MONSANTO RESEARCH CORPORATION

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Monsanto

DAYTON
LABORATORY

DAYTON, OHIO 45407

Quarterly Report No. 9
PHYSICAL AND RHEOLOGICAL PROPERTIES OF NITROSO RUBBERS
25 June 1965 through 24 September 1965

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12 October 1965

For

U. S. Army Natick Laboratories
Natick, Massachusetts

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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 molecular weight (light scattering) of a gum rubber fraction was determined to be 484,000 when examined in the perfluorocyclic ether solvent. Previous determinations on gums in perfluorotributylamine and Freon 113 indicated a molecular weight of 400,000 and 200,000, respectively. Respective intrinsic viscosities were 0.44, 0.41 and 0.18.

Mold cure plus a stepped post-cure was found necessary in order to obtain adequately vulcanized samples.

One part of carbon black in an amine-cured, SiO_2-filled nitroso rubber increased strength and hardness to produce a rubber of 267 psi strength, 420% elongation and 47 Shore A hardness units. Dynamic modulus measurements indicate a utility for the rubber well below -75°C.

A white precipitate found in earlier gums was not conclusively identified except that it was shown to be chemically different from the typical nitroso gum.

TABLE OF CONTENTS

		Page
I.	INTRODUCTION	1
II.	EXPERIMENTAL	2
	A. DETERMINATION OF MOLECULAR WEIGHT	2
	B. PHYSICAL PROPERTIES OF AMINE-CURED "PURIFIED" NITROSO COPOLYMER	3
	1. Compounding Formulations and Cure Conditions	3
	2. Tensile Strength, Elongation and Hardness	4
	3. 1000 Cycle Per Section Rebound Modulus	4
	4. 3 Cycle Per Section Forced Torsional Pendulum Modulus	5
	5. Clash Berg Torsional Modulus	5
	C. IDENTIFICATION OF WHITE PRECIPITATE FROM THIOKOL NITROSO GUM XP5702	6
III.	TECHNICAL DISCUSSION	7
	A. DETERMINATION OF MOLECULAR WEIGHT	7
	B. PHYSICAL PROPERTIES OF AMINE-CURED "PURIFIED" NITROSO COPOLYMER	8
	C. IDENTIFICATION OF WHITE PRECIPITATE FROM THIOKOL NITROSO GUM XP5702	9
IV.	SUMMARY AND CONCLUSIONS	10
V.	FUTURE PLANS	11
VI.	TIME AND FINANCIAL STATUS	12
VII.	REFERENCE	14
	APPENDIX - Figures	

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-XP 5675, XP5702, XP5812, XP5887, XP5807, XP5704, 0.2 C_2F_3H terpolymer, 0.5 C_2F_3H terpolymer, produced by the Thiokol Chemical Company, 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: determination of molecular weights by light scattering, characterization of the mechanical and dynamic mechanical properties of amine-cured nitroso gums, and identification of a white precipitate found in earlier nitroso gums.

II. EXPERIMENTAL

A. DETERMINATION OF MOLECULAR WEIGHT

Previously reported molecular weight values were determined by means of intrinsic viscosity data, the Mark-Houwink equation, and the constants of Morneau, Roth and Shultz (Ref. 1) for the trifluoronitrosomethane/tetrafluoroethylene copolymer in FC-43 (perfluorotributylamine) and in Freon 113. In addition, one determination of molecular weight by light scattering in the Brice Phoenix light-scattering photometer was performed in FC-43 solvent (Ref. 2).

The utility of FC-75 (isomers of perfluorocyclic ether) as a solvent for the CF_3NO/C_2F_4 copolymer has also been noted (Ref. 3).

As a start in determining the K and a constants for the Mark-Houwink equation of CF_3NO/C_2F_4 in FC-75, molecular weight determinations by light scattering were performed on the 3M-9690 (56703-3) gum. A plot of $HC/\tau(90°)$ versus concentration is shown in Figure 1 and a corresponding Zimm plot of HC/τ versus sin $\theta/2$ + 100C is shown in Figure 2. Extrapolating the concentration to zero, the reciprocal of HC/τ indicates the molecular weight to be 513,000.

The light-scattering determination was made at a wavelength of 436 mμ. The index of refraction, n, used was that reported by the 3M Company to be 1.281 (Ref. 4). $(n-n_o/C)$ was determined by the differential refractometer to be 0.0261. The weight average molecular weight was calculated by means of the Debye equation:

$$\frac{32\Pi^3 n^2 (dn/dc)^2}{3N_o\lambda^4} \qquad \frac{C}{\tau} = \frac{Hc}{\tau} = \frac{1}{M_w P(\theta)} + 2A_2 c + \ldots.$$

where n = refractive index of the solvent

dn/dc = refractive index increment of solution

λ = wavelength of light (436 mμ)

N_o = Avogadro's number (6.02×10^{23})

$P(\theta)$ = scattered light intensity

A_2 = Osmotic second virial coefficient

The shape of the Zimm plot for 3M-9690 in FC-75, as shown in Figure 2, was such that no extrapolation of a molecular weight could be determined.

As a basis for comparison, the molecular weight of National Bureau of Standards 706 broad molecular weight distribution polystyrene was measured on the identical light-scattering arrangement. The NBS reported the molecular weight to be 257,800 ± 930. A data plot of these determinations is shown in Figure 3 and a Zimm plot in Figure 4. The data plot extrapolated to Zero concentration resulted in a molecular weight value of 273,000, and the Zimm extrapolation resulted in a value of 272,000. These were 5.9 and 5.5 percent higher than the NBS reported values.

B. PHYSICAL PROPERTIES OF AMINE-CURED "PURIFIED" NITROSO COPOLYMER

1. Compounding Formulations and Cure Conditions

Eight samples of nitroso copolymer gum were prepared under various cure conditions to produce a cured rubber for physical characterization. A high-molecular-weight fraction (Thiokol Sample XP5812) was used throughout and was washed with Freon 113 to remove possible low-molecular-weight fractions. Except as noted the following formulation was used:

	Weight
"Purified" XP5812 nitroso gum	100.0
SiO_2 Filler	15.0
Triethylenetetramine	1.25
Hexamethylenediamine carbamate	2.50

Sample 9-1 - Cured at 250°F for 60 minutes under 1000 psig in 2.0 inch circular mold. Post cure at 212°F for 18 hours. Filler added to gum on mill.

Sample 9-2 - Cured at 250°F for 60 minutes under 1000 psig in 2.0 inch circular mold. Post cure at 212°F for 18 hours. All ingredients mixed prior to milling.

Sample 9-3 - Cured at 250°F for 60 minutes under 1000 psig in 2.0 inch circular mold. Post cure at 212°F for 18 hours. Ingredients added on mill roll.

Sample 9-4 - Cured at 250°F for 180 minutes under 1000 psig in 2.0 inch circular mold. Post cured in steps: 1 hr. at 120°F, 1 hr. at 150°F, 1 hr. at 185°F, and 18 hr. at 212°F. Ingredients added on mill roll.

Sample 9-5 - Prepared same as 9-4 except cure extended to 300 minutes.

Sample 9-6 - Formulation varied by the addition of 1 part of Elastopar. Cured at 250°F for 300 minutes at 800 psig. Post cure in steps of 30°F up to 212°F for 18 hours.

Sample 9-7 - Formulation varied by the replacement of the SiO_2 filler with Carbon Black (Sterling S). Cured at 250°F for 300 minutes at 1000 psig. Post cure in steps of 30°F up to 212°F for 18 hours.

Sample 9-8 - Formulation varied by the addition of 1.0 part Carbon Black (Sterling S). Cured at 250°F for 300 minutes at 1000 psig. Post cure in steps of 30°F up to 212°F for 18 hours.

2. Tensile Strength, Elongation and Hardness

Of the eight samples that were cured, numbers 9-2, 9-3, 9-6, and 9-7 did not exhibit adequate physical properties to be worthy of test. Sample 9-2 had a yellow appearance, did not seem cross-linked and probably had a strength of less than 100 psi. Sample 9-3 seemed well cured, with a hardness of 30 (Shore A) prior to post-cure and 33 after post-curing. Extreme blistering and porosity prevented adequate tensile tests. Sample 9-6 turned dark brown during cure indicating degradation had occurred. Tensile strength (not measured) would have been well below 100 psi. Sample 9-7 did not crosslink; however, the Carbon Black prevented the observability of any degradation. Its tensile strength would have been well below 100 psi also.

The tensile strength, elongation, and hardness of samples 9-1, 9-4, 9-5, and 9-8 are tabulated on the following page.

3. 1000 Cycle Per Second Rebound Modulus

The 1000 cps modulus for the uncured nitroso gum had previously been determined by the rebound technique (Ref. 5). The 1000 cps modulus of cured samples 9-4 and 9-5 is shown in Figure 5. Note the minimum rebound at temperatures of -12 and -7°C, respectively.

MECHANICAL PROPERTIES OF AMINE-CURED NITROSO RUBBER

Sample Number	Tensile* psi	Elongation*	Hardness (Shore A) Cured	Post Cured
9-1	130	100	-	27
9-4	∿210	∿400	20	38
9-5	232	420	32	39
9-8	267	420	-	47

* Microtensile specimen (1.0 inch gage) - average of 3 specimens.

The rebounding was from the surface of the rubber specimen as opposed to the technique of rebounding from an aluminum foil surface layer, as was necessary for the tacky gums.

4. 3 Cycle Per Second Forced Torsional Pendulum Modulus

Using a modified Nonius Torsion pendulum, as shown in Figure 6, the 3 cps loss modulus of samples 9-5 and 9-8 were determined. The torsion pendulum is modified by replacing the fine supporting wire with a rigid steel rod. The rigid rod provides a driving force which maintains the frequency of test at approximately 3 cps and allows testing at lower modulus values which a normal torsion pendulum, due to the required support of the specimen, would not allow. The damping values are of the steel/rubber composite system, so cannot be related to modulus terms developed merely on material properties. The temperature of maximum damping, however, is real and meaningful. The damping of rubber samples 9-5 and 9-8 is shown in Figure 7.

5. Clash Berg Torsional Modulus

The torsional modulus (static) by the more standard technique of Clash Berg is shown in Figure 8 for rubber sample 9-5. T_f (modulus at 4500 psi) is at -49°C and T_{665} (modulus at 665 psi) is at -32.5°C. This provides a Stifflex range of 16.5°C with a midpoint of -40.8°C.

C. IDENTIFICATION OF WHITE PRECIPITATE FROM THIOKOL NITROSO GUM XP5702

It had previously been reported that there were from 2 to 37 percent insolubles in nitroso gum samples XP5702 (Ref. 2). Part of this was in the form of a white precipitate which was centrifuged from the solution. It had been expected that this would be a known material because of its prominence. This evidently was not the case, so at this time an analysis of the white precipitate was initiated.

The IR spectrum of this material from 1 to 16 microns is shown in Figure 9.

III. TECHNICAL DISCUSSION

A. DETERMINATION OF MOLECULAR WEIGHT

The molecular weight of the 3M-9690 (56703-3) trifluoronitroso methane/tetrafluoroethylene copolymer was determined to be 513,000 when measured by light scattering in the perfluorocyclic ether solvent (FC-75). The intrinsic viscosity of this gum was previously determined to be 0.44 in the FC-75 solvent (Ref. 3).

NBS broad molecular weight distribution polystyrene, when evaluated on the identical apparatus, resulted in values about 5.5 percent higher than the NBS value, i.e., 272,000 compared to 257,800.

Assuming the 5.5% as a reasonable deviation of light-scattering measurement and applying this to the value of 513,000, the molecular weight of the 3M-9690 gum could fall into the range of 484,000. This may be compared to the value of 361,000 determined by intrinsic viscosity measurements in FC-43 (with the constants of Morneau, et al. (Ref. 1), a light scattering determination in FC-43 which resulted in a molecular weight of 400,000 (using an estimated index of refraction for FC-43), and a value of 200,000 determined by intrinsic viscosity measurements in Freon 113.

The corresponding intrinsic viscosities and molecular weights for the various solvents and the 3M-9690 gum are:

		M_W	
Solvent	[η]	Calculated	Measured
FC-75	0.44	-	484,000*
FC-43	0.41	361,000	400,000
Freon-113	0.18	200,000	-

*Corrected for apparatus deviation

As had been noted on light-scattering determinations of the 3M-9690 in FC-43, the Zimm plot (see Figure 2) was long and narrow with sufficient scatter-uncertainty to render it of dubious value for a determination of molecular weight.

B. PHYSICAL PROPERTIES OF AMINE-CURED "PURIFIED" NITROSO COPOLYMER

It was found that the cure cycle and post-cure cycle had to be modified greatly to obtain adequate rubber. The cure was extended to 300 minutes at 250°F and the post-cure applied in 30°F intervals for a period of 1 hour each up to 212°F, then held for 18 hours. Shorter cure times resulted in insufficient crosslinking, and the lack of stepped post-cure resulted in blistering and increased porosity.

The addition of a slight amount of Elastopar resulted in degradation of the gum during cure. Although no further studies were conducted, cures at lower temperatures may be possible. The use of Carbon Black as a filler was also found to be inadequate for reinforcing, and prevented crosslinking. The use of a higher-molecular-weight gum apparently improved the properties of the cured rubber. However, the results were not conclusive. Tensile strengths of 230 psi, 420% elongation, and a hardness of 39 were obtained with the amine-cured, SiO_2-reinforced nitroso rubber. An additional improvement of 35 psi in strength and 8 Shore hardness units resulted from the addition of 1 part of Carbon Black in the SiO_2-filled rubber. Thus a rubber of 267 psi tensile strength, 420% elongation, and 47 Shore A hardness units was obtained.

The temperature of maximum loss (minimum rebound), as determined by 1000 cps rebound modulus, was found to vary between -7 and -12°C for various amine cured specimens. The temperature of maximum loss for the 1000 cps modulus of the uncured gum had previously been determined to be -18°C (Ref. 5). Thus an expected increase in this temperature with curing was noted.

The temperature of maximum loss as determined by 3 cps torsional pendulum modulus was found to be in the range of -41 to -44°C for the amine-cured rubber. Relating the 3 cps modulus to the 1000 cps modulus would result in a translation of about 11°C per decade of frequency. This is a relatively high translation for a crosslinked rubber and further analysis will be needed to confirm it.

T_f (modulus at 4500 psi) as determined by the Clash Berg torsional modulus was -49°C with a Stifflex range of 16.5°C.

The various modulus measurements indicate the utility of the nitroso copolymer at low temperatures. Reasonable flexibility is available as low as -60°C and the rubber may well be utilized much below -75°C.

C. IDENTIFICATION OF WHITE PRECIPITATE FROM THIOKOL NITROSO GUM XP5702

The IR spectrum of the XP5702 gum, from which this precipitate was removed, was previously reported in our Sixth Quarterly report (Ref. 6). Figure 9, the IR spectrum of the white precipitate, is quite similar to the gum spectrum below 7.5 microns (7.5-15μ). However, a new band is observed at 11.3μ and the band at 12.0μ is not observed. The absorption at 12.0μ had been assigned to the CF_2-CF_2 bond.

For the white precipitate the region above 7.5 microns (2.5-7.5μ) is quite different from the nitroso rubber gums. Little or no absorption is usually observed in this region in the purified gums. In the precipitate, however, four strong bands are present. The bands at 2.9 and 6.15 microns are attributed to H_2O which the sample may have picked up over its storage life. The bands at 6.7 and 7.02 microns are unidentified. However, there is some indication that they may represent an amide structure. The effect of fluorine in an amide structure is unknown, but may have caused the slight frequency shift from that expected for a conventional amide structure. From these results the exact structure of the precipitate cannot be determined except to say that it is different from the nitroso gum.

IV. SUMMARY AND CONCLUSIONS

The molecular weight of a 3M-9690 trifluoronitrosomethane/tetrafluoroethylene copolymer was determined in perfluorocyclic ether (FC-75) to be 513,000. Applying a 5.5% deviation determined by evaluation of NBS polystyrene sample, the molecular weight is reported to be 484,000. The intrinsic viscosity of the gum in FC-75 was 0.44.

The molecular weight of 484,000 in FC-75 is compared to that measured in FC-43 to be 400,000 and calculated from Freon 113 data to be 200,000.

Cure cycles of up to 5 hours with step post curing to 212°F were required to obtain a suitable amine cured, SiO filled, nitroso rubber.

Elastopar and Carbon Black were found to be unsuitable to obtain a crosslinked rubber.

Tensile strengths of 230 psi, elongation of 420%, and a hardness of 39 were obtained with the amine-cured, SiO_2-filled nitroso rubber.

One part Carbon Black in the amine-cured, SiO_2-filled nitroso rubber increased the strength and hardness of the rubber to 267 psi and 47 Short A units with 420% elongation.

The peak damping of the 1000 cps rebound modulus was in the region of -7 to -12°C and the 3 cps peak was in the region of -41 to -44°C.

Clash Berg T_f modulus was -49°C with a Stifflex range of 16.5°C. The modulus values indicate a utility available to the rubber well below -75°C.

The white precipitate recovered from sample XP5702 was determined by IR to vary from the typical nitroso copolymer. Although an amide structure was indicated this was not proved conclusively.

V. FUTURE PLANS

For the purpose of characterization, a typical fluoronitroso copolymer gum has been defined as that portion having a high molecular weight distribution. Accordingly, we will proceed to characterize this typical gum completely. This will include an elution fractionation of the gum, determination of the molecular weights of the fractions, and point viscosities of these fractions. A limited curing program to produce nitroso rubber for characterization will be conducted and the rubber characterized. The molecular weight distribution of the "volatile" portion of the existing gums will be determined.

VI. TIME AND FINANCIAL STATUS

	Hours to 9/30/65
George L. Ball III, Research Specialist*	968
Ival O. Salyer, Research Manager, Polymer Applications	134
Harry S. Wilson, Research Group Leader	294
John V. Pustinger, Analytical Group Leader	31
F. Neil Hodgson, Research Analytical Chemist	22
Lucius Gilman, Manager, Plastics and Polymer Research	130
William R. Smith, Analytical Chemist	5
Professional, Mixed	8
Total	1592
Charlotte D. Fritsch, Research Technician	664
John E. Strobel, Research Technician	46
Richard L. Evers, Research Technician	24
Donald O. Douglas, Research Technician	33
Margaret S. Ross, Research Technician	223
Ralph R. Ferguson, Research Technician	29
Gary A. Clinehens, Research Technician	151
Rodrigue G. Thibodeau, Research Technicial	238
Conrad A. Cenerizio, Research Technician	26
Technical, Mixed	54
Total	1488
Grand Total	3080

* Project Leader

As of 30 September 1965 $42,494 has been spent. The contract, less fee, is for $59,335, leaving a balance of $16,841.00.

67% of work has been completed and 66.8% of the money spent. The time and money remaining on the contract is sufficient.

VII. REFERENCES

1. "Trifluoronitrosomethane/Tetrafluoroethylene Elastomers, Dilute Solution Properties and Molecular Weight", Morneau, G. A., Roth, P. I., and Shultz, A. R., J. Polymer Sci., 55, 609 (1961).

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

3. "Physical and Rheological Properties of Nitroso Rubbers", Quarterly Report No. 4, Contract No. DA19-129-AMC-151(N), (O.I. 9115), 24 July 1964.

4. 3M Company supplied information due to E. F. Beale, University of Maryland.

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

6. Physical and Rheological Properties of Nitroso Rubbers", Quarterly Report No. 6, Contract No. DA19-129-AMC-15(N), (O.I. 9115), 14 January 1965.

APPENDIX

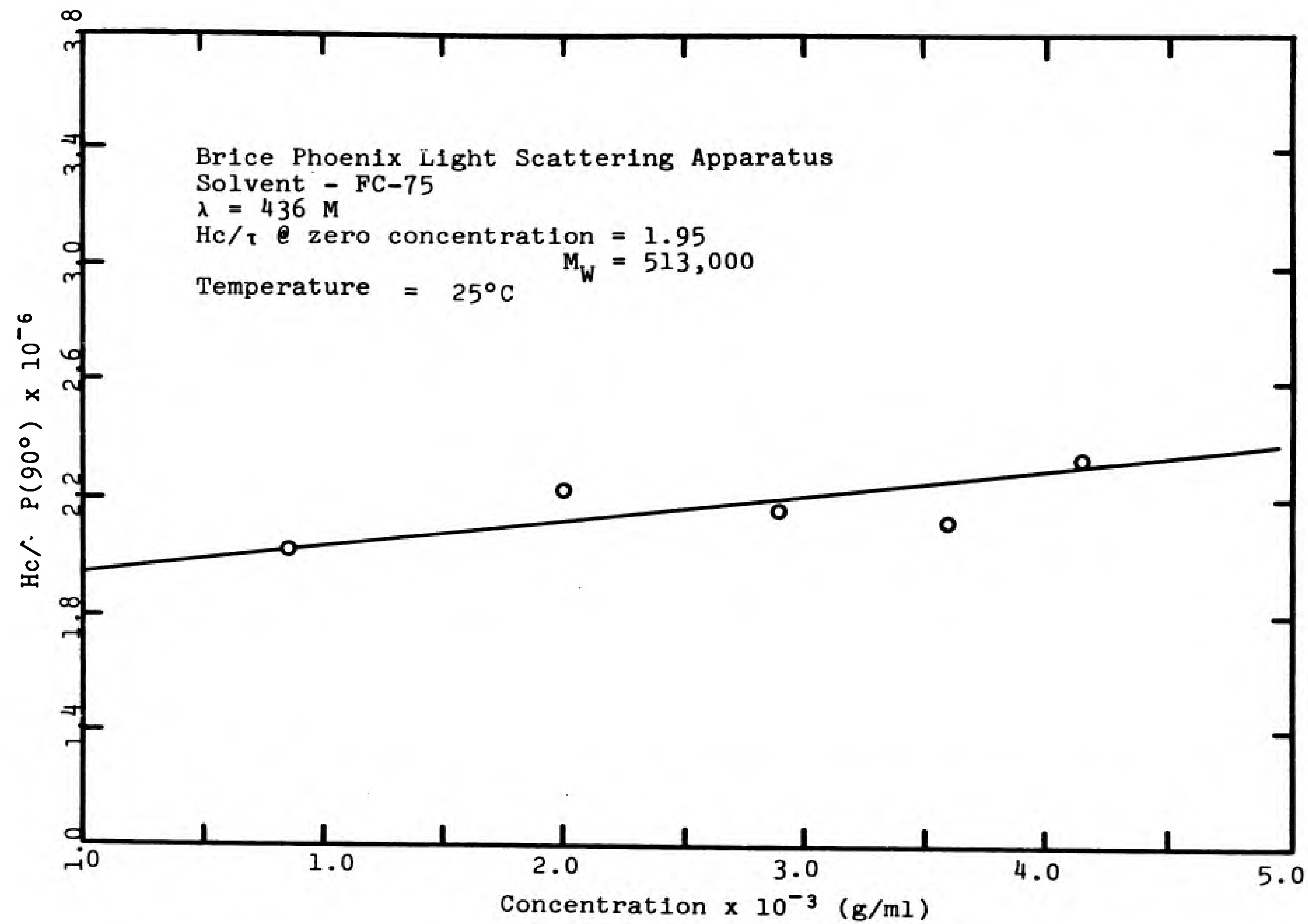

Figure 1. Light scattering data of 3M-9690 nitroso gum in FC-75

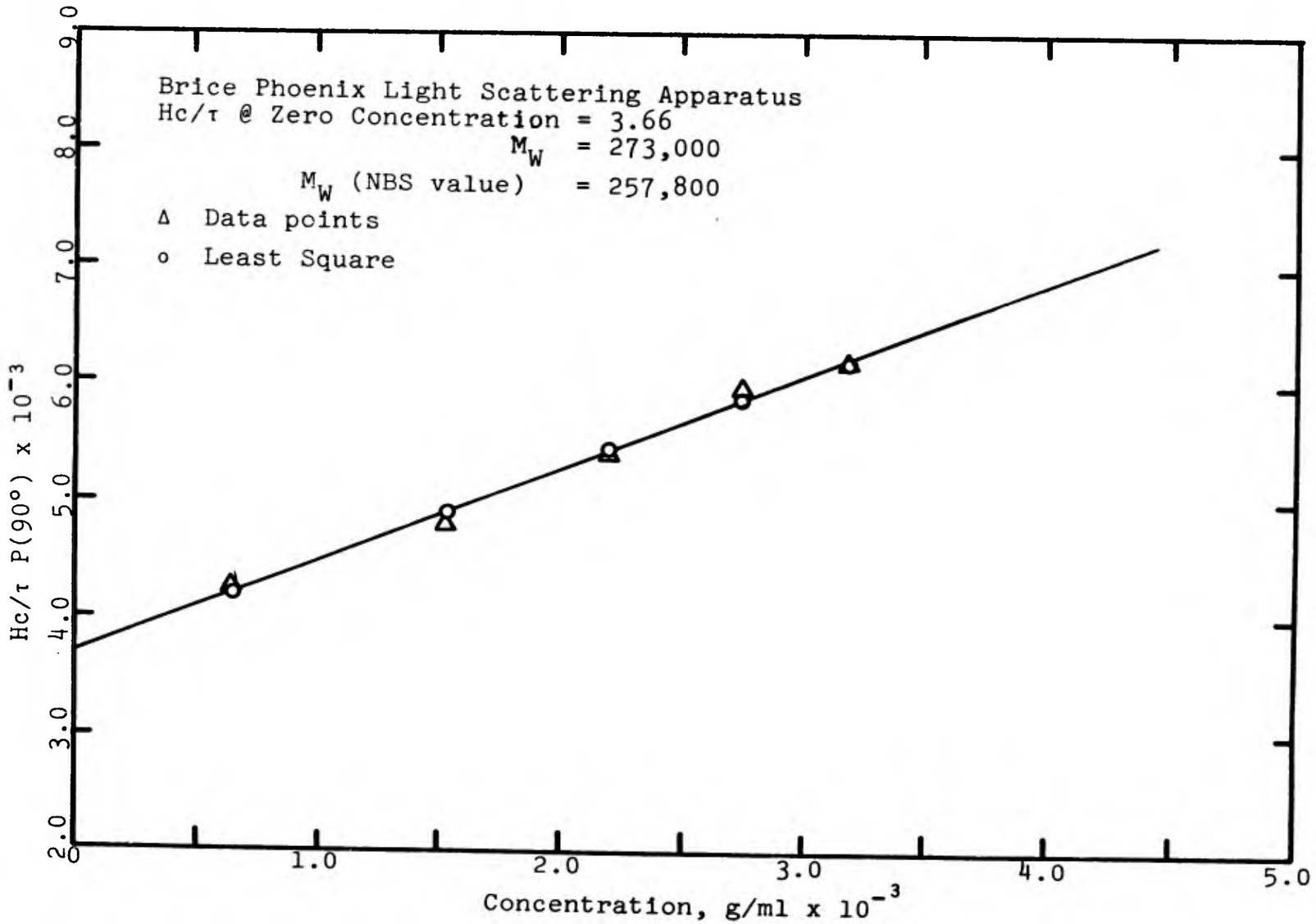

Figure 3. Light scattering data of NBS broad molecular weight polystyrene.

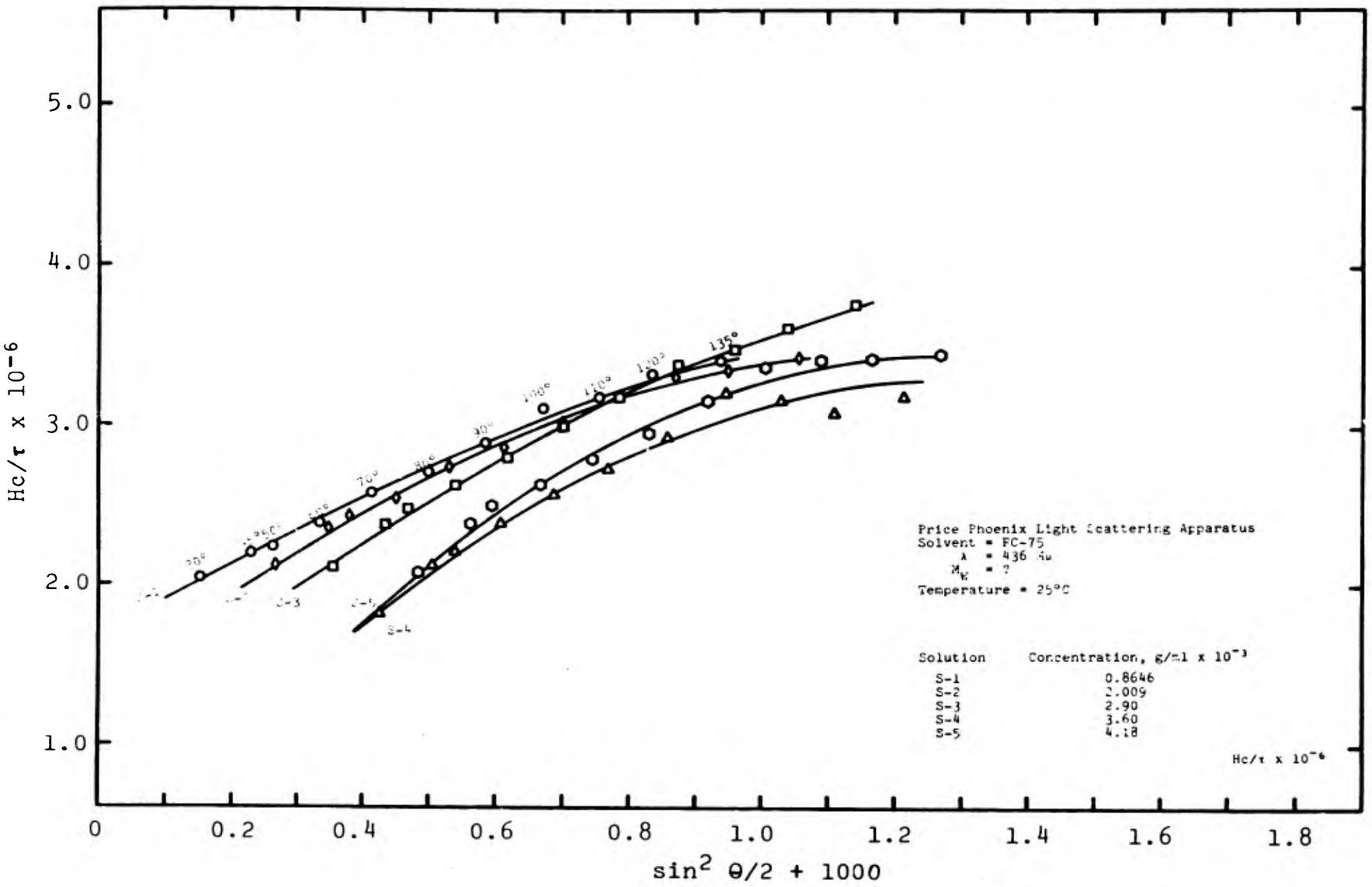

Figure 2. Zimm Plot of 3M-9690 nitroso gum in FC-75

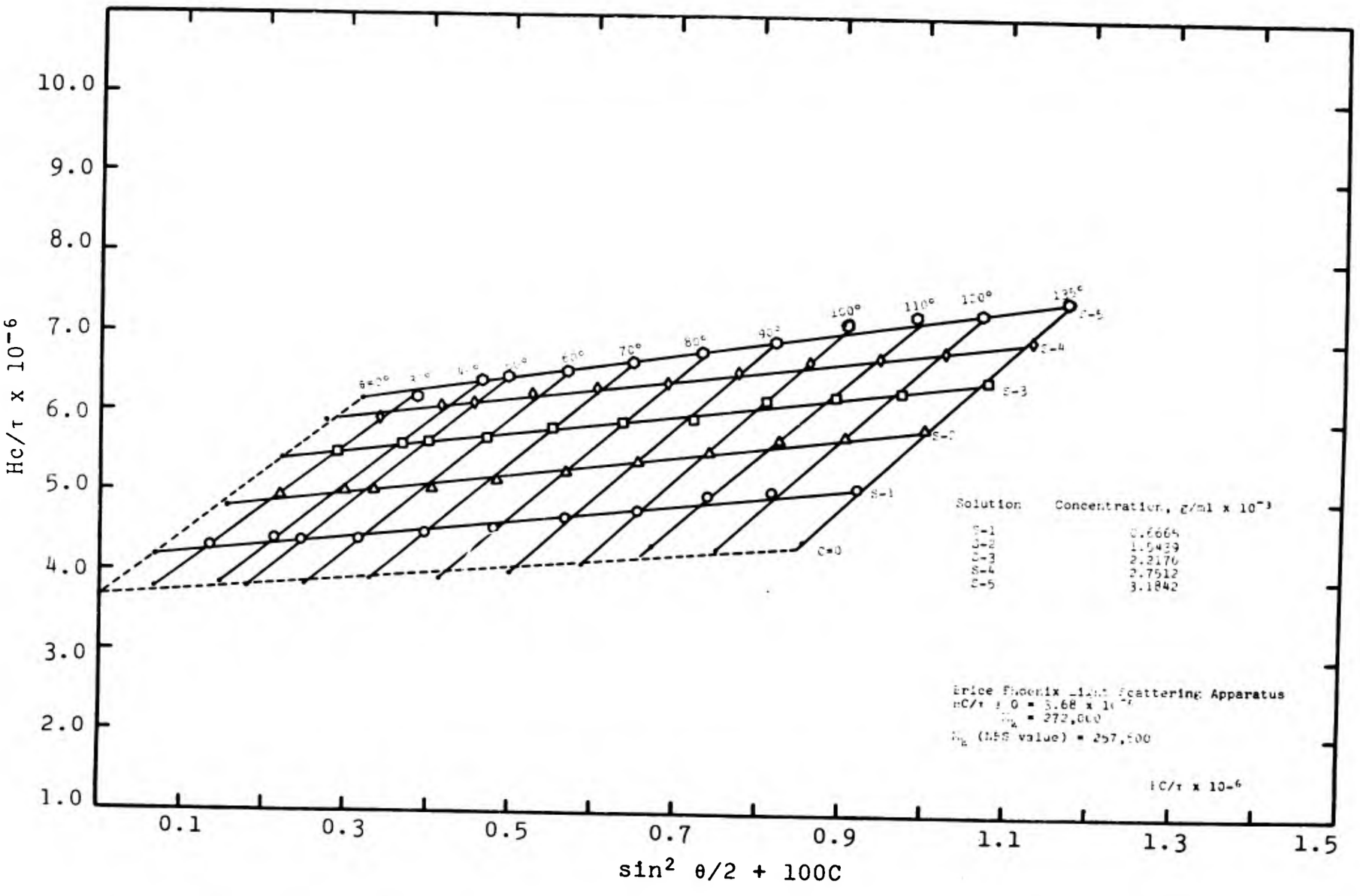

Figure 4. Zimm plot of NBS broad molecular weight polystyrene

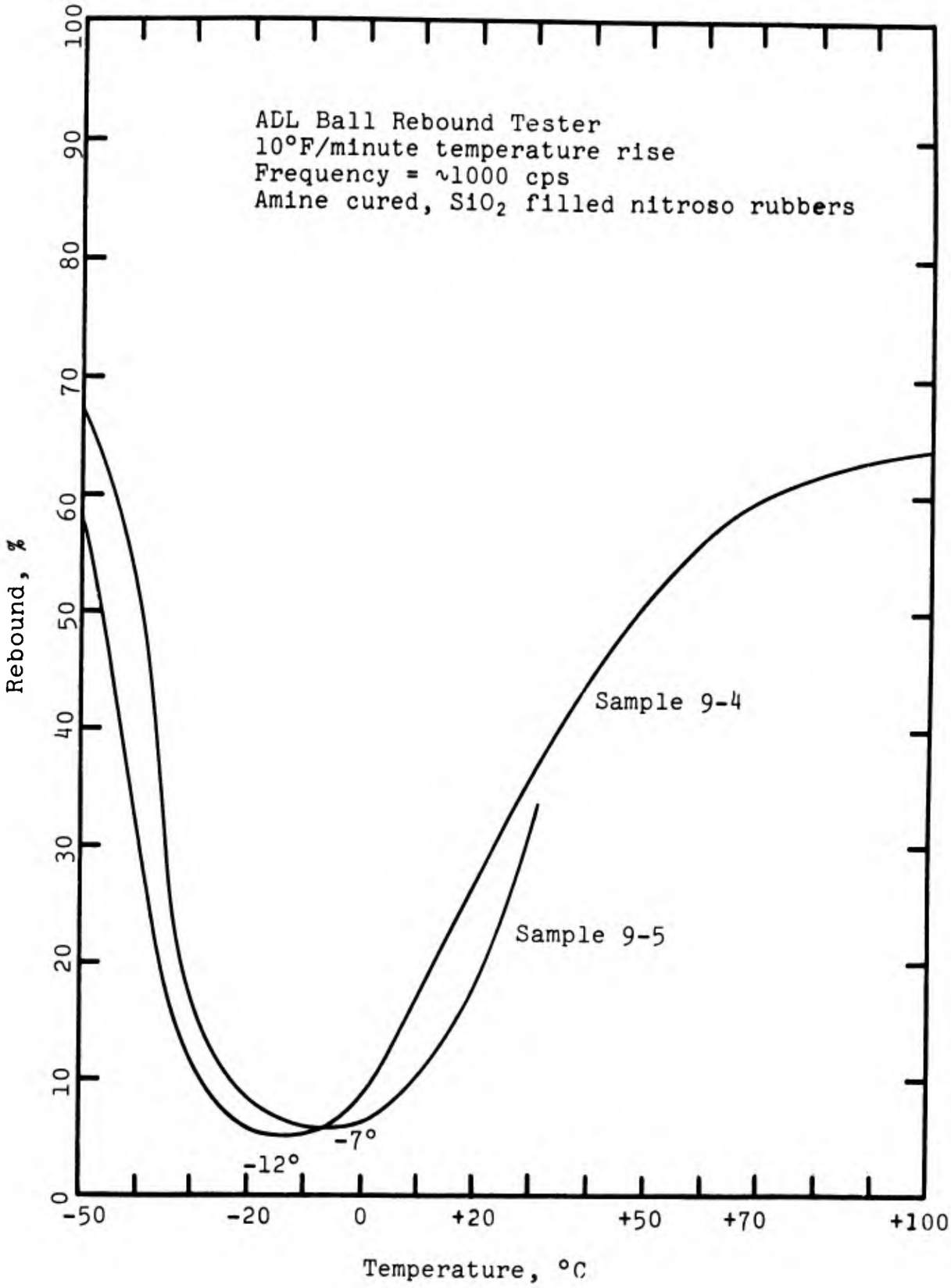

Figure 5. 1000 cps modulus of nitroso rubber samples 9-4 and 9-5

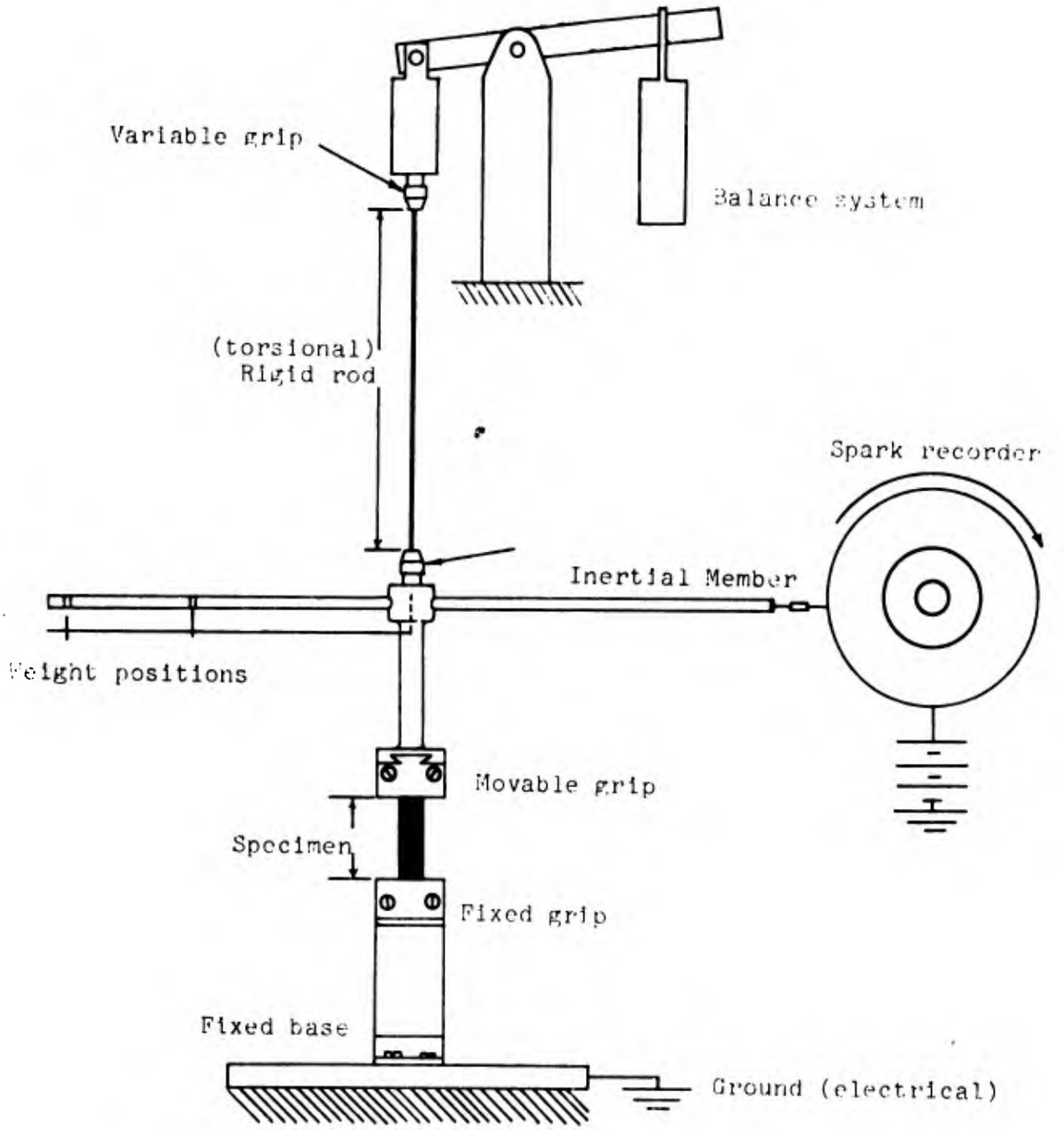

Figure 6. Modified nonius torsion pendulum apparatus for determination of 3 cps modulus

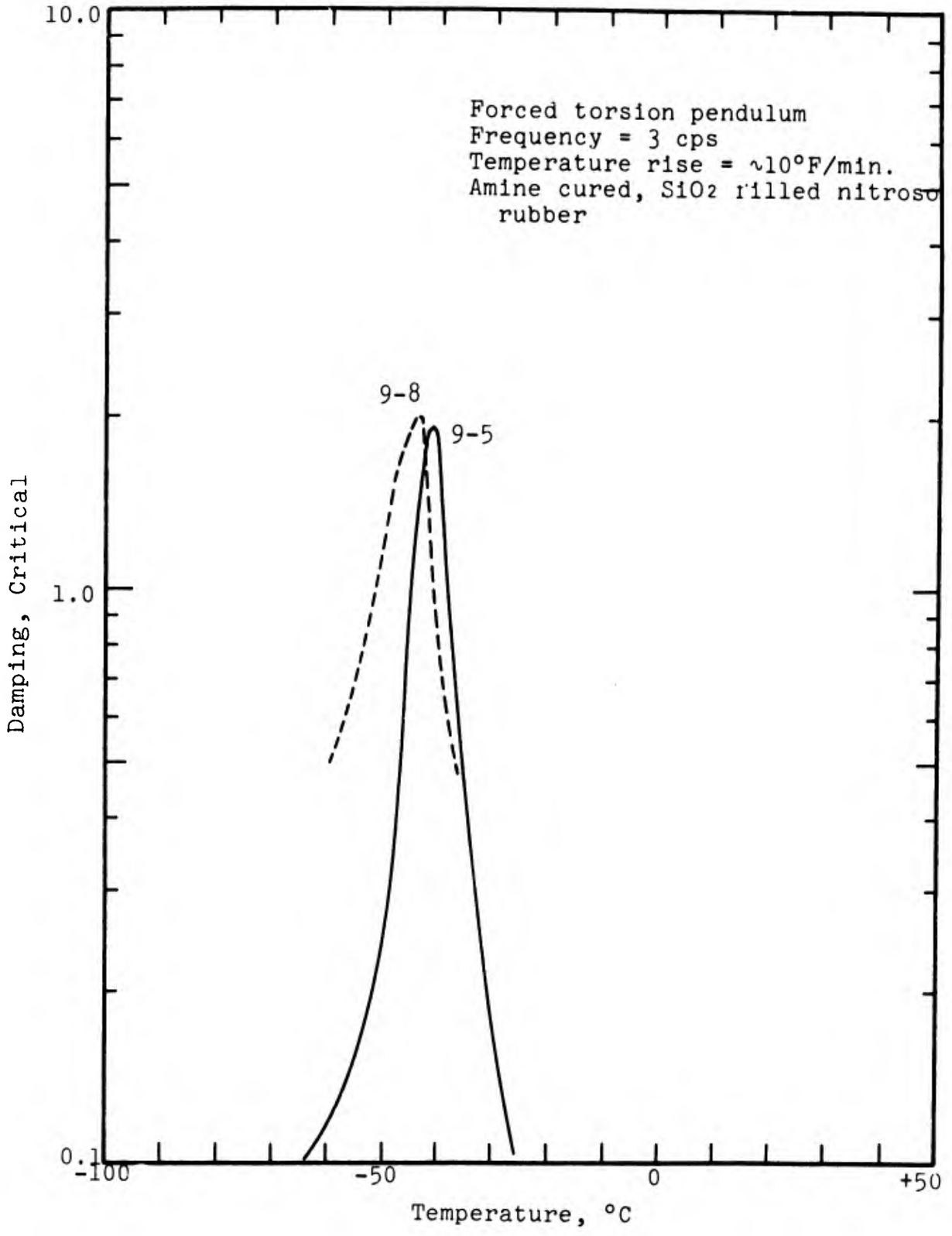

Figure 7. 3 cps modulus of nitroso rubber samples 9-5 and 9-8

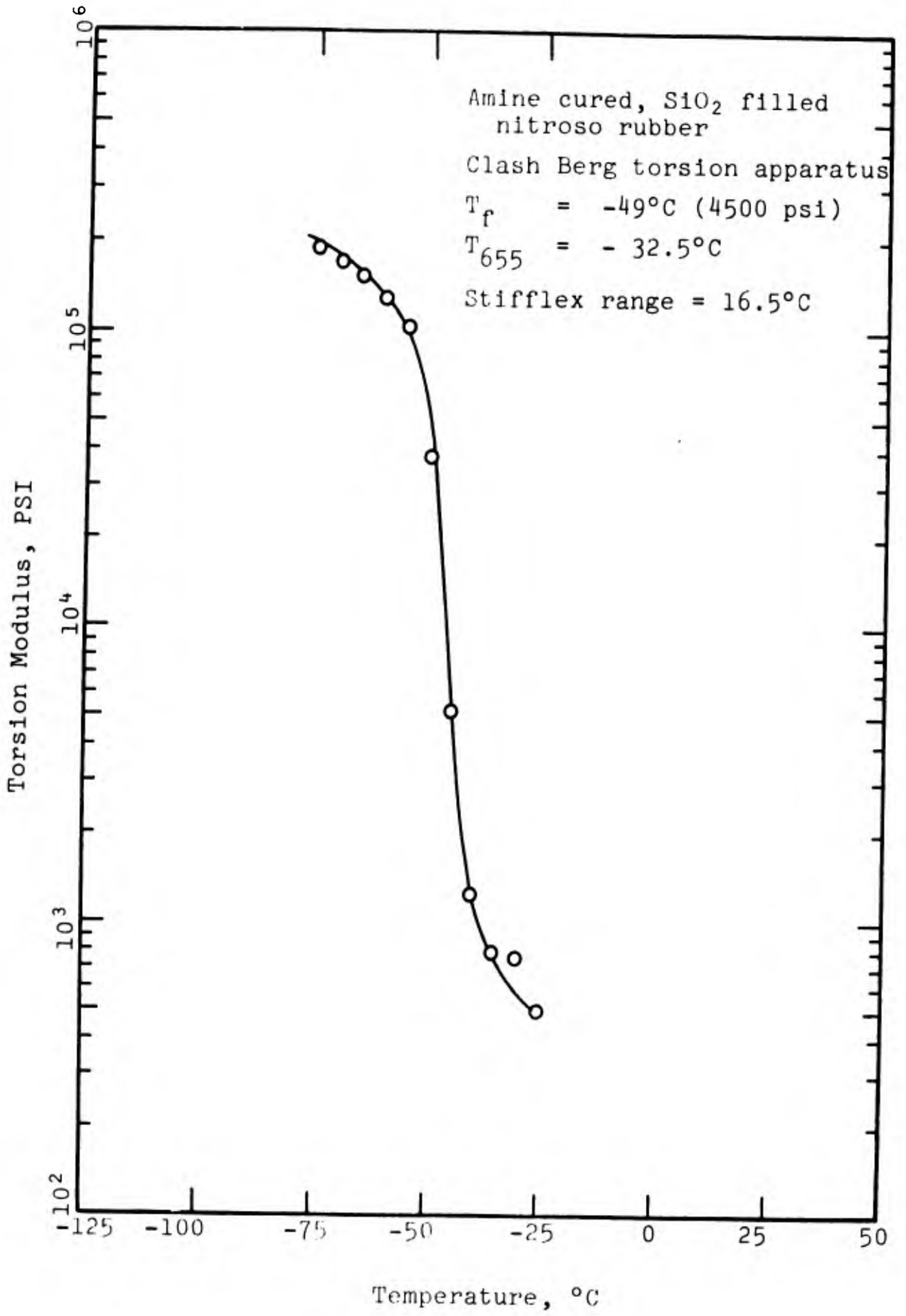

Figure 8. Clash Berg torsion modulus for nitroso rubber sample 9-5

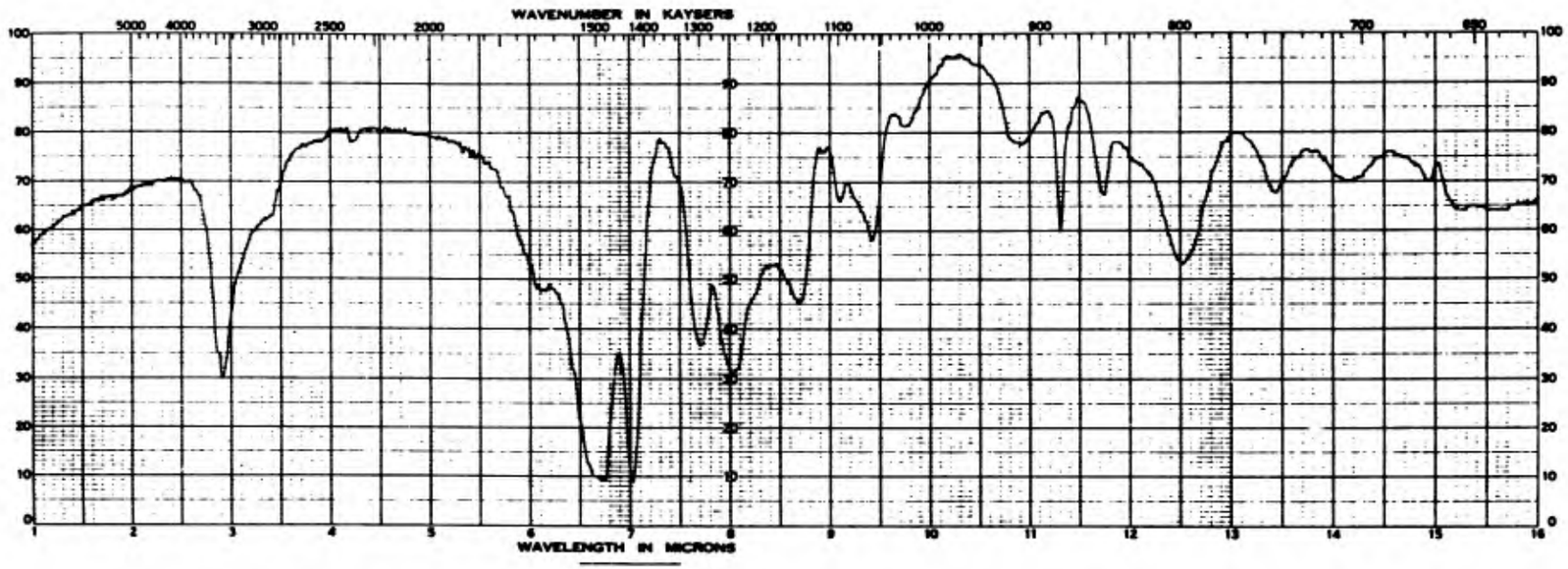

Figure 9. Infrared spectra of white precipitate from Thiokol nitroso gum sample XP5702

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Ball, G. L., III
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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 molecular weight (light scattering) of a gum rubber fraction was determined to be 484,000 when examined in the perfluorocyclic ether solvent. Previous determinations on gums in perfluorotributylamine and Freon 113 indicated a molecular weight of 400,000 and 200,000, respectively. Respective intrinsic viscosities were 0.44, 0.41 and 0.18.

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Rheology	8					
Nitroso rubber	9					

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