FINAL TECHNICAL REPORT

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SPACE ENVIRONMENTAL EFFECTS ON POLYMERIC MATERIALS

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ELECTRONS AND ULTRAVIOLET RADIATION

Introduction

Two of the major environmental hazards in the Geosynchronous Earth Orbit (GEO) are energetic charged particles and ultraviolet radiation. The charged particles, electrons and protons, range in energy from 0.1 to 4 MeV and each have a flux of 10^8 cm^{-2} sec^{-1}. Over a 30 year lifetime, materials in the GEO will have an absorbed dose from this radiation of 10^{10} rads. The ultraviolet radiation comes uninhibited from the sun with an irradiance of 1.4 Kw/m^2. Radiation is known to initiate chain sission and crosslinking in polymeric materials, both of which affect their structural properties. The 30-year dose level from the combined radiation in the GEO exceeds the threshold for measurable damage in most polymer systems studied.[2] Of further concern, is possible synergistic effects from the simultaneous irradiation with charged particles and ultraviolet radiation. Most studies on radiation effects on polymeric materials use either electrons or ultraviolet radiation alone, or in a sequential combination.[3]

Experimental

An apparatus built at the NASA Langley Research Center was used to investigate the possibliity of synergistic effects when polymer films are simultaneously irradiated with energetic electrons and ultraviolet radiation. The apparatus consists of a chamber 23 cm in diameter and 23 cm high into which a sample can be mounted. The chamber can be evacuated to a pressure of 10^{-8} torr. A Kimball Physics electron gun and an Inficon IQ 200 mass spectrometer are also incorporated in the vacuum system, and an Oriel 6141 system with a 1000 watt xenon-arc ultraviolet lamp is positioned in front of a quartz window so that the sample can be irradiated with electrons and ultraviolet radiation simultaneously or separately. The mass spectrometer can monitor the volatile products from the interactions of the radiations with the polymeric material. A schematic diagram of the apparatus is shown in figure 1.

We have obtained results for several polymer films irradiated separately, sequentially, and simultaneously with electrons and uv radiation. The polymers studied include: Lexan, a polycarbonate; P1700, a polysulfone; and Mylar, a polyethylene terephthalate. The repeat units of the three polymers are shown in figure 2. All three polymers had been studied extensively with both electrons and ultraviolet radiation separately but not simultaneously. Also, volatile products had not been monitored during irradiations of these materials. Film thicknesses were 127 μm (5 mil) for Lexan and Mylar, and 76 μm (3 mil) for the polysulfone.

Electron irradiations were made with 85 keV particles at a dose rate of 530 Mrads/hr. The high electron dose rate was used so that a sufficient

concentration of volatile products would be formed to yield a reasonable mass spectrum. The intensity of the ultraviolet lamp was set to 1.5 solar constants with a calibrated photometer. The electron dose rate corresponded to an energy flux of 0.014 J $cm^{-2} sec^{-1}$, while the intensity of the ultraviolet radiation corresponded to 0.21 J $cm^{-2} sec^{-1}$, a factor of 15 higher than that of the electrons. The temperature of the sample could be monitored with a thermocouple, but it could not be controlled. Thus, in the electron irradiations, the sample temperature was 35 to 40° C, while it went up to 110° C in the ultraviolet irradiations. All irradiations were for 8 hours.

Results and Discussion

The results of the mass spectrometer analyses are summarized in tables 1, 2, and 3. In all cases, the polymer films were irradaited with 85 keV electrons.It can be seen from table 1 that the mass spectrum of the polysulfone shows the largest peaks at mass 48 and at mass 64, which correspond to the chemical structures SO and SO_2. Other workers have shown by gas analysis experiments that SO_2 is a product of irradiation of polysulfone.[4] Since SO is not a stable chemical species, it would not be seen in an ordinary gas analysis experiment. It could be formed as a primary product of irradiation, or as a decomposition product of SO_2 at the mass spectrometer head. To determine the source of SO, pure SO_2 gas was introduced into the chamber and analyzed by the mass spectrometer without any radiation present. The mass spectrum clearly showed peaks at 48 and 64 in approximately the same ratio as observed in the radiation experiments. This indicates that SO_2 is a primary irradiation product, but SO is a decomposition product produced at the mass spectrometer head. The results for Mylar and Lexan show that the highest concentrations of volatile products had masses of 44, 28, 16, and 12, corresponding to products of CO_2, CO, O, and C.

Comparisons of separate, sequential, and simultaneous irradiations with electrons and ultraviolet radiation were made for each polymer. The radiation damage in each irradiation was monitored by noting changes in the uv/visible spectrum from the baseline spectrum taken on unirradiated films. Spectra were recorded with a Perkin-Elmer model 330 spectrophotometer and are shown in figures 3, 4, and 5 for P1700, Lexan, and Mylar respectively. Absorbance spectra were integrated by the spectrometer and the baseline area was subtracted from the area after irradiation. The results are shown in figures 6, 7, and 8 for P1700, Lexan, and Mylar respectively. The notation e-/uv means an 8 hour electron irradiation followed by an 8 hour ultraviolet irradiation. The notation, uv/e-, means the reverse. The Mylar film contained an ultraviolet inhibitor, the effect of which can be clearly seen in figures 5 and 8. However, when this Mylar film is subjected to electron irradiation followed by uv irradiation, the effect exceeds that of the simultaneous or separate electron irradiations by more than 17 percent. This

probably occurs because the electron irradaition degrades the ultraviolet inhibitor in addition to degrading the polymer.

The results shown in figures 6 and 7 are best explained in terms of a radical mechanism. It is well known that radicals are present during irradiations with both electrons[5] and uv radiation[6]. With the high radiation doses used in these studies, it is reasonable to assume that during the irradiation the radical concentration increases to a steady-state situation in which radicals are formed and destroyed at the same rate. In a simultaneous irradiation with electrons and uv radiation, the steady-state concentration of radicals would be attained more rapidly than in separate irradiations. Thus, in the absence of any synergistic effects, one would expect the effects of a simultaneous irradiation to be less than or equal to the sum of the effects of the separate irradiations, depending on the time necessary to achieve the radical steady-state condition. Figure 6 shows that this the case for the irradiations of P1700. For sequential irradiations, one would expect the effect to be equal to the sum of the effects from the separate irradiations. This is clearly not the case for P1700 as seen in Figure 6. It is possible that radicals formed during the initial irradiation do not decay in vacuum and thus a large radical concentration is present at the start of the sequential irradiation. This could be tested by interupting the vacuum between sequential irradiations. This does not explain the large difference between the two sequential irradiations. Clearly, some extra synergistic effect occurs when the electron irradiation is followed by the uv irradiation. At present, this difference is not understood.

Polycarbonates such as Lexan are known to be resistant to degradation by ultraviolet radiation.[7] This can be seen in figure 7. For Lexan, the effects in the sequential irradiations are roughly equal to the sum of the effects in the separate irradiations. This probably occurs because the effects arise mostly in the electron irradaitions. The effects from the simultaneous irradiation, however, are about 7 percent higher than any of the others. This may be a true synergistic effect which leads to an enhancement when both electron and uv radiation are used together.

Conclusions

The mass spectrometer data indicate that chain sission occurs primarily at the carbonyl group in Lexan and Mylar and primarily at the sulfone group in the polysulfone. These results are in line with studies reported in the literature.[8,7] The mass spectrum peak at mass 48 found during the irradiation of P1700, is due to SO formed at the mass spectrum head rather than as a primary degradation product.

The polysulfone, P1700, shows significantly more effect in sequential irradiations than in a simultaneous irradiation. In particular, the effect in the electron followed by uv irradiation is more than twice that of the simultaneous irradiation. The large difference likely has to do with residual

radicals from the initial irradiation being present at the start of the second irradiation. This will be checked by exposing the sample to air between irradiations which should quench many of the radicals formed in the initial irradiation. The reason for the large difference in effects between the two different sequential patterns (uv/e- vs. e-/uv) is not known at this time.

As a typical polycarbonate, Lexan shows little effect from uv irradiation. However, it shows an apparent synergistic effect when it undergoes simultaneous irradiation with 85 keV electrons and uv radiation. The effect is noted best in figure 6.

The ultraviolet inhibitor in the Mylar film is apparently degraded during electron irradiations. The effect is most readily seen when an 8 hour electron irradiation is followed by an 8 hour uv irradiation, but is also seen to a lesser extent in a simultaneous irradiation.

ATOMIC OXYGEN

Introduction

Atomic oxygen (AO), the most common atomic or molecular species at altitudes of Low Earth Orbit (LEO), presents a significant hazard to exposed organic materials on long-term missions in LEO. It is unusually reactive: AO quickly oxidizes most organic compounds, regardless of their complexity. Skylab and several shuttle missions have tested the effects of AO on polymeric films and coatings in orbit. Erosion takes place on the surfaces of these materials typically at rates that correspond to losses of 0.3 mm of material from forward facing surfaces (which receive the greatest flux) over ten years.[8,9] These high rates could jeopardize the utility of thermal blankets, adhesives, composite structural components, and protective coatings that have organic components.

The experiments carried out in LEO have shown that polymer films with fluorine or silicon atoms in their formulation are afforded some protection from AO.[9] For example, carbon atoms in the fluorocarbons TFE and FEP (both of which have the empirical formula CF_2) are already in a highly oxidized state and, consequently, these materials were found to be less vulnerable to AO.[10,11] Also, copolymers of an ether imide and a siloxane showed excellent resistance to erosion by AO, presumably because nonvolatile silicon oxides remain on the surface after a film has been exposed to AO and shield the unexposed material from further attack.[9]

For this study we proposed to simulate the effects of AO in LEO with an RF plasma generator. Our goals were (1) to collect the oxidation products from several polymer systems in order to learn more about the degradation

mechanism and (2) to test the capability of silicon-based diluents to reduce the susceptibility of polymers to oxidative degradation.

Experimental

Most samples studied were in the form of films which were prepared by casting from solutions that held about 15% solids. Chloroform was the solvent for polysulfone, a 60/40 chloroform-tetrachloroethane mixture (by weight) was used for Ultem, and toluene was the solvent for polystyrene. In preparing each film, the concentrated solution was poured onto plate glass, drawn with a doctor's blade, and placed in a dry box under flowing nitrogen for ca. 2 days during which time most of the solvent evaporated. The film was then heated gradually in an oven to temperatures higher than the boiling point of the solvent. In some instances a vacuum oven was used during the final heating to dry the sample at pressures below 1 Torr. It was necessary to move through the final steps slowly in order to avoid bubbles.

The effects of AO in space were simulated using an SPI Plasma Prep II Model 11005 plasma generator. The device is commonly referred to as an asher since it was designed to remove organic materials from glassware, etc. by rapid oxidation. Molecular oxygen flowed into the chamber (15 cm. long by 10 cm. in diameter) where it was dissociated by an RF field at 13.56 MHz. The pressure was maintained at ca. 200 mTorr by constant pumping with a mechanical pump. For some runs with the asher, three U-tubes were placed serially between the exhaust port of the chamber and the vacuum pump. The first tube was surrounded by a Dry Ice/acetone bath, while the other two were immersed in liquid nitrogen.

Most samples used in these experiments were circular films 2.2 cm. (7/8ths inch) in diameter. They were loosely clamped on top of the horizontal surface of a Pyrex microscope slide that was mounted on a Pyrex platform so that the samples were positioned near the center of the chamber. The flat faces of the samples were perpendicular to and facing the oxygen stream, i. e., they were in the ram position. Three samples, positioned side-by-side under the oxygen-inlet manifold, were exposed in each run with the asher. Mass losses for three identical samples in the same run typically varied by 10-20% owing the their positions in the chamber. Most runs lasted 90 min. In addition to films, some studies were made on carbon fibers (HMS batch 34-1) and on flat rectangular pieces of carbon/polyimide composite (C6000/Ultem 1000). The fiber samples were loosely coiled around a Pyrex holder. The 2.5x1.3-cm. composite samples lay flat on a Pyrex base; six such samples were exposed in each run along with one 2.2-cm diameter circle of Ultem film with the latter serving as a reference.

Results and Discussion

1. Product Collection Fully oxidized products were obtained during runs in which the cold traps were in line to collect condensable products in the exhaust from the asher. That is, in every such experiment the first U-tube at -78°C (Dry Ice/acetone) collected ice; the second tube at -196°C (liquid

N_2), carbon dioxide. When the sample in the asher was a polyimide (a nitrogen-containing compound), nitrogen dioxide (NO_2) was collected in the second tube. Experiments on polysulfone yielded sulfur dioxide (SO_2) in the second tube. Nothing was ever found in the third tube. These product compounds were identified by infrared spectroscopy. There was no evidence of other compounds. We conclude that even if other volatile species were formed in the reactions in the asher, they would have been further oxidized to H_2O, CO_2, NO_2, and/or SO_2 before they could be collected and analyzed. Because the cold traps were ineffective in isolating intermediate degradation products, they were removed from the system.

2. Comparison of Asher Results with STS-8 Results Mass-loss results for films without additives, for a graphite-polyimide composite (C6000/Ultem 1000), and for Nomex and Kevlar 49 fibers are shown on the right-hand side of figure 9. The results for the films were computed assuming that all the mass was lost from the upper side of the sample disk that was directly exposed to the oxygen (the under side of the disk having been protected by the glass holder on which it was resting). The rate of mass loss from the fiber was computed assuming that the fibers are infinitely long circular cylinders. (Measurements on the Kevlar and Nomex aramide fibers were made after the expiration of this grant and will be described in detail in the semi-annual status report for Cooperative Agreement covering these studies.) On the left-hand side of figure 9 are material losses reported for samples flown in LEO on STS-8.[8] This side of the figure was scaled so that the results for polystyrene are at approximately the same height as on the right-hand side.

While there were considerable uncertainties in the determinations of mass loss rates both aboard STS-8 and with the asher, it is apparent from figure 9 that the asher is useful in ordering these materials for their susceptibility to AO.

3. Effects of Adding PDMS Small quantities of Dow 220 polydimethylsiloxane (PDMS), a viscous fluid, were added to solutions of polystyrene, P-1700 polysulfone, and Ultem polyimide with chloroform usually serving as the solvent. In every instance films, prepared from these solutions as prescribed above, turned cloudy as the solvent evaporated indicating that the PDMS was not miscible with the polymers. Nevertheless, the PDMS caused a significant reduction in the rate of erosion. Below are ratios of the mass of sample lost during the second 90-min period of exposure of the sample to oxygen. The numerator is the mass loss between 90 and 180 min for film without PDMS; the denominator is the mass loss during the same period for the film impregnated with PDMS. (For each entry in the table the two films were in the asher at the same time.)

	mass loss for untreated film
Film	mass loss for PDMS-loaded film
Polystyrene	
0.4% PDMS	1.7
0.9% PDMS	4.0
Polysulfone (P1700)	
0.4% PDMS	1.1
1.8% PDMS	1.0
18.9% PDMS	6.7
Polyimide (Ultem)	
1.4% PDMS	2.0, 2.2
1.4% PDMS (film scraped after casting)	1.9
4.3% PDMS	2.6

Consequently, we concluded that, over the concentration ranges studied, the more PDMS added the greater the protection it offers.

Films without PDMS apparently degrade at an accelerating rate with time. For example, a 7/8-inch disk of Ultem polyimide whose original mass was 83.5 mg. was treated in the asher for 750 min during which period its mass was reduced to 20.4 mg. The first three columns in the tabulation below illustrate this acceleration phenomenon with this sample.

time (min)	Ultem w/o PDMS		Ultem + 4.3% PDMS	
	mass (mg)	loss rate (mg/hr)	mass (mg)	loss rate (mg/hr)
0	83.5		99.1	
90	78.3	3.5	93.7	3.6
180	72.6	3.8	91.5	1.5
270	66.0	4.4	88.9	1.7
360	59.3	4.5	86.4	1.7
450	50.6	5.8	84.0	1.6
750	20.4	6.0	75.5	1.7

We attribute the increasing mass-loss rates in the third column to changes in the surface as the Ultem is eroded in the asher. A less smooth surface is apparent in electron micrographs. On the other hand, as is evident from the final two columns, the addition of PDMS not only reduces the erosion rate, but the rate remains constant over time. The two samples described here were in the asher at the same time.

By itself, the PDMS fluid was hardly affected in the asher. A 1-gram sample of the fluid, spread out on the glass plate so that its exposed surface area was comparable to that of the 7/8-inch disks of film, lost only 4 mg during a 4-hour treatment.

Apparently, the PDMS in these experiments afforded protection to the films by undergoing oxidation to nonvolatile silicates which remained as a barrier between the film and the oxidizing medium. (See the results of EDAX studies described below.) This process took place even though the PDMS mixes very slightly with polyimide, polysulfone, and polystyrene. Presumably the PDMS is present throughout the film mostly as small encapsulated microdroplets.

4. **Effects of Adding Teflon or Glass Powder** In another series of experiments, glass (SiO_2) or Teflon powder was added to Ultem polyimide and P1700 polysulfone solutions before films of these polymers were cast. These additives were chosen because of their inertness to AO. In each of several exposures of these films in the asher, there was no evidence that the glass or Teflon, which made up as much as 5% of the mass of the films, reduced the degradation rate.

5. **Effects of Adding Low-Molecular Weight Organosilicon Compounds** It seemed likely that the kind of protection provided by PDMS could be improved if silicon-containing substances were more uniformly dispersed throughout the films. Accordingly, we sought nonvolatile silicon-containing compounds that are more miscible in the films than PDMS was found to be. The potential additives diphenylsilane, diphenylmethylsilane, and octaphenyl cyclotetrasiloxane proved to have greater solubilities, ranging from 2 to 10% in the polyimide. However, mass-loss measurements with the asher showed negligible differences between untreated Ultem and Ultem containing these additives. Additional experiments are planned with other organosilicon compounds as well as with organic compounds containing other group-IV elements.

6. **Measurements on Polyimide-Polysiloxane Copolymers** Five specialty films were supplied by the General Electric Company for testing in the asher. Although their formulations are incompletely known, all five copolymers apparently have an Ultem-like component. Three of these were copolymerized with PDMS, one was copolymerized with a diphenyl dimethyl siloxane, and one with a fluorinated siloxane. All five typically experienced mass losses in the asher that were only 30-40% of those for commercial Ultem reference samples (i. e., samples that had not been dissolved and recast) exposed at the same time.

Samples of one of the Ultem-PDMS copolymers were submitted to the NASA-LaRC SEM facility for an analysis by EDAX of the elements on the surfaces of the films after 0, 90, and 180-min treatments in the asher. Comparisons between Ultem film and a film of the Ultem-PDMS copolymer receiving similar exposures in the asher are shown in figure 10. It is apparent from these EDAX tracings that the surfaces of silicon-containing films have a build-up of silicon and oxygen at the expense of carbon when treated in the asher.

7. **Mass-Loss Experiments on Carbon Fiber-Ultem Composites** C6000/Ultem 1000 composites were tested with 4-hr runs in the asher. Within the reproducibility of the measurements (± 3 mg) the mass losses, which averaged

9.1 mg per sample, were independent of whether the fiber had been sized or not or whether the sample had been prepared with the fibers transverse or longitudinal to its exposed surface. The rate of mass loss divided by the area of the upper surface directly exposed to the incident oxygen was 12 μg/min/cm^2 . The commercial Ultem reference samples, that were in the asher at the same time, lost mass at 6 μg/min/cm^2 , i. e., half the rate for the composite.

8. Mass-Loss Experiments on Graphite Fibers Graphite fibers were found to oxidize rapidly in the asher. For example, a bundle of fibers, whose mass was 152 mg, lost mass at a rate of 0.2-0.4% per minute over the first four hours in the asher. At that point half the sample had eroded away. The erosion rate is a function of the thickness of the individual fibers, a value that was not determined for these experiments.

<u>Conclusions</u>

As is evident from figure 9, the degradation of polymer films in the asher tracks the degradation observed in experiments performed in LEO. We believe the asher to be a suitable screening device for qualitative estimates of the resistance of organic materials to AO, even though the oxidizing medium contains species other than AO. The effects of AO under conditions more closely resembling those in LEO can be studied more quantitatively using expensive AO generators being constructed for this purpose in several laboratories around the country.

The introduction of silicon-containing substances in films of polysulfone and polyimide can cause significant reduction in the degradation of the films resulting from a strongly oxidizing gaseous environment. The mechanism, which is corroborated by the EDAX studies on the Ultem-PDMS copolymer, involves the formation of a silicate deposit on the surface of the film which serves as a barrier between the vulnerable organic film and the oxidizing environment in the asher. We believe that a similar mechanism would be operative with the attack by AO in LEO.

It is the long-term goal of these studies to develop useful organic struc- tural materials that can be exposed to AO in LEO for long times without significant loss of integrity. The approach has been to seek additives, such as the silicon compounds described here, which permeate the material and which, on exposure to AO, form a protective coating. Such systems have the potential to be "self-healing" in the not-unlikely event of a nick or scratch that could occur during assembly or in collision with orbiting debris. The possibility of using siloxane copolymers has been considered; however, these have have been found to have unacceptably large coefficients of thermal expansion (CTE). In an effort to achieve the goal, subsequent studies will include a search for nonvolatile organic materials which (1) contain group-IV elements, (2) are soluble in polyimide and polysulfone films, (3) do not raise the CTE, and (4) do not seriously reduce the mechanical properties.

MISCELLANEOUS

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TABLE 1. MASS SPECTROMETER DATA TAKEN DURING
ELECTRON IRRADIATIONS AT 5.3×10^8 RADS/HR

POLYSULFONE P1700
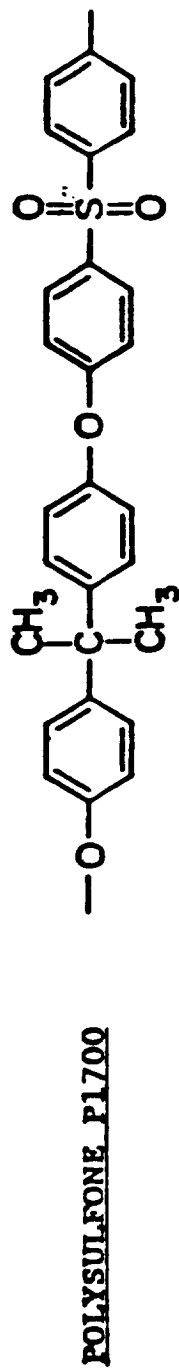
Mass	Relative Amplitude	Possible Products
48	5.6	SO
32	5.2	O_2, SO_2^{+2}
64	4.0	SO_2
12	1.5	C

TABLE 2. MASS SPECTROMETER DATA TAKEN DURING
ELECTRON IRRADIATIONS AT 5.3×10^8 RADS/HR
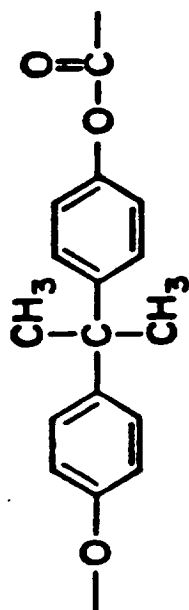
LEXAN

Mass	Relative Amplitude	Possible Products
28	8.0	CO, N_2
44	2.5	CO_2
16	1.5	O, CH_4
12	1.5	C
18	1.0	H_2O

TABLE 3. MASS SPECTROMETER DATA TAKEN DURING ELECTRON IRRADIATIONS AT 5.3×10^8 RADS/HR
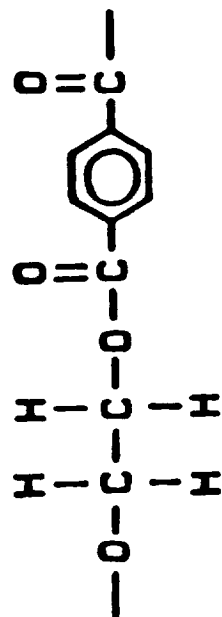
MYLAR

Mass	Relative Amplitude	Possible Products
44	12.0	CO_2, C_2H_4O
12	8.5	C
28	5.5	CO, N_2
16	5.2	O

14

Vacuum in Chamber 2 to 5x10^{-8} torr

Mass Spectrometer

Quartz Window

Ultraviolet Lamp

Sample

Sample Port
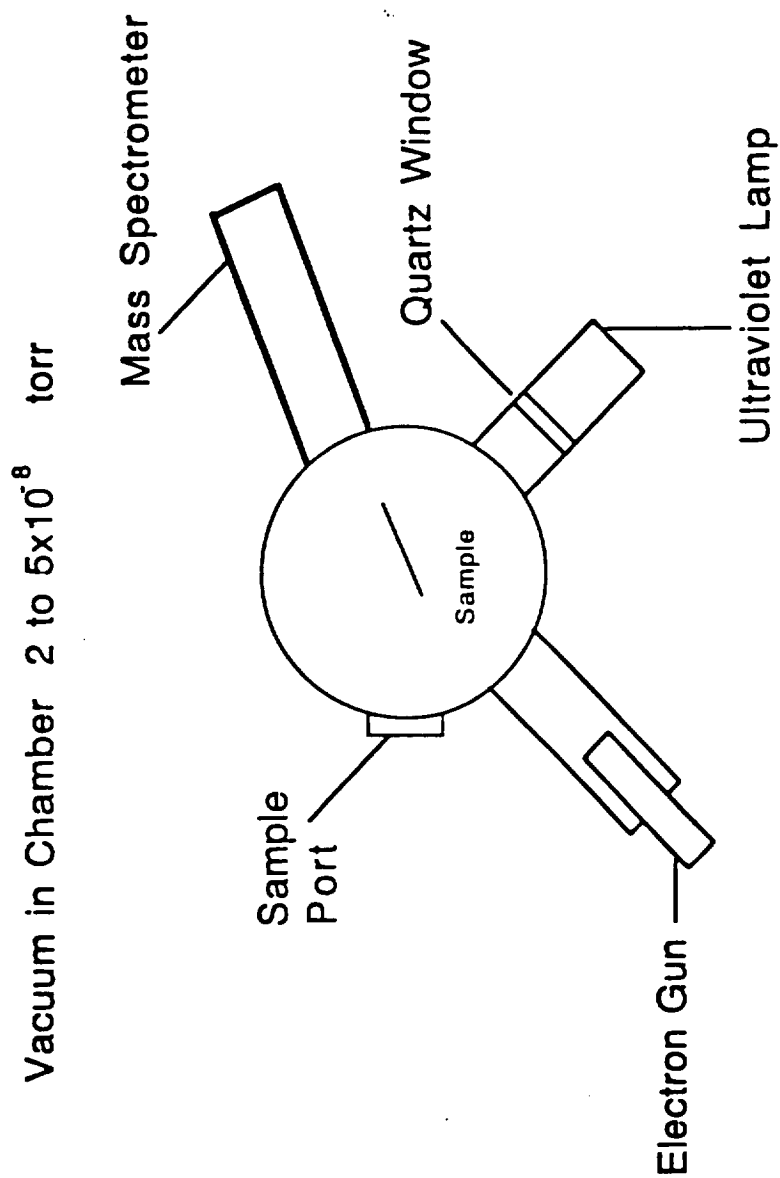
Electron Gun

Figure 1.   Schematic diagram of the apparatus for simultaneous electron and ultraviolet irradiations and mass spectrometry.
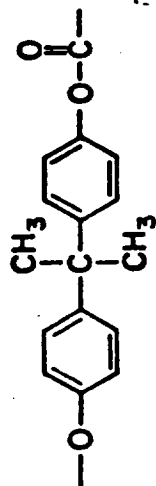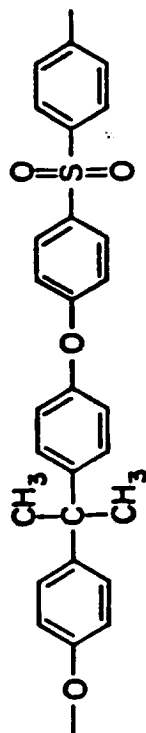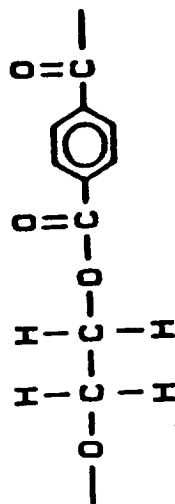
Figure 2. Repeat units of the polymeric materials studied.
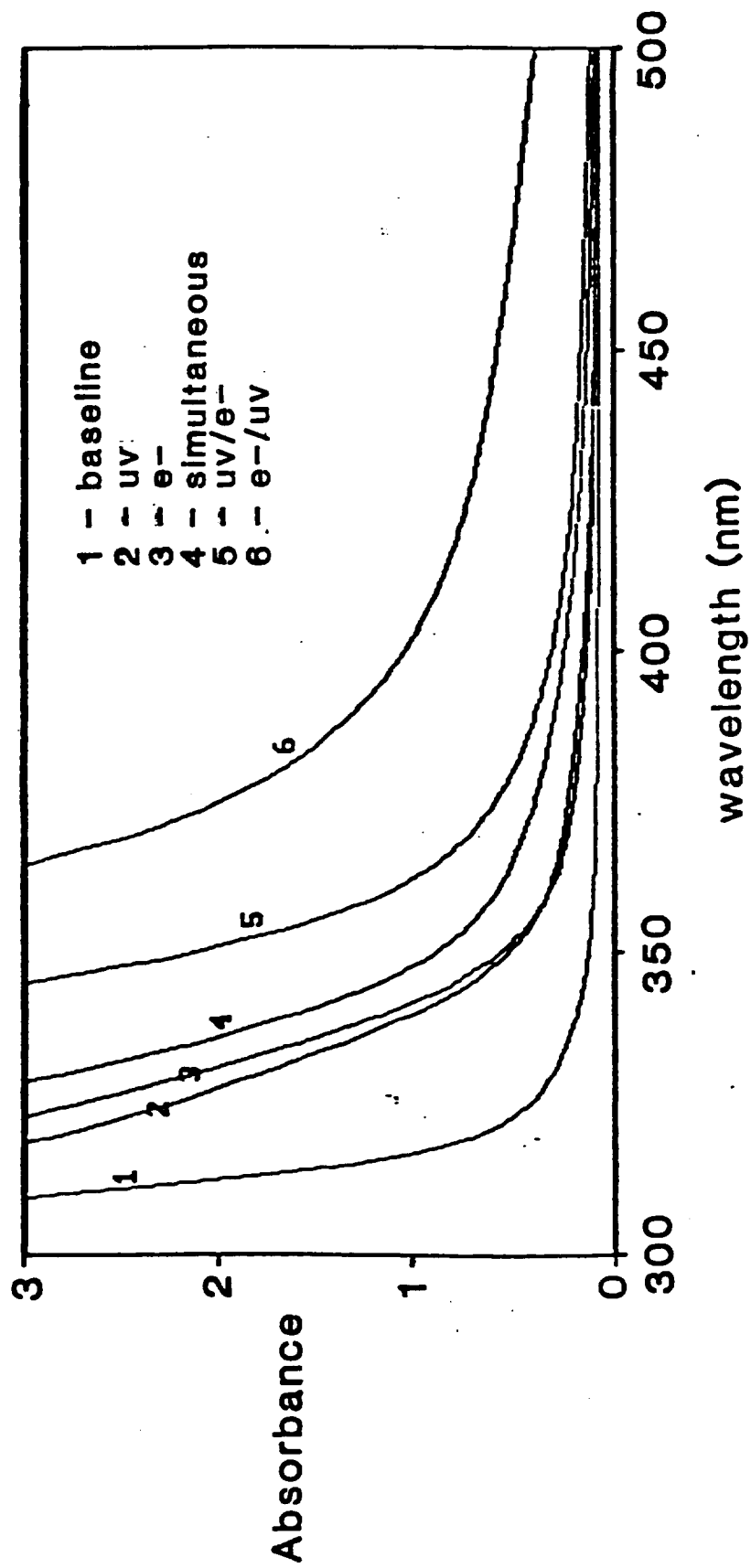
Figure 3. UV/visible spectra for P1700 polysulfone irradiations.
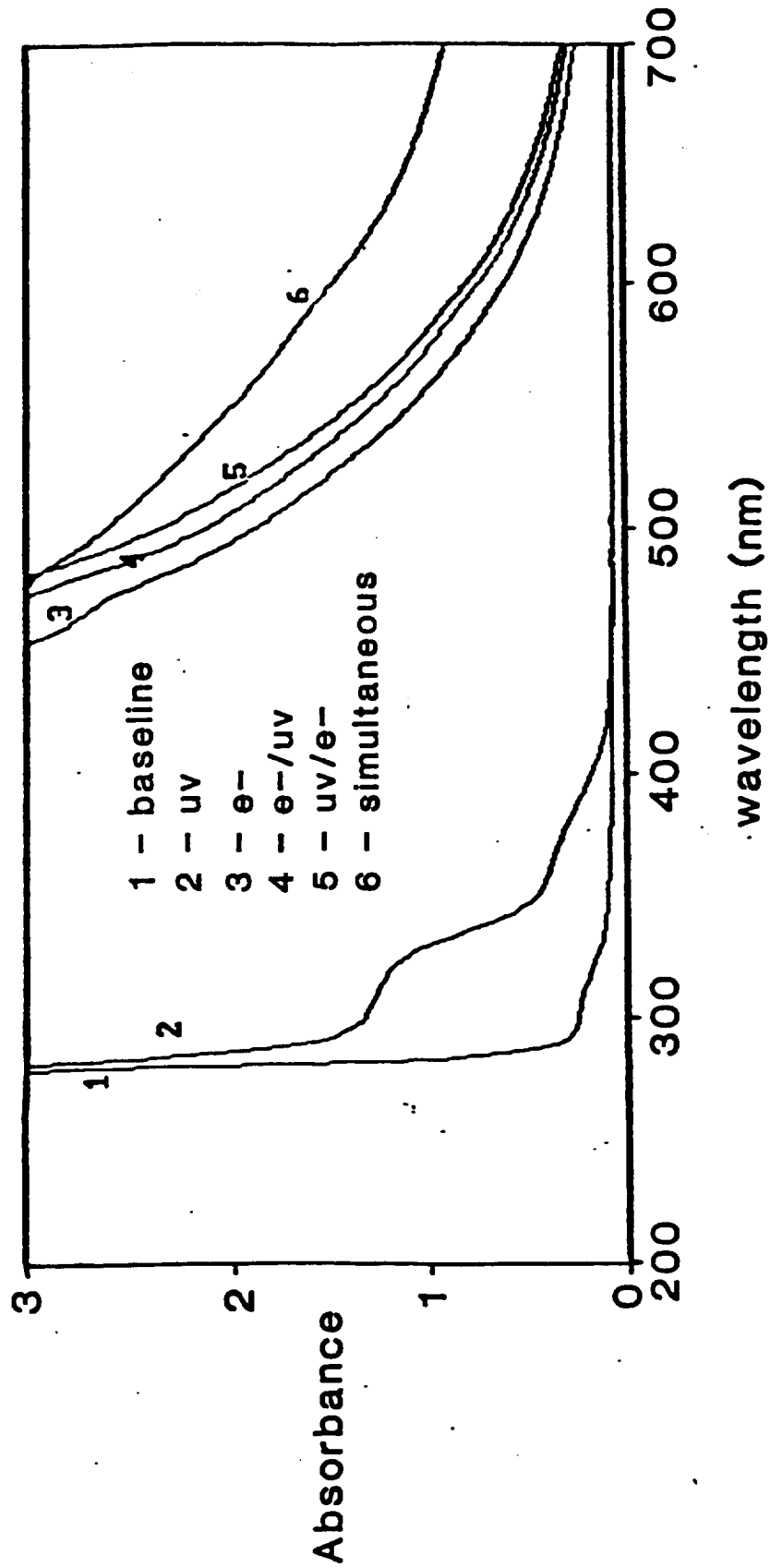
Figure 4. UV/visible spectra for Lexan irradiations.
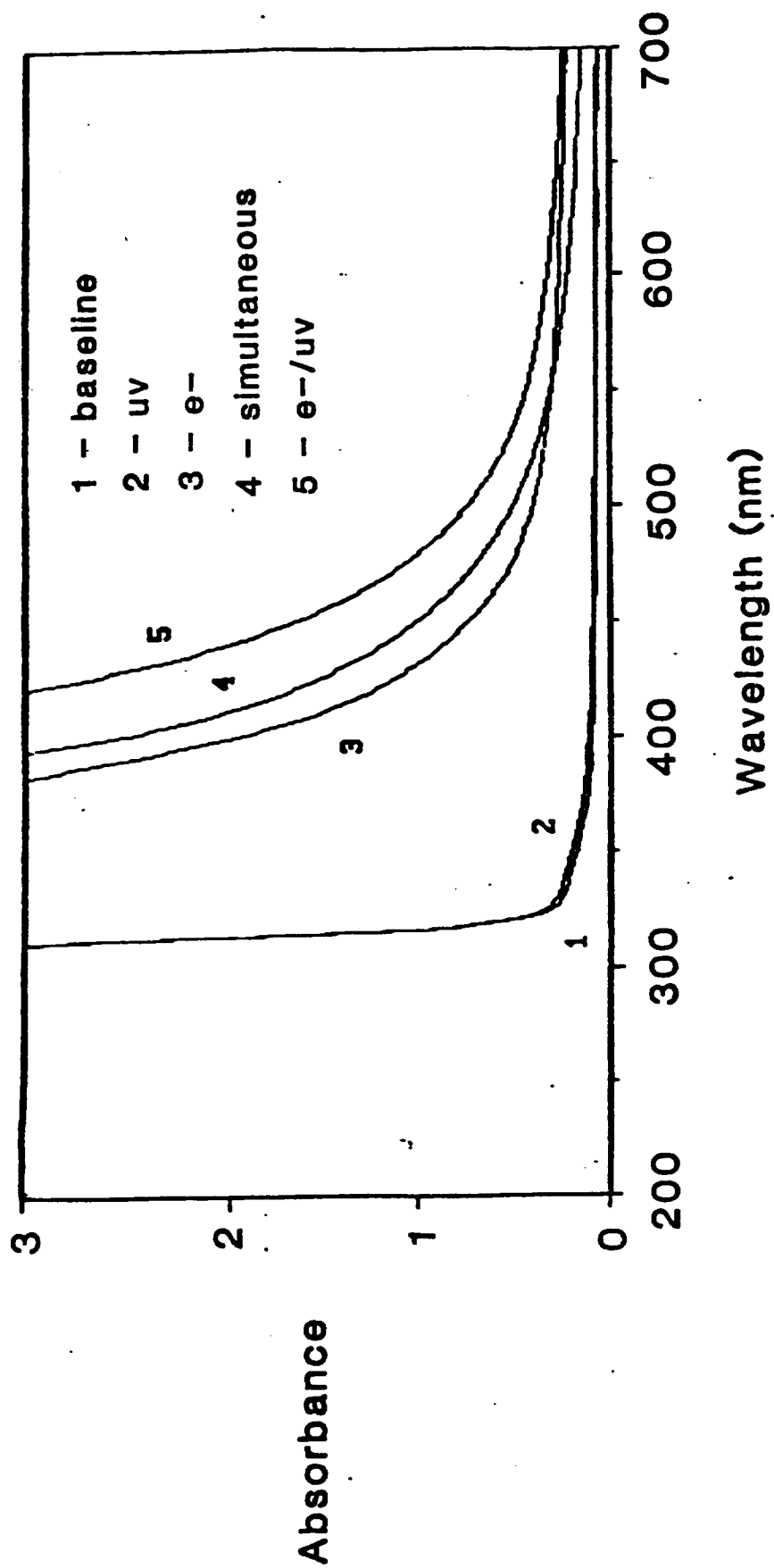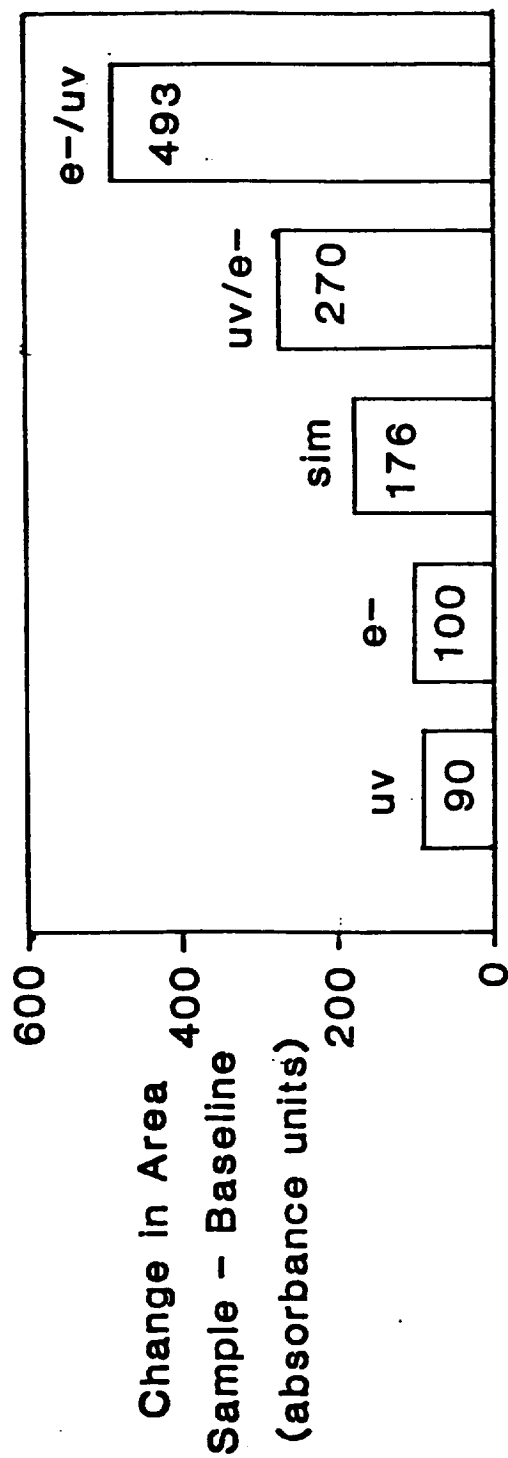
Figure 5. UV/visible spectra for Mylar irradiations.

Figure 6. Change in area in the UV/visible spectra
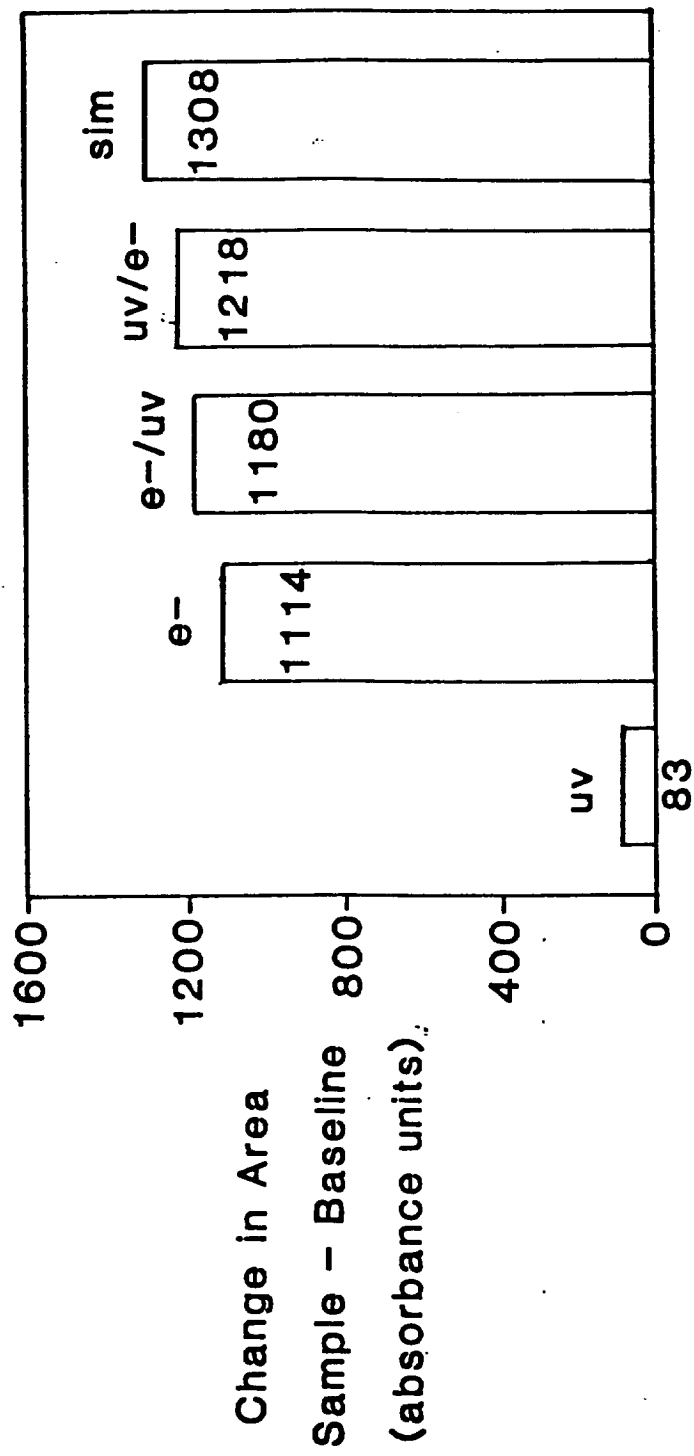for P1700 polysulfone irradiations.

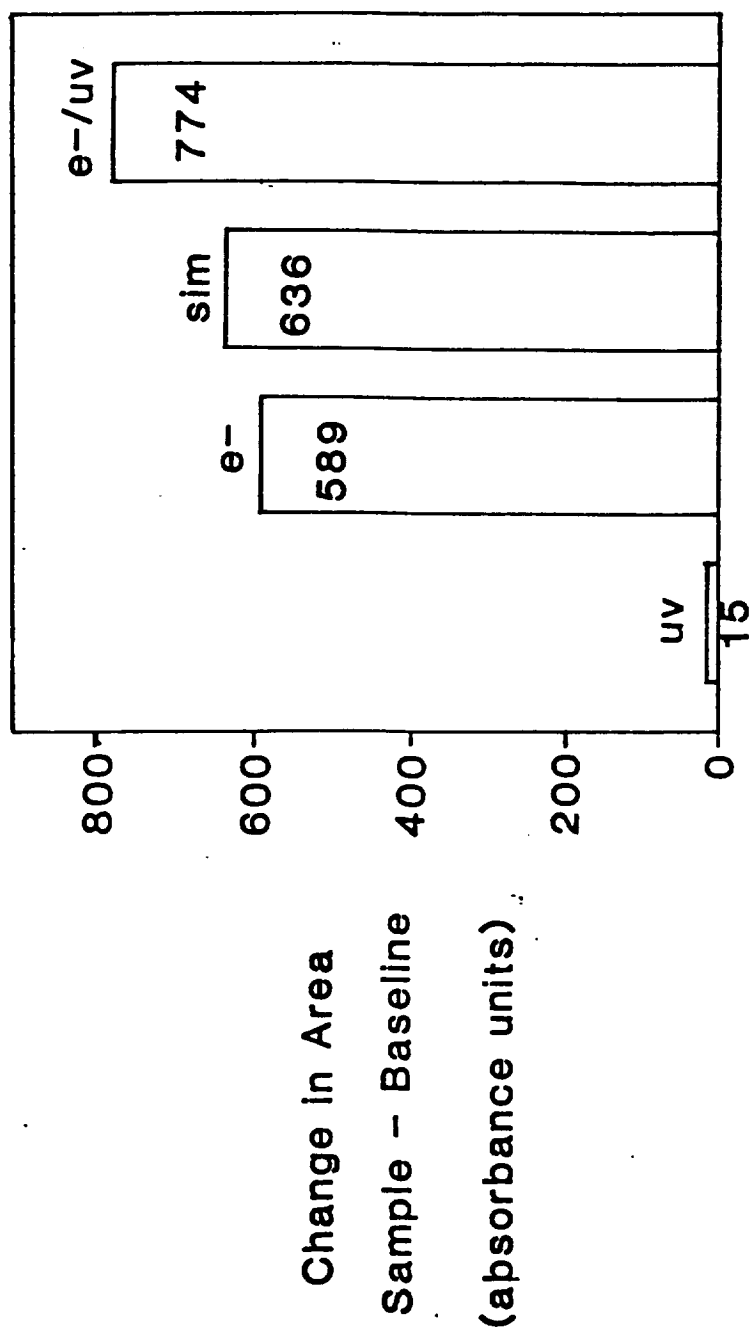Figure 7.   Change in area in the UV/visible spectra for Lexan irradiations.

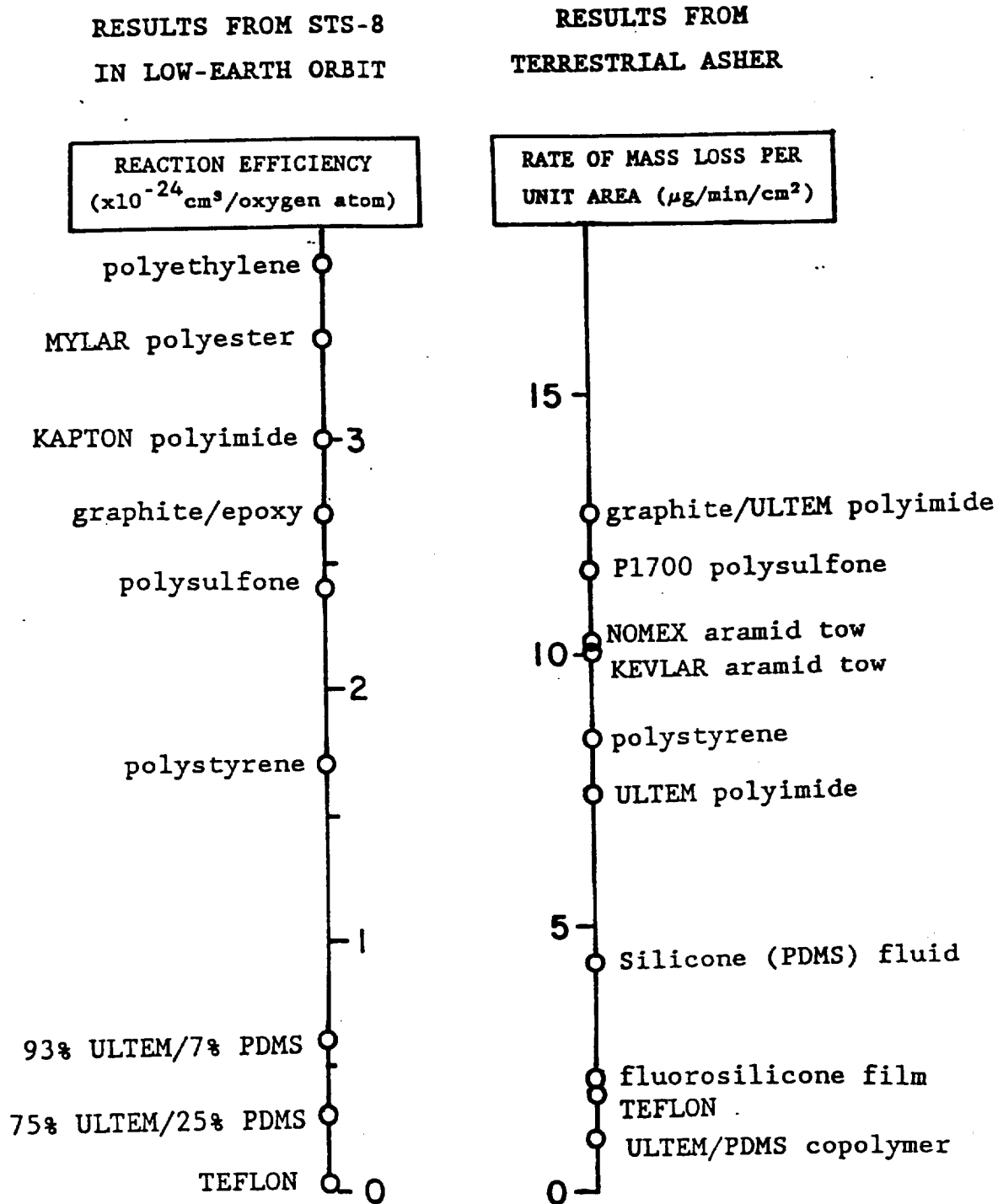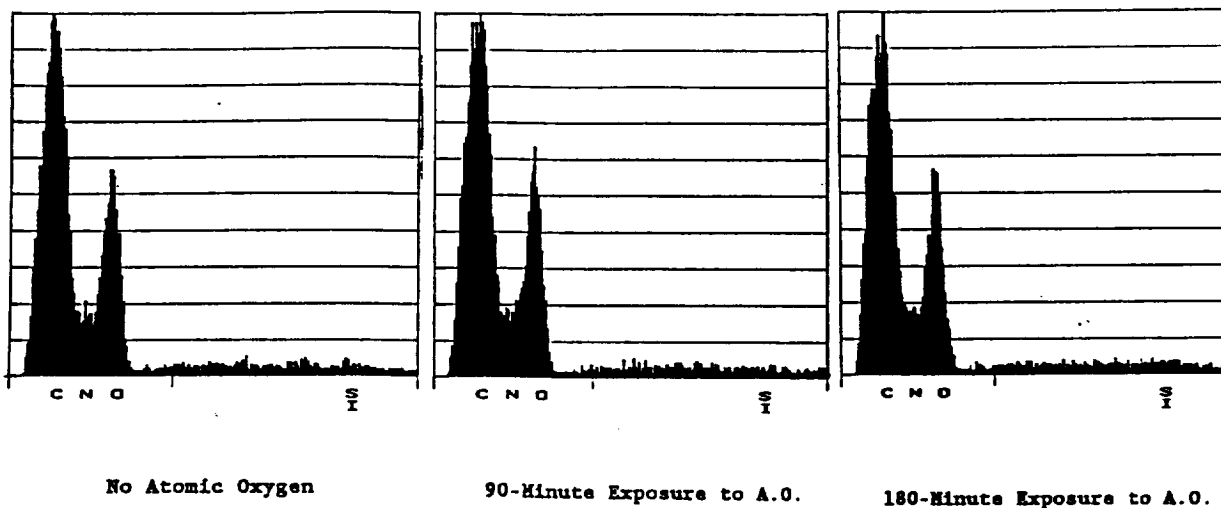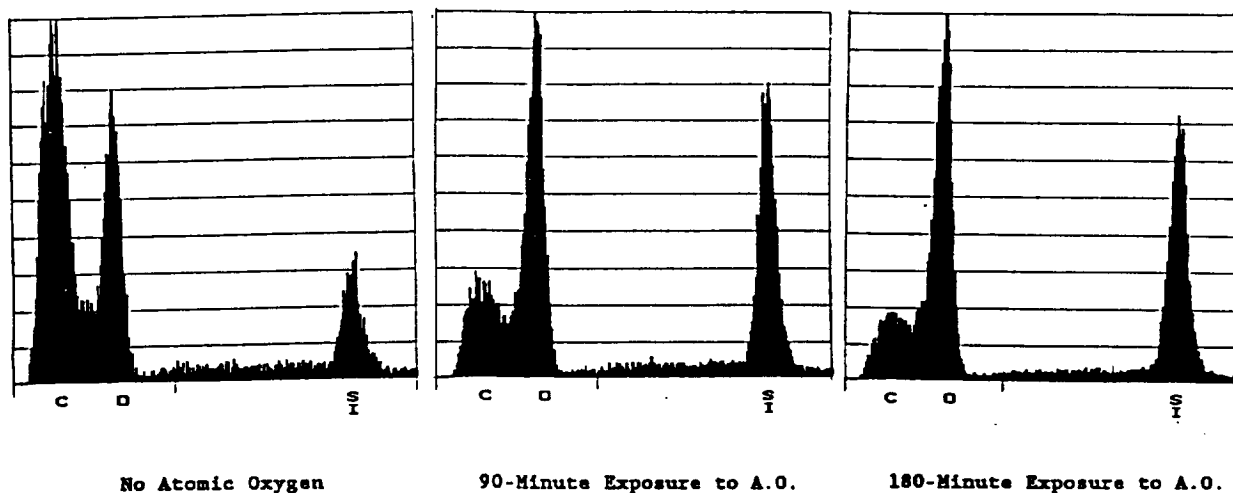Figure 8. Change in area in the UV/visible spectra for Mylar irradiations.

RESULTS FROM STS-8
IN LOW-EARTH ORBIT

RESULTS FROM
TERRESTRIAL ASHER

Figure 9. Comparison of reaction efficiency of polymeric materials measured
on STS-8 with the rate of mass loss in an asher.

No Atomic Oxygen   90-Minute Exposure to A.O.   180-Minute Exposure to A.O.

EFFECT OF ATOMIC OXYGEN ON THE SURFACE COMPOSITION OF ULTEM

Determined with EDAX

No Atomic Oxygen   90-Minute Exposure to A.O.   180-Minute Exposure to A.O.

EFFECT OF ATOMIC OXYGEN ON THE SURFACE COMPOSITION

OF ULTEM/POLY(DIMETHYL SILOXANE) COPOLYMER

Determined with EDAX

Figure 10.   EDAX results for Ultem polyimide film (top) and for Ultem-PDMS
copolymer film (bottom) before exposure to atomic oxygen, after
a 90-min exposure, and after a 180-min exposure.