MONSANTO RESEARCH CORPORATION
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DAYTON
LABORATORY

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PHYSICAL AND RHEOLOGICAL PROPERTIES OF NITROSO RUBBERS

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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 previously reported "volatile" portion of the fluoronitroso gums was identified as purely low molecular weight fractions. No other contaminants were found. Removal of this low molecular weight portion was demonstrated by solvent precipitation and vacuum drying techniques. The low molecular weight portion boils predominately in the 100°C region and is present up to 200°C. Except for average molecular weight no differences were noted between the present Thiokol gums and the earlier 3M gum. A usable solvent/non solvent system consisting of FC-75 and benzotrifluoride was determined and the useful solvent ratios and temperatures set. The 1000 cycle per second glass transition was determined to be -18°C. Amine cure of the Thiokol gum following a recommended procedure provided poor rubber specimens. Violent decomposition of the cured rubber occured at 270°C as had the gum.

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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 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: determining the amount of "volatile" material in the submitted gums, studying thermal decomposition modes by means of TGA and Mass Spectrograph, finding suitable solvent/non solvent systems for use in fractionating, measurement of the 1000 cps glass transition, and curing and evaluation of fluoronitroso rubber samples.

II. RESULTS

A. IDENTIFICATION OF THE VOLATILE PORTION OF THE TRIFLUORONITROSOMETHANE/TETRAFLUOROETHYLENE GUM

As has been previously reported, gum samples XP5675 and others exhibited a volatile portion at temperatures below 100°C (Ref 1). In order to identify this volatile portion and its relationship to the overall nitroso gum, a series of isothermal TGA's, isothermal mass spectrographs, NMR, and IR were conducted.

1. Isothermal Thermogravimetric Analyses

Figure 1 shows the isothermal runs at the various temperatures from 50°C up to 275°C and their corresponding weight losses. These were continuing runs on the identical specimen. Equilibrium weight was reached in most cases within 4.5 hours. A helium atmosphere was maintained which in combination with the little sample remaining at 275°C (.0061 g) avoided explosive decomposition.

Figure 2 shows the thermal weight loss of the nitroso gum residue which had been treated isothermally in 50°C increments (as shown in Figure 1) up to 275°C. The sample weighed 0.0061 grams and left a residue of 0.0003 grams at 500°C.

Figure 3 is a composite of the TGA's from 50°C up to 330°C showing the differential and total weight loss as a function of temperature for sample XP5675. Note the twin regions of weight loss with peak temperatures of 100°C and 250°C.

Also shown in Figure 3 is a sample of XP5675 which had been solvent precipitated (See Section C for method) to remove the low molecular weight portion which is volatile below 175°C. Vacuum drying at 100°C could also be utilized to accomplish this volatiles removal.

2. Isothermal Mass Spectrographic Analyses

Figure 4 shows a composite of the pressure (in microns) of a series of isothermal runs on the mass spectrograph. These data show the differential weight loss of sample XP5675, the precipitated XP5675, XP5812, and the 3M-56703-3 gum, and confirms the isothermal TGA results.

Figures 5 through 12 show the intensity of the ionic species most prominent in the mass spectrograph at 50°C intervals, from 50°C up to 275°C. These masses were 30, 31, 47, 50, 66, 69, 99, 100, 114, 119, 130, 133, 164, 180 and 199. Many other masses were present as shown in Figures 13 and 14 but were not prominent enough to be included in the already complicated figures. Suffice it to say that these additional masses were identical for all the samples tested.

3. Nuclear Magnetic Resonance

F^{19} NMR were run to determine differences in the precipitated gum and the volatile portion. H^1 NMR was conducted to determine the presence of any hydrogen in the nitroso gum.

The F^{19} spectra was the same as previously reported (Ref 2) and both the gum and volatile portion were identical. Slight differences were noted, however, due to the type of sample, i.e. film or solution. The H^1 NMR spectra was a flat base line indicating no hydrogen.

4. Infrared Analysis

In order to determine the presence of any hydrogen or CH_2 in the nitroso gum or its volatile portion, IR spectra in the 1-7 micron wavelength region was measured at high intensity. All spectra for specimens XP5675, precipitated XP5675, the volatile portion of XP5675 and 3M-56703-3 were identical and are shown in Figure 15.

B. THERMAL DECOMPOSITION OF THE NITROSO COPOLYMER BY THERMOGRAVIMETRIC AND MASS SPECTROGRAPHIC ANALYSES

The decomposition of the nitroso gum in an inert atmosphere (He) was shown in the series of isothermal TGA's, and in isothermal mass spectrographs.

Figure 3, which shows TGA weight loss with temperature, and Figure 1, which shows weight loss with time at a given temperature, indicate that a given weight loss is associated with a given temperature and reaches equilibrium with time. Significantly also no rapid or explosive weight loss was noted over the temperature range investigated.

The mass spectrographic data, by lack of any significant difference, indicate that the mode of decomposition is similar for all the specimens. Those masses of interest in the 250°C range are 66 and 133, the expected products of the trifluoronitrosomethane/tetrafluoroethylene mass of 199. These are shown in Figures 5 through 12.

C. SOLVENT/NON SOLVENT SYSTEMS FOR SEPARATION OF THE TRIFLUORONITROSOMETHANE/TETRAFLUOROETHYLENE COMPONENTS OF VARIOUS MOLECULAR WEIGHTS

In order to obtain various molecular weight fractions of the nitroso copolymer a solvent/non solvent system is necessary.

Four useful solvents had previously been selected (Ref 2). Of these, two were selected as having useful boiling points (less than 100°C), being relatively non-viscous, and having a miscible non solvent with similar properties.

The FC-43 [$(C_4F_9)_3N$] was too viscous (2.8 cs) and has a boiling point of 170°C. The Freon 112 doesn't melt or flow up to 25°C, therefore is most inconvenient as a fractionating solvent.

The Freon 113/Acetone, solvent/non solvent, system has been reported previously by others (Ref 3). It is very good except for the fact that the higher molecular weight nitroso portions are difficult to get into the Freon 113 solvent. The boiling point of 50°C also limits the temperature range usable to aid in separation of the fractions (by improving solubility).

Our preference for FC-75 [isomers of $C_8F_{16}O$] as a solvent has previously been noted (Ref 2). It also has a boiling point of ∿100°C and is relatively non viscous (0.65 cs) from room temperature up to its boiling point. Various non solvents miscible in the FC-75 were tried with benzotrifluoride being the best. Very few solvents are miscible in the FC-75. The benzotrifluoride has a boiling point of ∿100°C and about equal viscosity to the FC-75.

The precipitation point at 23°C and a range of useful concentrations and temperatures for fractionation by column elution have been determined for sample XP5702, a higher molecular weight gum portion. The concentrations and temperatures are as follows:

Solution = XP5702/FC-75 = (2.0 g/100 ml)

Temperature °C	Solution:benzotrifluoride ml:ml	Condition of Solution Visual
23	100:91.2	Polymer all out
80	100:91.2	Polymer all in
58	100:91.2	Hazy
43	100:91.2	Definite fraction drop out
40	100:91.2	Polymer all out

D. GLASS TRANSITION AT 1000 CYCLES PER SECOND BY REBOUND METHOD

Figure 16 shows the rebound tester used to determine the glass transition of a material at 1000 cps by measuring the rebound of a small (1/8 inch diameter) ball bearing from the surface of a specimen. Although complete correlation has not been made, the percent rebound as measured by the instrument has been shown to correlate to the logarithmic decrement of a material (Ref 4). Since the logarithmic decrement goes through a maximum where the elastic modulus decreases rapidly from a glassy state to a rubbery state (i.e., the glass transition) we may roughly predict the glass transition temperature at 1000 cps.

The need for such a test is a result of the inability to obtain any reasonably shaped or geometrically stable specimen. The rebound test requires only a flat non-sticky surface. The tackiness of the nitroso gum is no problem at low temperatures; however, from 0°C and up (where tackiness is a problem) a surface of aluminum can be used without altering the position of the transition region (only magnitude, of relative importance only, is affected).

Data of percent rebound versus temperature for specimen XP5675 are shown in Figure 17. A 0.5 mil aluminum foil covering was attached to the gum as a rebound surface. The specimen was tested from -100 up to 40°C.

E. PHYSICAL PROPERTIES OF AN AMINE CURED PURIFIED NITROSO COPOLYMER

In order to provide a nitroso copolymer rubber for characterization and to aid in determining the effect of the volatile nitroso portion on curing, the following formulation was compounded. This is the 3M recommended formulation (Ref 5).

	Weight
"Purified" XP5675*	100.0
SiO_2 filler**	15.0
Triethylenetetramine	1.25
Hexamethylenediamine carbamate	2.50

Cure: Press 60 minutes at 250°F.
oven 18 hours at 212°F

* Precipitated high molecular weight portion

** Hi Sil 101

1. Thermogravimetric Analysis

A TGA in helium and in air was conducted on the cured rubber, the results of which are shown in Figure 18. In air the rubber violently decomposed at 270°C forcing the remainder of the specimen out of the weighing crucible.

2. Tensile Strength

The specimen prepared as shown above was somewhat porous and layered causing difficulty in measurement of dimensions and causing doubt as to the validity of a tensile test. However, three microtensile (1.0 inch gage) specimens were tested. The average tensile strength at break was 130 psi with an average elongation at break of 100 per cent.

3. Hardness

By Shore A hardness the rubber measures 27 shore units.

4. Density

By displacement the density was 1.099 g/cc.

F. REFERENCES

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

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

4. Raphael, T. and C. D. Armeniades, "Correlation of Rebound Tester and Torsion Pendulum Data on the Round Robin Polymer Samples," Unpublished data of A.S.T.M., D-20, Section I-O, 5 February 1965.

5. Montermoso, J.C., C.B. Griffis, Angus Wilson and G.H. Crawford, "Vulcanisation and Properties of Nitroso Rubber," Rubber and Plastics Age, 42, 514(1961).

6. Shultz, A.R., N. Knoll, and G.A. Morneau, "Trifluoronitrosomethane/Tetrafluoroethylene Copolymer: Degradation by Heat and by Radiation," J. Polymer Sci., 62, 211 (1962).

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

III. TECHNICAL DISCUSSION AND CONCLUSIONS

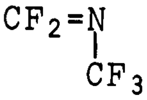
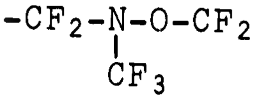
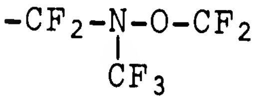
A. IDENTIFICATION OF THE VOLATILE PORTION OF THE TRIFLUORONITROSOMETHANE/TETRAFLUOROETHYLENE GUM

It had previously been reported that sample XP5675 contained a "volatile portion consisting of low molecular weight nitroso gum and a solvent" (Ref 1). In order to determine the nature and makeup of this volatile portion, isothermal TGA's and mass spectrographs were run, along with NMR and IR, to determine similarity and the existence, if any, of hydrogen or the CH_2 group which had been suspected.

Figure 3 shows the existence of the volatile which starts coming off at 50°C, has a maximum at 100°C and continues to be evolved above 200°C (below the decomposition temperature). The removal of this volatile portion was demonstrated on a solvent precipitated fraction which, following the expected thermal decomposition nature of a gum with a single distribution of molecular weights, started decomposing at 150°C, proceeded through a maximum at 250°C and continued to decompose the various molecular weight components up to 500°C. The volatile portion thus has the nature of a nitroso gum of low and normally distributed molecular weights.

Knowing the nature of the volatile product, a series of mass spectrographs were run isothermally to determine the make up. If solvents or other contaminants were present, it would be expected that these would show up coming off at temperatures in the volatile region adding to those masses expected for the nitroso copolymer. The results of the mass spectrographic data showed that there were no, even slightly significant, different masses given off in the volatile region and that the products given off in the volatile region were identical to those at elevated temperatures. The most prominent masses given off by the nitroso copolymer are those shown in figures 5 through 12. The most probably species to associate with those masses are:

Mass	Possible Species
30	NO
31	CF
47	CF=O

50	CF_2
66	$CF_2{=}O$
69	CF_3
99	CF_3NO
100	C_2F_4
114	C_2F_4N
119	C_2F_5
130	$(CF_2)_2NO$
133	 $CF_2{=}\underset{\begin{array}{c}\vert\\CF_3\end{array}}{N}$
164	$(CF_2)_2NCF_2$ (or C_6F_4O)
180	 $-CF_2-\underset{\begin{array}{c}\vert\\CF_3\end{array}}{N}-O-CF_2$
199	 $-CF_2-\underset{\begin{array}{c}\vert\\CF_3\end{array}}{N}-O-CF_2$

It should be noted that mass 100, which is associated with C_2F_4, is not present with the ratio of component masses which indicate tetrafluoroethylene. The C_2F_4 is a result of an ionic species created by the mass spectrograph from the polymer chain.

Figures 5 through 12 show that the XP5675, the precipitated XP5675, the 3M-56703-3, and the XP5812 do not differ in their composition. The difference in intensities is a function only of the molecular weight distribution.

The F^{19} NMR spectra confirmed this lack of difference. The H^1 NMR spectra and the IR spectra indicated the lack of any hydrogen or CH_2.

It is concluded, therefore, that the volatile portion of XP5675 is simply low molecular weight nitroso gum with no other impurities.

B. THERMAL DECOMPOSITION OF THE NITROSO COPOLYMER BY THERMOGRAVIMETRIC AND MASS SPECTROGRAPHIC ANALYSES

The isothermal thermogravimetric data shown in Figure 2 are most significant. The fact that a leveling off of weight loss occured at each temperature indicated that each weight loss is associated with a particular molecular weight fraction. No breaking down of higher molecular weight fractions occured which would produce shorter chain fractions which could then be lost (confirmed also by the mass spectrographic data).

We had hoped also to show that the mode of decomposition was, as shown by Shultz, et.al. (Ref 6):

$$CF_3NO/C_2F_4 \longrightarrow COF_2 + CF_3N{=}CF_2.$$

This would show up on the mass spectrographic data by an increase in masses 66 (COF_2) and 133 (CF_3NCF_2). Mass 66 did increase as expected (see Figure 5 for example); however, a corresponding increase in mass 133 was not demonstrated. The nature of the CF_3NCF_2 spectra is not known, but a further break up of this portion could account for the lack of the mass 133 increase. This data probably confirms the expected decomposition mode but could not establish it.

C. SOLVENT/NON SOLVENT SYSTEMS FOR SEPARATION OF THE TRIFLUORONITROSOMETHANE/TETRAFLUOROETHYLENE COMPONENTS OF VARIOUS MOLECULAR WEIGHTS

In order to obtain the molecular weight fractions of the nitroso copolymer, a solvent/non solvent system is necessary. Once such a system is determined, a means of fractionating may be employed such as solvent/non solvent precipitation or elution fractionation. Due to the time savings (without loss of accuracy) and the experience with the technique, we will conduct an elution fractionation (Ref 7).

The utility of the FC-75/benzotrifluoride system has been demonstrated in the results (II.C.). A range of 40°C is available between the condition that the polymer is all in solution and it is all precipitated out. The ratio of almost 1:1 of the two solvents before the gum is out of solution allows for a broad range of solvent ratios available for removal of the fractions from the elution column. The sensitivity of the gum solution to temperature and solvent ratio is high, however, so that great care is anticipated in preparation of the fraction.

D. GLASS TRANSITION OF THE NITROSO COPOLYMER GUM AT 1000 CYCLES PER SECOND BY REBOUND METHOD

Figure 17 shows the minimum rebound (maximum loss) to be in the region of -18°C. This is the 1000 cps glass transition temperature. Using an equivalence of 7°C per decade of frequency (which is purely an approximation at this stage of the knowledge of this gum) the 0.1 cps glass transition (such as determined by the torsion pendulum) would be -46°C.

This test and temperature will have to be validated by future runs but is a reasonable result especially since other dynamic tests are not usable.

E. PHYSICAL PROPERTIES OF AN AMINE CURED "PURIFIED" NITROSO COPOLYMER

The porous nature of the nitroso rubber indicated that the curing and post conditioning cycle recommended needs to be altered. The tensile strength of 130 psi and hardness of 27 shore A units were out of line due to the poor nature of the specimen.

The violent thermal decomposition in air at 270°C, which had previously been demonstrated on the gum (Ref 2), was shown to occur with the rubber. Decomposition of the rubber in helium occured over the temperature range from 150 through 320°C. Again we note that extreme caution should be used in handling this rubber at elevated temperatures (above 250°C).

The utility of the "purification" of the rubber by removal of the low molecular portions was not determined due to the poor cure, probably from an improper cure cycle.

The density of the rubber was 1.099 g/cc.

IV. SUMMARY

The "volatile" portions of the nitroso gums such as previously reported in sample XP5675 have been determined to be low molecular weight fractions of nitroso copolymer which evolve from 50 up through 200°C.

This low molecular weight portion can be removed by solution of the gum and precipitation of the high molecular weight portion or by vacuum drying.

This low molecular weight portion boils predominately in the 100°C region.

Except for average molecular weight, no differences were noted between the Thiokol gums and the 3M gum.

Weight losses in an inert atmosphere are associated with a corresponding molecular weight fraction.

The decomposition mode of:

$$CF_3NO/C_2F_4 \longrightarrow COF_2 \quad = CF_3N{=}CF_2$$

was indicated but not established.

A very usable solvent/non solvent system for the fractionation of the fluoronitroso copolymer has been determined. It consists of FC-75 and benzotrifluoride. Conditions for usable temperature ranges and solvent ratios have been set.

The 1000 cycle per second glass transition temperature (by rebound method) was determined to be -18°C. This corresponds to a 0.1 cps glass transition of -46°C.

An amine cure of a high molecular weight fraction of the nitroso copolymer provided a tensile strength of only 130 psi. Poor results were due probably to an improper cure cycle.

Violent decomposition of the nitroso rubber occured at 270°C, warranting <u>extreme caution</u> for use of this rubber at temperatures above 250°C.

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 completely characterize this typical gum. This will include first 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 6/31/65
George L. Ball III, Research Specialist*	830
Ival O. Salyer, Research Manager, Polymer Applications	109
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	7
Professional	1428
Charlotte D. Fritsch, Research Technician	546
John E. Strobel, Research Technician	46
Richard L. Evers, Research Technician	24
Donald O. Douglas, Research Technician	33
Margaret S. Ross, Research Technician	177
Ralph R. Ferguson, Research Technician	29
Gary A. Clinehens, Research Technician	71
Rodrigue G. Thibodeau, Research Technician	238
Conrad A. Cenerizio, Research Technician	26
Technical, Mixed	47
Technical	1237
Grand Total	2665

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* Project Leader

$37,296 has been spent as of 30 June 1965. The contract, less fee, is for $59,335, leaving a balance of $22,039.00.

60% of work has been completed and 58.7% of the money spent. The time and money remaining on the contract is sufficient.

APPENDIX

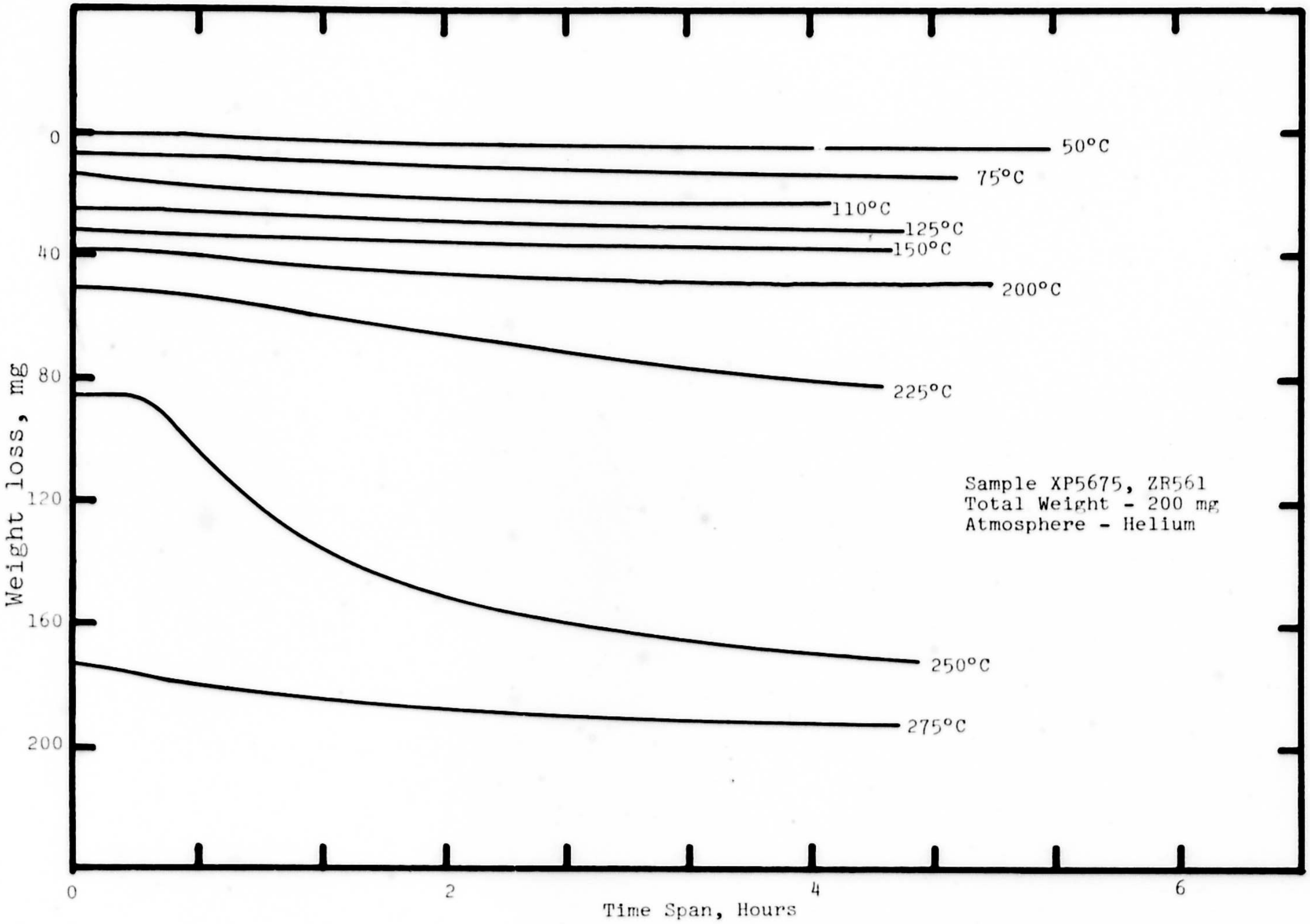

Figure 1. Isothermal thermogravimetric runs on fluoronitroso gum XP5675.

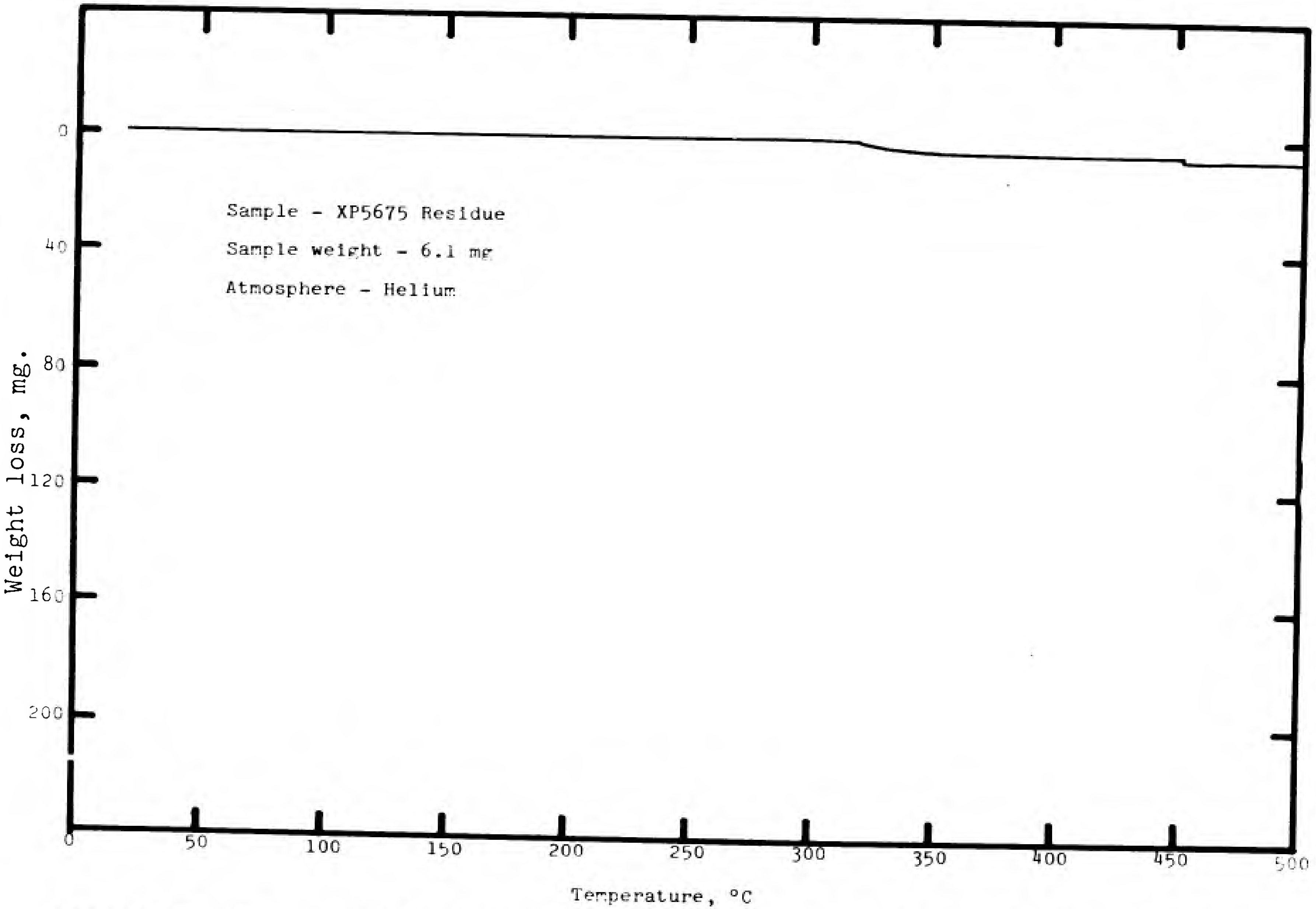

Figure 2. Thermogravimetric run on residue of XP5675 run isothermally up to 275°C.

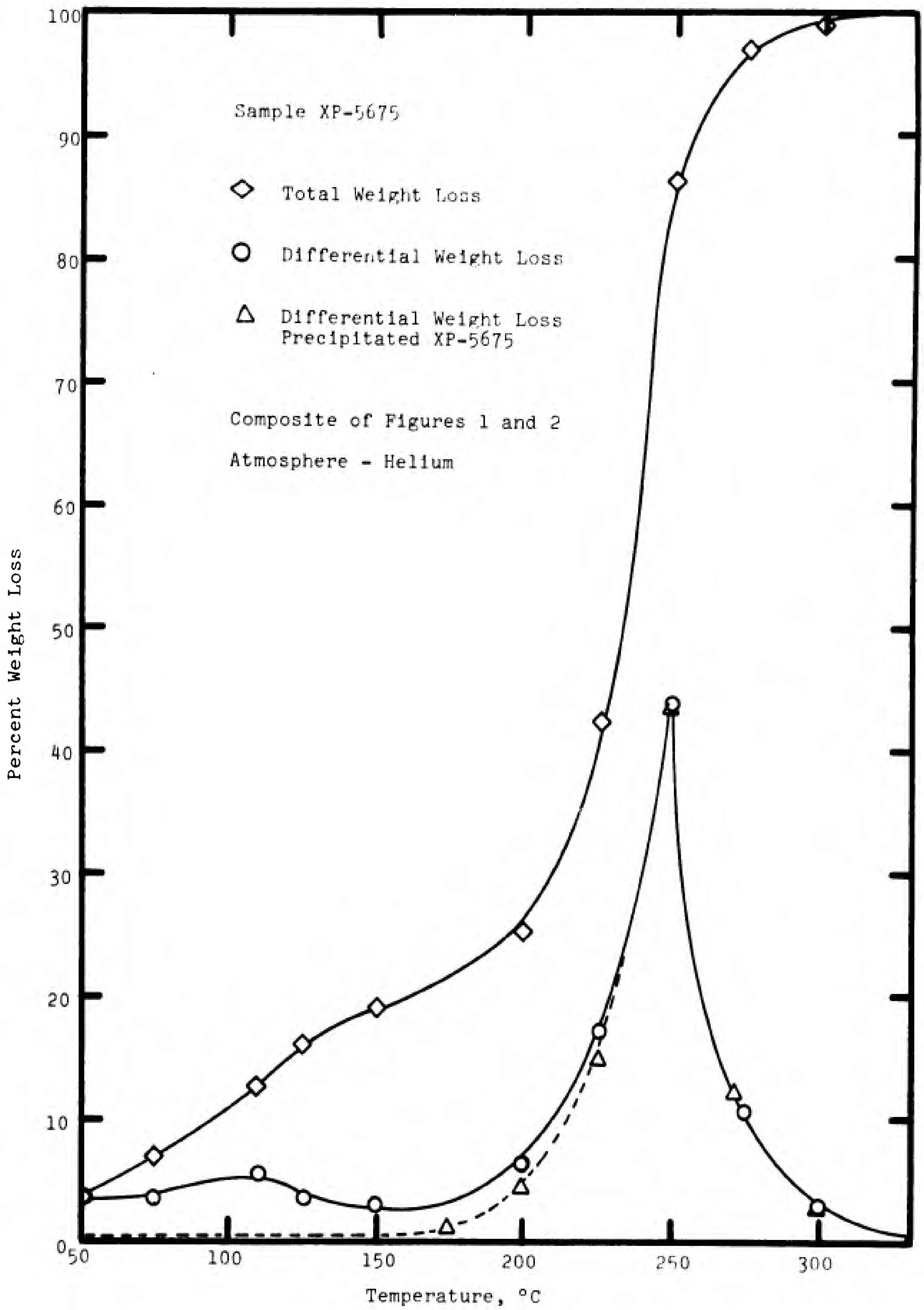

Figure 3. Composite of total and differential weight loss of sample XP5675 and precipitated XP5675.

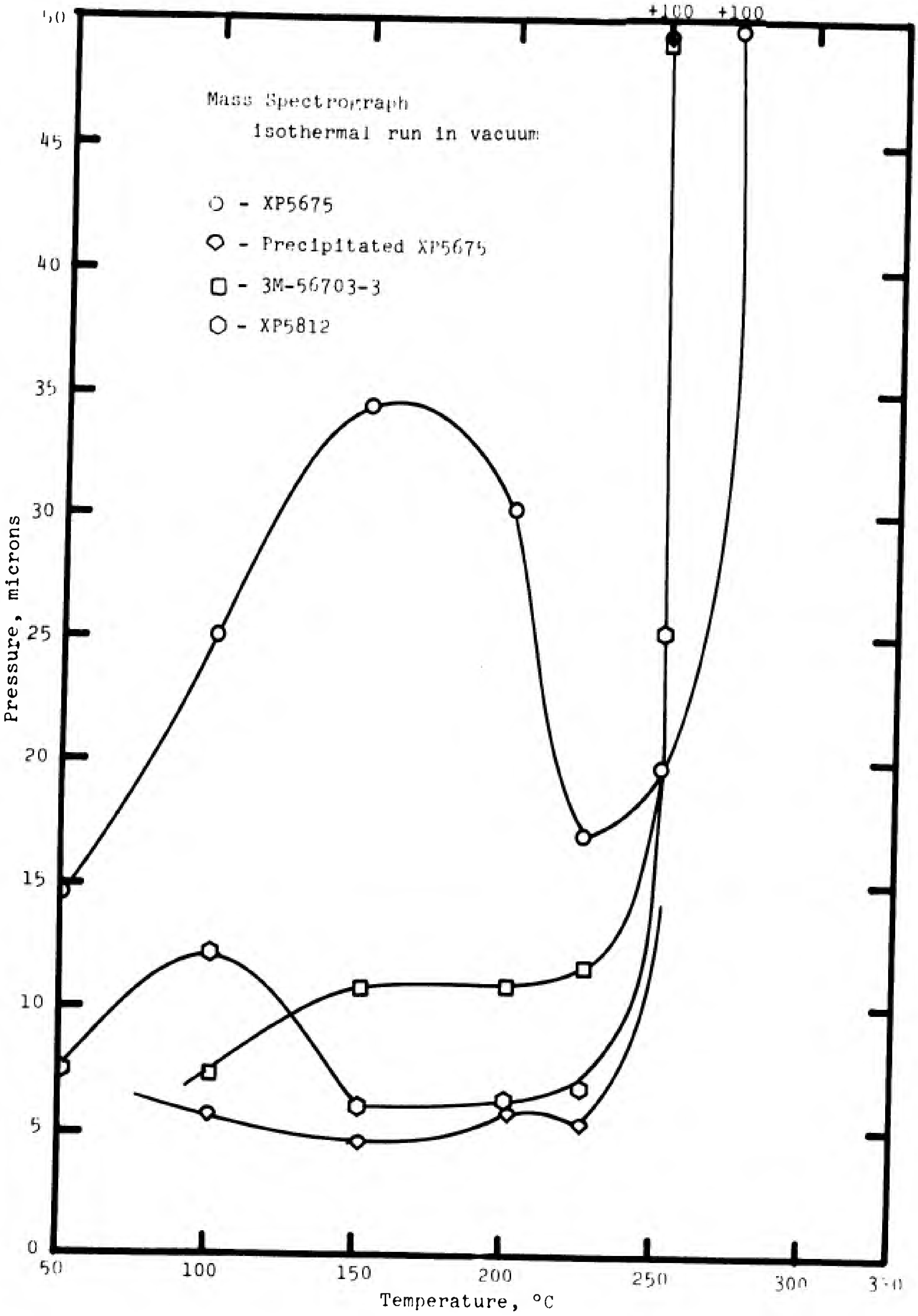

Figure 4. Mass spectrograph pressure during isothermal runs on samples XP5675, Precipitated XP5675, 3M-56703-3, and XP5812.

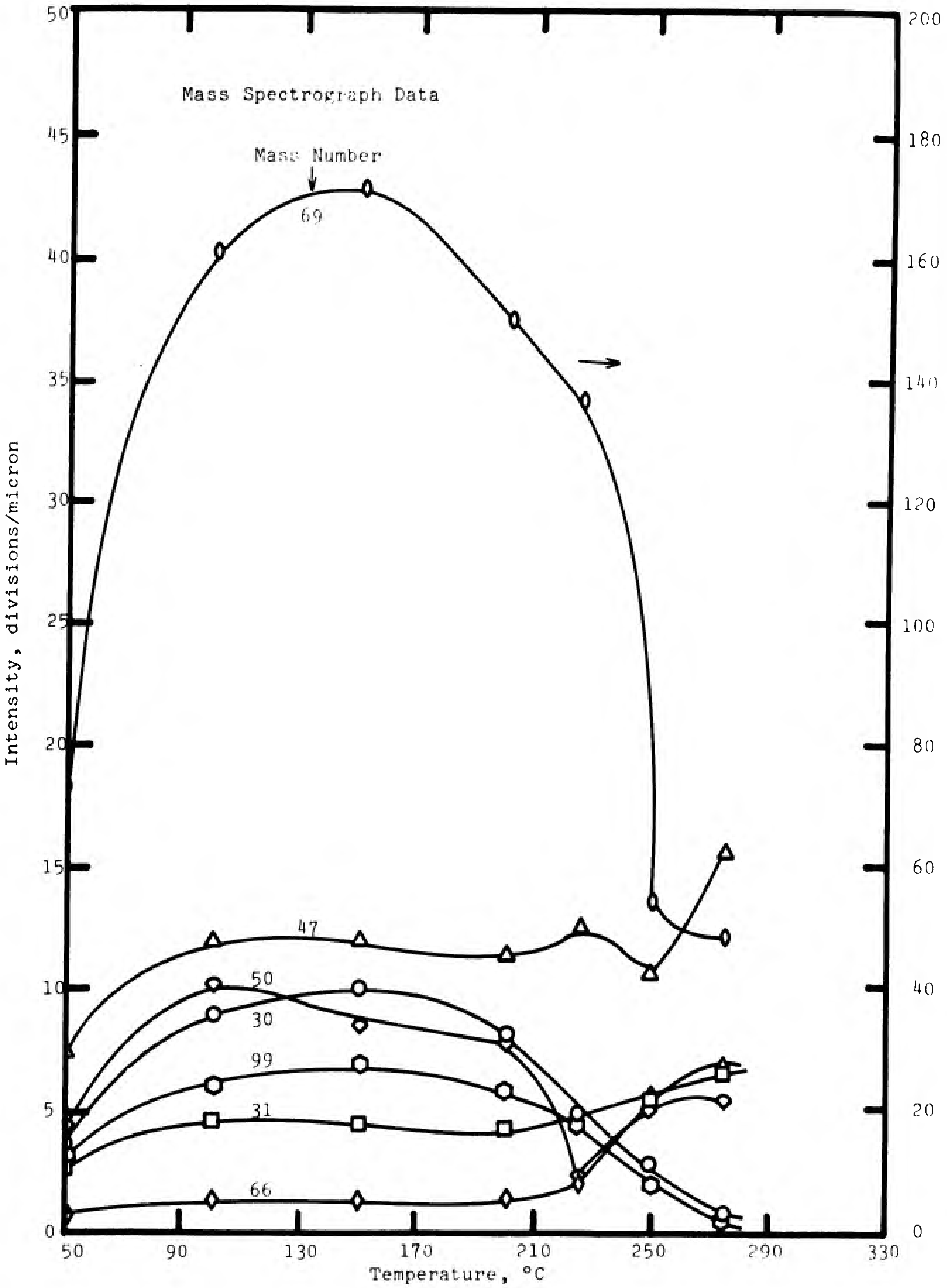

Figure 5. Most prominent ionic species from 50 up to 275°C of sample XP5675.

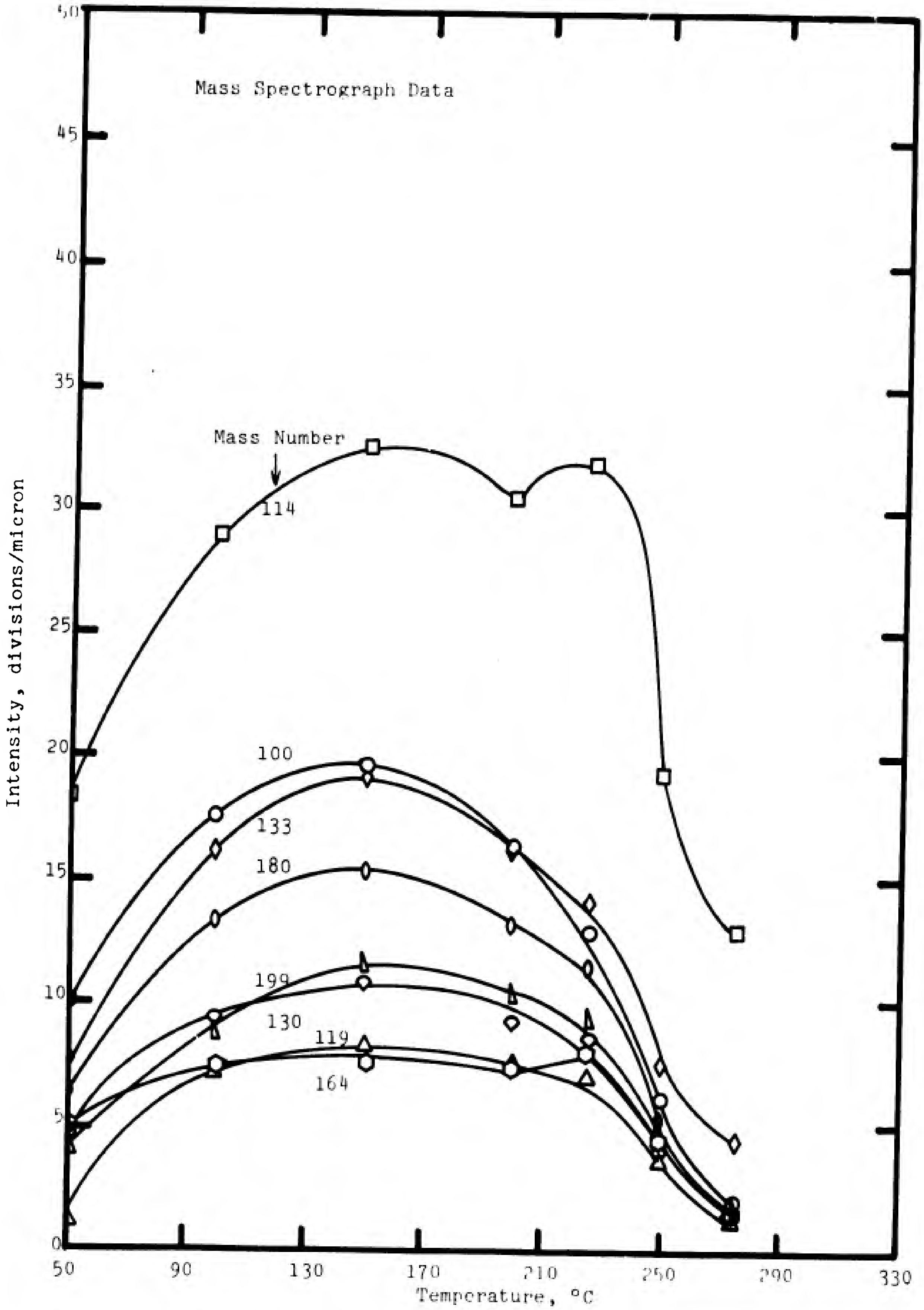

Figure 6. Most prominent ionic species from 50 up to 275°C of sample XP5675.

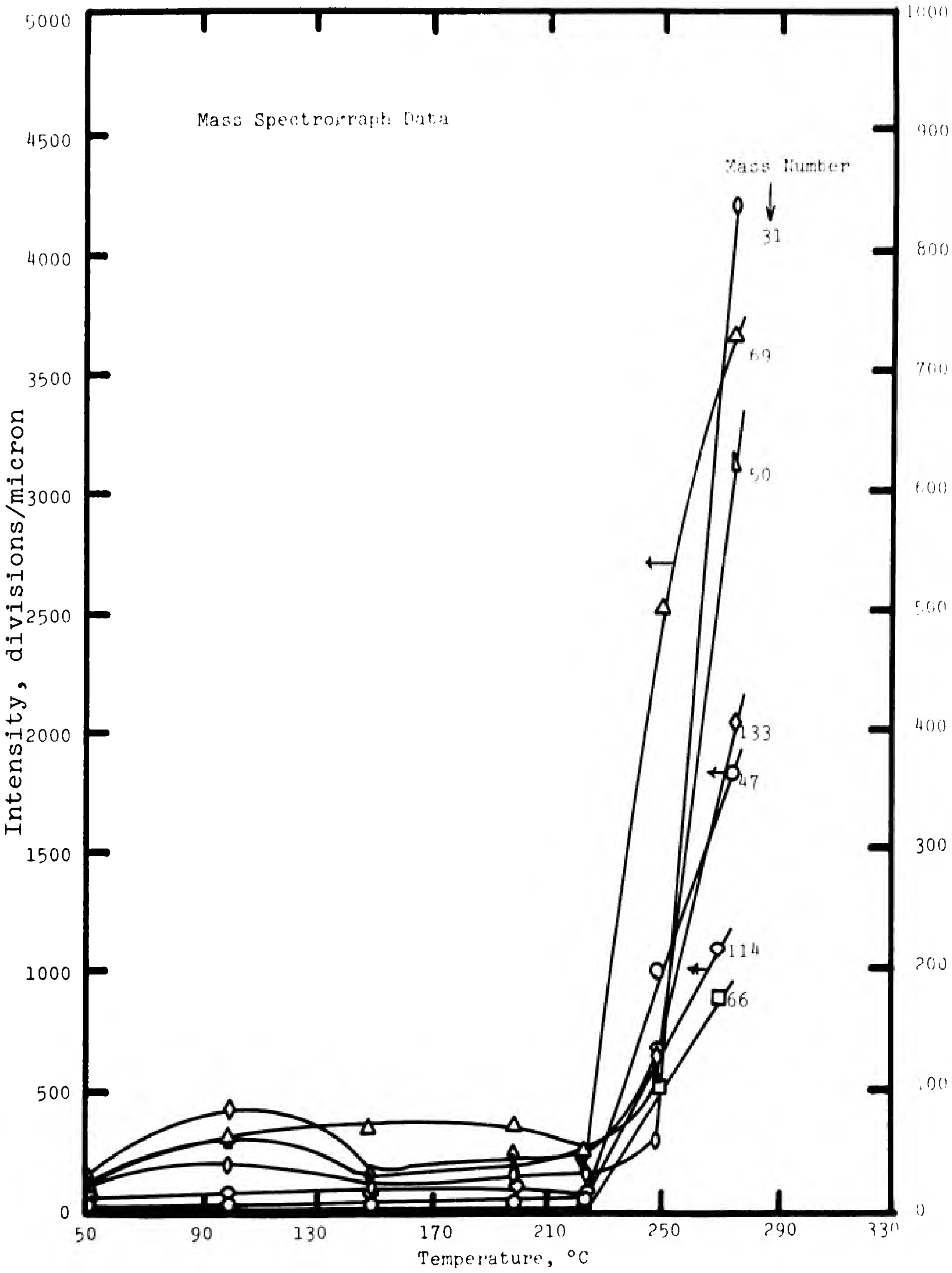

Figure 7. Most prominent ionic species from 50 up to 275°C of sample of precipitated XP5675.

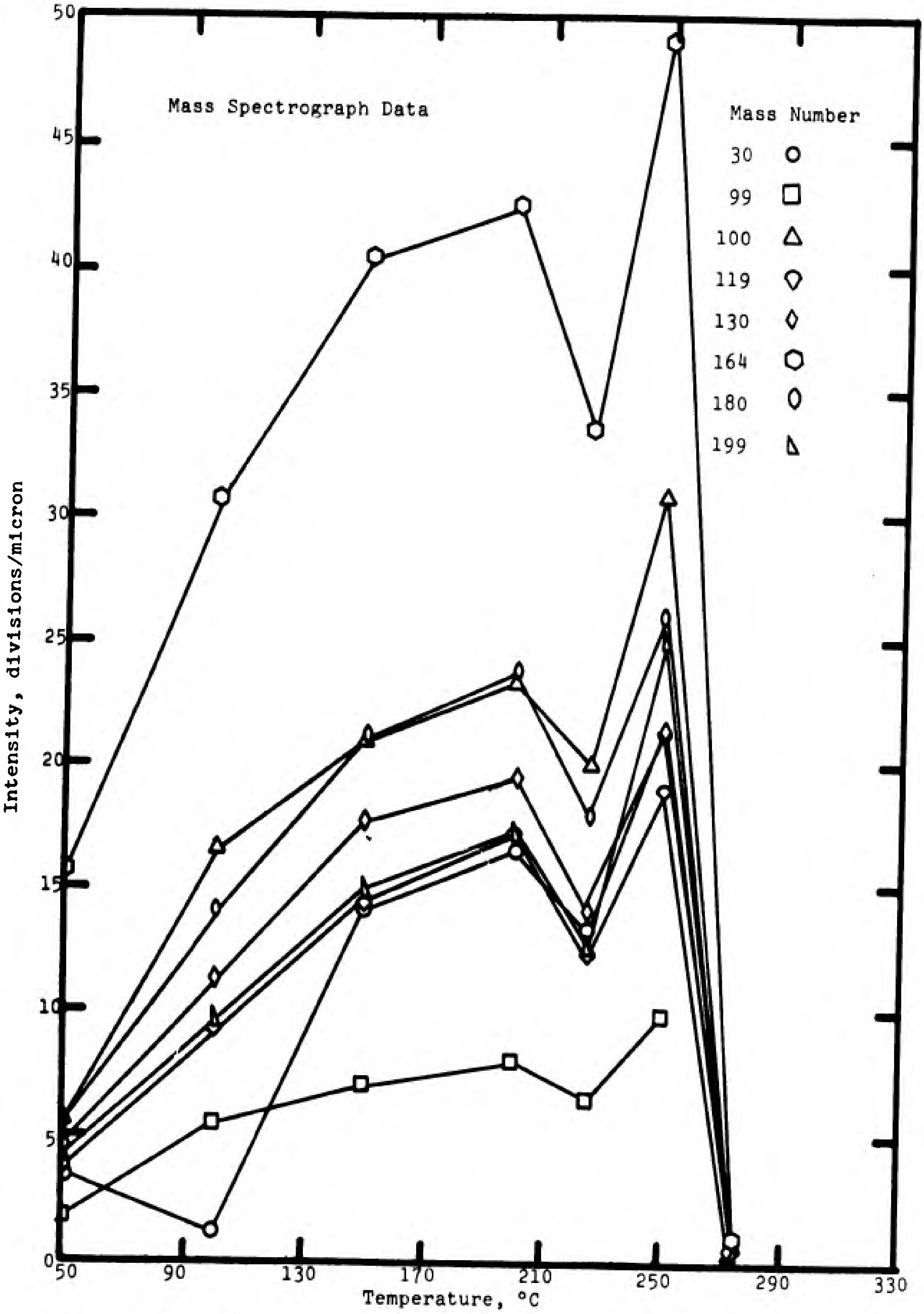

Figure 8. Most prominent ionic species from 50 up to 275°C of sample of precipitated XP5675.

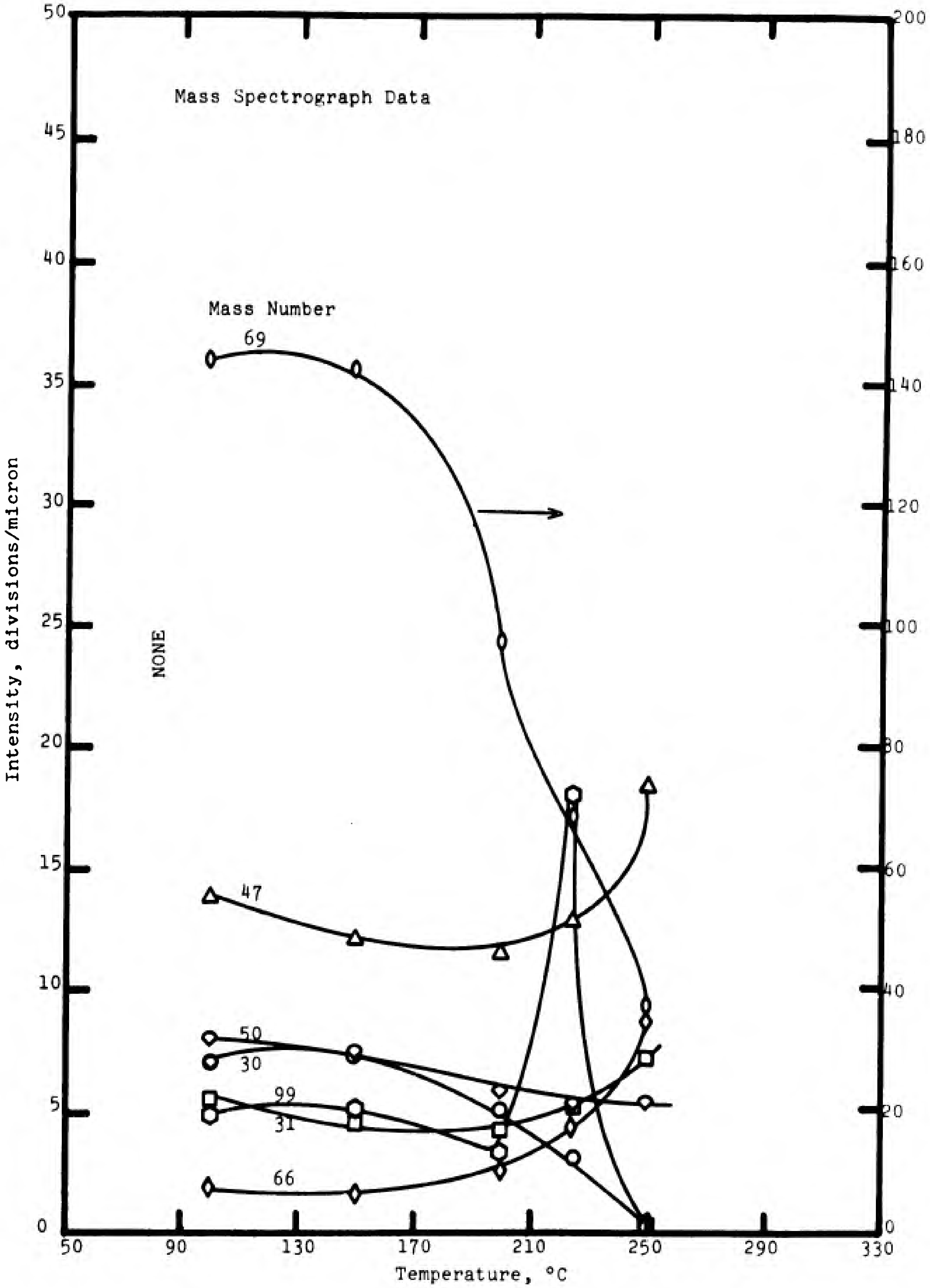

Figure 9. Most prominent ionic species from 50 up to 250°C of sample 3M-56703-3.

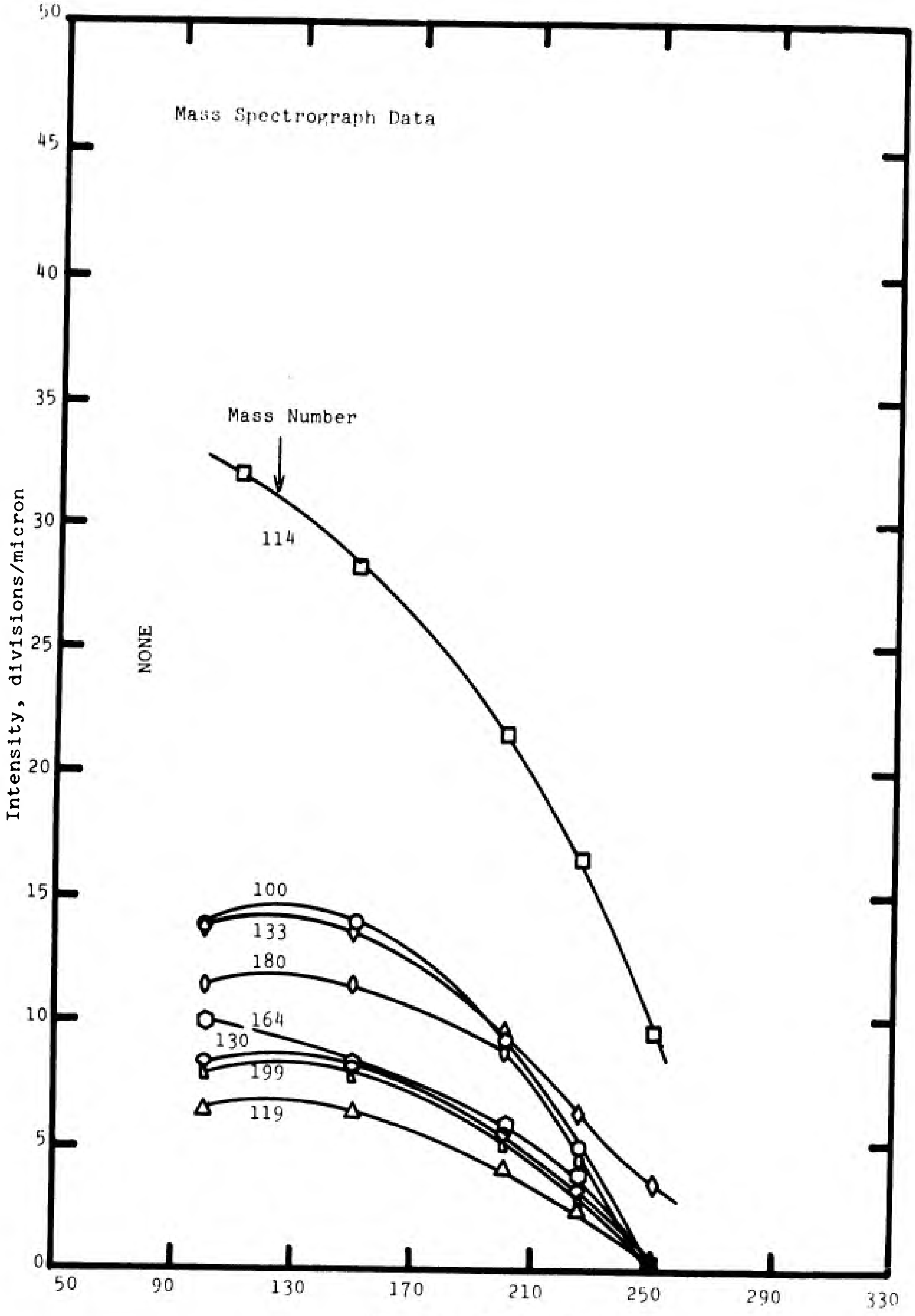

Figure 10. Most prominent ionic species from 50 up to 250°C of sample 3M-56703-3.

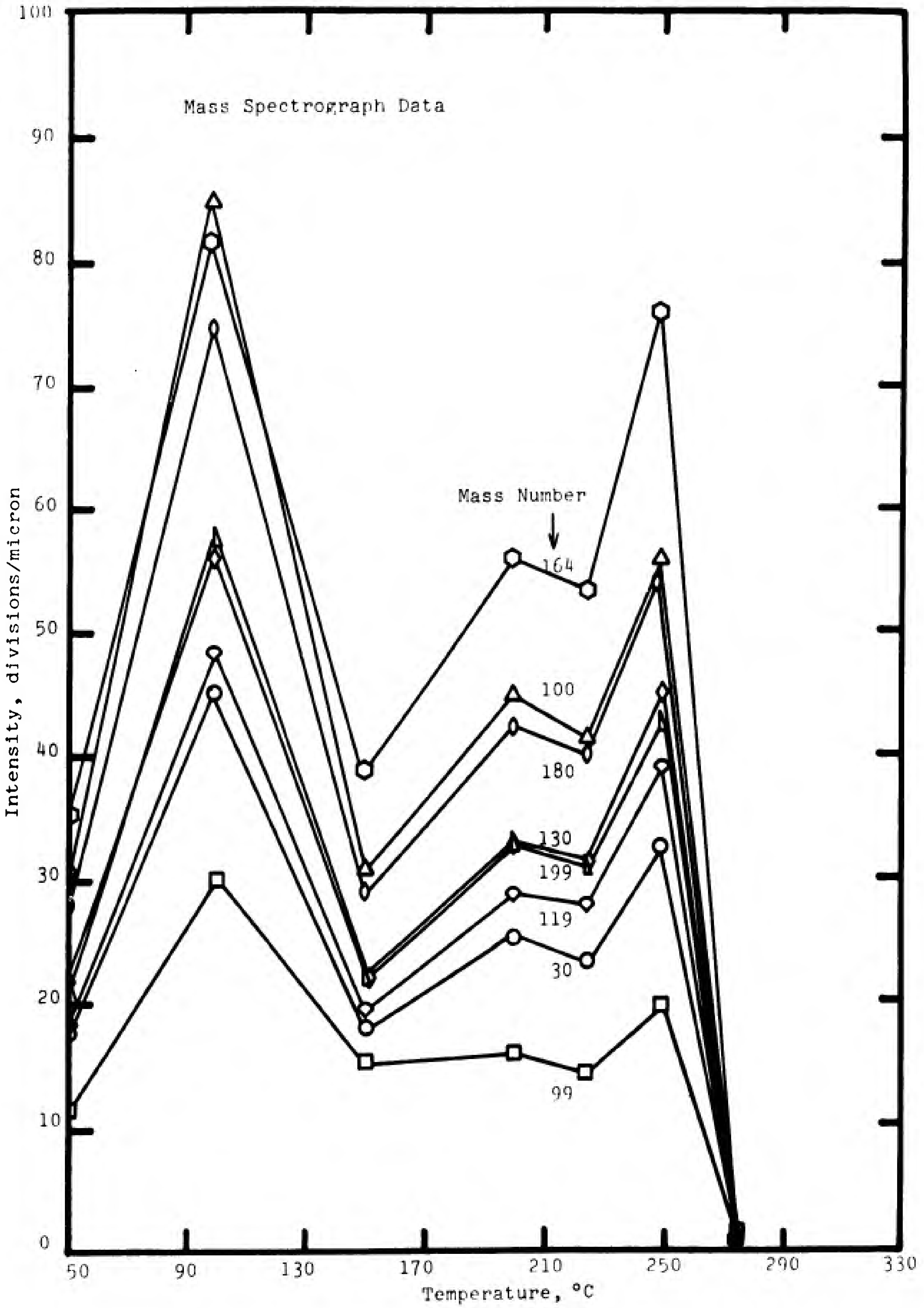

Figure 11. Most prominent ionic species from 50 up to 275°C for sample XP5812.

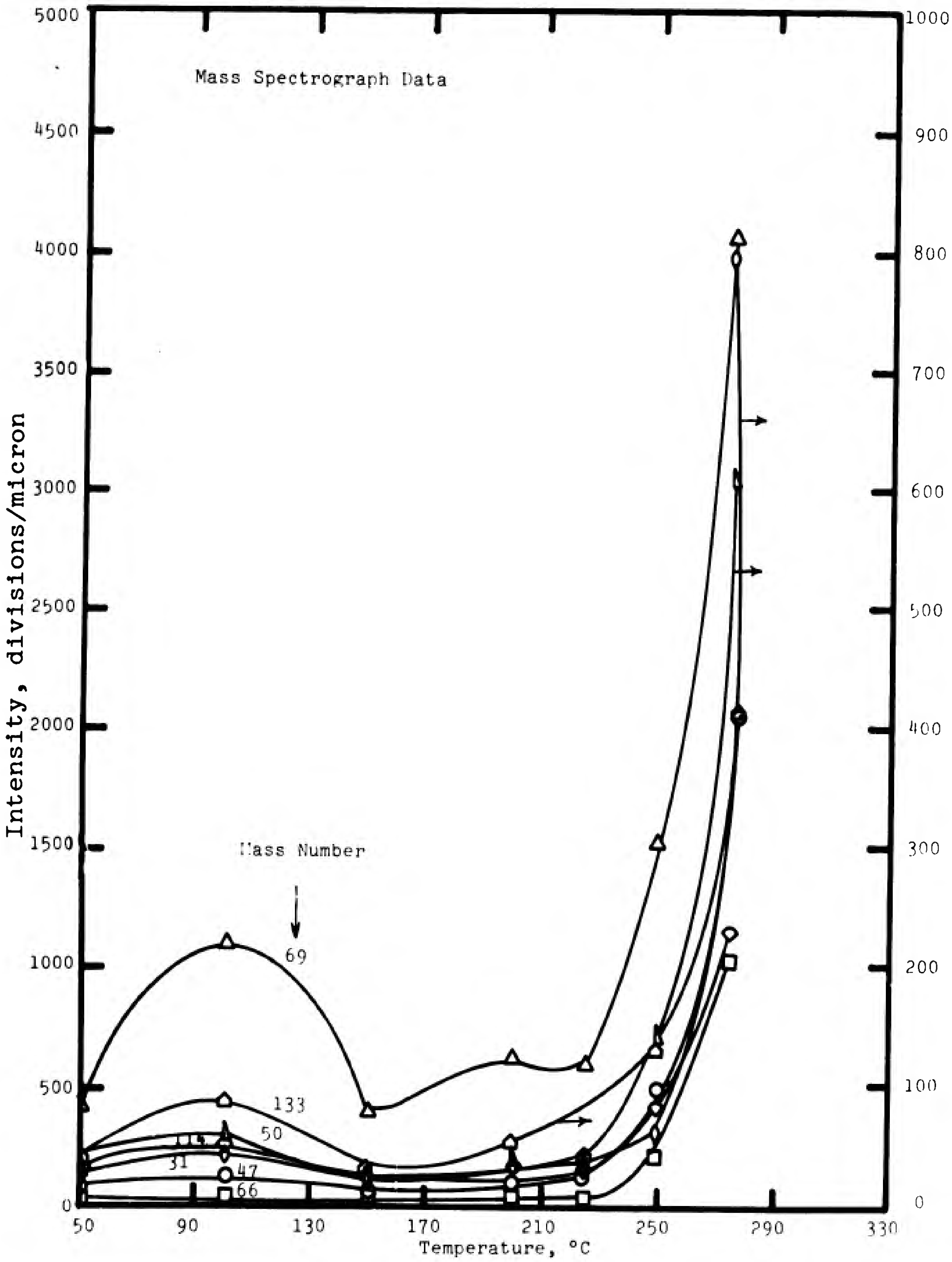

Figure 12. Most prominent ionic species from 50 up to 275°C for sample XP5812.

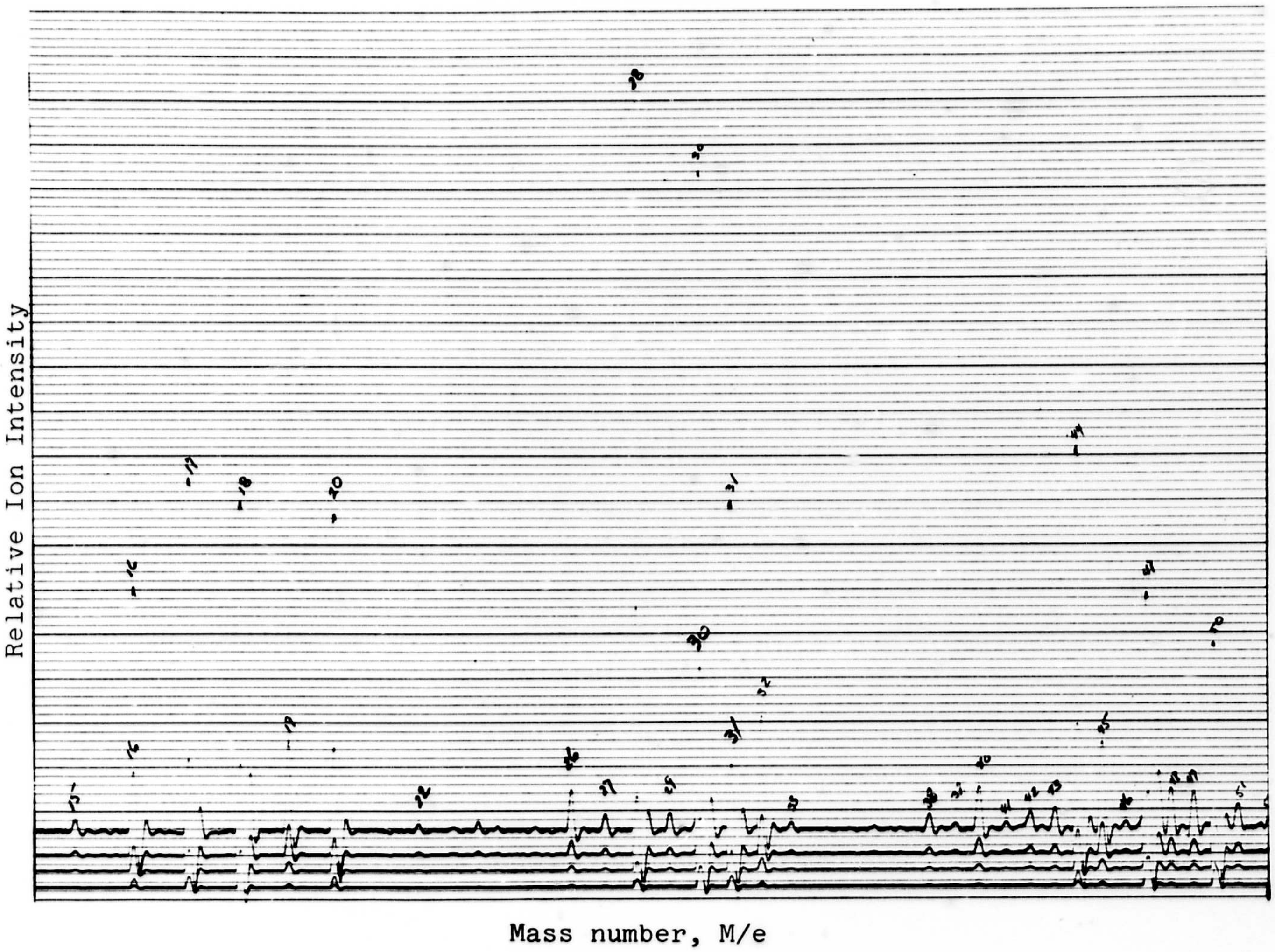

Figure 13. Actual mass spectra from mass 15 to 50 for XP5675 fluoronitroso gum (100°C).

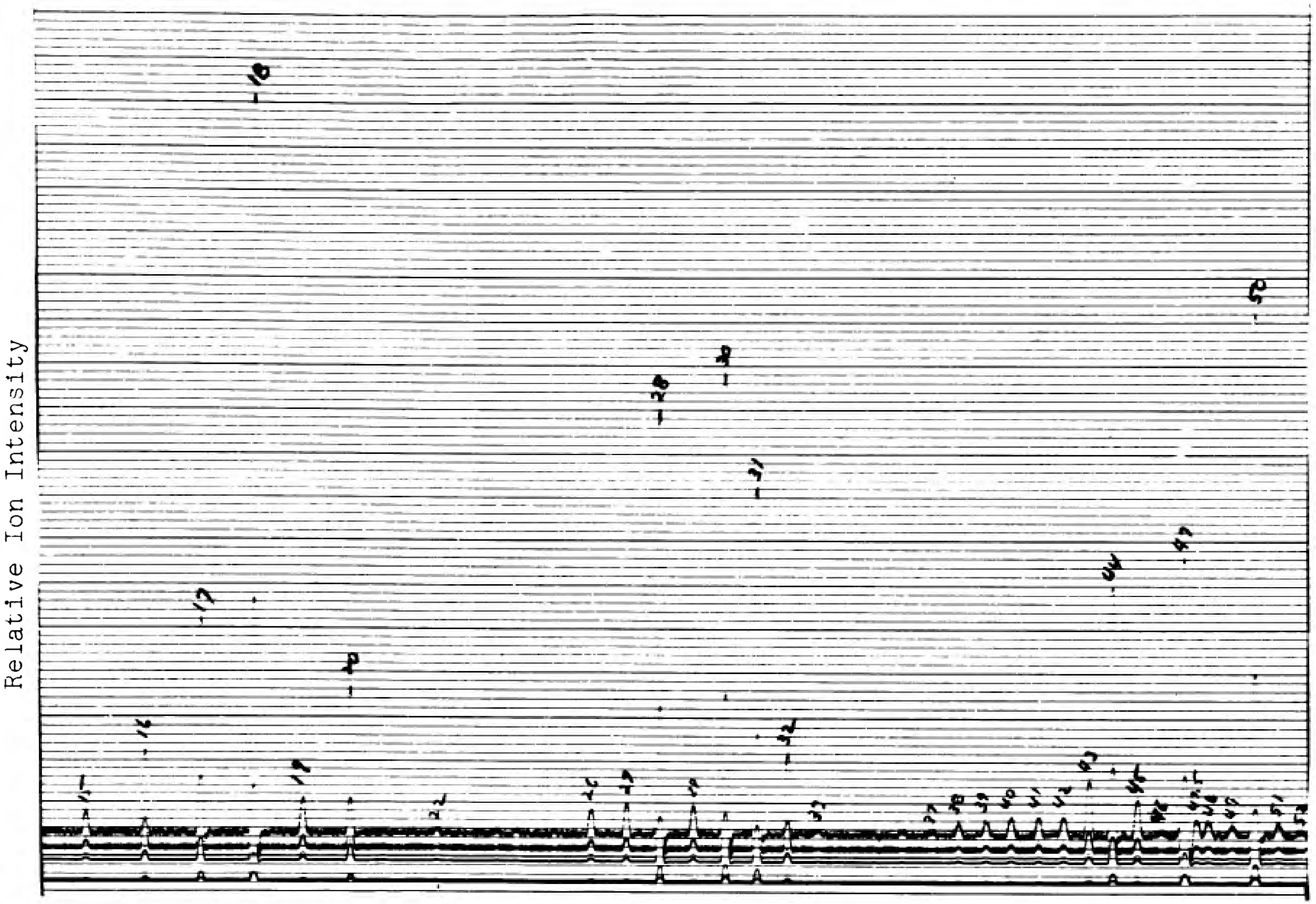

Figure 14. Actual mass spectra from mass 15 to 50 for 3M-56703-3 fluoronitroso gum (100°C).

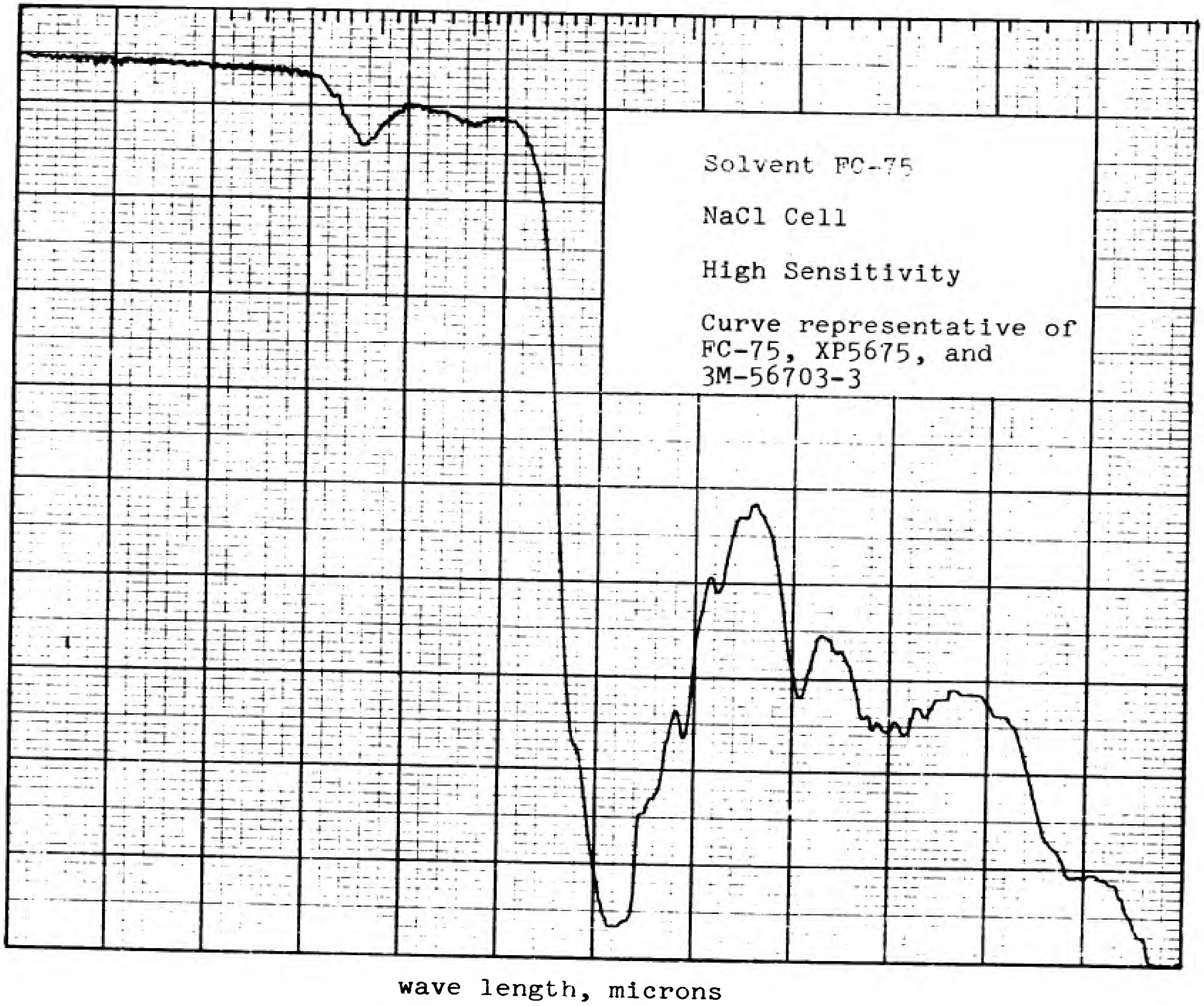

Figure 15. IR Spectra in the 1 to 7 micron region of 3M and thiokol fluoronitroso copolymer gum.

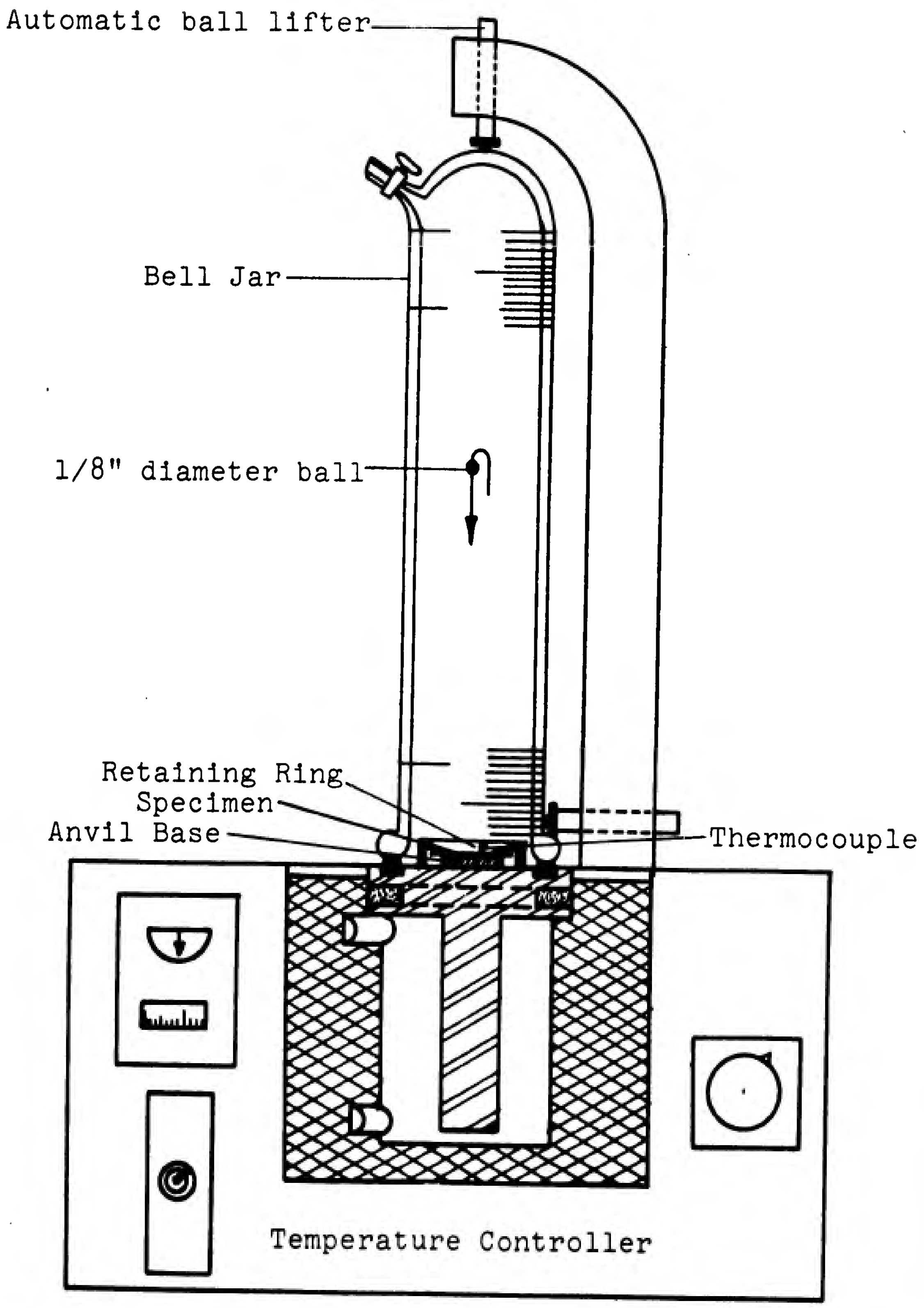

Figure 16. Rebound Tester for 1000 cycles per second modulus.

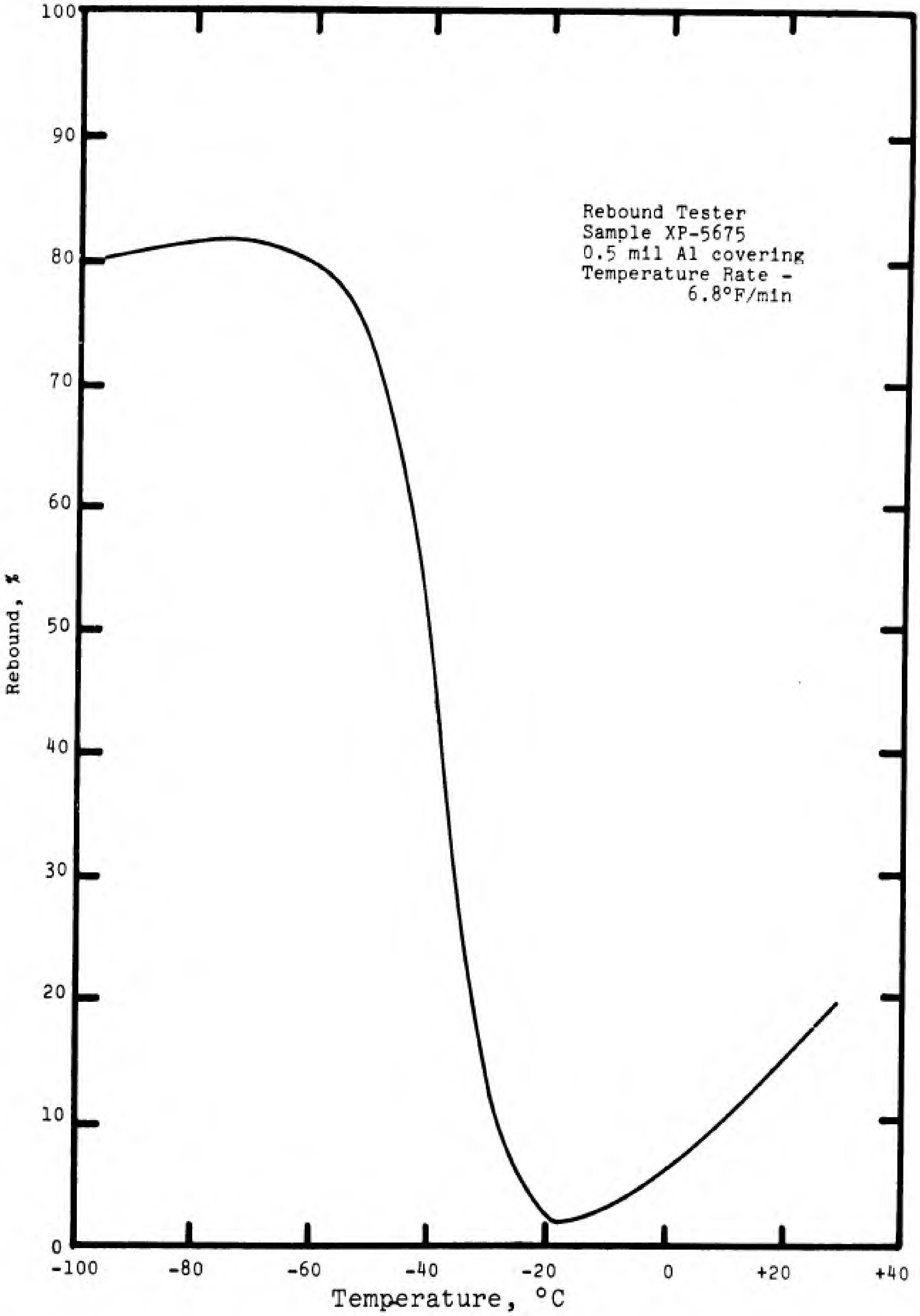

Figure 17. 1000 cycle per second modulus by rebound on sample XP5675.

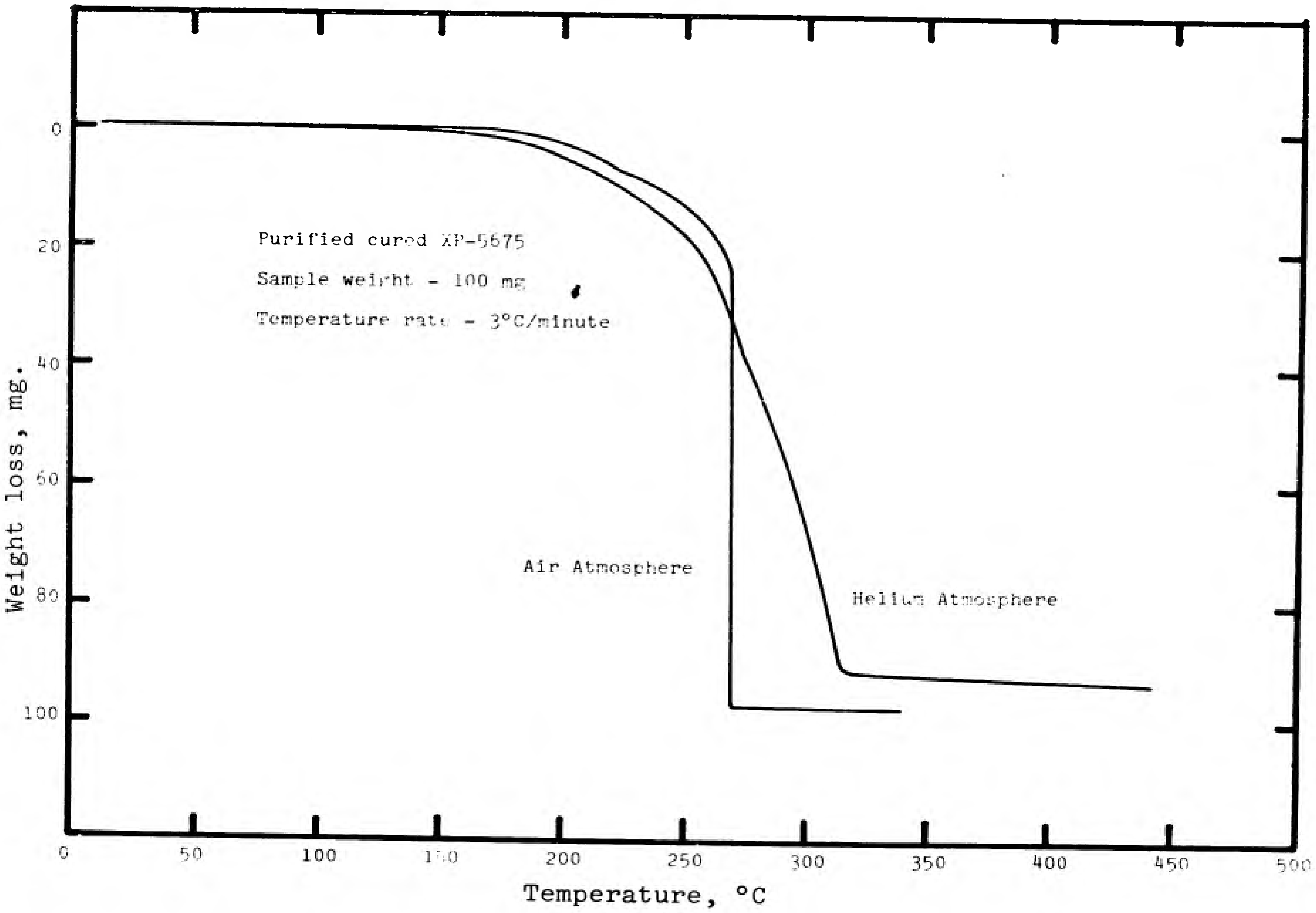

Figure 18. Thermogravimetric analysis in air and helium of an amine cured nitroso copolymer.

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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 previously reported "volatile" portion of the fluoronitroso gums was identified as purely low molecular weight fractions. No other contaminants were found. Removal of this low molecular weight portion was demonstrated by solvent precipitation and vacuum drying techniques. The low molecular weight portion boils predominately in the 100°C region and is present up to 200°C. Except for average molecular weight no differences were noted between the present Thiokol gums and the earlier 3M gum. A usable solvent/non solvent system consisting of FC-75 and benzotrifluoride was determined and the useful solvent ratios and temperatures set. The 1000 cycle per second glass transition was determined to be -18°C. Amine cure of the Thiokol gum following a recommended procedure provided poor rubber specimens. Violent decomposition of the cured rubber occured at 270°C as had the gum.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Physical properties	8					
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