NASA Contractor Report 198217

NASA-CR-198217
19960009412

Summary of GPC/DV Results for Space Exposed Poly(Arylene Ether Phosphine Oxide)s

Emilie J. Siochi
Lockheed Engineering & Sciences Company, Hampton, Virginia

Contract NAS1-19000

September 1995

National Aeronautics and
Space Administration
Langley Research Center
Hampton, Virginia 23681-0001

ABSTRACT

Gel Permeation Chromatography (GPC) was used to analyze poly(arylene ether phosphine oxide)s whose backbones were identical except for the ketone content and placement. These samples were exposed to low earth orbit environment (predominantly atomic oxygen) on space shuttle flights. The materials and their unexposed controls were then characterized by GPC to investigate the effect of atomic oxygen on the molecular weight distributions. Analysis of the soluble portion of the samples revealed that there was significant loss of high molecular weight species. The presence of insoluble material also suggested that crosslinking was induced by the atomic oxygen exposure and that this very likely occurred at the high molecular weight portion of the molecular weight distribution.

EXPERIMENTAL

Gel Permeation Chromatography was conducted on the poly(arylene ether phosphine oxide)s (PAEPO) controls and samples that had been exposed to low earth orbit (LEO) environment. These are shown in Table 1.

TABLE 1
Polymers Analyzed

SAMPLE	DESCRIPTION
JC 119-75	Control PAEPO with p-ketones
JC 119-75 LEO	LEO exposed JC 119-75
JC 119-76	Control PAEPO with 1 ketone unit
JC 119-76 LEO	LEO exposed JC 119-76
JGS 831-82-085	Control PAEPO with m-ketones
JGS 831-82-085 LEO	LEO exposed JGS 831-82-085

The experiments were conducted using distilled NMP with 0.02M LiBr dissolved in it. Samples were prepared at least 16 hours prior to analysis. They were filtered through a 0.5 μm Teflon® filter in series with a 0.2μm Teflon® filter prior to injection. Chromatography was performed on a Waters 150C gel permeation chromatograph which was equipped with a differential refractive index detector in parallel configuration with a Viscotek model 150R differential viscometer. A Waters Styragel HT 6E linear column covering a molecular weight range from 10^3 to 10^7 g/mol was used in series with a Styragel HT 3 column, which covers the range from 10^2 to 10^4 g/mol. A universal calibration curve was generated with narrow polystyrene standards with molecular weights ranging from 5×10^2 to 2.75×10^6 g/mol.

RESULTS AND DISCUSSION

None of the polymers analyzed above were completely soluble. The concentrations used in the calculation of molecular weights were corrected to reflect the actual injected concentration. This was determined by weighing the insoluble residue after it was rinsed with pure NMP and ethanol, then dried to constant weight. The solubilities determined in this manner are shown in Table 2.

The appearance of the insoluble portion of the controls and the LEO exposed samples were different. The insoluble portion of the LEO exposed samples had striations visible in the swollen state. These lines were absent in the swollen, insoluble portion of the controls. The presence of the striations in the exposed films suggest that exposure to LEO environment caused crosslinking to occur in the polymer films.

TABLE 2
SOLUBILITIES OF POLYMERS ANALYZED

SAMPLE	SOLUBILITY (%)	SAMPLE	SOLUBILITY (%)
JC 119-75 Control	94.2	JC 119-75 LEO	35.6
JC 119-76 Control	97.7	JC 119-76 LEO	37.0
JGS 831-82-085 Control	98.8	JGS 831-82-085 LEO	52.8

The results from GPC/DV are shown in Table 3.

TABLE 3
GPC/DV RESULTS

SAMPLE	M_n (g/mol)	M_w (g/mol)	M_z (g/mol)	INTRINSIC VISCOSITY (dL/g)
JC 119-75 Control	5204	495300	2781000	0.827
	5725	435700	2904000	0.808
JC 119-75 LEO	2650	107500	226400	0.499
	3127	110900	299500	0.530
JC 119-76 Control	6202	485500	6440000	0.932
	3591	478900	5968000	0.943
JC 119-76 LEO	8303	76090	216000	0.551
	7203	75910	184900	0.575
JGS 831-82-085 Control	2778	653500	7416000	0.983
	1220	651100	7771000	0.979
JGS 831-82-085 LEO	2692	224100	1200000	0.642
	1905	208700	1009000	0.673

Overlays of the molecular weight distributions of the controls and the soluble portion of the LEO exposed polymers are shown in Figures 1-3 for JC 119-75, JC 119-76 and JGS 831-82-085, respectively. They reveal that exposure to LEO environment resulted in the loss of a significant amount of high molecular weight material. The appearance of the swollen insoluble gel from the exposed materials suggests that crosslinking was the cause of the insolubility and the molecular weight characterization showed that the crosslinking occurred in the high molecular weight portion of the distribution. If the solubility data were to be used as an indicator of stability against crosslinking, it may be inferred that JGS 831-82-085, where the ketone groups are meta to each other, was the most stable of the three systems.

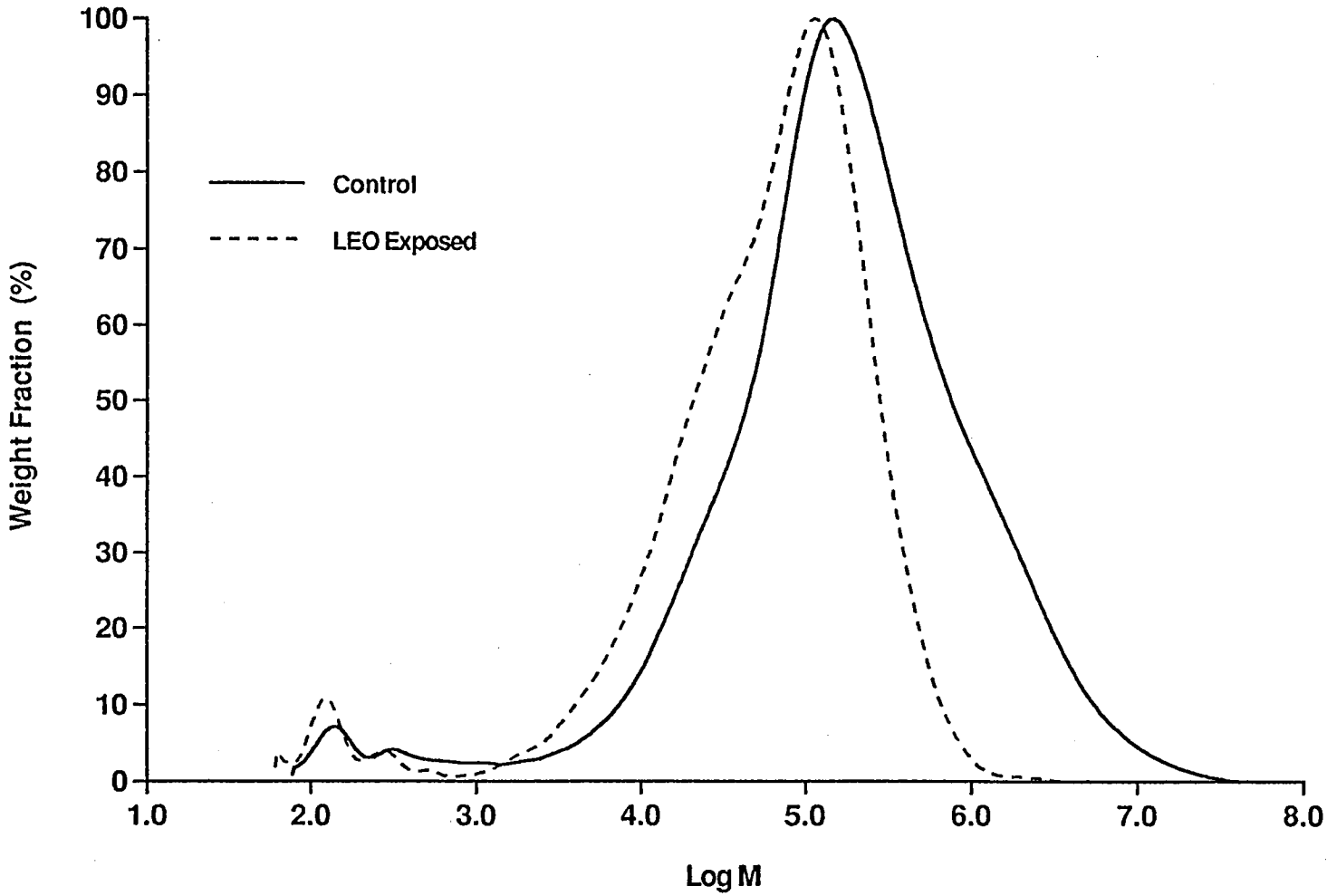

Figure 1: Overlay of molecular weight distributions of JC 119-75 control and LEO exposed.

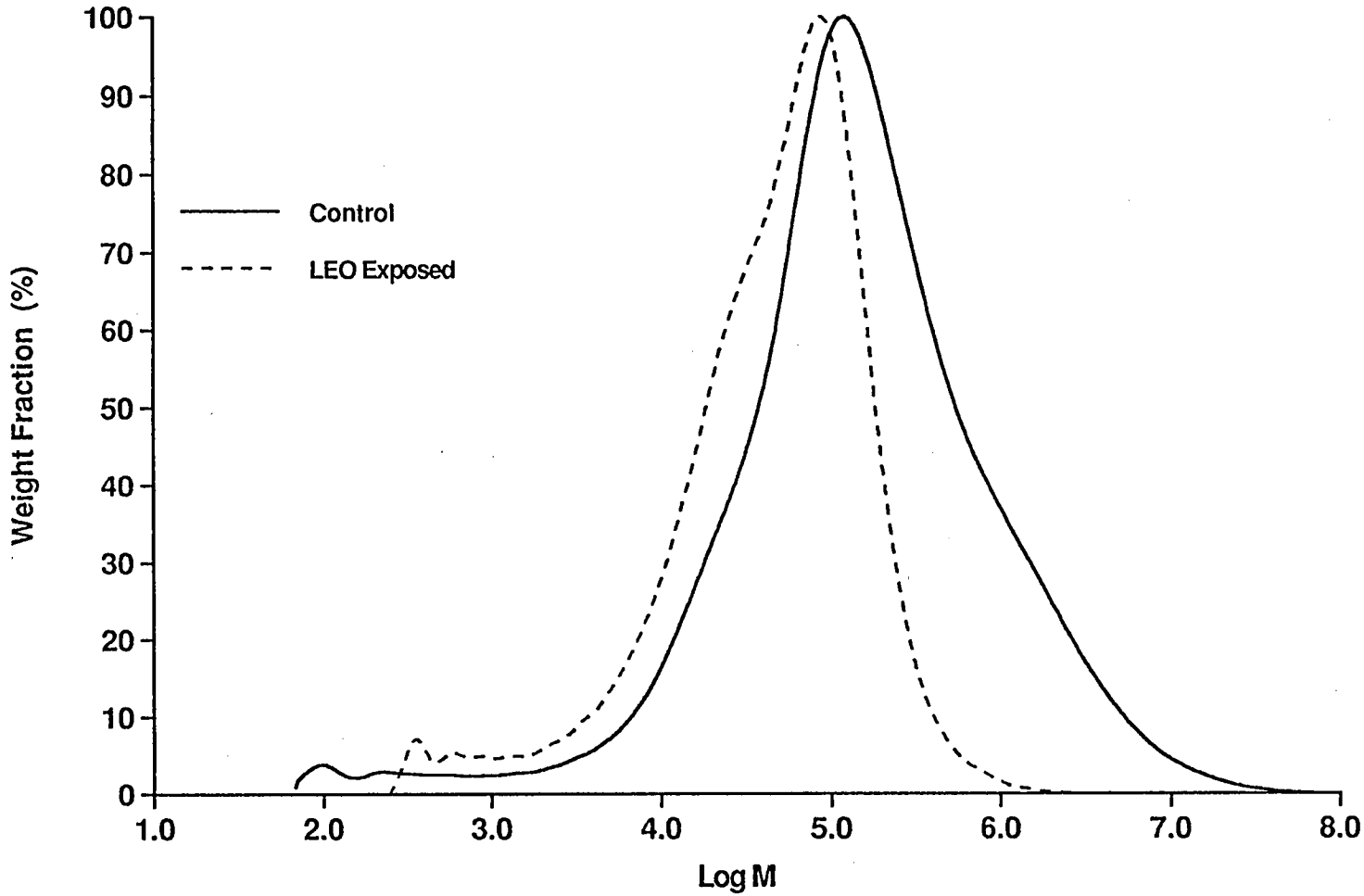

Figure 2: Overlay of molecular weight distributions of JC 119-76 control and LEO exposed.

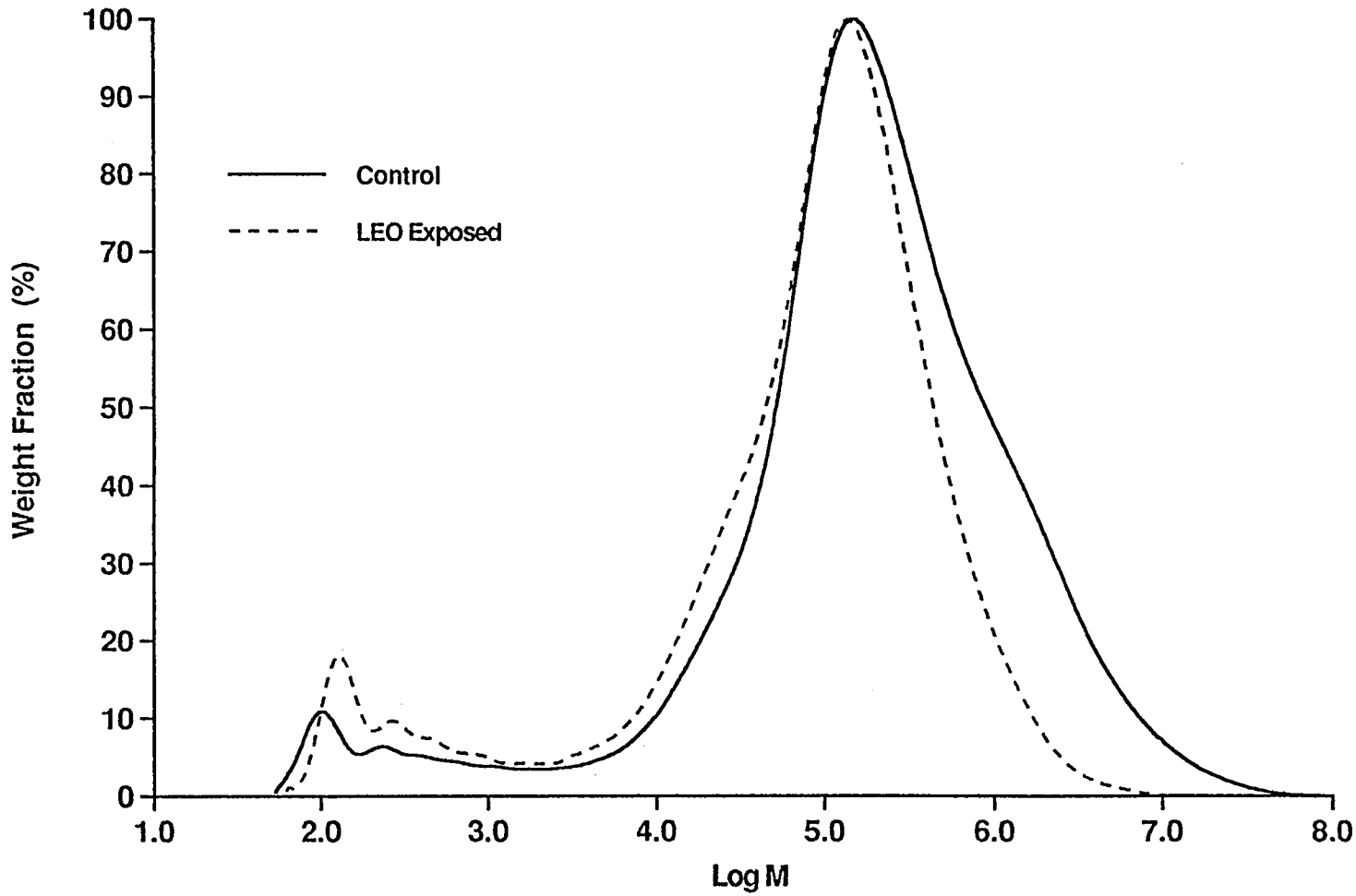

Figure 3: Overlay of molecular weight distributions of JGS 831-82-085 control and LEO exposed.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	September 1995	Contractor Report

4. TITLE AND SUBTITLE
Summary of GPC/DV Results for Space Exposed Poly(Arylene Ether Phosphine Oxide)s

5. FUNDING NUMBERS
NAS1-19000
505-63-50-01

6. AUTHOR(S)
Emilie J. Siochi

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Lockheed Engineering and Sciences
144 Research Drive
Hampton, VA 23666

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Langley Research Center
Hampton, VA 23681-0001

10. SPONSORING / MONITORING AGENCY REPORT NUMBER
NASA CR-198217

11. SUPPLEMENTARY NOTES
Langley Technical Monitor/Task Monitor: Fred Staggs/Terry St. Clair
Final Report

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Unclassified - Unlimited
Subject Category 27

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Gel Permeation Chromatography (GPC) was used to analyze poly(arylene ether phosphine oxide)s whose backbones were identical except for the ketone content and placement. These samples were exposed to low Earth orbit environment (predominantly atomic oxygen) on space shuttle flights. The materials and their unexposed controls were then characterized by GPC to investigate the effect of atomic oxygen on the molecular weight distributions. Analysis of the soluble portion of the samples revealed that there was significant loss of high molecular weight species. The presence of insoluble material also suggested that crosslinking was induced by the atomic oxygen exposure and that this very likely occurred at the high molecular weight portion of the molecular weight distribution.

14. SUBJECT TERMS
Gel Permeation Chromatography, Low Earth Orbit (LEO) environment, atomic oxygen, polymers, poly(arylene ether phosphine oxide)

15. NUMBER OF PAGES
7

16. PRICE CODE
A02

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified		