

## OUTGASSING OF FLOWN AND UNFLOWN MIR SOLAR CELLS

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**ABSTRACT**

A solar panel array with more than ten years space exposure was removed from the Mir core module in November 1997, and an eight panel section was returned to Earth in January 1998. Several solar cells were removed from panel eight of the returned array and placed in a high vacuum system with a residual gas analyzer (200 amu mass spectrometer) and a cold finger. Similar unflown solar cells of the same vintage were later obtained from Energia. Several of the unflown cells were also placed in the vacuum system and outgassed residues were collected on the LN<sub>2</sub> cold finger. Almost 3 mg of outgassed residue was collected from a string of three unflown solar cells over a period of 94 hours under vacuum. The collected residue was weighed with a microbalance, and then the residue was analyzed by FTIR spectroscopy, and by gas chromatograph-mass spectroscopy. About 25 outgassed constituents were separated by the gas chromatograph, and a high-resolution mass spectrum was obtained of each constituent. Molecular identifications have been made for the constituents. The constituents are primarily cyclic siloxanes, and several of the constituents are isomers of the same molecule. Most of the outgassed constituents have a molecular mass of about 500 amu.

Almost one mg of residue was extracted from one cm<sup>2</sup> of coverglass/adhesive from a flown solar cell by soaking in isopropyl alcohol for 30 minutes. The gas chromatograph separated about 20 constituents. The constituents are mostly cyclic siloxanes with linear branches, hydrocarbons, and phthalates. The typical molecular mass is about 600 amu.

These identifications of specific outgassing molecules have resulted in a more complete understanding of the SiO<sub>x</sub> contamination on the Mir solar cell coverglasses, and on the MEEP experiment trays and optical specimens during the Shuttle-Mir Phase One flight experiment program. Adjusted outgassing rates based on the data reported here, and/or measured outgassing rates and specific molecular identifications of ISS hardware samples are needed to input into model predictions of induced environment effects of the ISS.

**INTRODUCTION**

A photograph of the first-deployed solar-panel-array on Mir is shown in Figure 1. The solar-panel-array was still attached to the Mir core module during the STS 76 photographic survey, and a close-up on-orbit photograph of the mid-section of the array showing the eighth panel of the returned portion of the array is shown in Figure 2. The whole solar array was detached from the core module in November 1997, and one of four eight-panel sections was returned to Earth in January 1998. Seven of the eight panels were returned to Russia, and the eighth panel remained in the United States. The pronounced scattering of light (as seen as bright-white areas in Figure 2) by oxidized silicone films on the solar cell coverglasses is readily apparent in bright light, and has been previously reported and discussed (ref. 1).

Study of the returned section of the solar array is part of the International Space Station (ISS) Risk Mitigation Program. The investigation plan, schedule, and preliminary results have been reported by Visentine et al. (ref. 2). Initial hardware cleanliness studies of the returned array at NASA Langley Research Center were directed toward study of the detergent-like residues on the returned array similar to those on the Mir Environmental Effects Payload (MEEP) which have been reported by Harvey et al. (ref. 3).

A second, prevalent type of contamination was very thin films consisting of irregular shaped spots of millimeter size which are readily visible in brilliant colors when the solar cell coverglasses are viewed with a 50x brightfield microscope. These prolific, overlapping, and almost ubiquitous patterns strongly suggest wetting of the coverglass surfaces and have been attributed to hydrazine thrusters residue (refs. 4, 5, and 6).

A third type of contamination is the SiO<sub>x</sub> films produced by self-contamination from the silicone potting compound and adhesive. Silicone oils oozed from the potting compound; wetted the space-exposed coverglass surfaces; and was converted to SiO<sub>x</sub> by atomic oxygen in low Earth orbit (LEO)(ref. 1).

A fourth type of contamination is  $\text{SiO}_x$  particles and glass fibers resulting from the degradation of silicone potting and adhesive (ref. 4). The  $\text{SiO}_x$  films and the debris  $\text{SiO}_x$  particles show that the silicone potting and adhesive undergo changes when exposed to the LEO environment. The  $\text{SiO}_x$  films indicate a uniform creep or wetting of silicone oils across the coverglass with subsequent oxidation to  $\text{SiO}_x$ . Plumes of heavier and coarser  $\text{SiO}_x$  films at some coverglass edges (figure 3) indicate microeruptions of silicone gas and particles.

## OUTGASSING MEASUREMENTS AND CHEMICAL ANALYSES

Solar cell 9,5 was removed from panel eight of the returned array and placed in a small (less than 1 ft<sup>3</sup>) high vacuum chamber equipped with a 200 amu residual gas analyzer. A mass spectrum (figure 4) obtained during high vacuum exposure of this solar cell indicates a very small amount of silicone outgassing. Nine solar cells of the same vintage as the cells in the returned array were obtained from the Russian vender Energia. Three individual cells (#25, 26, and 27) were separately placed in the small high vacuum chamber, and mass spectra obtained at lower gain. Figure 5 is the mass spectrum of unflown cell #25. Unflown cells #26 and #27 had similar mass spectra. The mass spectra from cell 9,5 and the unflown cells indicate about two orders of magnitude higher outgassing from the unflown cells than from the flown cell.

Three of the unflown cells (string 1) were placed in the small vacuum chamber, and outgassed residue was collected on a  $\text{LN}_2$  cold finger during 95 hours of vacuum exposure. Almost 3 mg of outgassed residue was collected on the cold finger. The collected residue was measured with a Cahn microbalance, and then analyzed by FTIR spectroscopy. An FTIR spectrum of the unflown cells outgassed residue is shown in Figure 6. The initial outgassing,  $G = \text{mass/area-time}$  averaged over the 95 hours of vacuum exposure can be calculated. The initial outgassing rate was about  $1.40 \times 10^{-10} \text{ gm/cm}^2\text{-sec}$ .

The unflown outgassed residue was then analyzed by gas chromatography. A gas chromatogram of the unflown outgassed residue is shown in figure 7. Figure 7 shows that there are at least 26 constituents in the unflown outgassed residue-mixture.

The first constituent was eluted at 18.154 minutes, and has abundant mass fragments of 55 and 173 atomic mass units (amu), and may be an ether (figure 8). The second constituent was eluted at 18.384 minutes and has abundant 73 amu  $\text{Si}(\text{CH}_3)_3$  and 135 amu  $\text{Si}(\text{CH}_3)_2\text{C}_6\text{H}_5$  mass fragments and hence is a methyl-phenyl silicone. The 417 amu mass peak in figure 9 is probably the molecular ion. The molecular mass of most of the cyclic methyl silicones is the molecular ion plus the 15 amu methyl radical,  $\text{CH}_3$ . A possible molecular configuration of molecular mass 432 is shown in figure 10.

Constituents 4, 5, and 7 are clearly hydrocarbons with abundant alkyl mass fragments of 43, 57, 71, and 85 amu. Constituent 13 is a phthalate with the characteristic 149 amu abundant mass fragment. All of the other 21 constituents are methyl or methyl-phenyl silicones with probable molecular masses in the range 405 to 637 amu. Several isomers of molecular masses 405, 479, 503, and 553 are present. The elution times, probable molecular mass, and chemical class (including number of silicon atoms) of the 26 constituents are listed in Table 1.

Residue from one cm<sup>2</sup> of adhesive and scrim from flown solar cell 3,1 was extracted by soaking for 30 minutes in isopropyl alcohol. The alcohol was allowed to evaporate, and the extracted residue weighed (0.748 mg) with the Cahn microbalance. An FTIR spectrum of the extracted residue is shown in figure 10. The flown extracted residue was then analyzed by GC-MS. A gas chromatogram of the flown-extracted residue is shown in figure 11. Figure 11 shows that there are at least 19 constituents in the flown-extracted residue from solar cell 3,1.

The first constituent was eluted at 22.55 minutes, has an abundant mass fragment (base peak) at 149 amu, and hence is a phthalate. This is the same constituent as the 13<sup>th</sup> constituent of the unflown outgassed residue. Constituents 2 (figure 12) and 3 are hydrocarbons. Constituents 4 and 8 are phthalates. All of the other constituents are methyl (figure 12) or methyl-phenyl silicones with seven or eight silicon atoms. Table 2 lists the elution times, probable molecular mass, and chemical class of the extracted residue constituents.

## CONCLUSIONS

The initial (100-hour) outgassing rate of the Mir solar array was probably about  $1 \times 10^{-10} \text{ gm/cm}^2\text{-sec}$ . The outgassed residue was a mixture of about 25 chemical compounds, mostly methyl-phenyl silicone oils. Several isomers of some of the silicone compounds were present. A few hydrocarbons and a phthalate were also present. The average molecular mass of the outgassing constituents was about 500 amu.

The residual oil within the flown cells (i.e. potential outgassing residue) was similar to that of the unflown solar cells, but was much less abundant, typically had one or two more silicon atoms per molecule, and was about 100 amu larger mass.

This information can be appropriately adjusted to predict short term and long term outgassing of solar arrays on the ISS. GC-MS can be well suited for detailed study of some outgassing residues. Information regarding the number of constituents in an outgassing mixture, and molecular masses and chemistry can validate assumptions regarding outgassing decay, and space exposure effects such as oxidation and polymerization. Each constituent or chemical compound in an outgassing mixture has its own outgassing rate. The chromatograms provide information on the number of outgassing rates needed to accurately characterize the outgassing of the mixture over time. The molecular weight distribution and chemistry can be used to synthesize mixtures for detailed laboratory measurements under greatly varying conditions, and to use reference library and literature search data as inputs for calculated outgassing of specific hardware under expected conditions.

## REFERENCES

1. Harvey, G. A., et al., "Optical Characterization of Returned Mir Solar Cells," AIAA 99-4488, Space Technology Conference and Exposition, Albuquerque, NM, 1999.
2. Visentine, J., et al., "Mir Solar Array Return Experiment," AIAA 99-0100, 37<sup>th</sup> AIAA Aerospace Sciences Meeting and Exhibit, Reno, NV, 1999.
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5. Dever, J. A., et al., "Contamination and Space Environmental Effects on Solar Cells and Thermal Control Surfaces," J. of Spacecraft and Rockets, V 32, 5, pp850-855, 1995.
6. Koontz, S. et al., SPIFEX Contamination Studies," NASA JSC Report 27399, 1996.
7. Harvey, G. A. et al., "Particle Generation by Silicone Potting Compound of Returned Mir Solar Cells," SPIE 45<sup>th</sup> Annual Meeting, San Diego, CA, 2000.

TABLE 1- Unflown Outgassed Residue

GC peak	Elution time	Probable Mass	Chemical
1.	18.154	?	HC
2.	18.384	432	5SiMO
3.	19.659	504	6SiM
4.	19.744	?	HC
5.	19.873	?	HC
6.	20.729	506	6SiMO
7.	21.072	?	HC
8.	21.228	565	7SiMO+HC
9.	21.779	444	6SiM
10.	22.060	420	4SiMO
11.	22.269	420	4SiMO
12.	22.478	420	4SiMO
13.	22.546	?	phthalate
14.	22.587	420	4SiMO
15.	22.907	589	7SiMO
16.	23.665	518	7SiM
17.	23.804	494	5SiMO
18.	24.124	494	5SiMO
19.	24.292	494	5SiMO
20.	24.863	654	8SiMO
21.	25.404	518	7SiM
22.	25.638	568	6SiMO
23.	25.873	568	6SiMO
24.	25.969	568	6SiMO
25.	26.024	568	6SiMO
26.	26.093	568	6SiMO

TABLE 2- Flown Extracted Residue

GC peak	Elution time	Probable mass	Chemical
1.	22.551	?	phthalate
2.	23.647	479	HC
3.	25.866	?	HC
4.	26.693	?	phthalate
5.	26.990	518	7SiM
6.	28.138	579	7SiMO
7.	28.488	518	7SiM
8.	28.840	?	phthalate
9.	29.596	654	8SiMO
10.	29.833	518	7SiM
11.	30.930	654	8SiMO
12.	31.124	518	7SiM
13.	32.153	579	7SiMO
14.	32.332	518	7SiM
15.	33.324	654	8SiM
16.	33.324	518	7SiM
17.	34.428	579	7SiMO
18.	34.509	579	7SiMO
19.	35.566	579	7SiMO

Note: HC= hydrocarbon  
SiM = methyl silicone  
SiMO = methyl-phenyl silicone

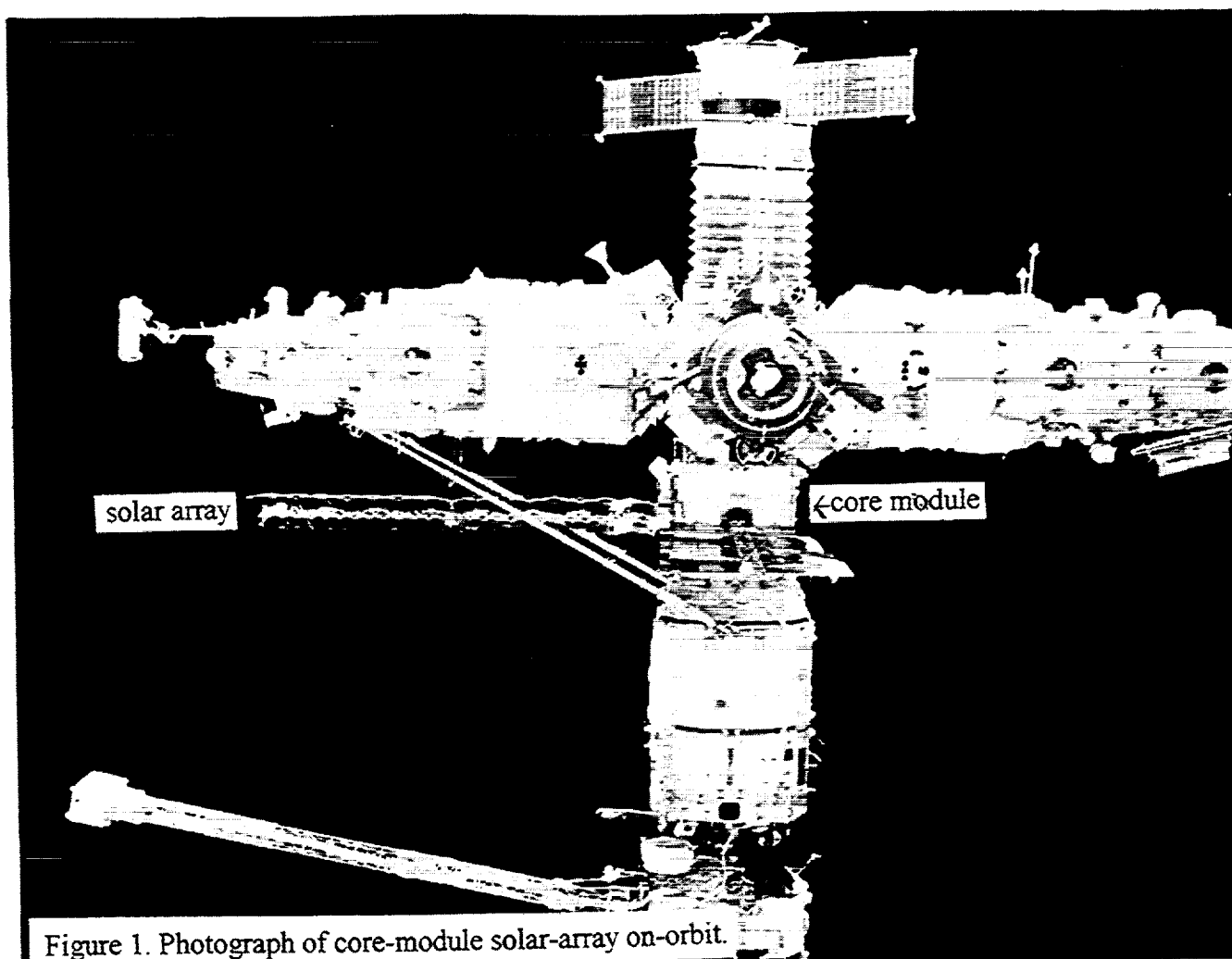


Figure 1. Photograph of core-module solar-array on-orbit.

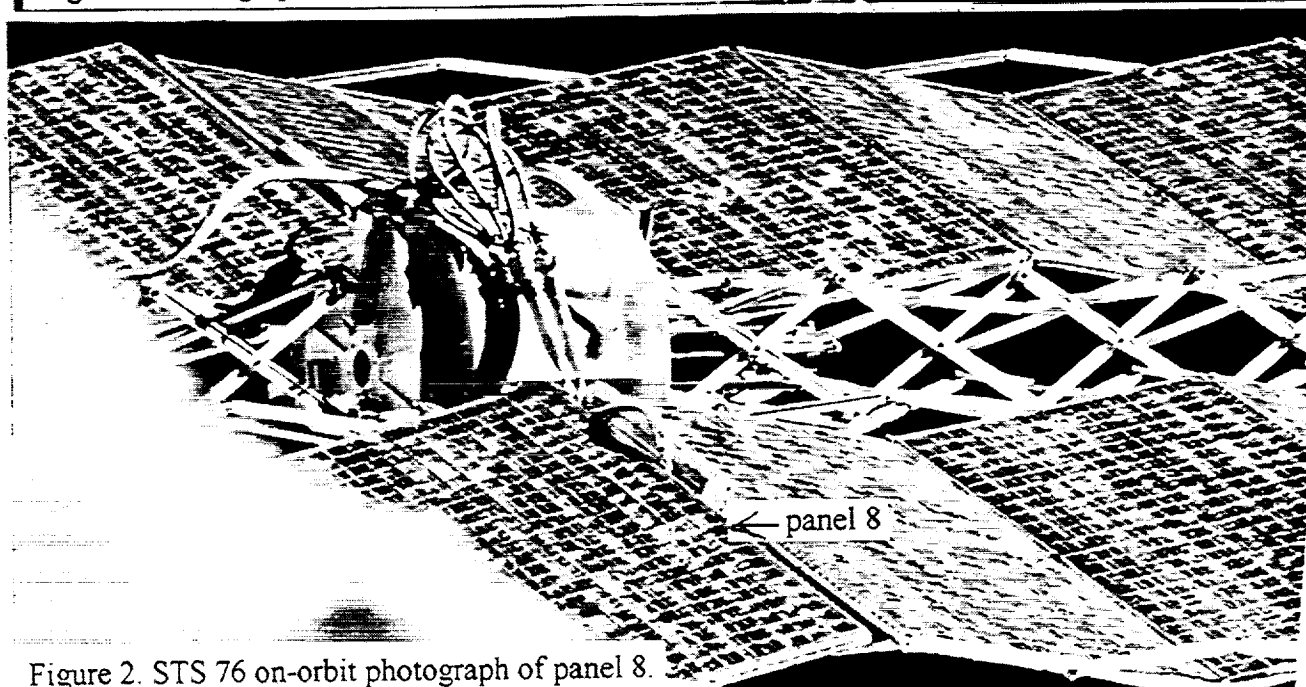


Figure 2. STS 76 on-orbit photograph of panel 8.

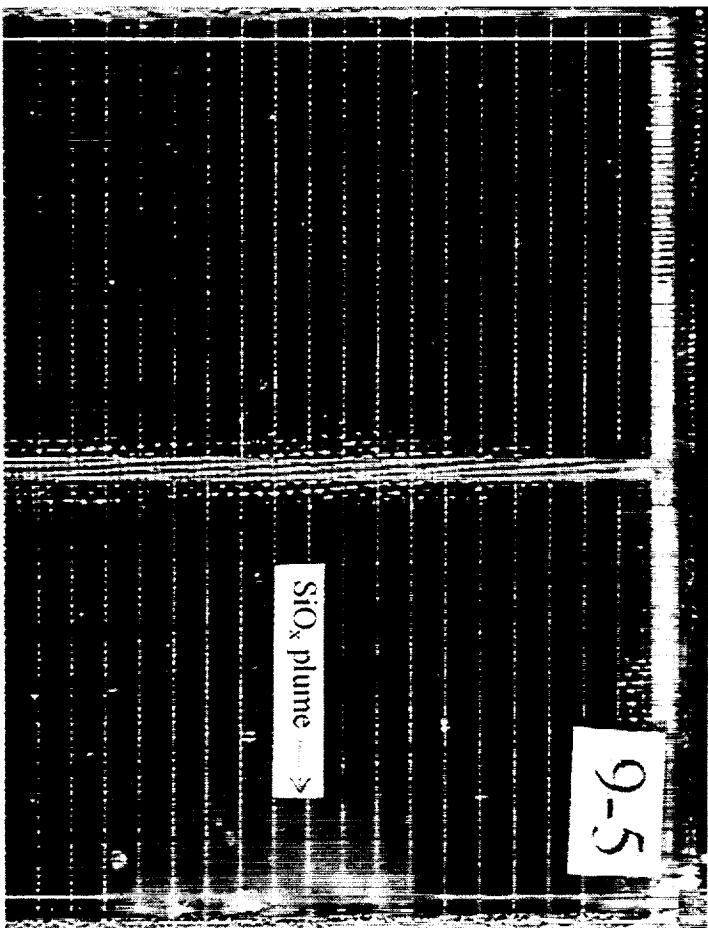


Figure 3.- Photograph of removed cell 9,5 with SiO<sub>x</sub> plume on coverglass.

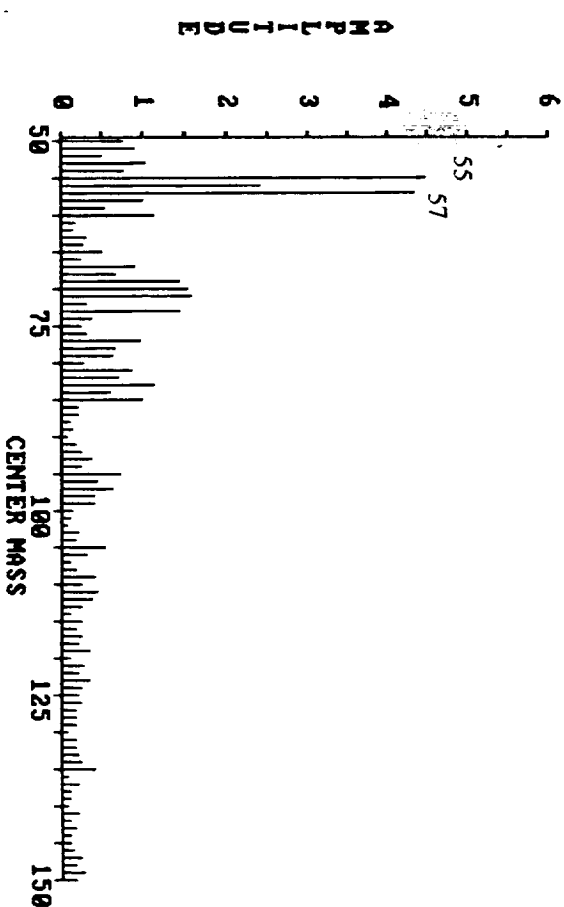


Figure 4.- RGA mass spectrum of outgassing from removed solar cell 9,5.

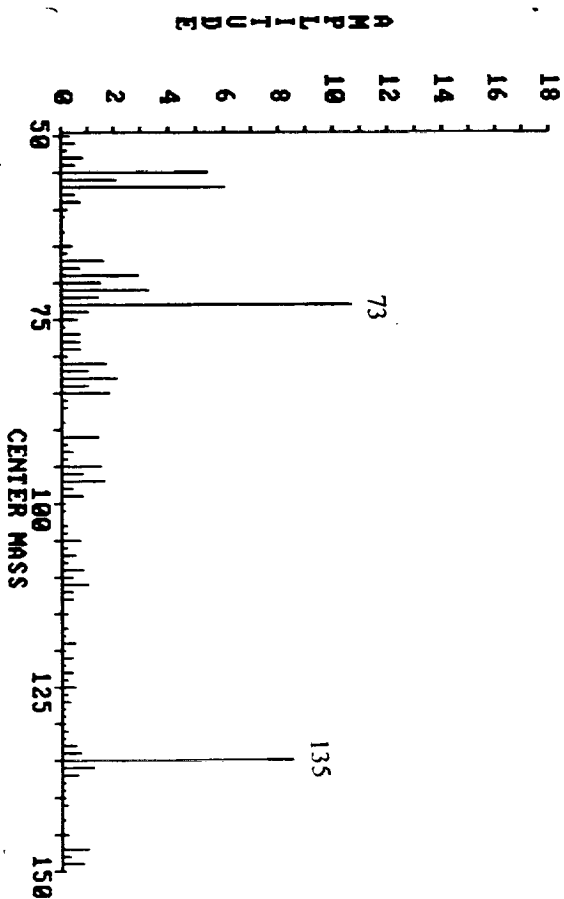


Figure 5.- RGA mass spectrum of outgassing from unflown solar cell #25.

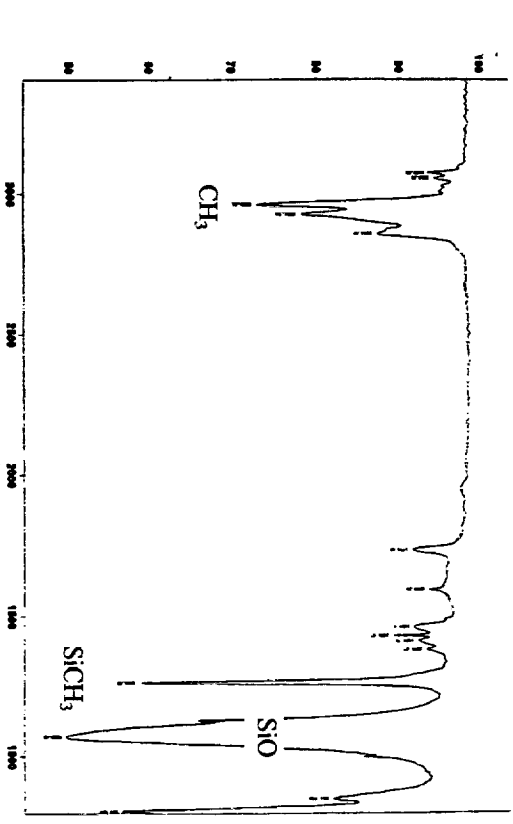


Figure 6.- FTIR spectrum of outgassed residue from three unflown solar cells.

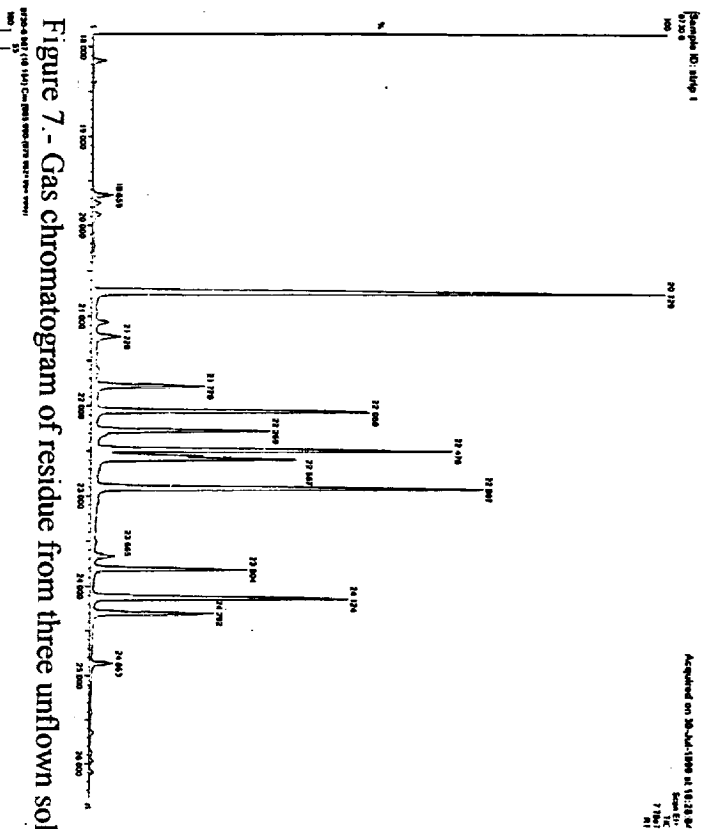


Figure 7. - Gas chromatogram of residue from three unflown solar cells.

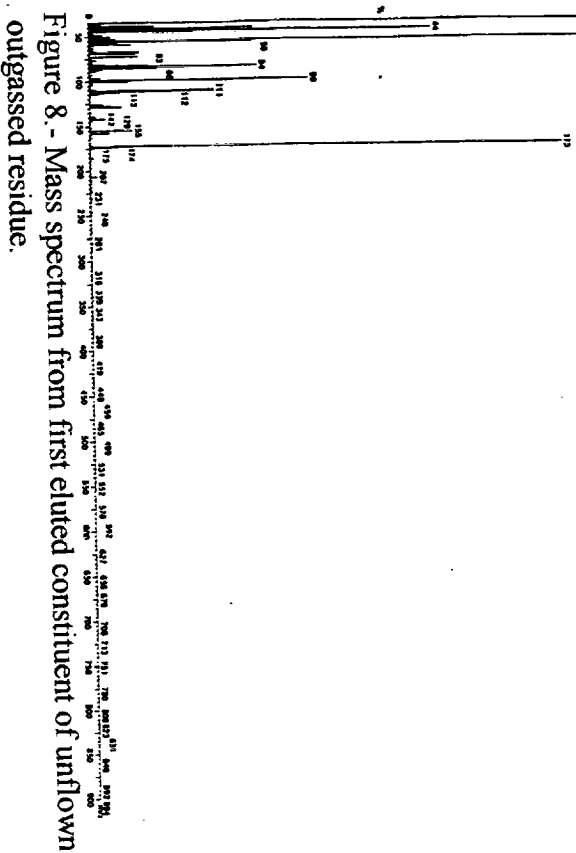
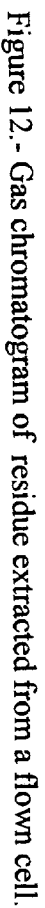




Figure 13. - Mass spectrum of seventh constituent eluted from residue extracted from a flown cell.

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