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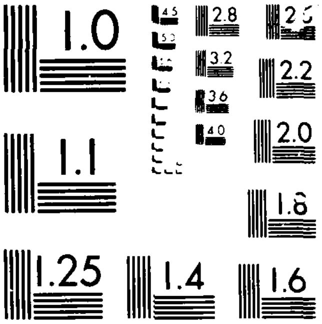

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MASS SPECTROMETRIC INVESTIGATION OF THE THERMAL DECOMPOSITION OF SEVERAL PROPELLANT AND EXPLOSIVE INGREDIENTS

FINAL REPORT

Contract N00014-80-C-0711

Department of the Navy
Office of Naval Research
Arlington, Virginia 22217

February 1986

Participants:
Milton Farber
S. P. Harris
R. D. Srivastava

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18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

dehydrofefo (DHF)
3,3 bis [(methyl nitramino methyl)] oxetane (BMNAMO)
poly (3-nitratomethyl-3-methyl) oxetane (p-NMMO)
50/50 methyl nitramino methyl methyl oxetane/azido methyl methyl oxetane (50/50 MNAMMO/AMMO)

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Thermal decomposition kinetics, including an elucidation of the mechanisms, were determined for several energetic polymeric materials. These materials included poly (3-nitratomethyl-3-methyl) oxetane (p-NMMO), the copolymer, methyl nitramino methyl methyl oxetane/azido methyl methyl oxetane (50/50 MNAMMO/AMMO), and the monomer 3,3 bis[(methyl nitramino methyl)] oxetane (BMNAMO). Experiments were performed on both stabilized and unstabilized dehydrofefo (DHF) to determine its decomposition and evaporation at 132 F.

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19. Key Words, continued...

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This report presents a discussion of the experimental results for several monomers and polymers obtained from thermal decomposition studies during the last twelve months of the contract period. A summary of work accomplished during the first four years of the contract is also presented.

I. THERMAL DECOMPOSITION STUDIES DURING CURRENT YEAR

Thermal decomposition kinetics, including an elucidation of the mechanisms, were determined for several energetic polymeric materials. These materials included poly(3-nitratomethyl-3-methyl) oxetane (p-NMMO), the copolymer, methyl nitramino methyl methyl oxetane/azido methyl methyl oxetane (50/50 MNAMMO/AMMO), and the monomer [3,3 bis(methyl nitramino methyl)] oxetane (BMNAMO). Samples of these materials were supplied by Dr. G. E. Manser. Experiments were performed on both stabilized and unstabilized dehydrofefo (DHF) to determine its decomposition and evaporation at 132 F. These samples were furnished by Dr. R. Reed.

The experiments were performed employing this laboratory's dual vacuum chamber furnace and mass spectrometer system.[1,2] The samples were contained in an alumina effusion cell 25 mm long, with an inside diameter of 6.8 mm and having an elongated orifice of 0.75 mm in diameter and 5.5 mm in length for beam collimation. Thus the products resulting from sublimation or evaporation and decomposition entering the beam were studied without further reaction. Gas products within the effusion cell could react with the condensed phase as well as the cell walls prior to leaving the cell and entering the high vacuum of the mass spectrometer ($\sim 10^{-7}$ mm Hg) for ion identification.

A. Monomers

1. Dehydrofefo (DHF)

Explosives containing DHF have been found to lose weight when stored at 132 F (56 C). Therefore, an investigation was undertaken at this temperature to determine whether the weight loss was due to DHF evaporation, thermal decomposition, or a combination of both.

Samples of DHF and DHF stabilized with approximately 3% MNA were heated to 132 F (56 C) and their vapor products examined mass spectrometrically. Both samples produced similar vapor species. Figure 1

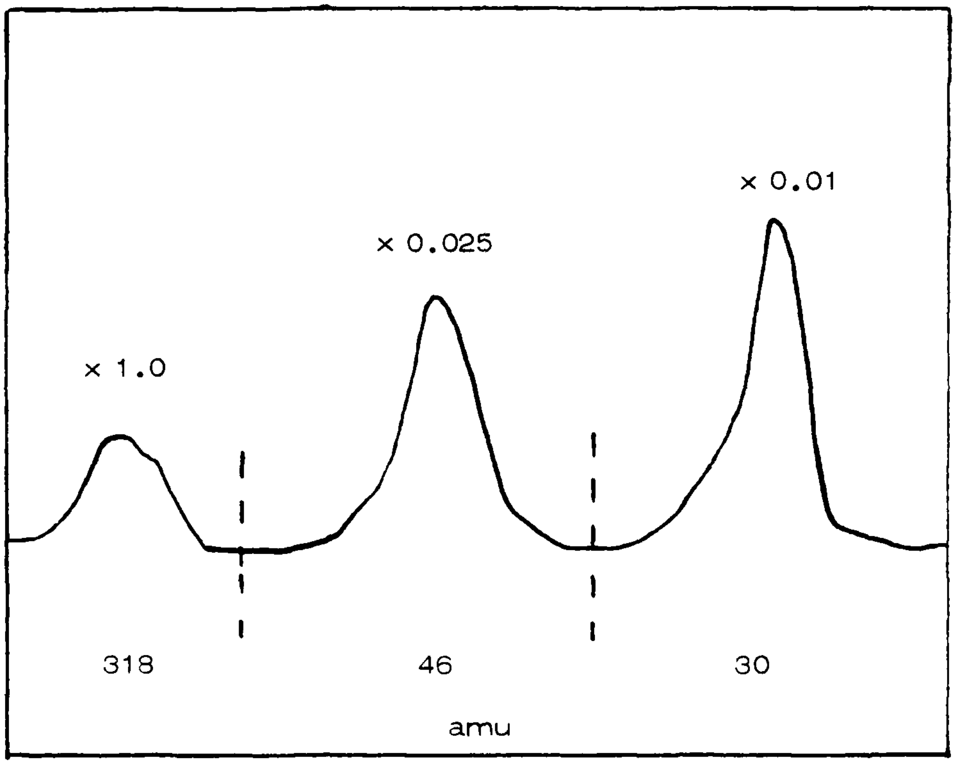
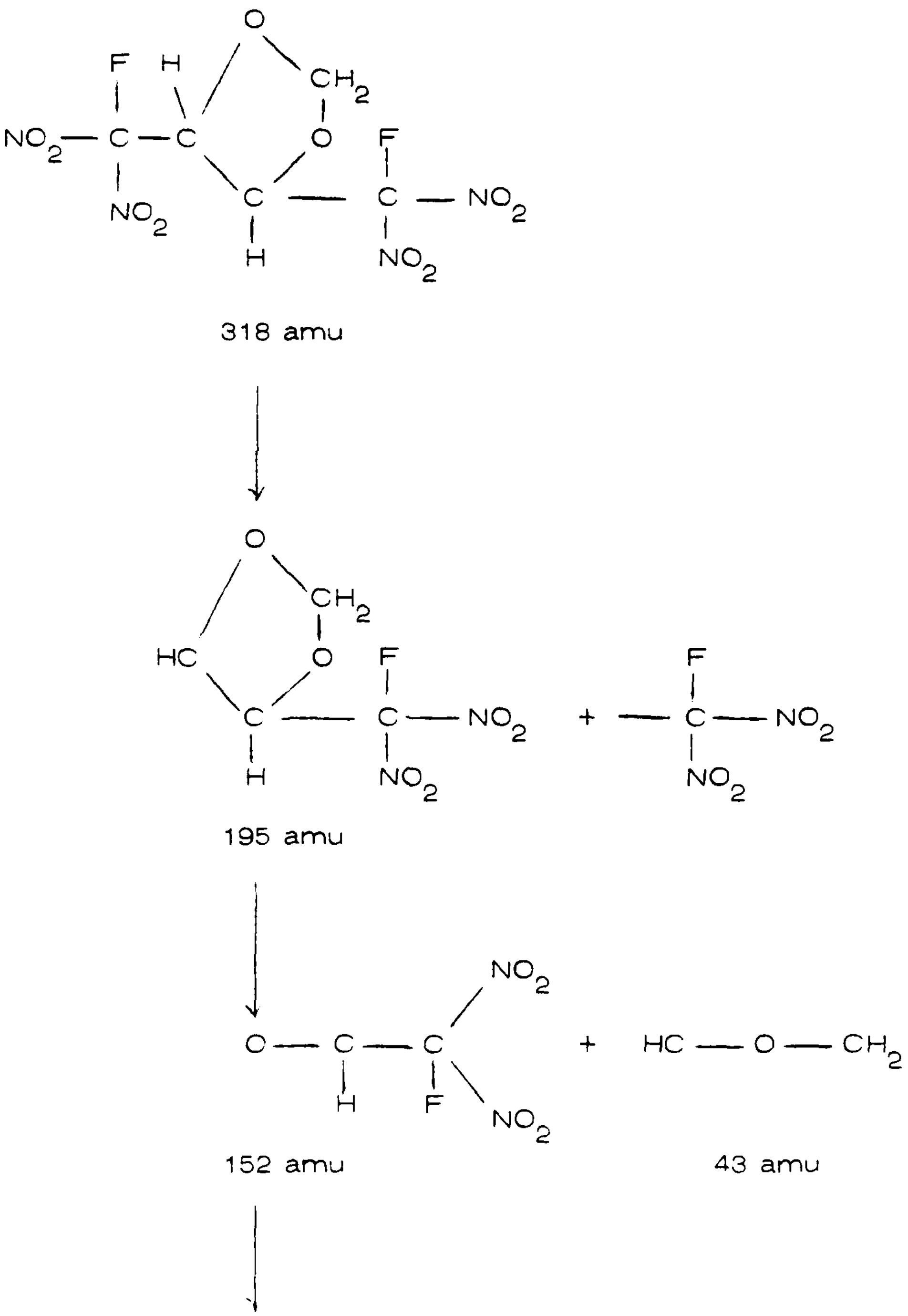
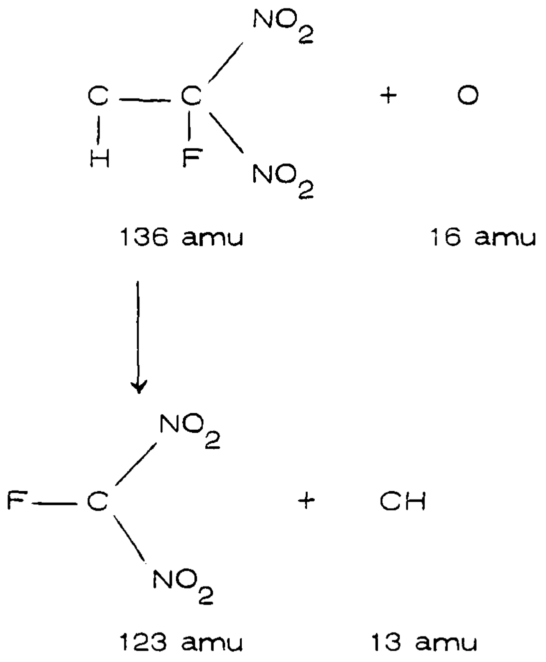


Fig. 1. Intensities due to DHF decomposition and evaporation at 132 F

shows decomposition peaks at 132 F. As can be seen, less than 10 percent DHF was observed at 318 amu. Decomposition of the stabilized and unstabilized samples was nearly identical, both producing two large molecular fragments at 123 and 152 amu (Fig. 2). The ratio of 123/152 is 5.7 for DHF, and 5.0 for DHF stabilized with MNA. Peaks at 195, 152, 136 and 123 amu were observed for the two samples, corresponding to the fragments:





An evaporation energy of approximately 165 kJ mol^{-1} (40 kcal/mol) was obtained from a decomposition study in the temperature range 30 - 60 C (86 - 140 F).

2. 3,3 bis [(methyl nitramino methyl)] oxetane (BMNAMO)

Energetic nitramino group materials are continually being prepared and investigated. Previous thermal decomposition studies involving both the methyl nitramino methyl methyl oxetane monomer and the homopolymer were reported in our Annual Report dated September 1984. The bis compound, having two nitramino groups, would appear to be more energetic and therefore warrants investigation as a possible propellant or explosive ingredient. BMNAMO is stable to approximately 120 C with decomposition commencing above this temperature. At 150 C prominent decomposition fragment peaks are observed (Fig. 3.a. and 3.b.). Figure 3.a. depicts the mass spectra in the 90-190 amu range at 120 C; figure 3.b. is the spectra in the same amu range at 150 C. A mass spectrum of the decomposition products in the 50 - 115 amu range at 150 C is shown in Fig. 4. Figure 5 is a composite of the mass spectra in the 60 - 90 amu range. From these spectra a scheme for the decomposition mechanism is proposed. The peaks at 145 and 89 amu suggest a decomposition path as the breaking of the central oxetane carbon to the nitroamino appendage as

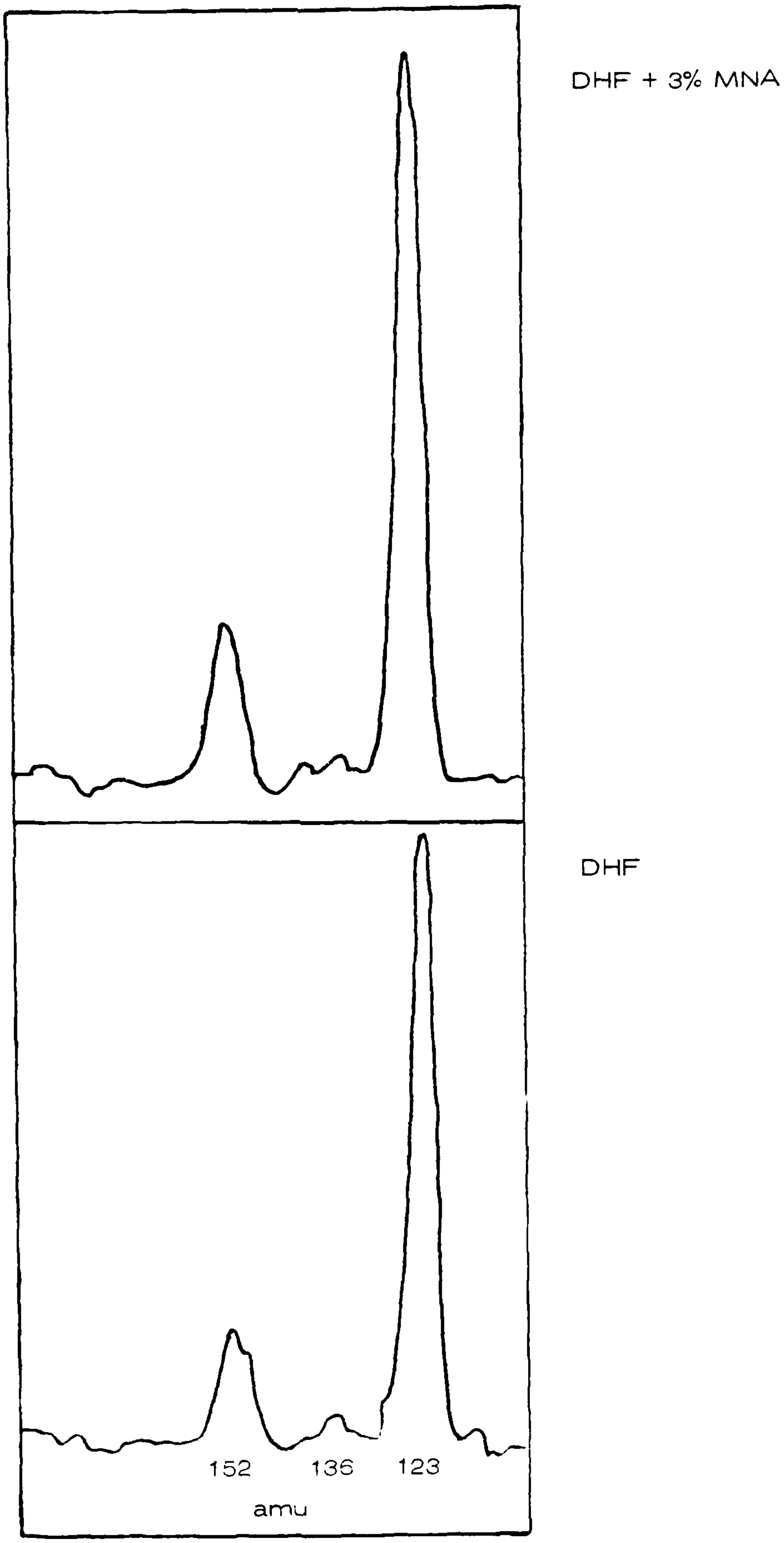


Fig. 2. Mass spectra comparison of pure DHF and DHF stabilized with 3% MNA at 60 C

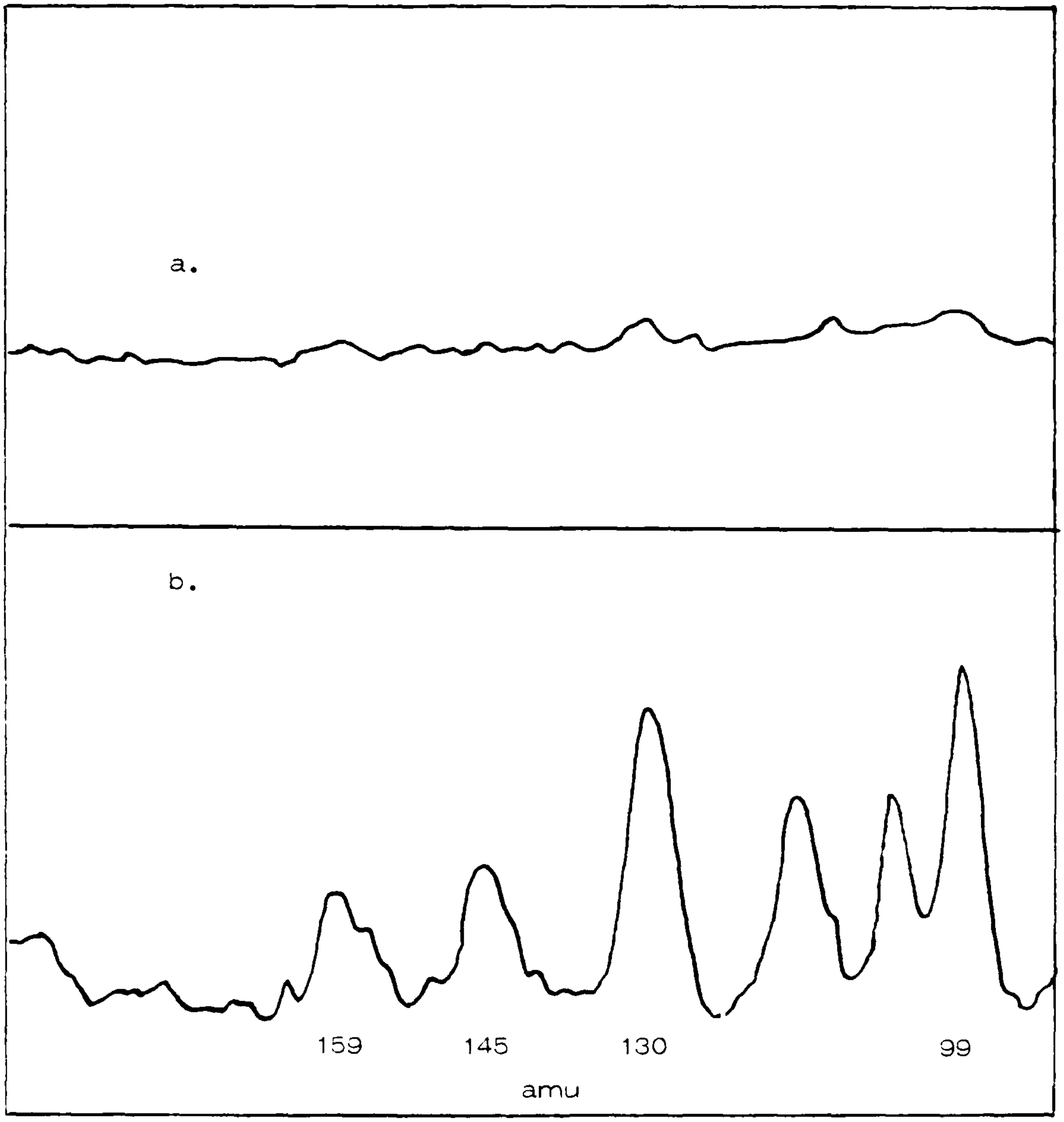


Fig. 3.a. Mass spectra in the amu range 90 - 190 for BMNAMO at 120 C

Fig. 3.b. Mass spectra in the amu range 90 - 190 for BMNAMO at 150 C showing considerable molecular decomposition

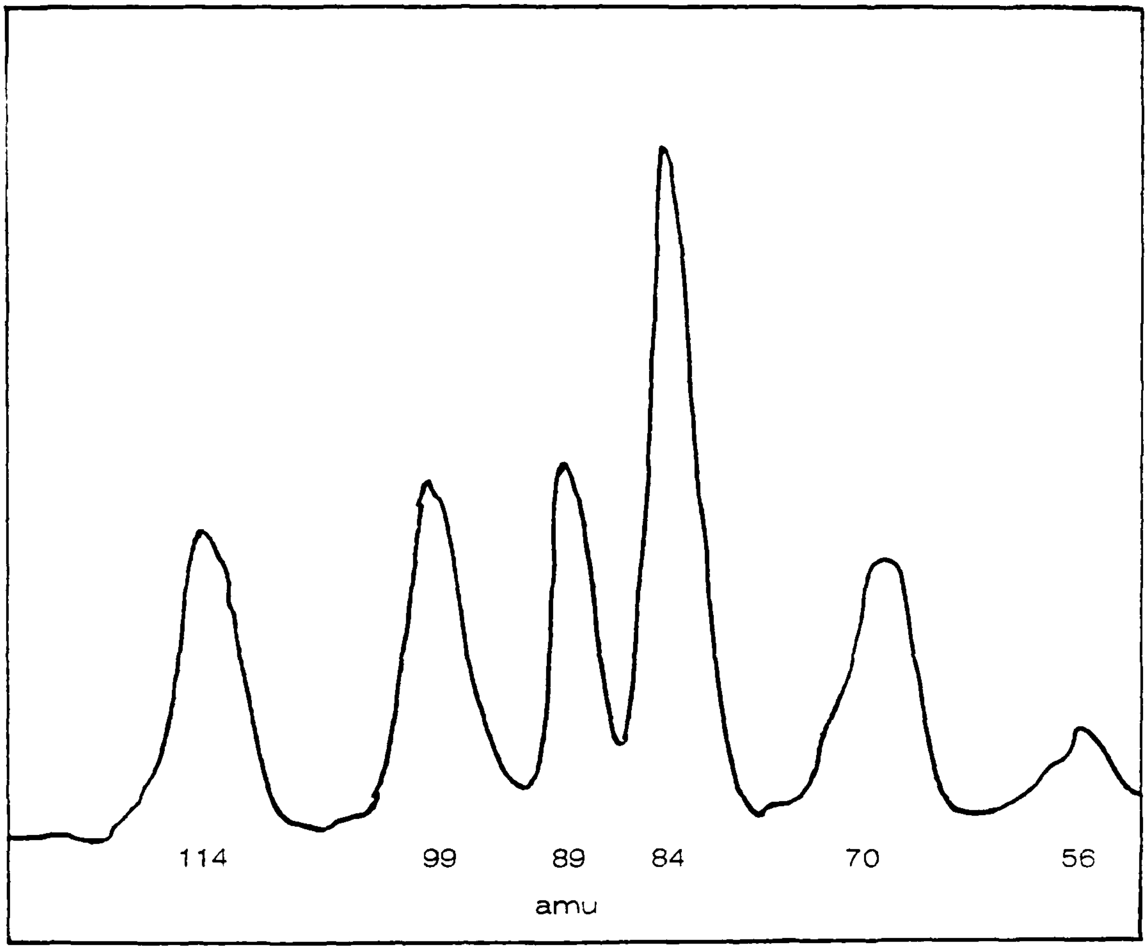


Fig. 4. Mass spectra in the 50 - 115 amu range for the decomposition products of BMNAMO at 150 C

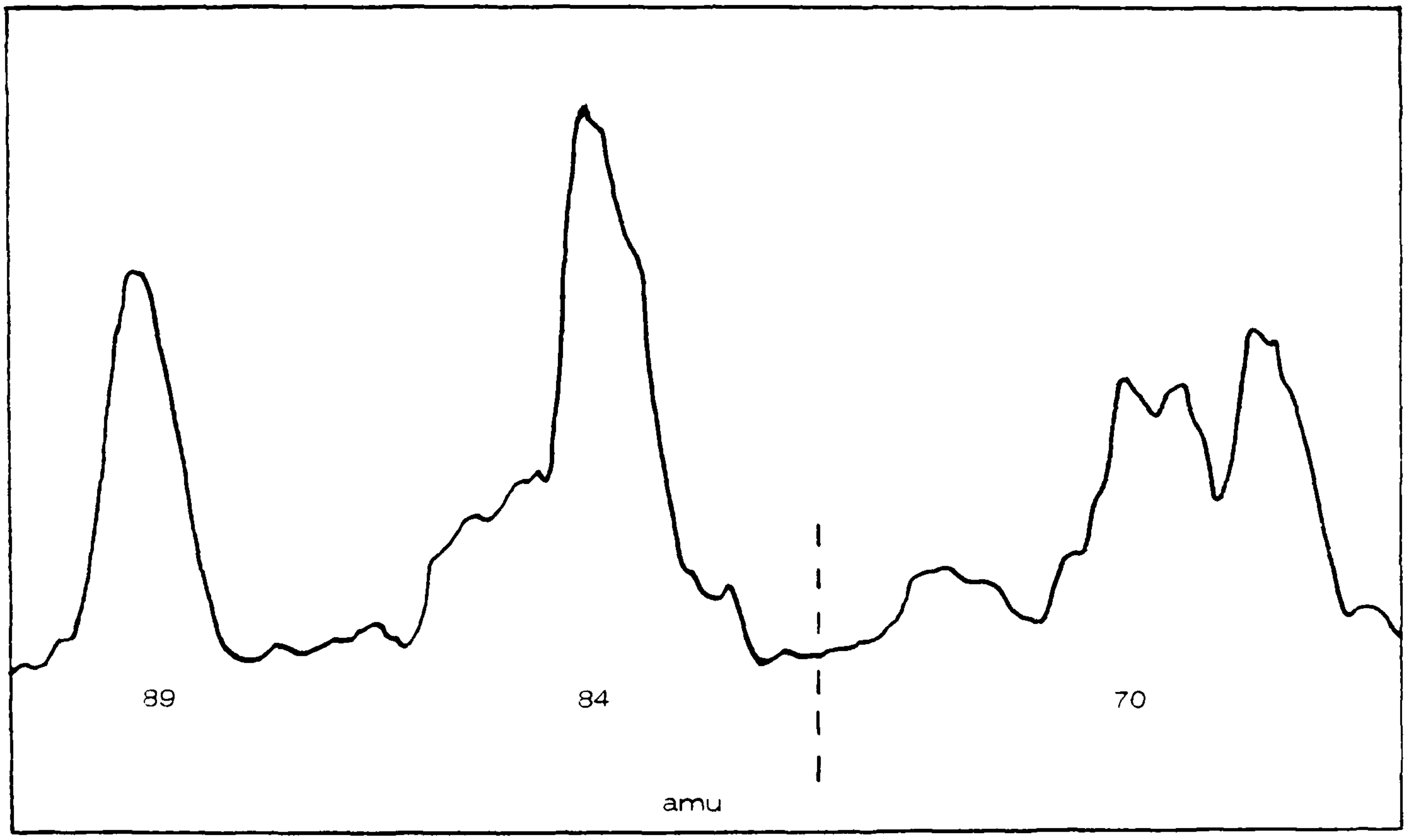
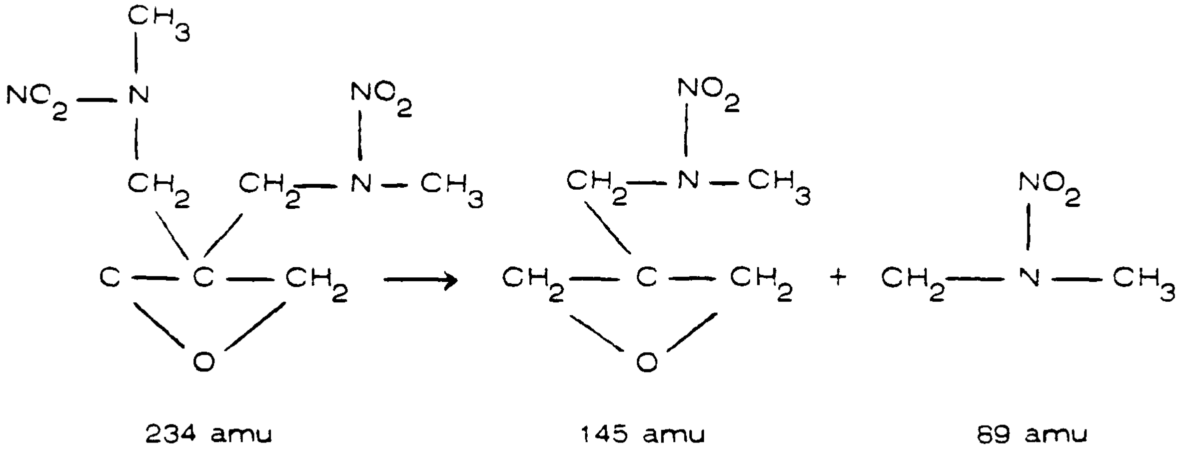
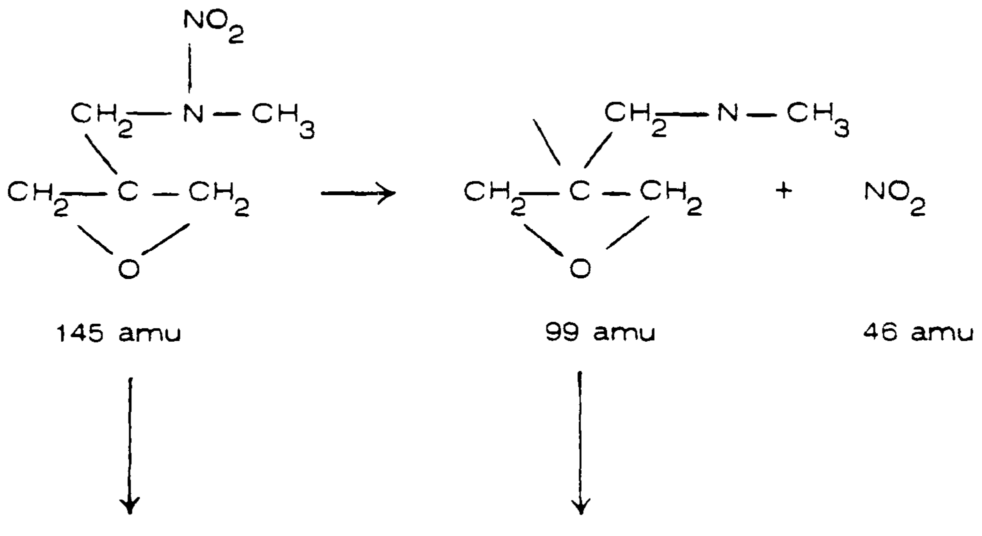
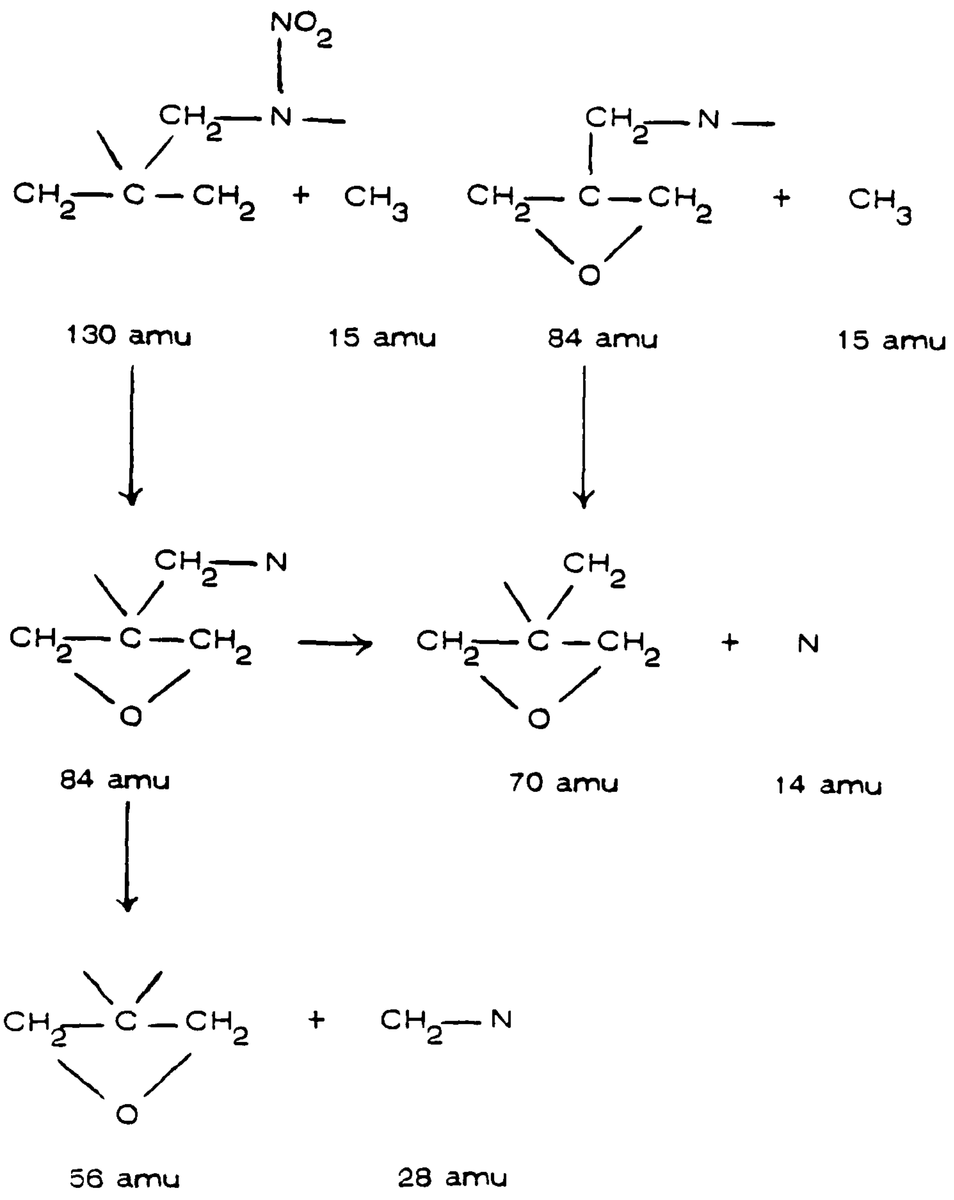


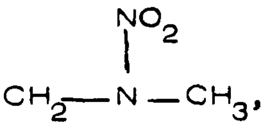
Fig. 5. Mass spectra in the 60 – 90 amu range for the decomposition products of BMNAMO at 150 C (spectra are continuous only within the dashed line)

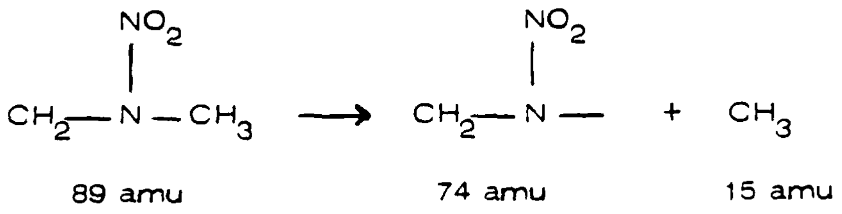


There is also an indication of C—N bond cleavage, with the observation of a small concentration of a 159 amu peak. Reactions continue within the effusion cell, with further decomposition of the larger fragments yielding the observed ion intensity peaks of 130, 99, 84, 75, 74, 70, 56, 46, 28 and 15 amu. Possible molecular compositions of these structures are





The appendage,  $CH_2-\overset{\overset{NO_2}{|}}{N}-CH_3$, also decomposes as

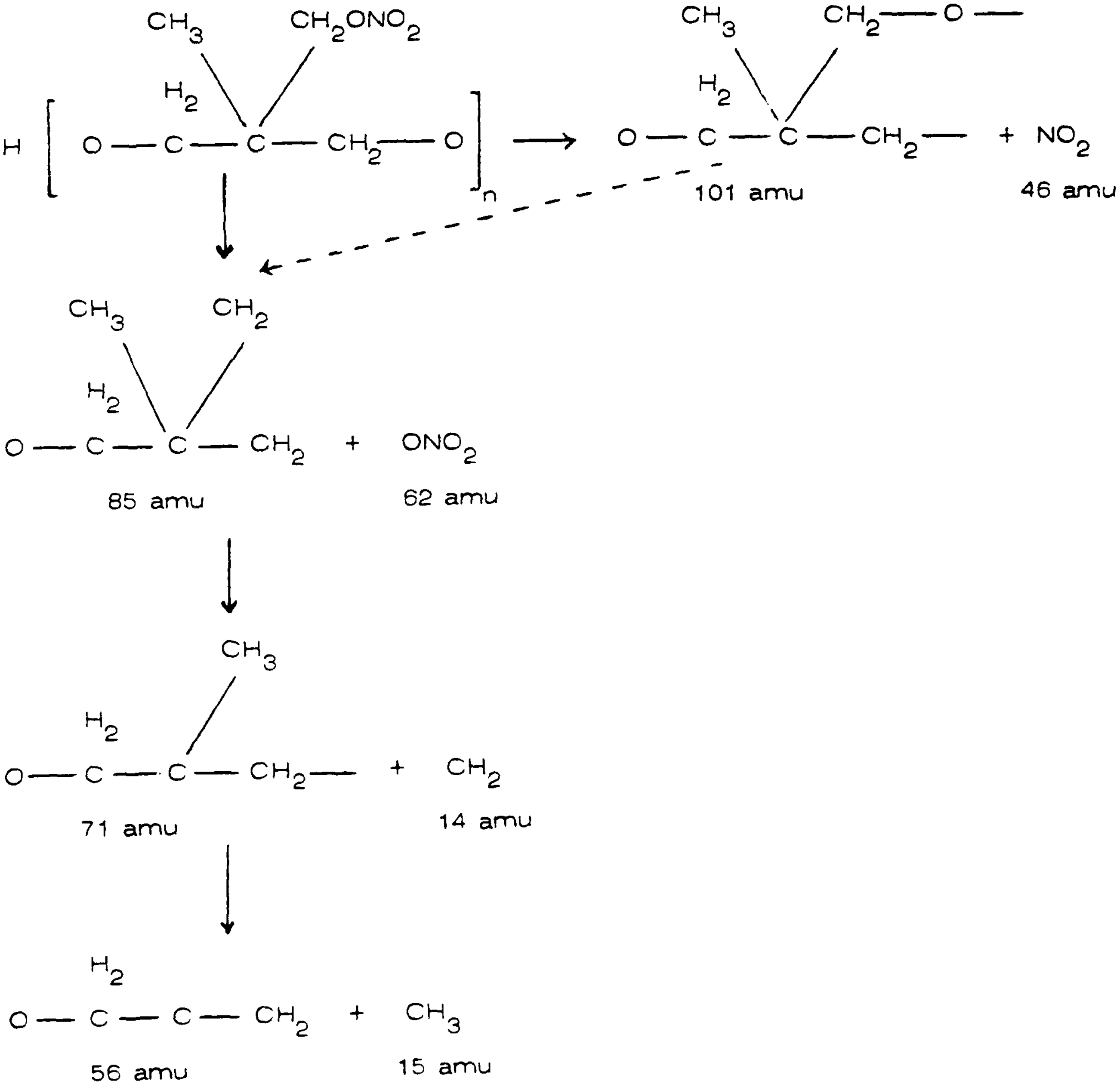


In addition, the usual small amu radicals and molecules observed in previous oxetane decomposition were seen.

B. Polymers

1. Poly (3-nitratomethyl-3-methyl) oxetane (p-NMMO)

A promising polymeric material appears to be poly (3-nitratomethyl-3-methyl) oxetane (p-NMMO). This material begins to decompose at approximately 100 C. Figure 6 shows the decomposition products in the amu range 14 - 105 at 150 C, where decomposition of the oxetane backbone is occurring. From these spectra the proposed decomposition methanism is:



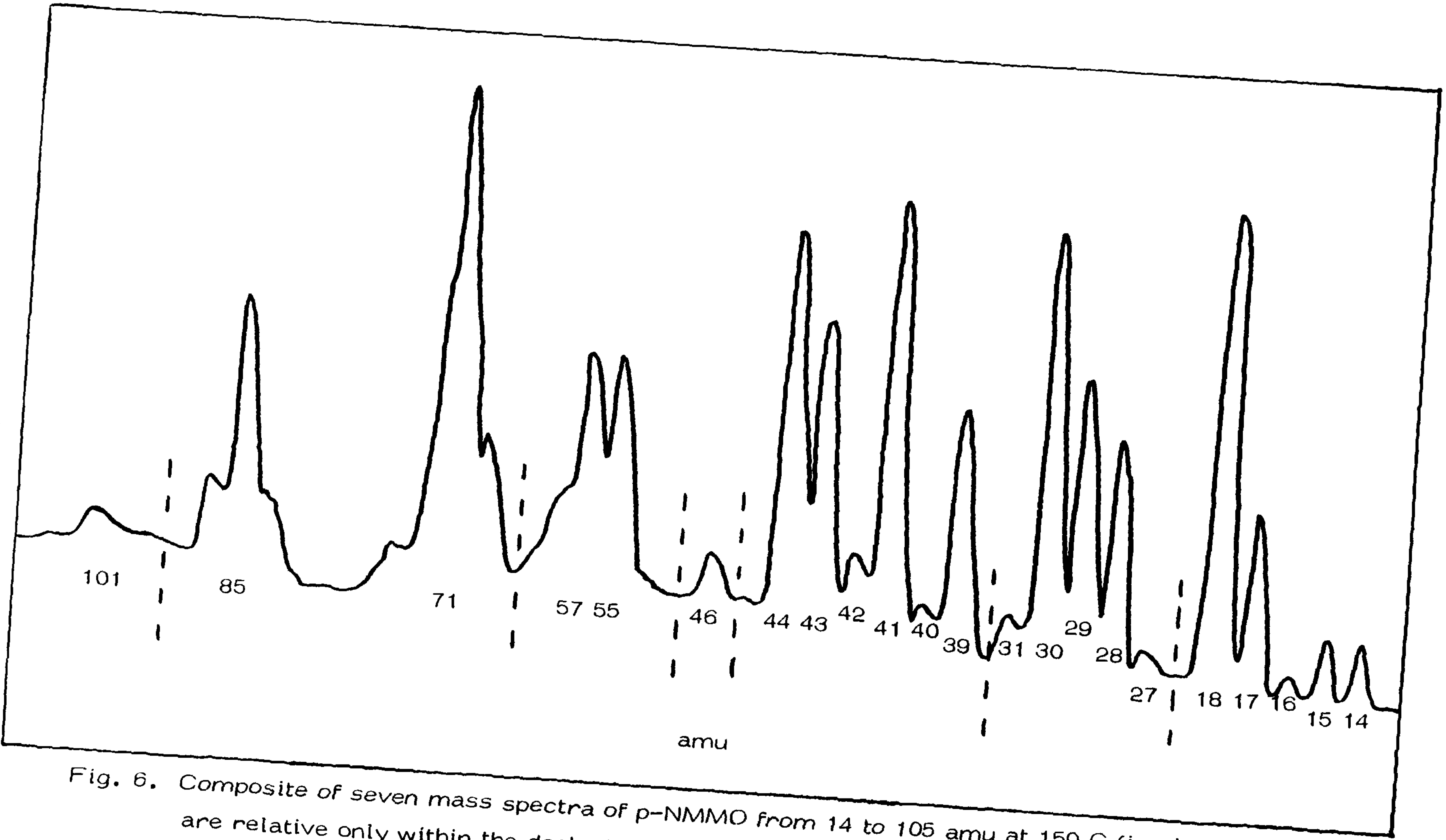


Fig. 6. Composite of seven mass spectra of p-NMMO from 14 to 105 amu at 150 C (ion intensities are relative only within the dashed lines)

A relatively small concentration of a product at 101 amu suggests a decomposition path with the release of NO_2 occurring from the rupture of the nitrate bond, $O{-}NO_2$. The low amu decomposition products are CH_2O at 30 amu, CHO at 29 amu, and CO at 28 amu. The prominent peaks at 14, 15 and 18 amu indicate CH_2, CH_3 and H_2O. The temperature dependence of the decomposition rate from 100 to 135 C is shown in Fig. 7. A 50-fold increase in relative ion intensities of NO_2 over this temperature range leads to an activation energy of 163 kJ mol^{-1} (39 kcal/mol).

Other laboratories are also investigating the thermal decomposition kinetics of p-NMMO.[5,6] DSC studies by Manser appear to suggest that nitrate ester decomposition is more thermally stable than the backbone cleavage. However, he reports that DSC kinetics employing the Ozawa multiple heat rate method do not support this observation. The FTIR investigation of Brill, made at higher temperatures (500 - 950 k) than those conducted at our laboratory (under 500 K), also indicates that the nitrate ester backbone decomposition is the primary mechanism, based on a high concentration of formaldehyde (65%) at 950 K. The method employed at our laboratory involves fast-time sampling (on the order of 100 microseconds or less) of the products mass spectrometrically. The initial gas products of the decomposition are thus allowed to enter the high vacuum of the mass spectrometer where they can be definitively identified. The method has proved successful in determining the thermal decomposition mechanism for HMX[3] and RDX.[4]

Since the results concerning the primary mechanism for p-NMMO (whether backbone cleavage or nitrate ester decomposition) are somewhat in disagreement, the three laboratories mentioned are planning to continue their efforts towards successfully elucidating the kinetics for this polymer.

2. Copolymer, 50/50 methyl nitramino methyl methyl oxetane/ azido methyl methyl oxetane (50/50 MNAMMO/AMMO)

The initial decomposition of the copolymer is quite similar to that of the individual homopolymers MNAMMO and AMMO. Decomposition gases evolving from the copolymer were observed as mass spectra at temperatures as low as 80 C. These gaseous products are from the

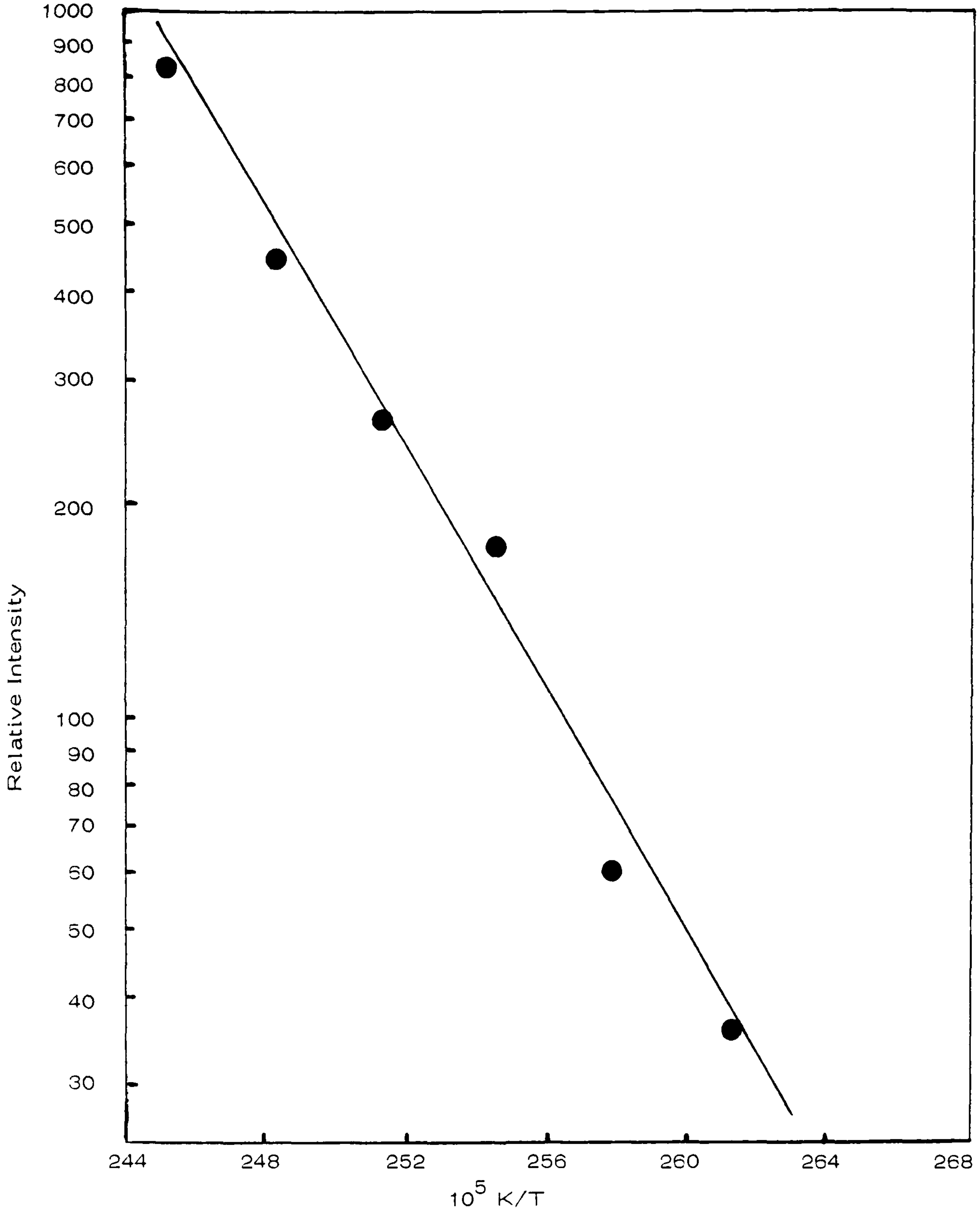
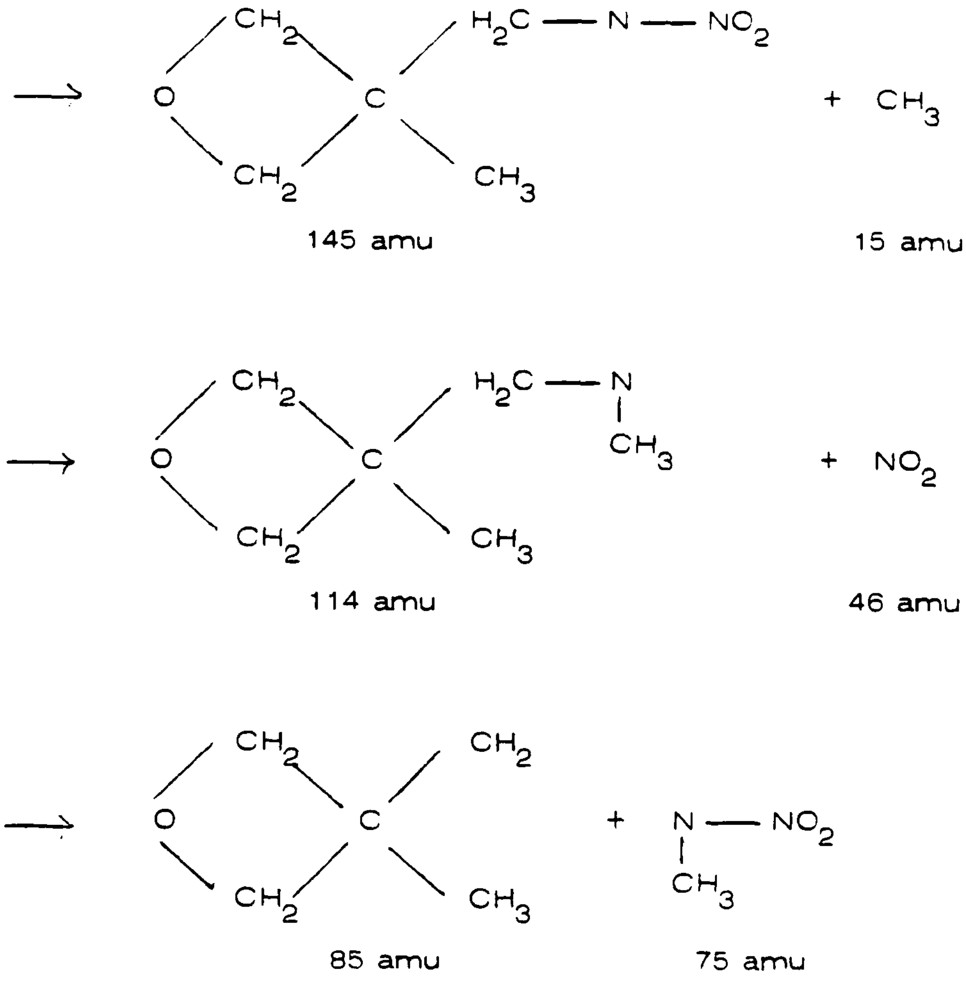


Fig. 7. Rate of decomposition of p-NMMO in the temperature range 100 to 135 C leading to an E_a of 163 kJ mol^{-1} (39 kcal/mole)

nitramine and agree with the decomposition peaks previously observed for the homopolymer. Figures 8.a. and 8.b. are comparison mass spectra and show the major ion intensities 85, 99, 114, 145, and 160 amu for the nitramine decomposition products at 120 C for both the copolymer (a.) and the homopolymer (b.). As can be seen, the relative intensities as well as the peaks are nearly identical for the homopolymer and the copolymer. This is substantial supporting evidence that the copolymer decomposes initially as separate homopolymers. The concentration of the nitramine fragment $CH_2\overset{CH_3}{N}—NO_2$ at amu 89 is quite temperature-dependent, as seen in Fig. 9. This figure shows the nitramine fragment increasing in intensity from its initial appearance at 70 C to a maximum at about 100 C, before its concentration commences to decrease. At 150 C a strong broad peak (81 - 83 amu) representing the oxetane backbone is observed. The mechanism for the nitramine polymer decomposition is the elimination of the entire nitramine arm or parts of the arm:



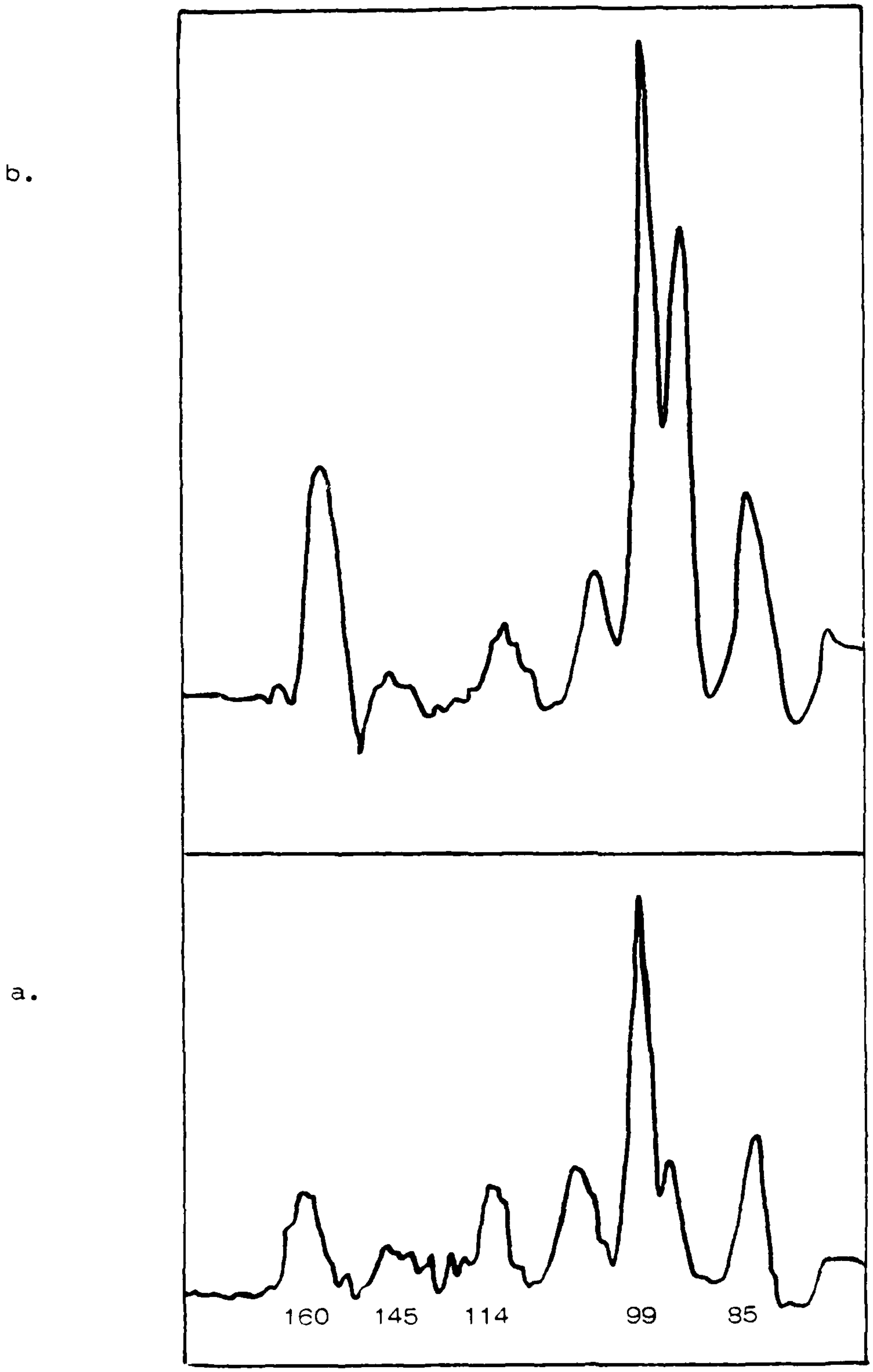


Fig. 8. Comparison of the thermal decomposition of 50/50 MNAMMO/AMMO (a) and of homopolymer p-MNAMMO (b) at 120 C. The relative intensities of the amu peaks in the 80 - 160 amu range indicate almost identical decomposition mechanisms for the copolymer and the homopolymer.

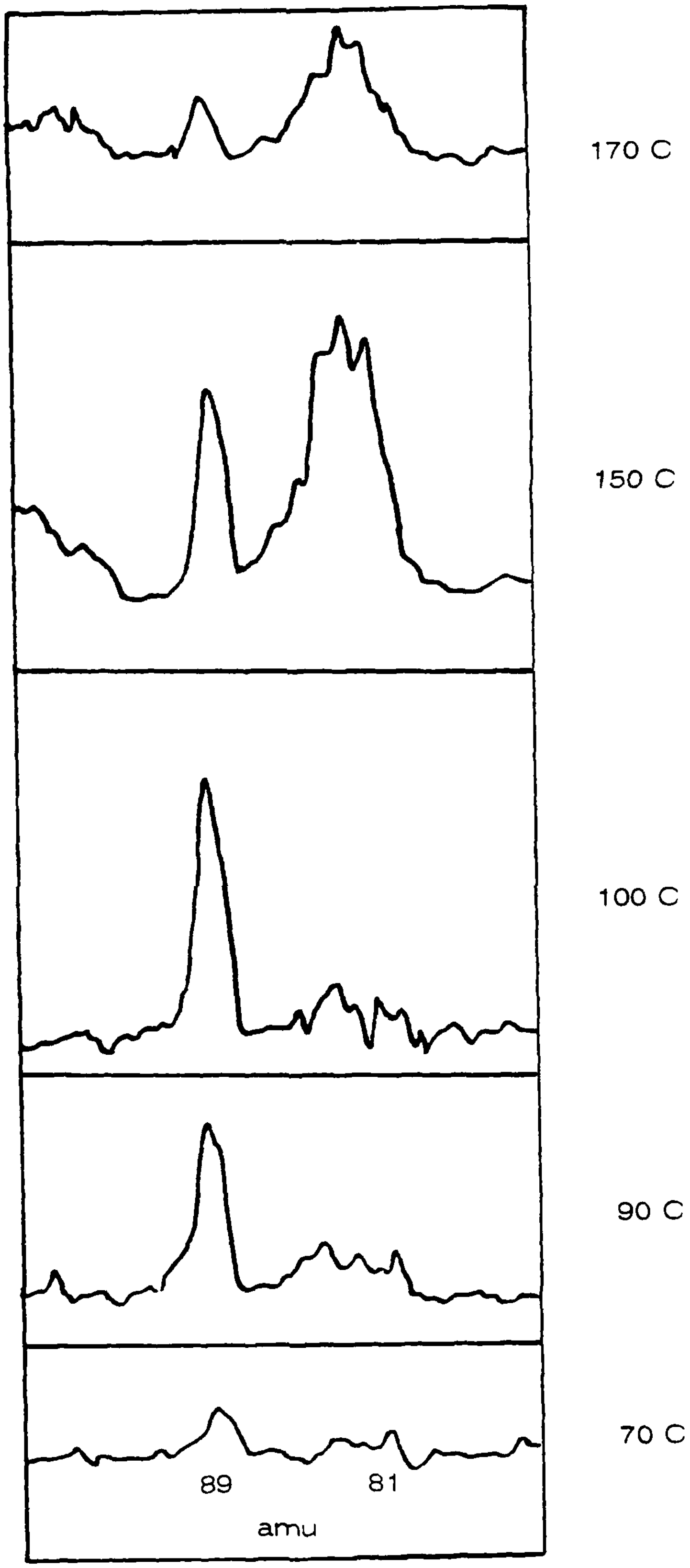


Fig. 9. Decomposition of 50/50 MNAMMO/AMMO as a function of temperature. The 89 amu peak represents the nitramino group whereas the 81 amu peak represents the oxetane backbone from AMMO decomposition.

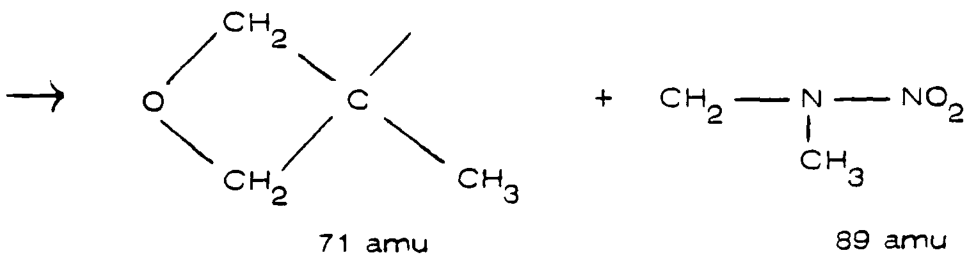


Figure 10 shows the high temperature (over 150 C) decomposition of the copolymer in the amu range 14 - 45. The spectrum of the backbone decomposition of the copolymer provides supporting evidence that once initial decomposition takes place (i.e., the nitramine arm of MNAMMO and the N_2 release of AMMO), the remaining condensed phase decomposes differently than either backbone of the two copolymers. This can be seen from an examination of the mass spectra in Figs. 10, 11 and 12. Figure 11 depicts the mass spectra in the amu range 14 - 45 for both the homopolymer MNAMMO (a.) and the homopolymer AMMO (b.). The backbone decomposition of the copolymer has individual peaks (Fig. 10) found in both homopolymers, whereas the homopolymer decomposition spectra, Fig. 11.a. and 11.b., are not identical. This can be seen clearly from Fig. 12.a. and 12.b. Figure 12.a. is the spectrum in the 14 - 18 amu range for the copolymer, while Fig. 12.b. shows the peaks in the same amu range for homo AMMO. Of interest is the methyl radical, CH_3, at amu 15, of considerably greater concentration in the copolymer backbone decomposition than that of the homo AMMO. Also, it can be seen that the relative ratios of OH/H_2O concentrations are reversed.

III. WORK ACCOMPLISHED DURING PREVIOUS CONTRACT PERIODS

Thermal decomposition kinetics of energetic and non-energetic monomers and polymers were reported in the Annual Summary Reports of 1981 through 1984, as well as in open literature publications.

Brief summaries of these annual studies are presented below.

A. Annual Summary Report - September 1981

The kinetics of thermal decomposition were determined for poly 3,3 bis (azido methyl) oxetane (BAMO), glycidyl azide polymer (GAP),

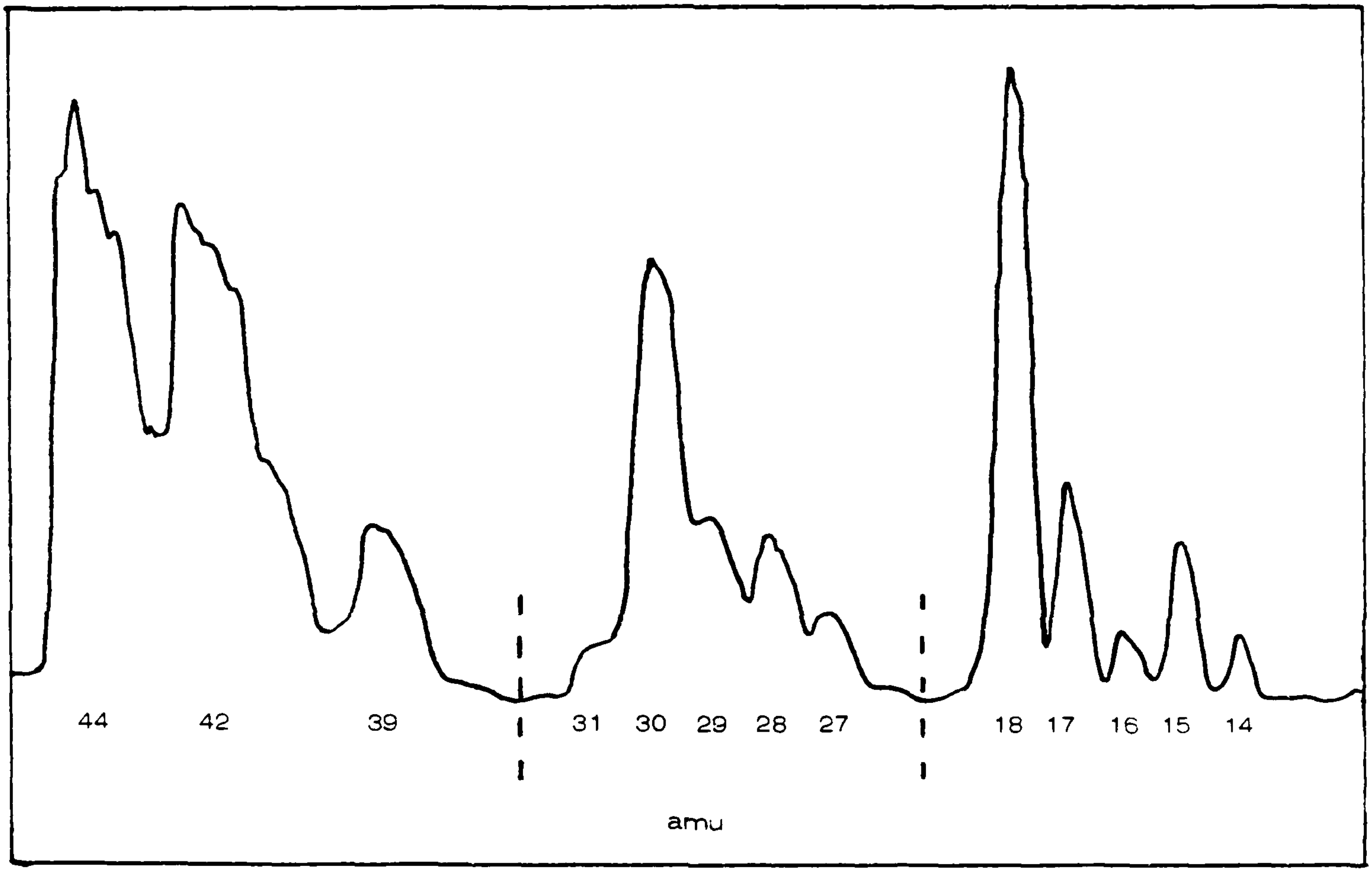


Fig. 10. High temperature thermal decomposition of 50/50 MNAMMO/AMMO at 160 C. The relative intensities are continuous only within the dashed lines.

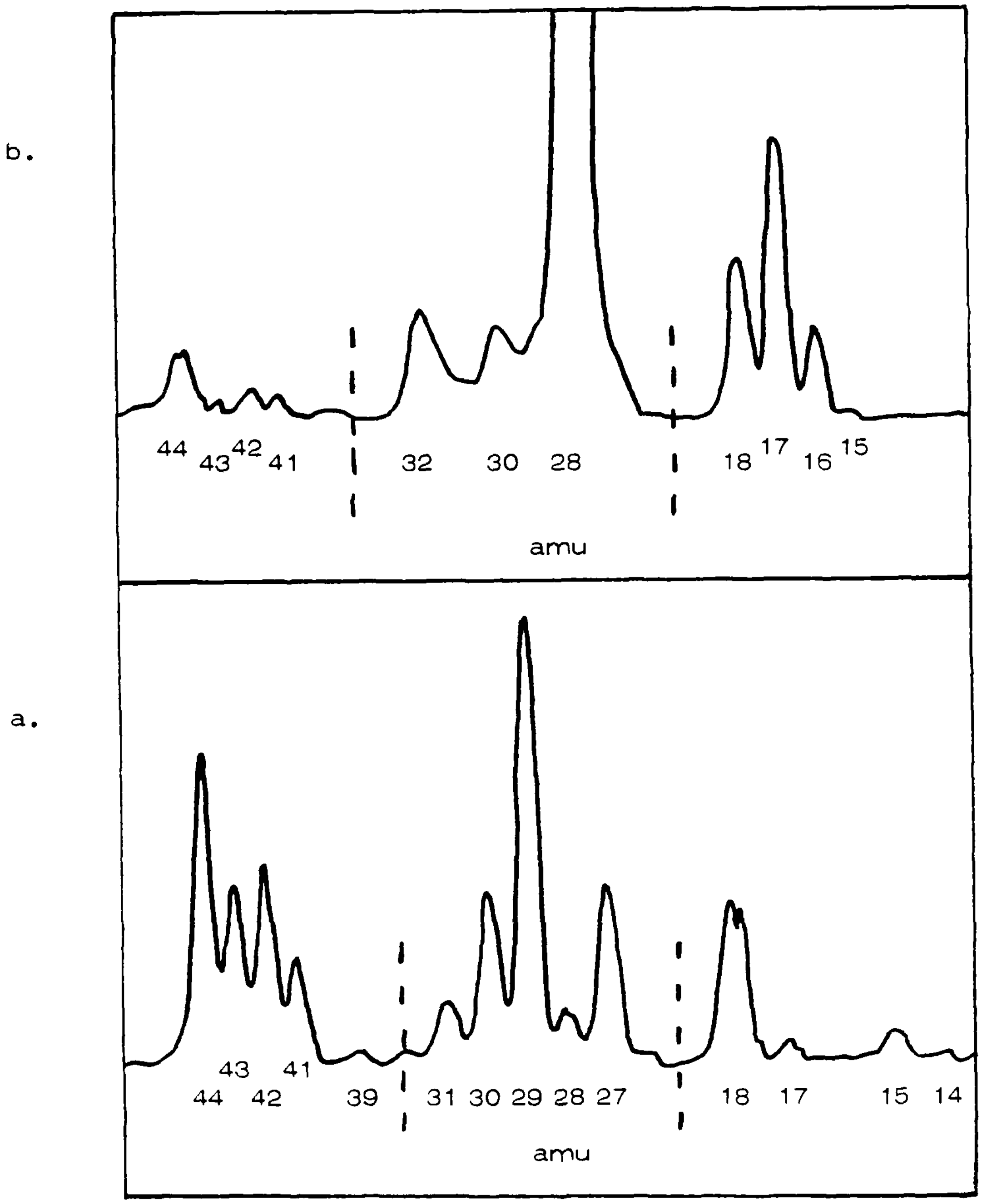


Fig. 11. High temperature thermal decomposition mass spectra of MNAMMO (a) and AMMO (b) in the 14 - 45 amu range (> 150 C)

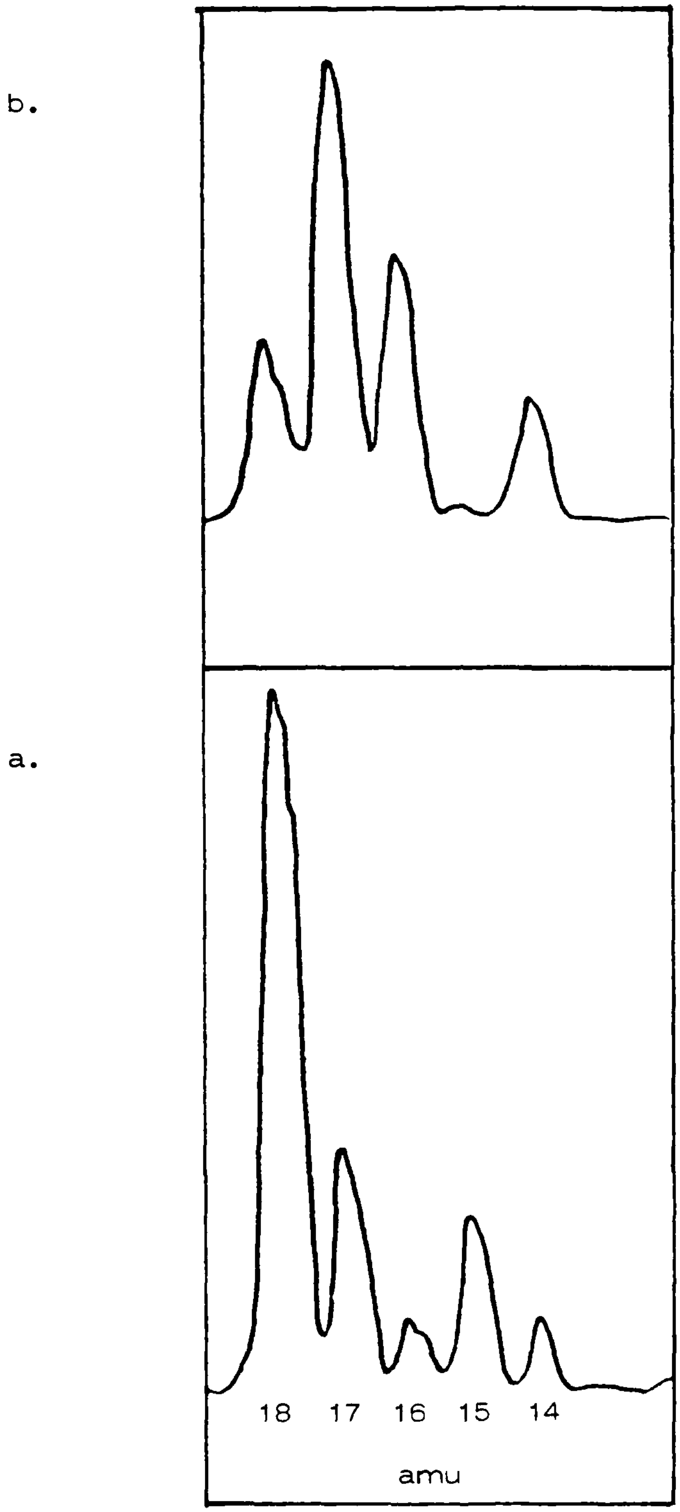


Fig. 12. High temperature thermal decomposition mass spectra of the copolymer 50/50 MNAMMO/AMMO (a) and the homopolymer AMMO (b) in the 14 - 18 amu range

and azido oxetane monomer (AZOX). Decomposition was initiated at approximately 120 C, with activation energies for the three materials approximately 170 kJ mol^{-1}. The primary mechanism for the decomposition is the release of molecular N_2. Secondary decomposition occurring at temperatures above 150 C included the fracturing of the three-carbon backbone of both the polymers and the monomer. Higher decomposition temperature species included HCN, HCO, and smaller amu fragments.

B. Annual Summary Report - September 1982

Thermal decomposition studies were conducted on AZOX and AMMO homopolymers and on 50-50% BAMO-AZOX and 50-50% BAMO-AMMO copolymers. Decomposition was initiated at approximately 120 C, with activation energies for the materials approximately 170 kJ mol^{-1}. The primary mechanism for the decomposition is the release of molecular N_2. Backbone decomposition occurs at temperatures above 150 C with thermal destruction of the three-carbon chain. A thermal decomposition study of a new copolymer consisting of an azido (BAMO) and nitrato ester, bis nitrato methyl oxetane (DNAO) showed both azide and nitrato group decomposition at differing rates. Preliminary mass spectrometric studies of the decomposition of a new amino nitro ring compound, $C_6H_8N_8O_{12}$, indicated NO_2 release with ring stability. Qualitative UV irradiation experiments at 2540 Å and 3660 Å wave lengths then were conducted on AZOX and AMMO homopolymers and 50-50% BAMO-AMMO and 50-50% BAMO-AZOX copolymers. Upwards of 25% of the polymeric N_2 was released with considerable cross-linking. The samples changed from viscous liquids to gummy, rubbery semi-solids. The irradiated samples were maintained for a number of weeks under ordinary atmospheric conditions as well as under vacuum without any indication of autocatalysis.

C. Annual Summary Report - September 1983

Thermal decomposition studies were performed on several azido and nitrato polymers and copolymers. Decomposition studies also included thermal plasticizers and novel nitramines. The 50-50% copolymer of BAMO-BNMO decomposes initially through the nitrato constituent at approximately 100 C followed by the azide decomposition. The thermal degradation mechanism for BEMO appears to be a stepwise breaking of the ethoxy-methyl bonds followed by the stripping of the methylene groups. BFMO is stable to temperatures greater than 200 C with initial decomposition products of HF, with considerable depolymerization occurring. The major decomposition product of the NSWC novel nitramine, $C_6H_8N_8O_{12}$, is the eight-membered nitramine ring less two NO_2 molecules, molecular weight 292. The decomposition product is stable in the gas phase at 300 C.

D. Annual Summary Report - September 1984

Effusion mass spectrometric investigations were performed on a new nitramine, 1,3,3 trinitro azetidine; a nitramine monomer and homopolymer, 3-methyl nitramino methyl 3-methyl oxetane, MNAMMO; poly (3-nitratomethyl 3-methyl) oxetane, NMMO; a copolymer consisting of 30% wt% random copolymer of bis (methoxymethyl) oxetane and poly-tetrahydrofuran, BEMO/p-THF/BMMO; and a cellulose acetate butyrate, CAB. From the mass spectra decomposition mechanisms were proposed and activation energies calculated. The degradation of the copolymer BEMO/BMMO/p-THF shows the influence of the individual homopolymers in that the decomposition products are those of the three constituents.

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6. T. Brill, University of Delaware, private communication to G. E. Manser (see reference 5 above).

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