EVA resin-based A9918 and similar formulations for PV encapsulation, some module producers, end-users, and investigators have reported a yellowing or browning phenomenon with EVA resin-based encapsulants in the field. While the incidence of this discoloration/degradation appeared at comparatively few sites at the time that this present program was conceived, it raised serious concern as to the long term reliability of EVA resin-based encapsulation systems.

Most notable was the browning degradation of the EVA resin-based encapsulant at Carrizo Solar Corporation's Carrisa Plains Photovoltaic Power Plant in California. This incident had a profound effect on the PV industry with respect to consumer confidence and reliability standards. The news of the browning and ultimate demise of this plant quickly transcended boundaries of small circulation PV industry newsletters, making headlines in national publications such as *Barron's*.

Surprisingly, while there had been considerable discussion of the discoloration/degradation of EVA resin-based encapsulants in the industry at the time this study was proposed to NREL, there was insufficient published information from which to draw meaningful conclusions regarding its cause(s).

Consequently, under the NREL PVMaT program, Springborn Laboratories proposed a comprehensive study of the discoloration problem and its possible solution(s). The goals of this encapsulant development program, as outlined in our proposal of January 1992, are to:

A. Zipser, "Solar Eclipse, Will the Mideast Crisis Make it a Hot Item Again?" *Barron's*, pp. 16,31 (August 20, 1990).

J. H. Wohlgemuth and R. C. Petersen, Solar Cells: Their Science, Technology, application and Economics, Solarex Experience with Ethylene Vinyl Acetate Encapsulation, (Elsevier Sequoia, 1991). pp 383-387.

- * Better define the problem of yellowing/browning of EVA-based encapsulants through examination of published papers, internal reports, case histories, and interviews with industry representatives.
- * Through laboratory investigation, determine probable mechanisms and the role of various parameters, such as heat, UV exposure, module construction, EVA interfaces, and EVA thickness, in the browning of EVA-based encapsulants.
- * Develop stabilization strategies, or a family of strategies for various module constructions, for protecting the encapsulant, based on EVA or other low-cost material, from degradative failure.
- * Conduct laboratory, accelerated outdoor, and field testing of encapsulant, laminated test coupons, and full modules in order to demonstrate the functional adequacy of the stabilization strategies.
- * Implement these strategies by: 1) wide dissemination of information developed under this program and 2) by making the improved or modified encapsulant formulation(s) available to the photovoltaic industry.
- * It is also a primary goal that the improved or modified encapsulant be low in cost (ie. competitively priced with present EVA resin based formulations), amenable to lamination with existing production lamination equipment with only minor modifications to processing conditions, and when combined with cover material will allow a minimum of 90% light transmission over the range of 350 to 1100 nm.

Prior to December 1992, the discoloration of EVA resin-based encapsulant as reported at the Carrisa Plains facility had been attributed to high operating temperatures, approximately 90°C, and increased light intensity resulting from mirror-enhanced light exposure. It is interesting that field survey findings on modules operated at normal conditions of temperature and light intensity were consistent with this speculation - that is, the discoloration was not nearly as severe with these other modules as that experienced at Carrisa Plains.

The Phase I survey of case histories of EVA-based encapsulant discoloration in fielded modules in the U.S., conducted during the first year of this program, revealed that the problem is limited to areas of the West and Southwest that have comparatively high solar insolation and ambient temperature. There has been only one confirmed case of discolored EVA-based encapsulant from modules fielded in the Northeast, and that occurred after 12 years in Maryland.

The absence of hard data regarding module operating temperatures, solar insolation, onset of discoloration, and quantitative evaluation has made correlations difficult if not impossible. However, the degree of discoloration does appear to correlate with increasing average daily direct normal solar radiation and approximate maximum module operating temperature, as estimated from maximum ambient temperatures.

Also, it is clear that the discoloration problem is not limited to the modules of any one manufacturer, however, the rate and degree of discoloration do appear to vary from company to company. And discoloration is not limited to EVA resin-based encapsulant sheet from any one supplier.

Over the course of the first year of this program, an accelerated U.V. aging method was selected. On careful review of the various types of accelerated U.V. aging equipment available, an Atlas Ci35A Weather-Ometer Xenon Exposure System was selected as appropriate equipment for this work. To summarize, some of the more significant advantages of the Ci35 A include:

I.. The spectral irradiance of filtered xenon light in the UV and visible range of the spectrum closely resembles that of natural sunlight, with no large spikes as are often found with mercury lamps or carbon arc sources.

II.. The xenon-arc source is widely accepted by industry (e.g. textile, automotive, plastic) and Government for accelerated weathering.

III.. The Ci35A is flexible in that the operator is able to set, monitor, and control the irradiance, temperature, humidity, and water spray.

IV.. The Ci35A has the capacity for many more samples than the table top models -more than sixty 2.6 x 5.0 inch samples, and twice that number if 2.6 x 2.5 inch samples are used. This is particularly important considering the scope not only of the Task 2 problem definition studies, but also the Task 5 evaluation of improved encapsulant formulations.

Disadvantages of the xenon-arc include degradation of filters and the air-cooled lamp, requiring periodic replacement of both, and comparatively high cost of the equipment and replacement parts.

On balance, however, this device was superior to any others. Consequently, a Ci35A device was purchased for utilization on the program.

The following report summarizes how this accelerated aging technique was used, first to further define the problem by studying various compositional and process parameters, then to investigate possible chemical mechanisms of discoloration, and subsequently to develop a family of solutions to the discoloration problem.

2.0 FURTHER PROBLEM DEFINITION - LABORATORY AGING STUDIES

2.1: Purpose: Task 2 of this PVMaT project involved conducting laboratory problem definition work with an emphasis on controlled accelerated U.V. aging studies (AAS) to evaluate the influence of various compositional, processing, and operating parameters on A9918P EVA resin-based encapsulant discoloration. In support of these AAS of coupon-size EVA-based A9918P and 15295P laminates, an Atlas xenon arc Ci35A Weather-Ometer was procured, installed, and calibrated for temperature and irradiance (See Annual report under this subcontract, for the period December 30, 1992 to March 31, 1994).

2.2: Sample Preparation: For these comparative AAS, coupon-size laminates, measuring 2.7 x 2.75 inches and preferably less than 0.255 inches thick, were used. These were prepared by vacuum lamination using the commercial time/temperature/vacuum schedules recommended for encapsulation work when using either EVA-based formulation A9918P or 15295P, except when the cure schedule was purposely varied in order to assess its effect. A laboratory-scale vacuum laminator and pump were used for this work. A data logger and multiple thermocouples were used to verify the temperature profile of samples cured in the laminator. For most of this work, glass/glass laminates were used to facilitate visual, colorimetric, and spectrographic measurements. However, some laminates contained cells in order to investigate such effects as cell/encapsulant and metallization/encapsulant interfacial chemical changes.

2.3: Superstrates: The superstrate used was low iron glass so as to allow the maximum amount of UV-B light through (ie. 285 to 350 nm), that wavelength region suspected as being responsible for EVA resin-based encapsulant discoloration/degradation. Also, low iron glass is the superstrate most commonly used in those fielded modules which have shown a discoloration problem. TPE refers to standard Tedlar/polyester/EVA laminate backing material, where the EVA layer is a non-conformal 4% vinyl acetate content grade (ie. this EVA is a tie layer rather than an encapsulant).

Low iron glasses used in this task (see table at the top of the following page) included Solite (AFG - samples from the mid 1980s), Starphire (PPG), Solatex II (AFG - containing cerium oxide, estimated to be at less than 4% by weight), and Solarphire (formerly Airphire: PPG - also containing cerium oxide). Solarphire and Solatex II benefit this application by removing most of the UV-B radiation between 280 and 330 nm, a region known to be detrimental to polymer stability (see annual report under this subcontract, for period from December 30, 1992 to March 31, 1994 for further details on these glass superstrates).

<u>Trade Name</u>	<u>Manufacturer</u>	<u>Contains Cerium</u>	<u>Currently Available</u>
Solite	AFG	No	No
Starphire	PPG	No	Yes
Solarphire (formerly Airphire)	PPG	Yes	Yes
Solatex II	AFG	Yes	Yes

2.4: Sample Exposure Conditions: Coupon laminates were exposed to 0.55 watts/square meter/nm (taken @ 340 nm) and 100°C in a Ci35A Atlas xenon-arc Weather-Ometer using quartz/borosilicate glass filters. The nominal lower end U.V. cut-off was 285 nm. Samples were exposed for a minimum of 17 weeks or until significant degradation/discoloration of the EVA had occurred. In all cases, samples were exposed in duplicate.

Calibration work on the Ci35A revealed that slight temperature and irradiance variations exist between locations in the top, middle, and bottom racks. Some samples were placed so as to assess the effect of this variation. However, since the purpose of the testing was to develop comparative data, most of the samples were rotated between racks, from top to bottom on a weekly basis, to normalize any minor temperature/irradiance (T/I) differences.

Again, it should be emphasized that the Weather-Ometer is being used only as a screening tool - that is, a laboratory technique for conducting preliminary evaluation of various module encapsulation materials/constructions in order to assess their relative U.V. aging resistance and to obtain a relative ranking of those materials/constructions. There will be no attempt to develop firm correlations between accelerated aging results and field information or to develop definitive acceleration factors, both of which are beyond the scope of this investigation.

2.5: Sample Evaluation: Coupons were monitored for color/chemical changes during the exposure. Non-destructive tests on the samples included visual fluorescence at 360 nm under a hand held "mineral light" (qualitative test for development of chemical change in the encapsulant), Yellowness Index per ASTM D-1925, and percent light transmission (%T) between 250 and 900 nm by U.V.-VIS spectrophotometer.

Some destructive testing for analysis of additive concentrations and vinyl acetate content was also done. At the conclusion of the accelerated UV exposure period, selected samples were forwarded to NREL for fluorescence spectroscopy and to UCONN/IMS for Task 3 analysis (see section 3.0 of this report for details).

2.6 Aging Results: Quantitative yellowing results can be found in Table 1. Based on these results, browning appears to be related to the various formulation components as follows:

EVA Resin (Elvax 150 & Elvax 3185): Neat EVA resin, containing 33% vinyl acetate comonomer by weight, as received from the supplier without additional additives, showed little to no color development following 17 weeks exposure in the Ci35A (see samples in Table 1.). These laminates, which used either Solite or Starphire as the superstrates, developed a 0.7 to 1.4 Yellowness Index and no visual yellowing.

By contrast, control samples containing the "Standard-Cure" A9918P EVA-based encapsulant showed significant color change over the same period (Table 1.), again with Solite or Starphire oxide glass, i.e. 39.1 to 49.6 color index and, visually a medium brown color (see photographs - Figure 1.). After 22 weeks aging, these samples developed a color index of 53.6 to 64.0, and the Solite covered sample had a color index of 82.8 after 36 weeks in the Ci35A Weather-Ometer, (Table 1.).

These results suggest that the browning might be related only to the additives of A9918P and not to the EVA resin at all. Analytical results strongly support this hypothesis (see Section 3.0 of this report for details).

Peroxide Crosslinker-Lupersol 101: Surprisingly, samples using A9918P EVA-based encapsulant, but without the peroxide crosslinker [Lupersol 101; 2,5-dimethyl-2,5-di (t-butylperoxy) hexane] developed comparatively little color after 10 weeks of exposure (color index of 5.4 to 7.1 versus 18.8 to 19.6 for fully formulated A9918P). This data suggested that Lupersol 101 peroxide is playing a significant role in the discoloration. However, the color index for these samples (5.4 to 7.1) would appear to indicate that one or more of the other additives also play a role in the encapsulant's discoloration.

For comparison, a set of samples was prepared and exposed using EVA-based encapsulant 15295P and Solite superstrate. The difference between 15295P and A9918P is the use of Lupersol TBEC in place of Lupersol 101 to provide a "faster cure". After 17 weeks in the Ci35A, these samples developed a color index of only 14.2 to 16.7 (Table 1.) compared to 39.1 to 40.4 for the A9918P. And after 26 weeks, the 15295P-based laminate had a color index of 33.7 versus 63.5 for the A9918P-based sample (Table 1.).

Again, this comparison implicates the peroxide, Lupersol 101, [2,5-dimethyl-2,5-di (t-butylperoxy) hexane], and to a lesser extent Lupersol TBEC, [00-tert-butyl 0-(2-ethylhexyl) monoperoxy- carbonate], as a contributor, along with one or more of the stabilization-package additives, in the color development process.

Stabilization-Package Additives: A number of sample laminates were constructed using Starphire or Solite glass superstrate/encapsulant/glass, in which the various stabilizers, Naugard P, Cyasorb UV531 and Tinuvin 770, were systematically removed in order to determine each additive's contribution towards the encapsulant's discoloration. Once again the accelerated UV aging was

accomplished using the Ci35A Xenon Arc Weather-Ometer. The results of those experimental efforts caused us to suspect that the color formation was influenced by an interaction of two or more additives with the Lupersol 101 peroxide, leading to chromophore formation (discoloration).

Glass superstrates (Solite versus Solatex II, Starphire and Solarphire): After 17 weeks of aging, some significant differences in discoloration showed up in samples of A9918P using different types of glass superstrate (Table 1.). Solarphire, containing cerium oxide, appears to be the most effective at slowing the rate of discoloration (color index of 9.3 at 17 weeks), but Solatex II, also containing cerium oxide, is also very effective (color index of 9.8 to 10.3), and the results between samples using these two glasses may be within experimental error. After 35 weeks aging, the Solarphire-covered laminate had a color index of 17.8, while the sample prepared with Solatex II developed a color index of 23.8 (Table 1.).

The control, Solite, allowed a 17-week color index of 39.1 to 40.4, while A9918P discoloration with Starphire glass superstrate was somewhat worse, with a color index of 48.7 to 49.6. After 35 weeks in the Ci35A, the Solite-covered sample showed a color index of 81.9 (Table 1), compared with 17.8 to 23.8 for samples prepared with either Solarphire or Solatex II, as noted in the preceding paragraph.

It is noteworthy that Starphire transmits more light in the 280 to 340 nm wavelength region than does Solite, and this could account for the difference in discoloration rate (see Figures 8-11 in the annual Report under this subcontract for the period December 30, 1992 to March 31, 1994).

Breathable Substrates and Superstrates: When an oxygen permeable substrate or superstrate is used in place of one of the layers of glass, comparatively little color development occurs. For example, A9918P-based laminates prepared with Solite glass and no backing showed Yellowness Index, after 17 weeks of Weather-Ometer aging, of only 3.1 to 3.3 and only 4.5 after 26 weeks (Table 1). Also, samples prepared with 10 mil Tedlar™ backing, had no visible discoloration after 17 weeks of aging (Table 1.).

When Tedlar™ or Tefzel™ was used as the superstrate, with low iron glass as the substrate and A9918P as the encapsulant, Yellowness Index readings of only 2.0 to 2.9 were recorded after 17 weeks in the Ci35A (Table 1). And after 32 weeks, the Tefzel-covered sample had a Yellowness Index of only 3.9 (Table 1).

Analytical work reveals that oxidation is taking place in the encapsulant in such permeable areas, and we speculate that either chromophores are being oxidized as well, or their formation is being inhibited by the presence of oxygen. (See section 3.0 of this report for details)

2.7: Outdoor EMMA Accelerated Weathering Tests: For added confirmation of laboratory accelerated aging data based on Atlas Ci35A xenon-arc Weather-Ometer exposure, selected samples were submitted to DSET Laboratories Inc., Phoenix, Arizona, where they are being

subjected to EMMA (Equatorial Mount with Mirrors for Acceleration) accelerated outdoor exposure. Samples, prepared in duplicate, include:

Solite (circa 1980s)/A9918P/Starphire (substrate)

Solatex II/A9918P/Starphire

Tefzel/A9918P/Starphire

Solite (circa 1980s)/15295P (with Lupersol TBEC)/Starphire

Solatex II/15295P/Starphire

Solite/A9918P/cells/Tedlar film laminate

These samples are being evaluated monthly by DSET Labs for Yellowness Index (see Table 2). The control material, A9918P with either Solite or Starphire glass superstrate, has developed measurable yellowing (ie. a Yellowness Index of 13.8 to 15.7 after 36 weeks of exposure).

2.8: Conclusions from Aging Studies: These Task 2 results suggest that the photochemistry of EVA-based encapsulant A9918P is related to the stabilization-package additives interacting with residual Lupersol 101 peroxide. The EVA base resin, Elvax 3185, does not appear to be a significant contributor to color, unless the reaction products of the stabilization package with the Lupersol 101 are in turn involving the polymer in some way.

Analytical results corroborate these finding, as discussed on page 10, Section 3.0 of this report.

The use of cerium oxide-containing glass, Solarphire or Solatex II, or window glass greatly reduces the rate of discoloration of EVA-based A9918P, presumably by filtering out much of the UV-B radiation (ie. 280 to 340 nm).

Also the use of Lupersol TBEC, as a replacement for Lupersol 101, significantly reduces the rate of encapsulant discoloration.

And, when Lupersol TBEC-based encapsulant is employed with Solarphire or Solatex II, color development is nearly eliminated. After 29 weeks in the Ci35A Weather-Ometer, laminates prepared with either Solatex II or Solarphire show no visible discoloration, and a Yellowness Index of only 5.1 to 7.9 (see Table 1.).

3.0 DEFINE POSSIBLE DEGRADATION MECHANISMS

3.1; Task 3 involves instrumental analysis and polymer characterization to verify suspected chemical degradation mechanisms. Both field-aged and laboratory Weather-Ometer-aged modules and laminates prepared with EVA-based encapsulant were evaluated by a variety of analytical methods including GC/MS (gas chromatography/mass spectrometry) for UV absorbers, GC, FTIR (fourier transform infrared spectroscopy) for unsaturation and evidence of oxidation, thermogravimetric analysis for vinyl acetate content of the EVA resin, DSC (differential scanning calorimetry) for residual peroxide, and microscopy for morphological changes.

The following summarizes conclusions that have been drawn from the work to date. A detailed Task 3 report on this work was prepared and submitted to NREL February 6, 1995:

1. University of Connecticut, Institute of Materials Science, Storrs, CT, has been unable to verify that discoloration of EVA-based encapsulant in photovoltaic modules is related to long chains of conjugated unsaturation (i.e. polyenes).

a. Infrared spectroscopy does not indicate the presence of significant amounts of unsaturation in discolored EVA-based encapsulant. IR spectra of unaged and highly discolored encapsulants were practically superimposable with no perceptible absorption in the region of the spectra where double bonds would have been expected to have absorbed.

b. The vinyl acetate contents of all samples of EVA-based encapsulants analyzed, which included both A9918P and 15295P grades, field aged and xenon-arc Weather-Ometer aged, and all levels of discoloration, were virtually identical and at the expected levels (see Table 3). Consequently, there appears to be little double bond formation from photolysis of vinyl acetate. And if there is little basis for double bond formation at any level, there is even less reason to expect conjugated double bond sequences of eight or more as required to develop color in the EVA copolymer.

c. EVA-resin samples without additives when laminated between low iron glass did not discolor on exposure in the Ci35A Weather-Ometer (see page 5).

d. It is difficult to envision the generation of chains of conjugated unsaturation containing more than eight double bonds and in sufficient quantity so as to result in the degree of discoloration that has been evidenced, especially at Carrisa Plains. After all, Elvax 3185 contains only 15 mole percent vinyl acetate and 85 mole percent ethylene, and the reactivity ratios for ethylene and vinyl acetate favor a completely random copolymer.

e. When treated with peracetic acid, discolored EVA-based encapsulant did not lose any of its color. But a PVC control, which had been purposely degraded to create conjugated unsaturation and a dark brown, nearly black color, was bleached to a translucent white when this conjugated unsaturation was oxidized by peracetic acid using the same method. Once again, this supports the hypothesis that the brown color in field aged EVA-based encapsulant arises from some other cause than polyenes.

2. Unreacted Lupersol 101 peroxide remaining after curing exhibited a significant effect on the concentrations of stabilizing additives.

a. Cyasorb UV 531 concentrations suffered little reduction in concentration when a glass/encapsulant/glass laminate of EVA resin without Lupersol 101 was exposed in the Ci35A Weather-Ometer for ten weeks, whereas samples with the usual amount of Lupersol exhibited a 40% reduction in Cyasorb UV 531 concentration during twelve weeks Weather-Ometer exposure. Also, the later showed significant yellowing, while the former did not.

b. The concentration of Tinuvin 770 behaved similarly showing 61% reduction during twelve weeks exposure.

3. Transformation products of BHT (butylated hydroxytoluene, a hindered phenol-type antioxidant added to the EVA resin by DuPont to protect it during processing), Cyasorb UV 531, and nonyl phenol (from reactions of Naugard P), arising from reactions with alkoxy radicals from the photolysis of Lupersol 101, might play an important role in the discoloration of EVA-based encapsulant. Investigations with laboratory Weather-Ometer aged glass/encapsulant/glass laminates showed correlation between color development and stabilizing additives/Lupersol 101 interactions (Table 6).

4. IR evidence indicates light colored encapsulant areas of modules recovered from the field have experienced oxidation. This suggests the backing is permeable to oxygen which diffuses into the rear of the modules and migrates through regions where there are no silicon cell barriers. Regions reached by oxygen might be "photo-oxidatively bleached." Colorless regions in the vicinity of cracks in the cells exhibit this same phenomenon, that is, the crack provides access of oxygen to the discolored EVA-based encapsulant above the cells (Figures 2 through 5).

4.0 DEVELOP ENCAPSULATION STRATEGIES FOR REDUCED DISCOLORATION AND/OR DEGRADATION AND CONDUCT ACCELERATED TESTING OF LAMINATES

4.1: Purpose: Using the results of Tasks 2 and 3, as discussed previously in sections 2 and 3 of this report, the purpose of this Task 4 and 5 effort is to: 1) develop possible approaches for stabilizing the EVA-based encapsulant against discoloration/degradation and consider alternate encapsulation systems that might be more inherently resistant to discoloration/degradation, and 2) evaluate the performance of promising systems by AAS (accelerated aging studies) using xenon-arc Weather-Ometer.

Strategies being considered include cerous and uranium salts as UVA (UV absorbers), metallocene compounds as UVA, other organic compounds with the ability to absorb strongly radiation in the 285 to 350 nm range as UVA, other hindered amine light stabilizers (HALS) as alternatives to Tinuvin 770, higher concentrations of existing additives, no phosphite or alternate phosphites to Naugard P as peroxide decomposers, UV absorbing coatings on the glass superstrate, and UV-absorbing glass superstrates.

Also being investigated are other low-cost polyolefin-based resins as alternatives to EVA resin Elvax 3185.

4.2: Sample Preparation: Additives are being compounded into Elvax 3185 and other base resins on a laboratory-sized, differential-speed, two-roll rubber mill. Sheet samples of these compounds are being compression molded using laboratory hydraulic presses equipped with electrically heated platens.

Then using these molded sheets, glass/encapsulant/glass samples are being prepared in a laboratory-scale laminator, as detailed in section 2.2 of this report, p. 4.

4.3: Sample Evaluation: Coupon-size laminates are being exposed to an irradiance of 0.55 watts per square meter/nm (@ 340 nm) in the Ci35A Weather-Ometer, as detailed in section 2.4 of this report, p. 4. On a weekly basis, samples are being evaluated for Yellowness Index per ASTM D-1925. Selected samples will also be checked periodically for %T (see section 2.5 of this report, p 5)

4.4: Results of AAS: With the Task 2 and 3 work indicating an interaction of Lupersol 101 with the stabilization-package additives as a primary cause of the development of chromophores, most of the Task 4 studies have focused on replacement additives. A number of alternate additives were evaluated in Elvax 3185 in combination with Lupersol 101. For the purpose of evaluation, the UVA were added at higher concentrations, 1.0 phr (parts by weight per hundred parts of resin) UVA and 1.5 phr Lupersol 101, to exacerbate any possible interactions.

Glass/encapsulant/glass laminates were subjected to AAS as before. Starphire was used as the superstrate, because of lack of availability of the older Solite material.

The following additives have been evaluated in both "standard" cure and "fast" cure EVA-based formulations:

UV Absorbers

Cyasorb UV 1164	Cyasorb UV 531 (control)
Cyasorb UV 5411	Givisorb UV-1
Tinuvin 328	Tinuvin P
Uvinul 3039	Sanduvor VSU
Eastman inhibitor	Phenyl 2 hydroxybenzoate

Hindered Amine Light Stabilizers (HALS)

Tinuvin 622LD	Tinuvin 770 (control)
Cyasorb UV 3581	Cyasorb 3346
Cyasorb UV 2908	Cyasorb 3668
Chimmasorb 944	Voidox
Sandostab D-EPA	Sanduvor 3225

Other Stabilizers/Antioxidants

Sandostab P-EPQ	Sandostab 3225
Irganox 1010	Irganox 1076
Irganox 245	Cyanox 1790
Ethanox 398	

On the basis of these investigations, we have developed four experimental formulations, X9903P, X9923P and X9933P, EVA-based encapsulants which cure under the same conditions as A9918P and X15303P, a material which cures under the same conditions as 15295P. After 14 weeks in the Ci35A Weather-Ometer, glass/encapsulant/glass laminates prepared with both X9903P and X15303P show a negligible 1.6 Yellowness Index (Table 4). Figure 1 provides a graphical comparison with A9918P and 15295P after 10 weeks of Weather-Ometer exposure.

It should be noted that both experimental encapsulants were laminated with Starphire glass, which is slightly more transparent to UV-B than Solite glass and thereby represents a "worst-case" for simulating outdoor exposure. Also, both experimental grades are based on the same Elvax 3185 EVA resin used in 15295P and A9918P.

Experimental formulation X9903P is also being subjected to EMMA testing at DSET laboratories in glass/encapsulant/glass laminates. Results will appear in the next semi-annual report.

5.0 CONCLUSIONS AND FUTURE WORK

Conclusions: A detailed study of compositional and aging variables and degradation mechanisms, has revealed several important insights:

* While "standard cure" A9918P EVA resin-based encapsulant, laminated between low iron glass, showed significant yellowing after 17 weeks in an Atlas Ci35A xenon-arc Weather-Ometer, "neat" Elvax™ 3185, EVA resin with no additives, showed little or no yellowing after the same exposure.

* When similar laminates were prepared and exposed in the Weather-Ometer, using A9918P with no Lupersol™ 101, color development after 10 weeks was reduced by approximately 2/3. This result strongly implicated Lupersol 101 in the discoloration process. And when Lupersol TBEC was substituted for Lupersol 101 in the encapsulant (i.e. "fast cure" 15295P formulation rather than "standard cure" A9918P), the rate of yellowing was reduced by a factor of approximately 2.5 based on 17 weeks in the Weather-Ometer.

* Subsequent controlled Weather-Ometer aging studies of other laminates, prepared using A9918P with various combinations of additives, provided strong correlational evidence suggesting that A9918P discoloration arises primarily from stabilizing additives and their interaction with Lupersol 101. It appears that transformation products of these additives are giving rise to yellowing, rather than Elvax 3185, the base resin in A9918P.

* While it was not a goal of this program to fully explore all facets of the yellowing mechanism, instrumental analysis suggests that additive reactions and their interactions are a key step in the overall discoloration mechanism. Therefore, development work has focused on eliminating or interrupting this step.

Development work has yielded three strategies which limit photothermal browning:

* **Use of cerium-oxide-containing glass superstrates, especially with "fast-cure" 15295P encapsulant:** 30 weeks exposure in the Weather-Ometer (0.55 watts/m² /nm @ 340 nm, 100C) of glass/EVA/glass samples with cerium-containing glass gave a Yellowness Index of 5.2, barely detectable by eye, and one year exposure produced a 13 Index. By comparison, after the same 30 weeks exposure a 15295P control with Starphire™ low-iron glass had an Index of 65. Also, 36 weeks EMMA exposure in Phoenix resulted in almost no color for samples with cerium glass, versus a -0.7 Index with Solite™ glass and 13.8 with Solite glass when combined with "standard-cure" A9918P encapsulant.

* **Use of a Tefzel™ cover film in place of glass:** After 60 weeks exposure in the Ci35A, a Tefzel/A9918P/glass laminate had a Yellowness Index of 2.0, which is undetectable by eye, while 40 weeks of EMMA gave no measurable yellowing.

* **Use of one of four new EVA-based encapsulants, three "standard-cure" (X9903P, X9923P, and X9933P) and one that processes under "fast-cure" conditions (X15303P):** After 14 weeks in the Ci35A, laminates of these encapsulants with Starphire, a glass highly transparent in the UV-B region of the spectrum, developed Yellowness Indexes of only 1.6.

For added protection of these EVA-based encapsulants, a cerium-oxide-containing glass, either Solatex II™ or Solarphire™ should be used as the superstrate. These glasses should contain a sufficient quantity of cerium oxide to provide a lower end light transmission cut-off of approximately 325 nm.

Finally, the new encapsulants have the following features: - Employ the same Elvax 3185 used in A9918P and 15295P; - Handle, laminate, and cure under the same conditions as A9918P or 15295P; - Are comparable in cost.

Future Work: During Phase III of this PVMaT program, Springborn Labs will:

1. Conduct laboratory, accelerated outdoor, and field testing of encapsulant, laminated test coupons, and full modules in order to demonstrate the functional adequacy of the stabilization strategies. Approximately ten team members, domestic PV module manufacturers will prepare coupon-size mini-modules as well as full size modules. Some of the mini-modules will be subjected to Ci35A Weather-Ometer exposure, while the remainder will be sent to DSET Labs for EMMA testing in Phoenix.

Arizona State University will subject full size modules to qualification testing, and will oversee the installation of an array on a two axis tracker at Arizona Public Service's STAR facility in Tempe, AZ. Modules will be monitored regularly by ASU for light and dark IV, module temperatures, etc.

2. Implement these strategies by: a) wide dissemination of information developed under this program. Springborn Labs will continue to present and submit for publication technical papers which detail the results of this work. Conferences will include the NREL Photovoltaic Advanced Research & Development Project Review Meeting, the IEEE Photovoltaic Specialist Conference, the NREL Photovoltaic Performance and Reliability Workshop, and the European Photovoltaic Solar Energy Conference.

and b) by making the improved or modified encapsulant formulation(s) available to the photovoltaic industry. Springborn Labs will continue to manufacture the new encapsulant sheet and sample interested PV manufacturers.

TABLE 1
(page 2 of 2)

Yellowness Index Measurements (not corrected for yellowness due to 100°C Oven Aging)
For Glass/EVA/Glass Laminates (1)

Samples	Construction	21weeks	22weeks	23weeks	24weeks	25weeks	26weeks	27weeks	28weeks	29weeks	30 weeks	31 weeks	32 weeks	32 weeks	34 weeks	35 weeks	35 weeks	37 weeks	38 weeks	39 weeks	40 weeks
No Backing A0918P																					
29187-2b	A0918P/Solite/no back	3.7	3.9	3.5	3.9	4.0	4.5	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29188-3c	Solatex II (Cerium)/no back	4.7	5.1	4.8	4.7	4.7	5.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29188-4a	Solatex II (Cerium)/no back	4.4	4.6	3.9	4.0	3.9	5.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
A0918P Standard																					
29188-1b	Solatex II (Cerium)	11.8	12.8	12.5	13.8	14.1	14.9	15.8	16.1	17.1	17.8	20.2	20.5	19.9	22.1	23.8	24.2	28.0	27.2	28.4	28.7
29178-5b	Window Glass	17.5	18.2	20.0	21.1	22.7	24.4	25.8	27.3	29.1	29.1	33.4	35.1	36.8	39.3	40.7	-	46.5	48.2	48.4	50.5
29178-4a	Airphire (Cerium)	10.8	10.7	11.5	11.7	12.2	12.4	12.7	13.3	13.8	13.8	15.8	15.8	16.5	17.2	17.8	-	21.3	19.8	20.4	21.5
29188-1a	Solite	51.9	53.8	55.7	58.8	60.8	63.5	66.0	67.8	70.1	72.0	75.0	76.8	78.4	79.3	81.9	82.8	-	88.2	88.6	86.1
Alumina Core Materials																					
29202-1a	Tedlar Cover, TUT20BG3	9.4	9.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29202-2b	Tedlar Cover, TUT20BG3	9.4	9.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29202-3a	Teflon TFM-C cover	2.4	2.2	2.1	2.3	3.0	3.1	2.6	2.2	2.2	2.4	3.9	4.0	4.0	4.7	4.9	2.9	2.7	2.7	2.7	2.5
S94 Control																					
29201-14b	A0918P/Sapphire Control S/S/94	61.4	64.0	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
A0918P Formulations																					
28937-1	Solatex II	3.3	3.1	3.7	4.5	4.3	4.1	4.4	4.9	5.1	5.4	5.8	5.9	5.9	6.9	6.8	7.1	6.9	6.9	6.2	6.2
28937-2	Solatex II	3.3	3.0	3.2	5.2	4.3	4.2	4.7	5.0	5.2	-	5.8	-	-	-	-	-	-	-	-	-
28937-3	Sapphire	37.0	40.3	44.4	48.9	51.3	54.7	57.2	59.4	62.6	67.0	69.4	70.4	71.6	73.8	75.4	75.4	75.4	75.4	78.6	78.6
28937-5	Solite	24.0	24.4	27.9	31.2	34.2	33.7	39.2	44.7	45.4	-	49.3	54.8	54.3	62.9	-	-	-	-	-	-
28937-7	Tedlar cover	4.6	5.5	6.0	7.8	7.3	7.0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
28937-9	Airphire	4.4	4.5	5.3	6.4	6.4	6.2	6.4	7.3	7.9	-	6.4	9.4	9.8	10.8	11.8	11.8	11.8	-	13.4	-
A0918P Sapphire cover																					
28938-1	10 mil Tedlar Back	3.6	4.1	4.2	5.5	4.7	4.5	4.6	4.5	4.9	4.5	4.4	4.5	4.8	4.8	4.4	4.3	4.4	4.4	4.8	4.8

* Solite is scratched
(1) ASTM D1925-70 Standard Test Method for Yellowness Index of Plastics, Byk Gardner Colorgard System 1000

Table 2

The Average Yellowness Index of Cured EVA Laminates
After EMMA Aging ⁽¹⁾

<u>Samples</u>	<u>Construction</u>	<u>Color Index</u> ⁽²⁾					<u>0 to 36</u> ⁽³⁾ <u>weeks</u> <u>Δ</u>
		<u>0 weeks</u>	<u>4 weeks</u>	<u>8 weeks</u>	<u>12 weeks</u>	<u>36 weeks</u>	
29214-1,2	Solite/A9918P/Starphire	-1.3	0.5	1.2	1.6	13.8	15.1
29214-3,4	Solatex II/A9918P/Starphire	-1.2	0.0	-0.1	-0.0	1.0	2.2
29214-5,6	Starphire/A9918P/Starphire	-1.6	-0.7	0.2	1.1	15.7	17.3
29214-7,8	Tefzel/A9918P/Starphire	-0.1	-0.6	-0.8	-0.9	-0.9	---
29214-9,10	Solite/15295P/Starphire	-2.6	-1.4	-1.2	-1.0	-0.7	1.9
29214-11,1	Solatex II/15295P/Starphire	-2.3	-1.9	-1.9	-2.0	-1.5	0.8
	Starphire/X9903P/Starphire	-1.4	-1.7	---	---	---	---

(1) EMMA Aging (Equatorial mount with mirrors for acceleration) by DSET Laboratories, Phoenix, AZ; nominal 5 suns

(2) By ASTM D1925-70, Standard Test Method for Yellowness Index of Plastics

(3) * 3.5 years in AZ

Table 3

**ANALYSIS OF EVA-BASED ENCAPSULANTS SHOWS NO
SIGNIFICANT LOSS OF ACETIC ACID**

% VINYL ACETATE CONTENT BY TGA (1)

<u>Standard Cure A9918P</u>		<u>Fast Cure 15295P</u>	
<u>Description</u>	<u>% VA (2)</u>	<u>Description</u>	<u>% VA (2)</u>
Uncured sheet	33.2	Uncured sheet	32.4
Cured, unaged	34.9	Cured, unaged	32.8
Weather-Ometer aged, 12 weeks (light yellow)	33.5	Weather-Ometer aged, 25 weeks (yellow)	32.1/32.6
Carrizo modules (brown)	33.9 to 35.5	Weather-Ometer aged, 34 weeks (amber)	3.0/33.6
<hr/>			
Dupont value for virgin 3185	33.0		
<hr/>			

(1) by complete thermolysis of the acetate group: $\pm 0.5\%$

(2) Corrected for ash residue

Table 4

AVERAGE YELLOWNESS INDEX OF CURED
ENCAPSULANT/GLASS LAMINATES WITH WEATHER-OMETER AGING (1)

<u>Sample Construction (2)</u>	<u>Yellowness Index (3)</u>					<u>Diff. 0-14 wks</u>
	<u>0 weeks</u>	<u>4 weeks</u>	<u>8 weeks</u>	<u>12 weeks</u>	<u>14weeks</u>	
"Standard Cure" Encapsulants						
X9903P/Starphire	0.2	2.6	2.3	1.8	1.8	1.6
A9918P/Starphire (Control)	0.7	7.0	16.7	30.6	--	> 30
A9918P/Solatex II or Solarphire	1.2	6.8	8.0	9.2	9.6	8.4
"Fast Cure" Encapsulants						
X15303P/Starphire	0.4	2.5	2.3	1.3	2.0	1.6
15295P/Starphire (Control)	-0.4	0.4	2.2	5.7	9.0	9.4
15295P/Solatex II	0.0	1.3	1.8	2.2	2.5	2.5

(1) Ci35A xenon-arc Weather-Ometer, 100 degrees C, 0.55 watts/m²/nm at 340 nm

(2) Glass/encapsulant/Glass laminates with Starphire on the back side

(3) By ASTM D1925-70, "Standard Test Method for Yellowness Index of Plastics"

TABLE 5
UV531 AND TINUVIN 770 CONTENT OF GLASS/EVA/GLASS 18 MIL A9918P
AGED 9 - 11 WEEKS ⁽¹⁾

			UV531				TINUVIN 770			
Sample NB No.	History	SL Yellowness Index	GC/MS area/mg x 10 ⁻⁷	Avg.	% Remaining	% Consumed	GC/MS area/mg x 10 ⁻⁷	Avg.	% Remaining	% Consumed
28948-9	Unaged cured full formulation	0	1.48 1.48 1.54	1.50	100	0	1.09 1.21 1.08	1.13	100	0
29154-20	Nine weeks Starphire	19.3	0.64 0.67	0.66	44	56	0.43 0.72	0.58	51	49
29154-61	Nine weeks Starphire	19.6	0.57 0.64	0.61	41	59	0.60 0.61	0.61	54	46
29154-4M	Eleven weeks Starphire	28.4	0.71 0.56	0.64	43	57	0.38 0.32	0.35	31	69
29154-11M	Eleven weeks Solite	24.4	0.80 0.73 0.67	0.73	49	51	0.53 0.62 0.37	0.51	45	55

(1) By Xenon-Arc Weather-Ometer, 0.55 w/m² at 100°C

TABLE 6
CYASORB UV531, TINUVIN 770 AND LUPERSOL 101 CONTENT OF GLASS/EVA/GLASS LAMINATED
UNAGED AND UV-AGED IN LABORATORY

			UV531		TINUVIN 770		LUPERSOL 101	
Sample No.	Composition and History	SL Yellowness Index	GC/FID area/mg x 10 ⁻⁴	% Consumed	GC/FID area/mg x 10 ⁻³	% Consumed	DSC joules/g	phr 101 Present
28950-2A	UV531 100% Std		6.20		8.88			
28950-4A	UV531 50% Std		2.38		9.44			
28948-9	Full Form, Unaged		5.17		9.39		3.47	0.11
29230-4	Full Form Aged 2 Wks		5.04	3 (rel. to 28948-9)	8.70	7 (rel. to 28948-9)	0.39	0.01
29227-4	101 Only, Unaged						3.16	0.10
29227-3	No 101, Unaged	0.5	5.30		17.1		0.72	0.02
29163-3	No 101, Aged 10 Wks	5.4	5.29	0	16.2	5		
29227-6	101 + UV531, Unaged	1.7	5.39				2.92	0.09
29208-5	101 + UV531 Aged 12 Wks	13.5	3.22	40				
29227-7	UV 531 x 2, Unaged	0.6	13.4		10.6		2.01	0.05
28938-9	UV531 x 2 Aged 11 Wks	28.3	6.32	53	3.72	65		
29227-5	101+770 (no UV531) Unaged	0.5			9.18		2.76	0.08
29208-3	Aged 12 Wks	1			3.56	61		
29227-1	770 Only, Unaged	-0.3			14.9			
29203-6	Aged 6 Wks	-0.8			15.6	+5		
29227-2	UV531 Only, Unaged	-0.2	5.99					
29203-11	Aged 11 Wks	-0.9	6.09	0				

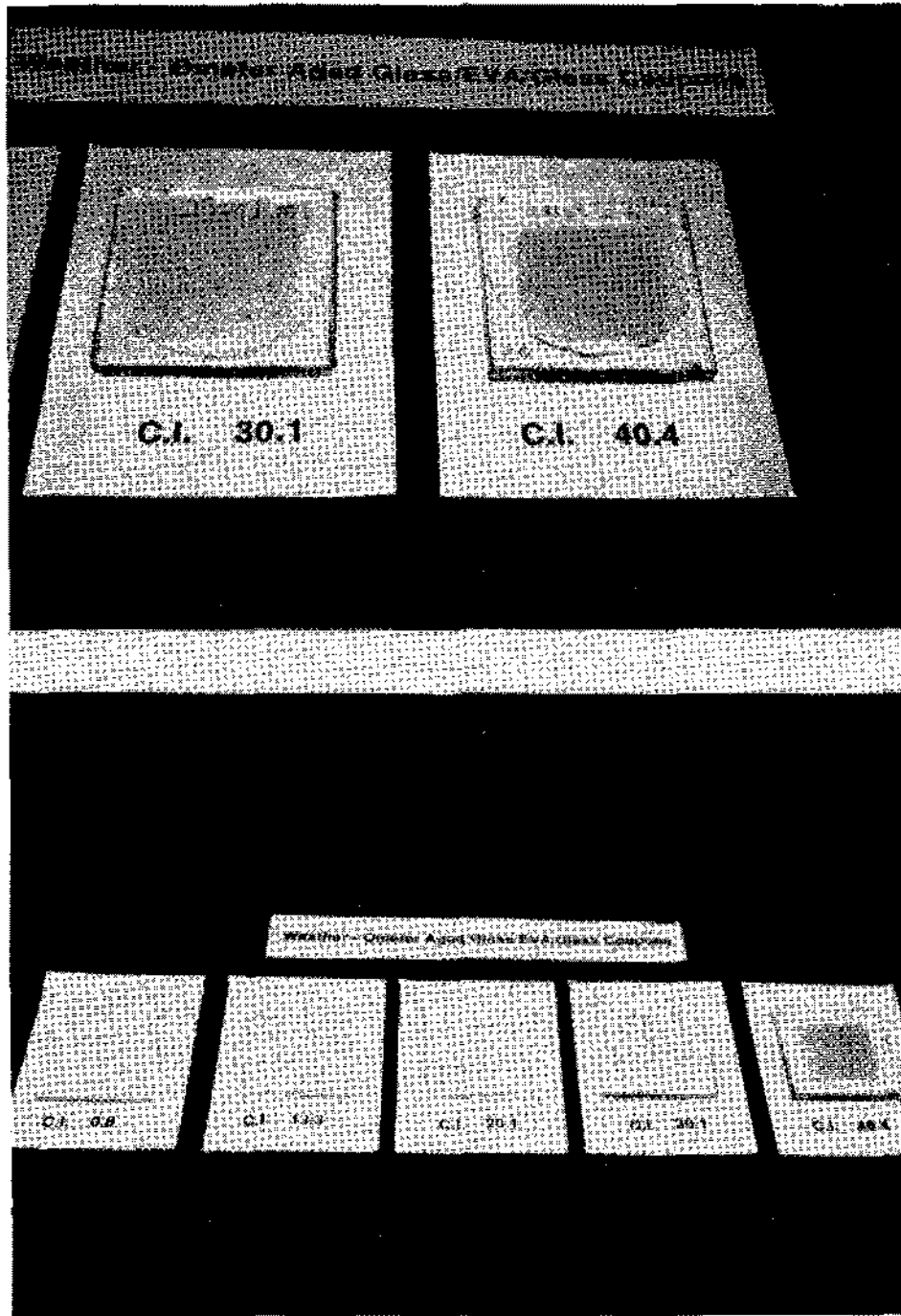


FIGURE 1: Color Indexes: 0.8 through 40.4

Fourier Transform / Infrared Spectroscopy

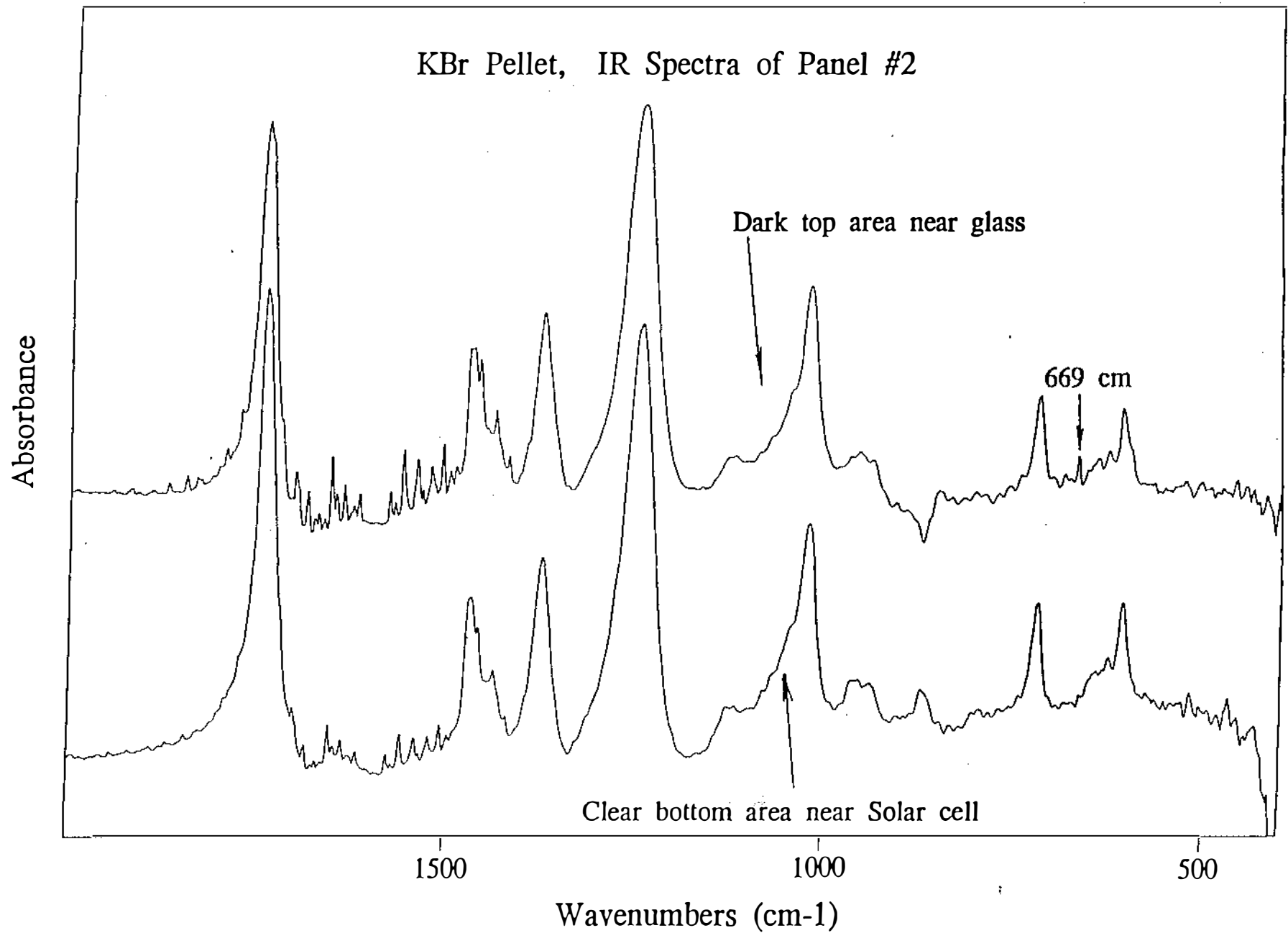


FIGURE 2

Micro Fourier Transform / Infrared Spectroscopy

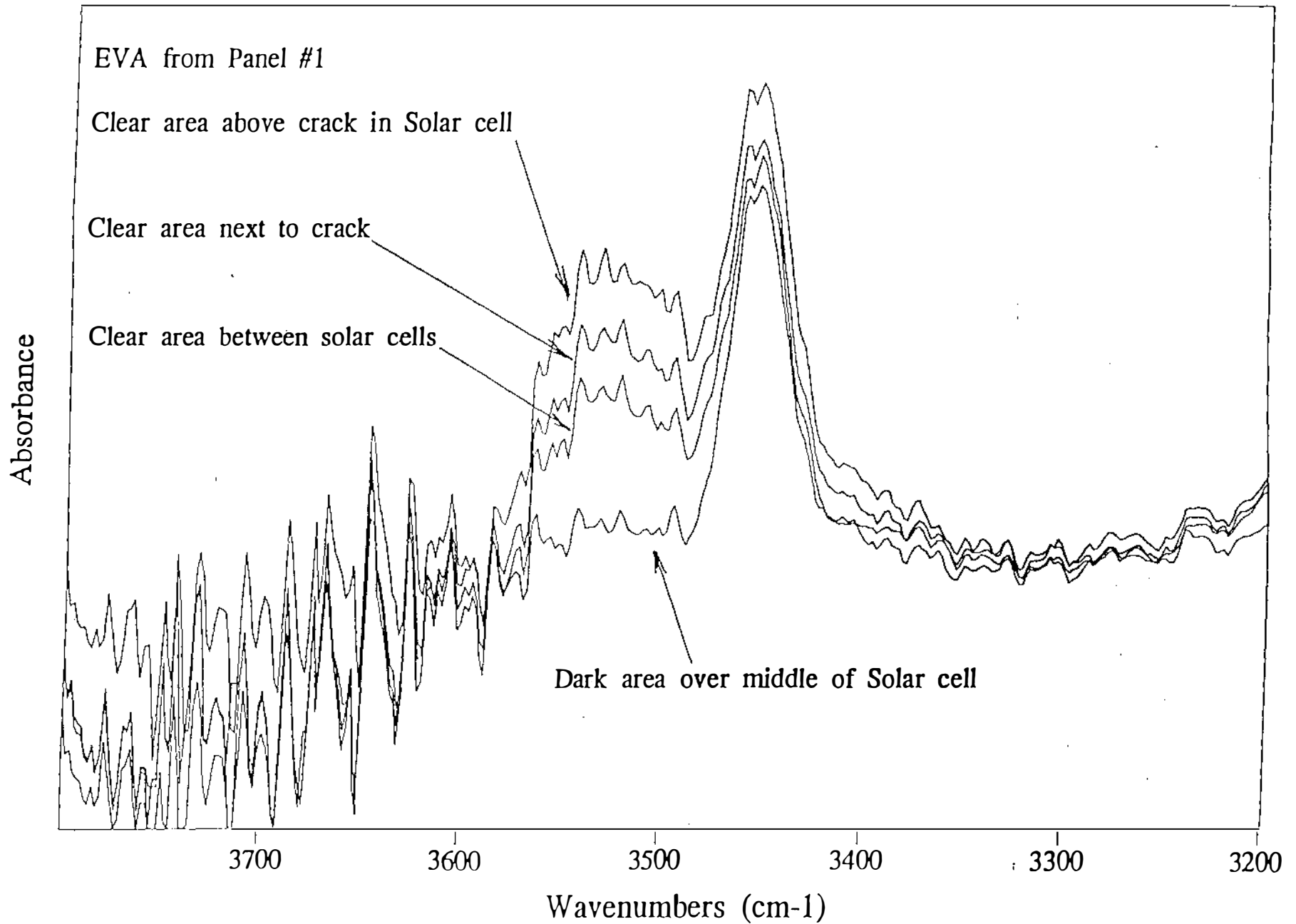


FIGURE 3

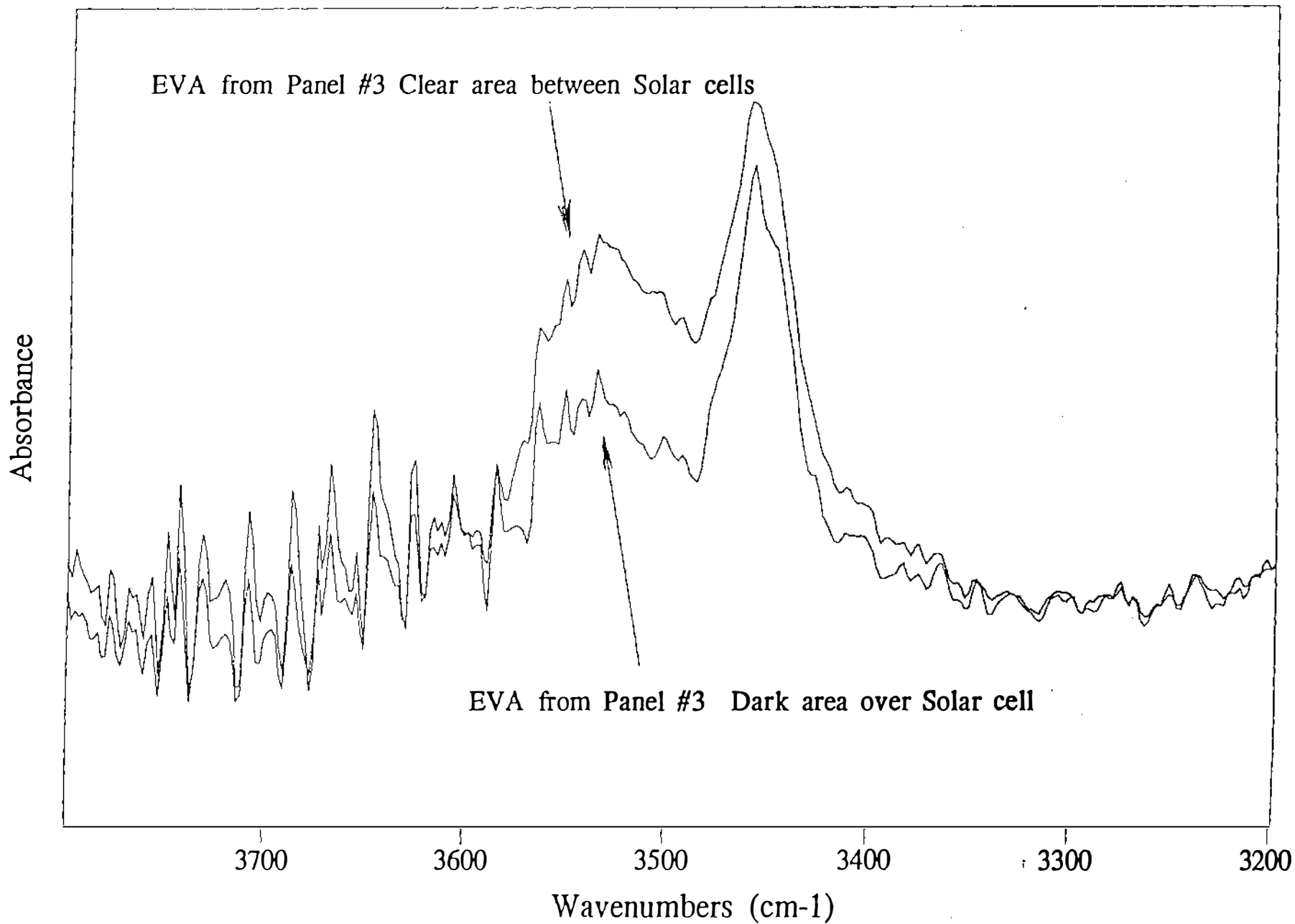
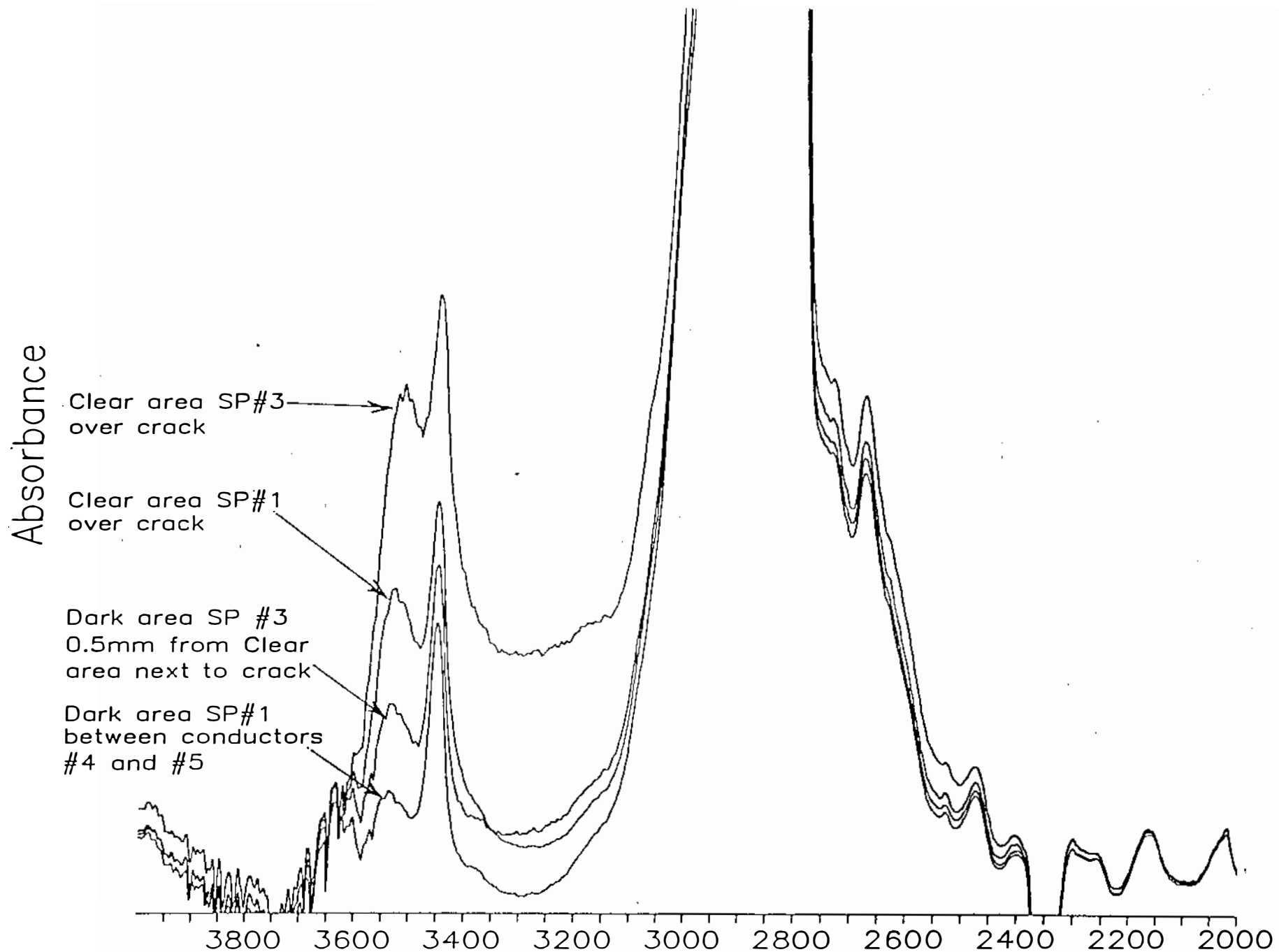


FIGURE 4



Gary Lavigne
U-Conn IMS

Wavenumbers
Resolution (4cm-1)

09/26/94
CLEAR3CK

FIGURE 5

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13. ABSTRACT (<i>Maximum 200 words</i>) This report describes work performed under a subcontract to the National Renewable Energy Laboratory under the Photovoltaic Manufacturing Technology Project. The objectives of this subcontract are to (1) define the problem of yellowing/browning of EVA-based encapsulants; (2) determine probable mechanisms and the role of various parameters such as heat, UV exposure, module construction, EVA interfaces, and EVA thickness, in the browning of EVA-based encapsulants; (3) develop stabilization strategies for various module constructions to protect the encapsulant from degradative failure; (4) conduct laboratory, accelerated outdoor, and field testing of encapsulant, laminated test coupons, and full modules to demonstrate the functional adequacy of the stabilization strategies; and (5) implement these strategies. This report summarizes the accomplishments related to the above goals for the reporting period.				
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