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MONSANTO RESEARCH CORPORATION

A SUBSIDIARY OF MONSANTO COMPANY

DAYTON LABORATORY

DAYTON, OHIO 45407

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Quarterly Report No. 10
PHYSICAL AND RHEOLOGICAL PROPERTIES OF NITROSO RUBBERS
25 September 1965 through 24 December 1965

Contract No. DA19-129-AMC-151(N)
(O.I. 9115)

12 January 1966

.For

U. S. Army Natick Laboratories
Natick, Massachusetts

Authors

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ABSTRACT

Characterization of the trifluoronitrosomethane/tetrafluoroethylene copolymer produced by the Thiokol Chemical Corporation and supplied by the U.S. Army Natick Laboratories was continued. The CF_3NO/C_2F_4 copolymer was found to be adaptable to elution fractionation in a perfluorocyclic ether and a benzotrifluoride system, even though 85% of the polymer eluted in a narrow viscosity range of 1.3 to 2.0. Values of K' and K" of the copolymer in FC-75 were confirmed to be 0.353 and 0.147, respectively. Thermal decomposition in air and in an inert gas was shown to be identical. The glass transition of the copolymer, as measured by DTA, is in the region of -41° to -46°C with no more than 2°C difference between production lots. The white insoluble material present in the production lots was identified to be basic magnesium carbonate trihydrate.

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I. INTRODUCTION

The fluorinated nitroso rubber to be characterized in this program is considered to be a highly solvent-resistant, stable, low- and high-temperature rubber. The degree of its worth in these respects can only be determined through a characterization of its basic physical properties. The purpose of the characterization is to describe the rubber for its use and further improvement or modification.

Nine nitroso gum samples, listed as ZR-561-XP5675, XP5702, XP5812, XP5887, XP5807, XP5704, 0.2 C_2F_3H terpolymer, 0.5 C_2F_3H terpolymer, produced by the Thiokol Chemical Corporation, and a 3M-produced gum were delivered to Monsanto Research Corporation via the Natick Laboratories for characterization.

Research completed during this period of work includes: elution fractionation of the XP5702 copolymer, description of the elution fractions, thermogravimetric analysis of the copolymer in air and an inert gas, low temperature differential thermal analysis, terminal group analysis by NMR, and identification of the white precipitate present in the production lots.

II. EXPERIMENTAL

A. ELUTION FRACTIONATION OF XP5702 TRIFLUORO-NITROSOMETHANE/TETRAFLUOROETHYLENE COPOLYMER

A purified sample of the XP5702 copolymer, which has previously been partially described (Ref. 1), was fractionated by the elution technique (Ref. 2).

The sample was cleaned up by putting it into solution in FC-75 solvent (isomers of perfluorocyclic ether), heating to 80°C, cooling, filtering, centrifuging, decanting of the clear solution, evaporating the solvent, and recovering the soluble gum. A large portion (25%) of insoluble gel was noted as removed from the gum. 1.0661 grams of polymer was recovered.

The polymer was again put into solution in FC-75 (1.0661 g in 29 ml at 85°C). A solvent/non-solvent system was then prepared based on prior determinations (Ref. 3) of solvent/non-solvent ratios. The system consisted of a ratio of 3.2303 g polymer/ 100 ml FC-75/73 ml benzotrifluoride. Under these conditions, the polymer was all in solution at 70°C and precipitated at 25°C.

The elution column consisted of cleaned, pure silica sand of from -40 to +200 mesh particle size. N_2 gas was used as a back pressure to regulate the flow of the solvent over the sand. This column is described in Reference 2 and is shown in Figure 1.

The column was maintained at 78°C by refluxing etnanol in the jacket, and the polymer solution was heated to 80°C prior to pouring it on the column. The actual solution added consisted of 1.0661 g XP5702 (purified), 33 ml of FC-75, and 24 ml benzotri-fluoride. After the solution was added, the column was cooled slowly in order to cause selective deposition of the polymer.

Removal of the polymer fractions was performed by eluting with solvent/non-solvent mixtures of FC-75 and benzotrifluoride. The column was first flushed with 250 ml of benzotrifluoride to remove any impurities and set the polymer to the substrate. Progressive elutions were then performed by solvent/non-solvent mixtures, where the solvent was varied from 5% to 100% in 5% volume increments. One-hundred ml of the solution was eluted each three times over the column, with an elution time of 30 minutes. In this way, 15 fractions were recovered as shown in Table 1.

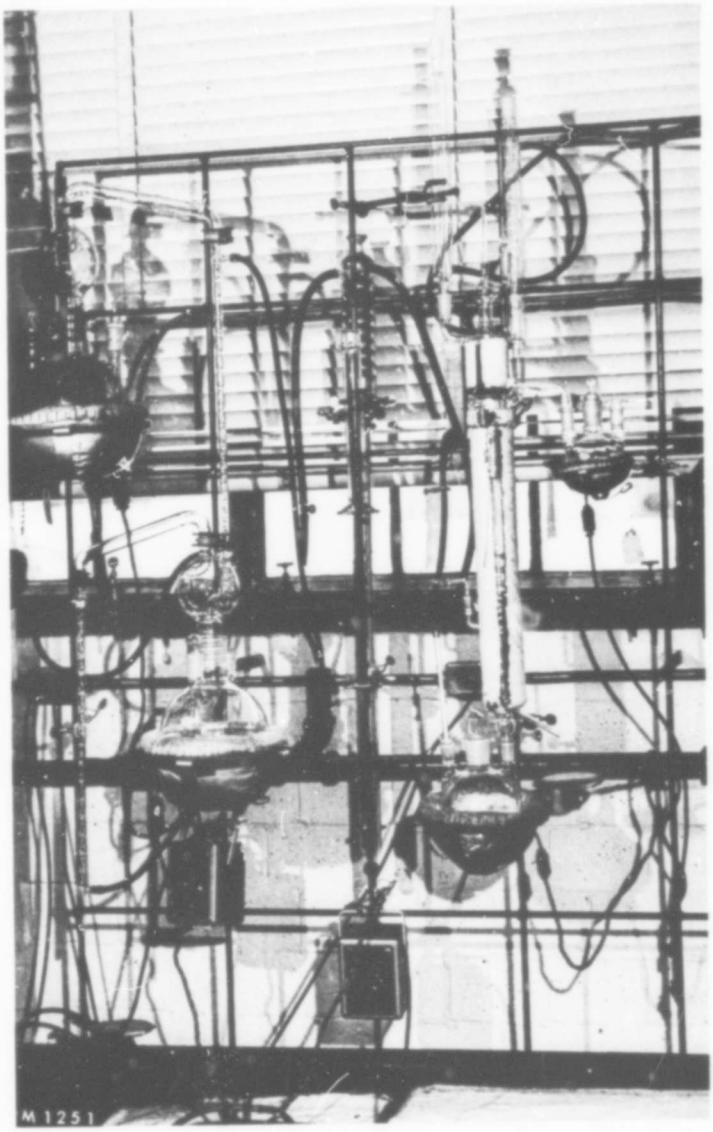


Figure 1. Laboratory one-gram elution fractionation column

TABLE 1

ELUTION FRACTIONATION OF FLURONITROSO GUM XP5702
ROLLOPING SELECTIVE DEPOSITION

	Solvent Mixture										
	FC-75	Benzo trifluoride	Weight of fraction	Polymer eluted	Cumulative polymer eluted	η_{sp} [2]	η_{sp}/C [2]	η_{sp} [4]	η_{sp}/C [4]	$[\eta]$ [5]	$[\eta]$ [6]
Fraction	ml	ml	g	g [1]	g [1]						
Flush	0	250	0.0119	1.22	1.22	- [3]	-				
1	5	95	0.0228	2.34	3.56	- [3]	-				
2	10	90	0.0083	0.85	4.41	- [3]	-				
3	15	85	0.0052	0.53	4.94	- [3]	-				
4	20	80	0.0057	0.59	5.53	- [3]	-				
5	25	75	0.0074	0.76	6.29	- [3]	-				
6	30	70	0.0222	2.28	8.57	- [3]	-				
7	35	65	0.0199	2.05	10.62	- [3]	-				
8	40	60	0.2986	30.71	41.33	0.138	1.38	0.127	1.27	1.38	
9	45	55	0.2711	27.88	69.21	0.191	1.91			1.78	
10	50	50	0.1132	11.64	80.85	0.162	1.62				
11	55	45	0.1402	14.42	95.27	0.186	1.86	0.165	1.65		
12	60	40	0.0351	3.61	98.88						
13	70	30	0.0057	0.59	99.47	- [3]	-				
14	100	0	0.0027	0.28	99.75	- [3]	-				
15	100	0	0.0022	0.23	99.98	- [3]	-				
Unrecovered	-	-	0.0939	-	-	- [3]	-				
			1.0661		99.98						
Whole Polymer	-	-	-	-	-	-	1.15-1.44	-	1.03	1.1-1.38	0.99

1 based on recovered amount of 0.9722g
2 0.1% solution in FC-75
3 insufficient to recover or measure
4 0.1% solution in FC-43
5 in FC-75
6 in FC-43

The polymer was recovered by evaporation of the solvent at 50°C. The weights of the recovered fractions are shown in Table 1.

Specific viscosities of the fractions, where sufficient sample was available, were conducted in a 0.1% solution of FC-75. These data are shown in Table 1 for Fractions 8, 9, 10, 11 and 12. Specific viscosities of Fractions 8 and 11 were also measured in a 0.1% solution of FC-43, and the intrinsic viscosity of Fractions 8 and 9 were measured in FC-75. (See Figure 2.)

The viscosity of the whole polymer had previously been determined (Ref. 1) and is reported here for comparison. The variation noted is due to a non-homogeneity of the uncleaned whole polymer.

Values of K' and K" were calculated for the CF_3NO/C_2F_4 in FC-75 and were, respectively, 0.353 and 0.147.

B. ISOTHERMAL THERMOGRAVIMETRIC ANALYSIS OF SAMPLE XP5675 IN AIR VERSUS INERT GAS

Isothermal thermogravimetric analysis of the XP5675 CF_3NO/C_2F_4 copolymer in an inert gas (helium) had previously been performed (Ref. 3). The results are shown in Figure 3.

An identical analysis was performed in an atmosphere of air. Isothermal temperatures were maintained to equilibrium (usually a period of 4.5 hours) at 25°C intervals from 50°C up to 330°C. A composite of the isothermal runs is shown as a dashed line in Figure 3.

No explosive decomposition occurred during volatilization of the complete sample.

C. LOW-TEMPERATURE DIFFERENTIAL THERMAL ANALYSIS OF THE CF_3NO/C_2F_4 COPOLYMER

Differential thermal analyses were performed on Samples 3M-9690, XP5675 and XP5702 from -100°C up to 100°C, on an MRC-designed DTA apparatus utilizing the Aminco TGA readout unit and a version of the duPont DTA cell. (See Figure 4.) The DTA plots are shown in Figure 5. Sample size was ~0.04 g, not diluted with alumina.

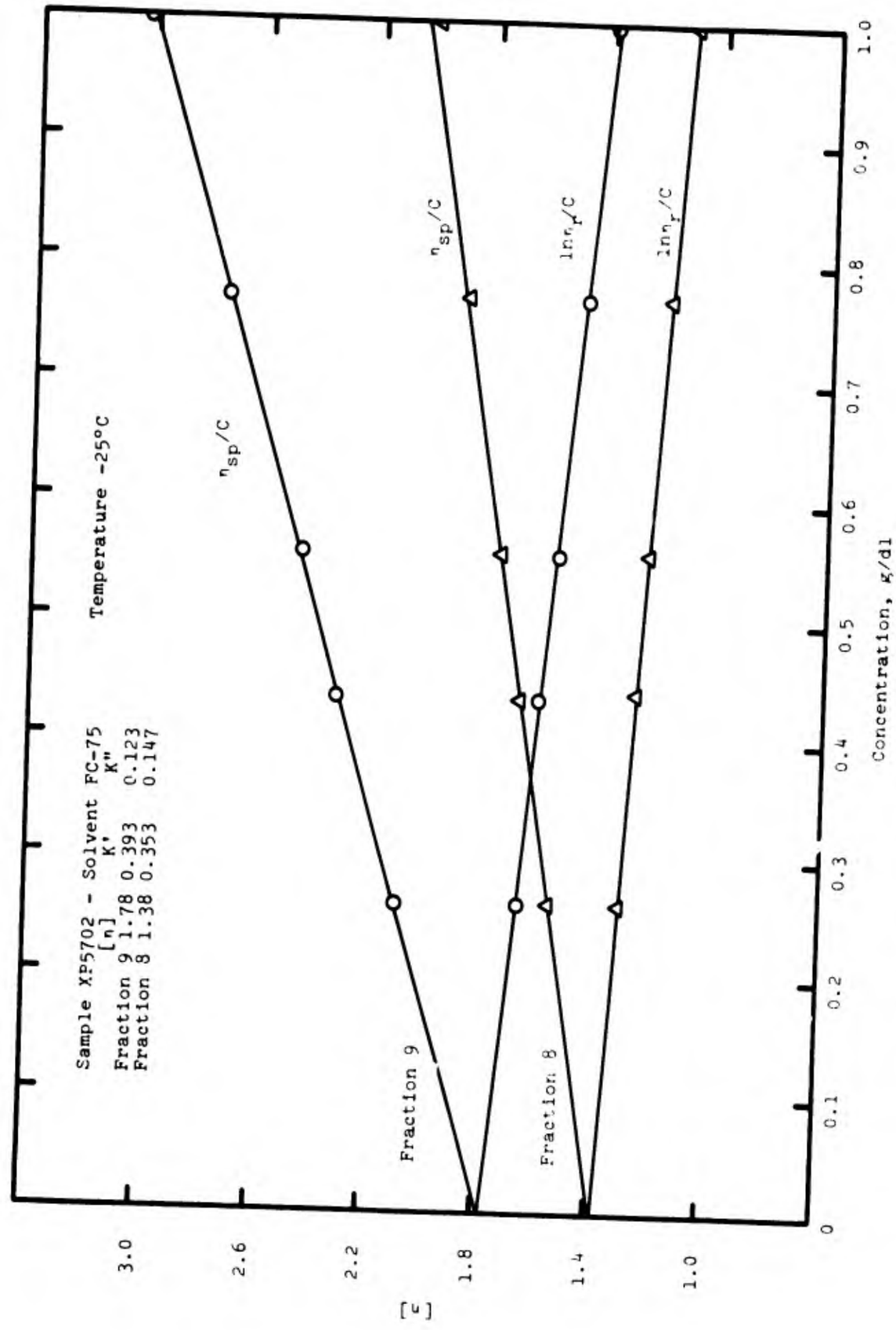


Figure 2. Solution viscosities of two fractions of CF_3NO/C_2F_4 sample XP5702 in FC-75

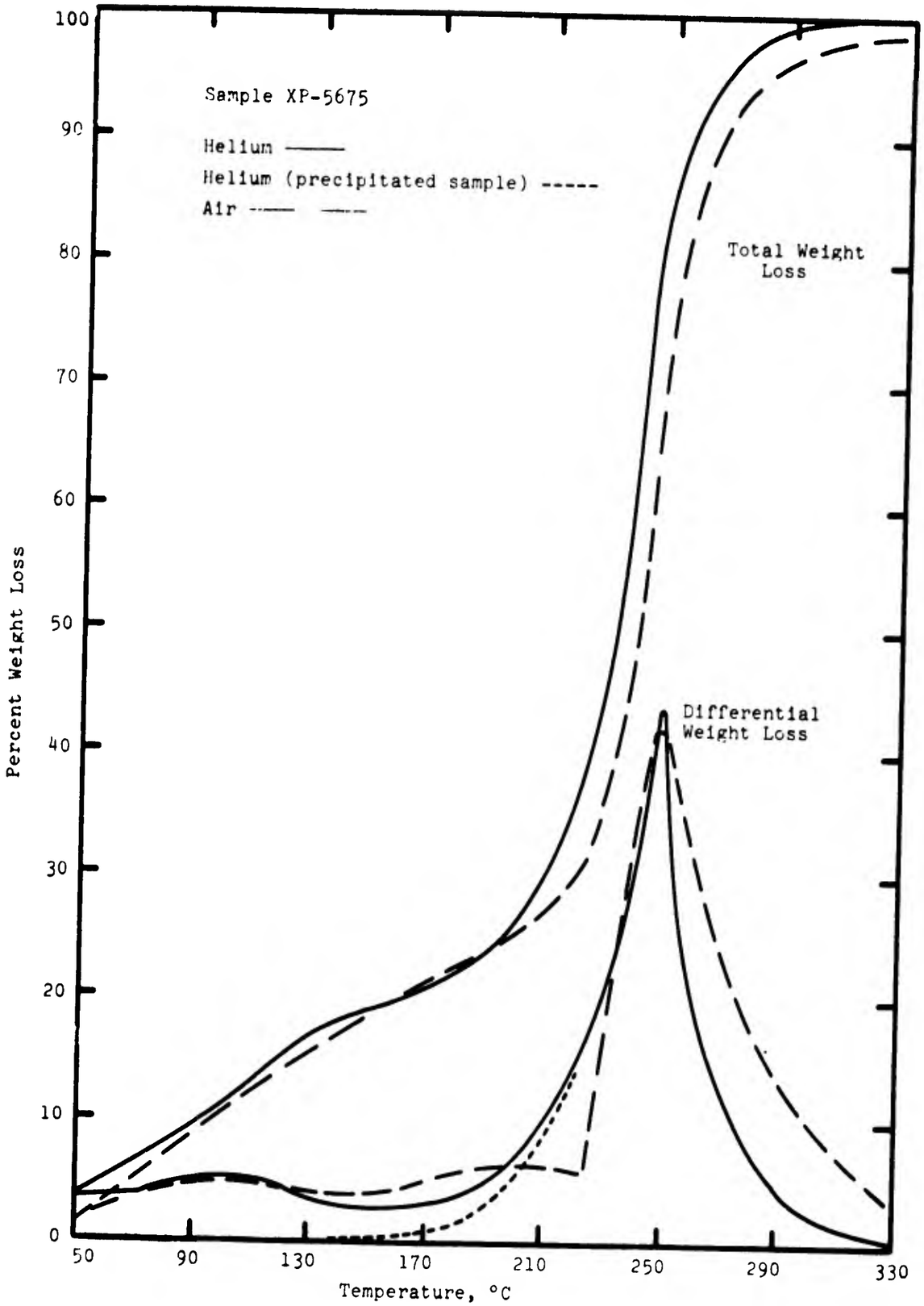


Figure 3. Composite of total and differential weight loss of sample XP5675 in helium and air

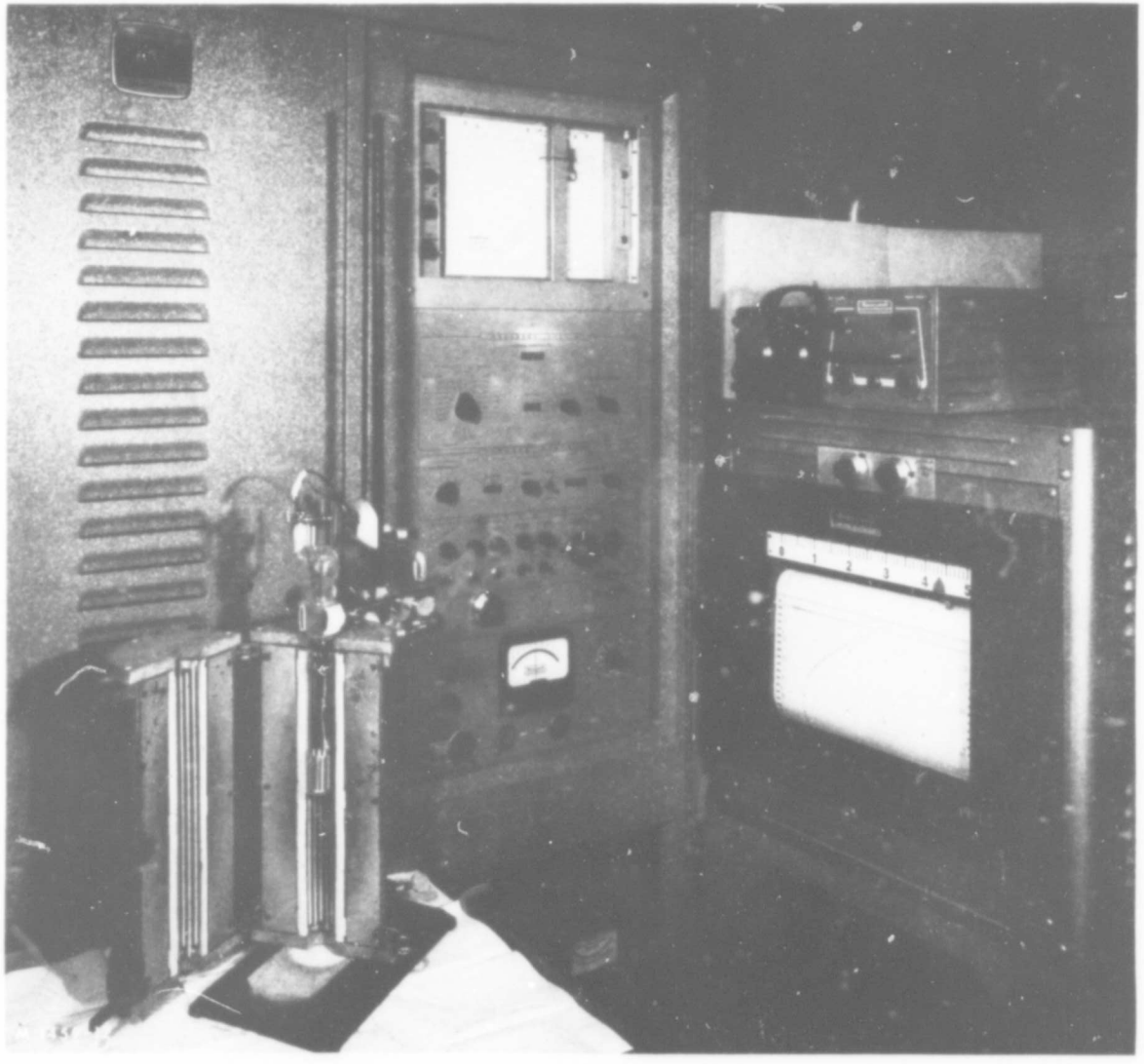


Figure 4. MRC-Aminco Differential Thermal Analyzer for temperatures from -150° to 600°C

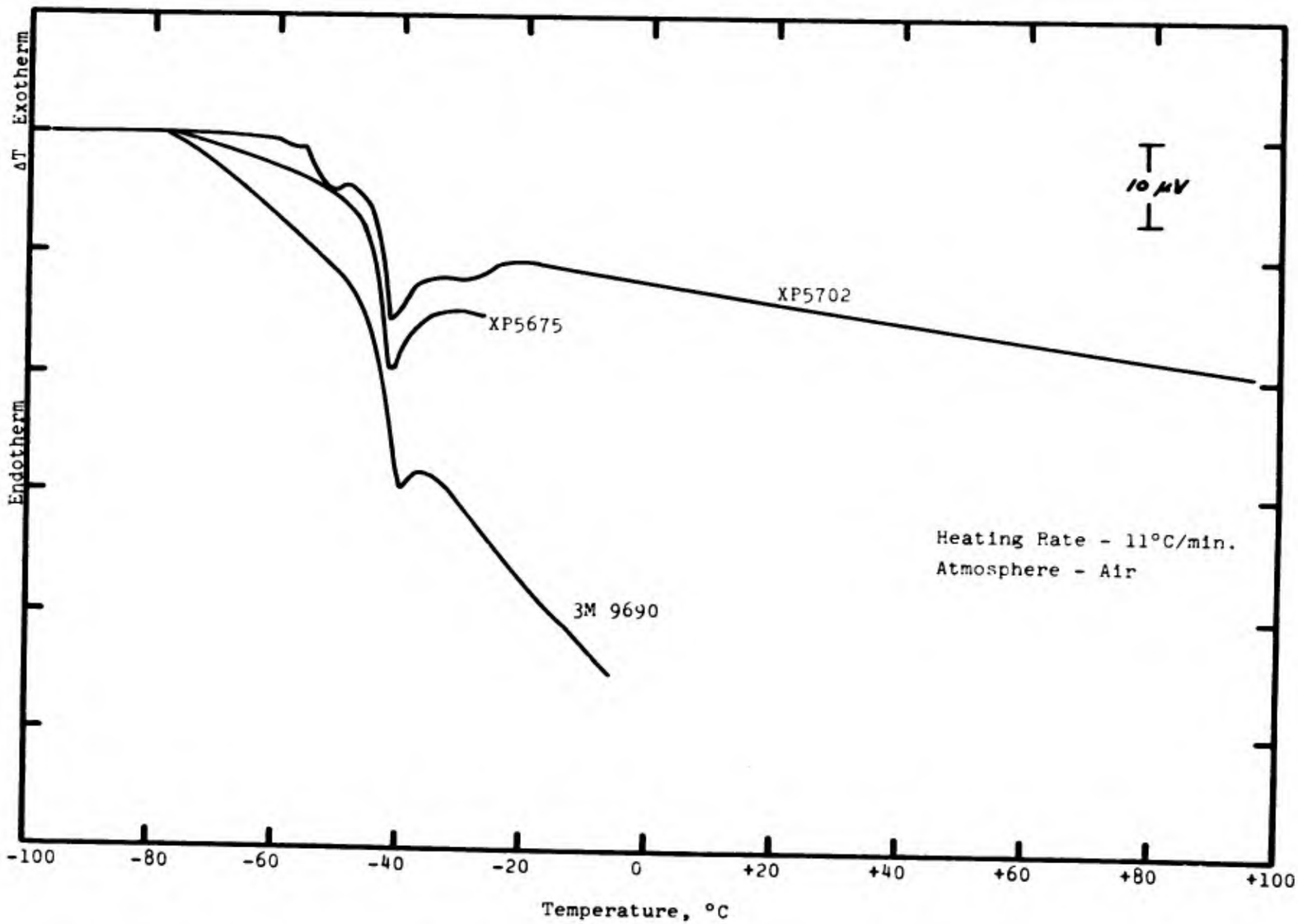


Figure 5. Differential thermographs of three CF_3NO/C_2F_4 copolymers (low temperature)

The reference was alumina. Glass cells were used, and a programed temperature rise of 11°C/min. was maintained.

The DTA's were performed primarily for the low temperature characterization; therefore, the analyses were not conducted to degradation, which probably would have been explosive (Ref. 4).

D. NUCLEAR MAGNETIC RESONANCE OF LOW-, MEDIUM-, AND HIGH-MOLECULAR-WEIGHT FRACTIONS OF CF_3NO/C_2F_4

Proton and F^{19} nuclear magnetic resonance spectra of three CF_3NO/C_2F_4 copolymers of apparent molecular-weight difference were determined. The three samples were XP5807, XP5702 and XP5887, in order of increasing molecular weight.

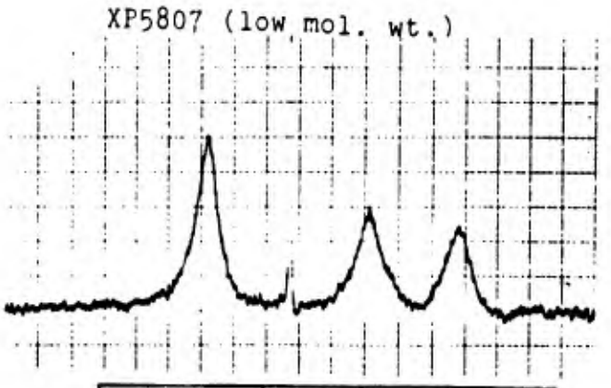
The three F^{19} spectra are shown in Figure 6. The spiked peak is the trifluoroacetic acid reference. The position of the measured peaks and their relative area ratios are calculated based on the largest area arbitrarily being set at 3.0. The RF frequency was 40 Mc, and the gain-and-sweep rate, fast for maximum resolution.

High-gain proton resonance NMR measurements exhibited no spectrum on any of the samples or in the FC-75 solvent used as a carrier.

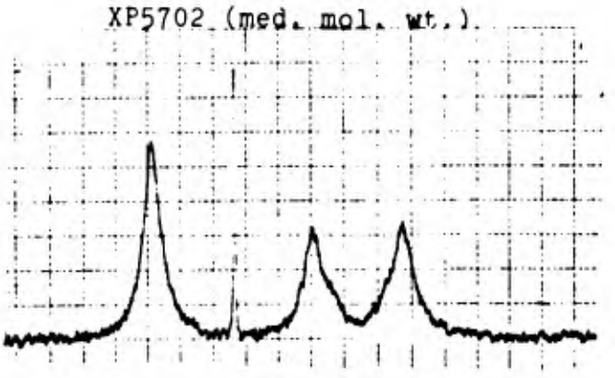
E. IDENTIFICATION OF WHITE INSOLUBLE MATERIAL FROM THIOKOL CF_3NO/C_2F_4 GUM XP5702

The IR spectrum of the white insoluble material from Sample XP5702 was reported in our Ninth Quarterly Report (Ref. 5), as well as in the IR spectrum of the XP5702 gum reported in the Sixth Quarterly Report (Ref. 4).

Emission analysis has since been performed, indicating the presence of magnesium.



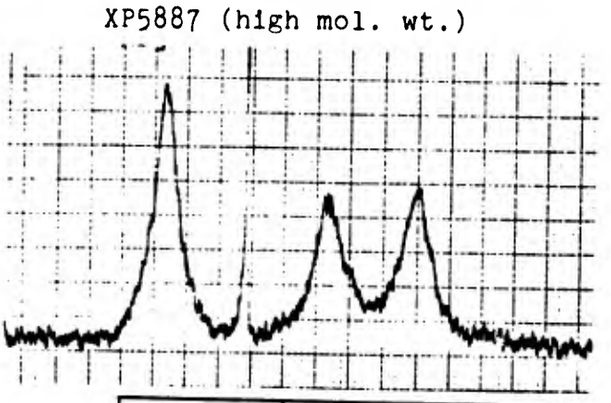
Peak	1	2	3
PPM	-12.3	+11.7	+24.6
Area Ratio	3.0	2.3	2.1



Reference
Trifluoro-acetic
Acid

40.0 Mc

Peak	1	2	3
PPM	-12.4	+12.1	+25.3
Area Ratio	3.0	2.2	2.2



Peak	1	2	3
PPM	-12.4	+12.2	+25.0
Area Ratio	3.0	2.0	1.9

Figure 6. F^{19} NMR spectra of three CF_3NO/C_2F_4 copolymers having different molecular weights

F. REFERENCES

1. "Physical and Rheological Properties of Nitroso Rubbers," Quarterly Report No. 5, Contract No. DA19-129-AMC-151(N), (O.I. 9115), 24 October 1964.

2. Kenyon, A. S., and Salyer, I. O., "Elution Fractionation of Crystalline and Amorphous Polymers," J. Polymer Sci., XLIII, 427 (1960).

3. "Physical and Rheological Properties of Nitroso Rubbers," Quarterly Report No. 8, Contract No. DA19-129-AMC-151(N), (O.I. 9115), 15 July 1965.

4. "Physical and Rheological Properties of Nitroso Rubbers," Quarterly Report No. 6, Contract No. DA19-129-AMC-151(N), (O.I. 9115), 14 January 1965.

5. "Physical and Rheological Properties of Nitroso Rubbers," Quarterly Report No. 9, Contract No. DA19-129-AMC-151(N), (O.I. 9115), 12 October 1965.

III. TECHNICAL DISCUSSION AND CONCLUSIONS

A. FRACTIONATION OF THE TRIFLUORONITROSOMETHANE/TETRAFLUOROETHYLENE COPOLYMER

It was demonstrated that the CF_3NO/C_2F_4 copolymer is adaptable to elution fractionation by selective deposition in an FC-75 benzotrifluoride solvent/non-solvent system.

Reversals of fractions occurred which indicated that this particular elution fractionation was not perfect (i.e., see viscosities of Fractions 8, 9 and 10). However, this was to be expected, since the region of primary elution had not previously been described, and, as it turned out, 85% of the polymer eluted in a narrow region of [η] from 1.3 to 2.0. (Other rubbers would span the region from 0.5 to 5.0.)

The results describe the route to follow for future fractionations. Elution would be started at Fraction 6 so that the first cut would result in 8% of the polymer. Progressive cuts would be made at 2% solvent/non-solvent intervals (as opposed to the 5% used), so that Fractions 8 and 9 would be broken up into 5 fractions. Working toward about 15 fractions, Fractions 12 through 15 would be combined.

Utilizing this technique, it is expected that at least 15 good fractions would result which could be characterized as to viscosity, as well as, in a few cases where sufficient quantity is obtained, molecular weight determinations by light scattering may be performed.

The fact that viscosities greater than that of the whole polymer resulted from the various fractions indicated that the fractionation was successful and values of K' and K" were in excellent agreement with previously determined values on the whole polymer. This allowed conversion of reduced viscosities to intrinsic viscosities.

B. THERMAL DECOMPOSITION OF THE CF_3NO/C_2F_4 COPOLYMER (SAMPLE XP5675)

A comparison of the thermal decomposition of the CF_3NO/C_2F_4 copolymer in air and in an inert gas indicates no effect of oxygen on the thermal failure.

In both cases, a volatile portion (which has been shown to be removable) is evident from 50°C up to 200°C with a peak at 100°C. Primary decomposition occurs at 250°C (from 150°C up to 450°C).

Again, no violent decomposition occurred during decomposition of a relatively small specimen.

A final isothermal thermogravimetric analysis performed in a vacuum would be informative to show the translation of the loss of the volatile portion to lower temperatures, while the primary degradation should be relatively unaffected.

C. LOW-TEMPERATURE DIFFERENTIAL THERMAL ANALYSIS OF THE CF_3NO/C_2F_4 COPOLYMER

As shown in Figure 5, the glass transition by DTA of the CF_3NO/C_2F_4 copolymer is in the region of -41°to -46°C. The transitions of the three samples, 3M-9690, XP5675 and XP5702, are within 2°C of each other. The DTA method does not allow further refinement of the difference in the transitions of the various samples. The change in slope in the region of -51°to -56°C of Sample XP5702 is undefined, and there presently is doubt as to whether it is real or not. The possibility of this being the lower-molecular-weight gum is considered, but not proven or entirely anticipated. The various slopes shown in Figure 5 are probably more a result of the technique than a characteristic of the polymer. The change in slopes, however, is real and has been confirmed by repeated analyses.

D. TERMINAL GROUP ANALYSIS BY NUCLEAR MAGNETIC RESONANCE OF THE CF_3NO/C_2F_4 COPOLYMER

As shown in Figure 5, no difference was demonstrated between the F^{19} spectra of the lowest- and highest-molecular-weight gums, and there was no difference, as well as no hydrogen spectrum, present. Therefore, no indication of end groups was able to be determined.

E. IDENTIFICATION OF WHITE INSOLUBLE MATERIAL FROM THIOKOL CF_3NO/C_2F_4 GUM XP5702

Previously (Ref. 5) it had been reported that an amide structure was indicated in the white insoluble material found in XP5702 as well as other Thiokol samples.

Further analysis of the IR data and emission analysis alters this conclusion. The material is a mixture of nitroso gum similar to XP5675 and basic magnesium carbonate trihydrate (Sadtler #1740).

The assignments now given to the absorption bands of the infrared spectrum are:

Sample XP5702 IR Assignments

(microns)	Assignment
2.9 6.1	H_2O
6.7 7.0	Carbonate ion
7.7 8.0 8.7 9.1 9.4 9.75 10.9	Nitroso rubber
11.3 11.7 12.5	Basic magnesium carbonate trihydrate
13.5 14.2	Basic magnesium carbonate trihydrate and nitroso rubber

The presence of magnesium was substantiated by emission analysis.

IV. SUMMARY

Adaptability of the CF_3NO/C_2F_4 copolymer to elution fractionation in FC-75 and benzotrifluoride was demonstrated.

Eight-five percent of the polymer eluted in a narrow viscosity region of from 1.3 to 2.0, preventing a usable molecular-weight characterization of one particular polymer.

Values of K' and K" of CF_3NO/C_2F_4 in FC-75 were confirmed as being 0.353 and 0.147, respectively.

Thermal decomposition modes of the CF_3NO/C_2F_4 copolymer were the same in air and in an inert gas with no explosive exotherms occurring on heating of a small sample.

The glass transition of the CF_3NO/C_2F_4 copolymer, as measured by DTA, is in the region of -41° to -46°C.

There was no more than a 2°C difference in the glass transitions of various production lots and a 3M gum.

Nuclear magnetic resonance did not provide an indication of terminal groups.

The white insoluble material present in most of the production lots was identified to be basic magnesium carbonate trihydrate in CF_3NO/C_2F_4 gum.

V. FUTURE PLANS

For the purpose of characterization, a typical fluoronitroso copolymer gum has been define as that portion having a high molecular weight distribution. We will proceed to characterize this typical gum completely. This will include an elution fractionation of the gum and determination of the molecular weights and point viscosities of these fractions.

A definition of the low temperature properties and solvent resistance will be attempted.

VI. TIME AND FINANCIAL STATUS

	Hours to 12/31/65	
George L. Ball III, Research Group Leader*	1,044	
Ival O. Salyer, Research Manager, Polymer Physical Chemistry and Applications	167	
John V. Pustinger, Analytical Group Leader	34	
F. Neil Hodgson, Research Analytical Chemist	22	
Professional, unspecified	444	
Total	1,711	
Charlotte D. Fritsch, Research Technician	766	
John E. Strobel, Research Technician	57	
Richard L. Evers, Research Technician	24	
Donald Q. Douglas, Research Technician	33	
Margaret J. Ross, Research Technician	259	
Ralph R. Ferguson, Research Technician	33	
Gary A. Clinehens, Research Technician	157	
Technical, unspecified	354	
Total	1,683	
Grand Total		3,394

As of 31 December 1965, $46,980 (including fee) has been spent. The contract, less fee, is for $59,335, leaving a balance of $12,355.

Seventy-four percent of the work has been completed and 74% of the money spent. The time and money remaining on the contract are sufficient.

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* Project Leader

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Dayton Laboratory
Dayton, Ohio 45407

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13. ABSTRACT

Characterization of the trifluoronitrosomethane/tetrafluoroethylene copolymer produced by the Thiokol Chemical Corporation and supplied by the U.S. Army Natick Laboratories was continued. The CF_3NO/C_2F_4 copolymer was found to be adaptable to elution fractionation in a perfluorocyclic ether and benzotrifluoride system, even though 85% of the polymer eluted in a narrow viscosity range of 1.3 to 2.0. Values of K' and K" of the copolymer in FC-75 were confirmed to be 0.353 and 0.147, respectively. Thermal decomposition in air and in an inert gas was shown to be identical. The glass transition of the copolymer, as measured by DTA, is in the region of -41° to $-46^{\circ}C$ with no more than $2^{\circ}C$ difference between production lots. The white insoluble material present in the production lots was identified to be basic magnesium carbonate trihydrate.

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Physical properties	8					
Rheology	8					
Nitroso rubber	9					

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