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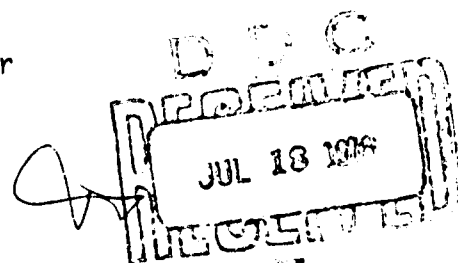
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REPORT

MRL-R-710

THERMAL DECOMPOSITION OF TETRAZENE AT 90°C

R. Bird and A.J. Power



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1. INTRODUCTION

$$\begin{array}{c} \text{N} - \text{N} = \\ || \\ \text{N} - \text{N} - \text{C} - \text{N} = \text{N} - (\text{N}_2\text{H}_2) - \text{C} = \text{NH} \\ | \\ \text{H} \end{array} \text{NH}_2 \cdot \text{H}_2\text{O}$$

(1)

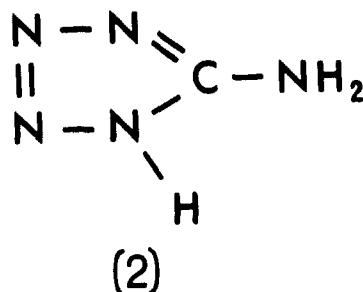
* The (N₂H₂) moiety in structure (1) permits either of the suggested part structures N-(NH-NH)-C and N-(N-NH₂)-C (refs. [3] and [4], respectively).

since pressure build-up from such a process could be sufficient to cause rupture of the seals. It was also considered expedient to determine the major decomposition products in an attempt to elucidate the mechanism of thermal decomposition.

Tetrazene decomposes too slowly at 60°C for the purposes of short-term experimentation [2]. It was found, however, that tetrazene decomposed almost completely after approximately one week at 90°C, and this temperature was selected for the accelerated thermal studies reported here.

2. RESULTS

A sample of tetrazene was heated in an oven at 90°C and its rate of thermal decomposition was monitored by infrared (i.r.) and ultraviolet (u.v.) spectroscopy. After 6 days the tetrazene had been decomposed and the solid residue consisted almost entirely of 5-aminotetrazole (2). The rate of formation of this substance was obtained from a calibration graph of u.v. absorbance against 5-aminotetrazole concentration (Figure 1) and is illustrated in Figure 2. At the completion of the experiment 1.7 mole of 5-aminotetrazole had been formed from 1.0 mole of tetrazene. The i.r. data indicated that guanyl azide was formed as an intermediate during the reaction. Its spontaneous conversion to 5-aminotetrazole is discussed in Section 3.



From these results it is clear that tetrazene decomposes quite rapidly at 90°C and is therefore unsatisfactory for use in any situation where this temperature may be approached. I.r. and u.v. monitoring data indicate that tetrazene stored at 90°C would lose its stab-sensitising properties in less than 3 days.

The rate of gas evolution from the decomposition of tetrazene at 90°C was measured separately, and the results of this determination are shown in Figure 3. Gaseous products equivalent to a total of 8.44 cm³ at STP were formed from 0.1 g of tetrazene and it was calculated that within the confines of an experimental hermetically-sealed detonator (Figure 4), in which the stab-sensitising layer comprises a mixture incorporating 5% tetrazene (0.030 g), decomposition of the tetrazene at 90°C would create a pressure of approximately 1 MPa (140 psi)*. This pressure should be sufficient to cause

* This calculation was based on the volume of gaseous products which would be evolved from 0.030 g tetrazene at 90°C occupying the available free space inside the detonator (i.e. voids between crystals as determined from known crystal densities of the lead azide and RDX increments).

initial swelling of the detonator cup and ultimate rupture of the seal. However, this temperature is unlikely to be reached in normal service storage environments and the situation at lower temperatures must be considered.

The shape of the graph for gas formation against time (Figure 3) indicates that the thermal decomposition reaction is autocatalytic and it is reasonable to suggest that the same mechanism may be operative at considerably lower temperatures. In the temperature range encountered in routine storage, however, the rate of reaction would be expected to be very slow and the guanyl azide intermediate would probably be more stable, producing a relatively larger amount of 5-aminotetrazole from the side chain (see Section 3) and less gaseous products than at 90°C. It is therefore unlikely that the hermetically-sealed detonators containing low percentages of tetrazene would rupture under long-term storage conditions.

Several tetrazene-sensitised compositions are available, and satisfactory initiation can be achieved by incorporating as little as 2% tetrazene in the stab-sensitive layer (e.g. VH2). It is clear that in the event of gas evolution occurring during long-term ambient storage of the detonators, less pressure would result from a composition with 2% tetrazene than one such as NOL 130 which contains 5%. At first sight this makes VH2 a more attractive selection, but if it is assumed that gas evolution is accompanied by 5-aminotetrazole formation there would be a larger amount of residual unreacted tetrazene in NOL 130 after similar storage periods. Consequently, eventual ignition failure would be more likely with detonators containing VH2 where the percentage of tetrazene could drop significantly below 2%.

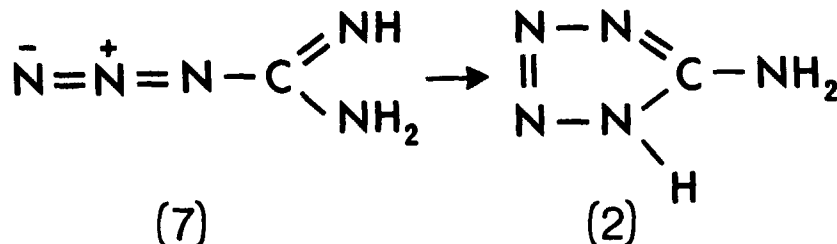
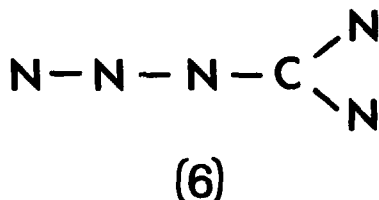
In balancing the probability of seal rupture against the probability of ignition failure we must emphasise that the amount of gaseous products formed from tetrazene at 90°C was far less than had been expected from such a thermally unstable compound. This was due to the fortuitous formation of a high yield of the solid product 5-aminotetrazole from the side chain of tetrazene.

Moreover, in view of the advantages of hermetically sealing the lead azide component from hydrolysis by atmospheric water vapour, weighed against the anticipated very slow decomposition of tetrazene at ambient temperatures, we consider that it would be worthwhile developing the proposed sealed detonators. It is clearly advisable, nevertheless, that the detonators should always be stored in the lowest temperature environment available to minimise the possibility of tetrazene decomposition.

3. DISCUSSION

The observed stoichiometry of 1.7:1.0 of 5-aminotetrazole formed from tetrazene in the thermal decomposition experiment described above indicates that this product may be derived from both the tetrazole ring system and the side chain in tetrazene. For the purpose of studying this reaction further, three tetrazene samples (3), (4) and (5) with ¹⁵N labels at specific sites were synthesised through diazotisation reactions using ¹⁵N-labelled sodium nitrite.

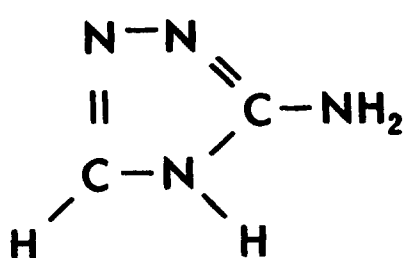
It is shown in Section 4.5 that at 140°C the tetrazole ring remains intact, since only the chain-labelled tetrazene (3) released $N^{15}N$ (m.w.29) while its ring-labelled counterpart (4) yielded only N_2 (m.w.28). It is reasonable to assume that at 90°C the tetrazole ring is similarly undisturbed and would be involved quantitatively in the formation of 1.0 mole of 5-aminotetrazole. The remaining 0.7 mole of 5-aminotetrazole must therefore be derived by cleavage of the azo bond in tetrazene to give an intermediate with skeleton structure (6), which then cyclises.



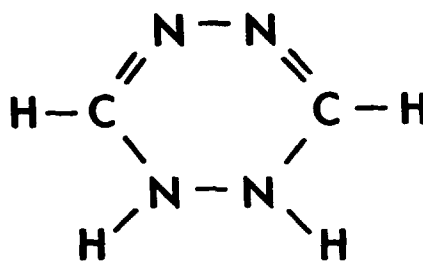
This implies that guanyl azide (7) is the intermediate species. The spontaneous cyclisation of this azide to 5-aminotetrazole is well documented [5]. The formation of guanyl azide during the reaction is supported by the i.r. data presented in Table 1, which shows qualitatively that after 3 days the azide and 5-aminotetrazole have formed at the expense of tetrazene, with the final residue showing only the main absorptions of 5-aminotetrazole.

Corroborative evidence for the derivation of this compound from both the tetrazole system and the side chain is provided by the mass-spectral data shown in Figure 5. While the major product of the thermal decomposition of the mono-labelled tetrazenes (3) and (4) have almost identical spectra with parent ions at m/e 85 and 86 corresponding to unlabelled and labelled 5-aminotetrazole molecules (2) and (8), respectively, the double-labelled tetrazene (5) has decomposed to yield only (8) (see Scheme 1).

A compound with m.w.84 detected in the residues from the thermal decomposition of unlabelled tetrazene did not incorporate the label on decomposition of any of the labelled tetrazene models, suggesting that it is formed via reaction of two formamidine end groups to give (9) or (10). Due to its low yield, no attempt was made to establish the structure of this compound.



(9)



(10)

4. EXPERIMENTAL

4.1 Introduction

Infrared spectra were recorded on a Unicam SP 1000 Spectrophotometer as KBr discs. Ultraviolet spectra were determined in the range 275-200 nm using a Unicam SP 800 Spectrophotometer with 1 cm cells of solutions in benzene-free u.v. grade ethanol. Mass spectra were measured with a Varian MAT III GC/MS System, using the direct insertion probe inlet (unless otherwise stated). Isotopically labelled sodium nitrite ($\text{Na}^{15}\text{NO}_2$) was from Stohler Isotope Chemicals and had a stated purity of 99.5%.

4.2 Thermal Decomposition of Tetrazene at 90°C

A sample of tetrazene was heated at 90°C for 7 days in a gravity convection oven and specimens were removed periodically for i.r. and u.v. analysis. At the end of this period the residue was dissolved in hot ethanol and filtered to remove traces of insoluble material. The solution was cooled and white needles were obtained with m.p. 194-196°C which was not depressed in admixture with authentic 5-aminotetrazole (2) (m.p. 199°C, lit. [6] m.p. 203°C). The i.r. spectra of the reaction product and (2) were identical.

4.2.1 Spectral Analysis (i.r. and u.v.)

The thermal decomposition of tetrazene was monitored by studying the i.r. and u.v. spectra obtained from the samples taken during the experiment (Section 4.2). After 3 days the characteristic i.r. absorbances of tetrazene

were greatly diminished and the major absorption peaks indicated that 5-aminotetrazole was being formed. In addition, a very strong peak near 2100 cm^{-1} indicated the presence of an organic azide. As the periodic sampling continued the spectra indicated further decomposition of tetrazene, and after 6 days the peak near 2100 cm^{-1} was no longer present. Only the spectrum of 5-aminotetrazole remained (see Section 4.2).

The u.v. spectrum of authentic 5-aminotetrazole (2) shows a single strong absorbance at 219 nm. The u.v. spectra of progressive samples obtained from thermal decomposition at 90°C showed the growth of an absorbance at 219 nm. This was attributed to the formation of (2) and was measured quantitatively as described in Section 4.2.2.

4.2.2 Quantitative Determination of 5-Aminotetrazole Formation

Ethanol solutions containing known various concentrations of authentic recrystallised 5-aminotetrazole were prepared, their u.v. spectra recorded, and a calibration curve of concentration against absorbance at 219 nm was drawn (Figure 1). The u.v. spectra of residues from the thermal decomposition of tetrazene were also recorded and by interpolation from Figure 1 it was possible to determine the rate of formation of 5-aminotetrazole. This is shown graphically in Figure 2. After 6 days ca 1.7 moles of 5-aminotetrazole had been formed from 1.0 mole of tetrazene.

4.2.3 Measurement of Gas Evolution During Decomposition of Tetrazene at 90°C

A sample of tetrazene (0.1 g) was heated at 90°C in a vacuum tube connected to a mercury manometer. The initial pressure inside the system was 5×10^{-2} Torr (7 Pa) and the pressure increase during reaction was converted to give the increase in gas volume. A graph of volume of gas against reaction time was plotted (Figure 3) and a total of 8.44 cm^3 of gas was evolved.

At the completion of the experiment the solid residue was removed, dried and weighed (0.0769 g). Calculation showed that 1.7 moles of 5-aminotetrazole had formed from 1.0 mole of tetrazene; this result is in agreement with that in Section 4.2.2.

4.2.4 Preparation of Guanyl Azide Nitrite

This compound was synthesised for the purpose of observing the formation of the organic azide during thermal decomposition of tetrazene (Section 4.2.1 and Table 1). The azide was prepared by addition of a solution of sodium nitrite to aminoguanidine sulphate in water in the presence of nitric acid [7].

4.3 Synthesis of ^{15}N - labelled Tetrazenes

Tetrazenes (3), (4) and (5) incorporating an ^{15}N label at specific sites in the molecule were prepared for mass-spectrometric analysis of the thermal decomposition products of tetrazene.

4.3.1 Tetrazene (3): ^{15}N in Azo Group

$\text{Na}^{15}\text{NO}_2$ (0.030 g) was added to an aqueous solution of aminoguanidine sulphate (0.1 ml, 250 g l^{-1}) and 5-aminotetrazole (2) (0.4 ml, 42.4 g l^{-1}) and the reaction mixture was heated to 60°C over a period of 15 min. Tetrazene (3) then precipitated out of solution. The site of the ^{15}N is unambiguous since the reaction proceeds via diazotisation of the amino group of (2).

4.3.2 Tetrazene (4): ^{15}N in Tetrazole Ring

^{15}N - labelled 5-aminotetrazole (8) was prepared as follows : $\text{Na}^{15}\text{NO}_2$ (0.19 g) was added to a solution of aminoguanidine sulphate (0.35 g) and conc. HNO_3 (0.23 g) in water (1 ml) at 15°C . Sodium acetate (0.4 g) was added, and the solution was heated to 90°C for 5 min. On cooling to 5°C , the tetrazole (8) precipitated from solution. Diazotisation of the amino group in aminoguanidine sulphate followed by cyclisation places the ^{15}N label unambiguously in the 2-position. The tetrazole (8) was then reacted with aminoguanidine sulphate and NaNO_2 under the conditions described in 4.3.1 to yield tetrazene (4).

4.3.3 Tetrazene (5): ^{15}N in Azo Group and Tetrazole Ring

^{15}N - labelled 5-aminotetrazole (8) was reacted with aminoguanidine sulphate and $\text{Na}^{15}\text{NO}_2$ under the conditions described in Section 4.3.1 to produce the double ^{15}N - labelled tetrazene (5).

4.4 Mass-Spectrometric Analysis of Solid Residues from Thermal Decomposition of Labelled Tetrazenes

Mass spectra of the solid residues from thermal decomposition (9 days, 90°C) of tetrazene (1) and its ^{15}N - labelled relatives (3), (4) and (5) were recorded. By slow heating of the probe, a compound with m.w.84 (mass spectrum: m/e 26 (18%), 27 (83%), 29 (80%), 41 (25%), 42 (87%), 43 (71%), 57 (84%), 84 (100%)) was observed, and its spectrum persisted during the recording of the mass spectra of the 5-aminotetrazoles at higher temperatures. Since the spectrum of authentic 5-aminotetrazole (2) (m.w.85) has no peak at m/e 84, contributions from this substance were subtracted from the subsequent spectra which are reproduced in Figure 5 and discussed in Section 3. Note that the spectra of (2) and (8) in Figure 5 were identical with those of authentic 5-aminotetrazole and ^{15}N - labelled 5-aminotetrazole, respectively.

4.5 Thermal Decomposition of Tetrazene at 140°C

Tetrazene (1) decomposes very rapidly at 140°C which is just below its ignition temperature of 143°C . Identification of the gaseous products of decomposition at that temperature was accomplished by one-line pyrolysis/gas chromatography/mass spectrometry. The pyrolysis unit comprises a constant temperature furnace connected directly to the injection port of the gas chromatograph (GC) of the Varian MAT III GC/MS System. The furnace houses a silica tube which transports helium carrier gas to the GC column.

Samples of chain- and ring-labelled tetrazenes (3) and (4), respectively, were introduced into the tube by an SGE solids injector through a silicone rubber septum, and the gaseous decomposition products were separated

on the GC column (Porapak Q, 2 m x 3 mm o.d., 50-150°C at 6°C/min) and identified by the mass spectrometer. The two major products were water and nitrogen and the mass spectra yielded the following information.

	<u>Peak 1</u>	<u>Peak 2</u>
Tetrazene (3) (chain-labelled)	M^{+} 18 (H_2O)	28 (N_2) and 29 ($N^{15}N$)
Tetrazene (4) (ring-labelled)	M^{+} 18 (H_2O)	28 (N_2)

The absence of $N^{15}N$ from (4) indicates that at 140°C the tetrazole ring remains intact and decomposition of the sidechain occurs. At temperatures higher than 143°C (e.g. 155°C) destruction of the tetrazole ring was demonstrated by the formation of $N^{15}N$ from (4).

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T A B L E 1

CHANGES IN MAJOR i.r. ABSORPTIONS (cm⁻¹) OBSERVED
DURING THERMAL DECOMPOSITION OF TETRAZENE AT 90°C

Reaction Time (days)			Reference Compounds	
0 ^a	3	6	5-amino-tetrazole	guanyl azide nitrite
3350 B/S ^b	3400 B/S	3400 B/S	3400 B/S	
3000 B/S				
	2100 B/S			2100 S/S
1710 S/S ^c				
	1670 B/S	1670 B/S	1670 B/S	1670 B/S
1630 S/S				
	1590 S/S			
1490 S/S				
1420 S/S				
				1385 ^d B/S

a - Spectrum of pure tetrazene

b - B/S - broad, strong

c - S/S - sharp, strong

d - N-O stretch from NO₂ group

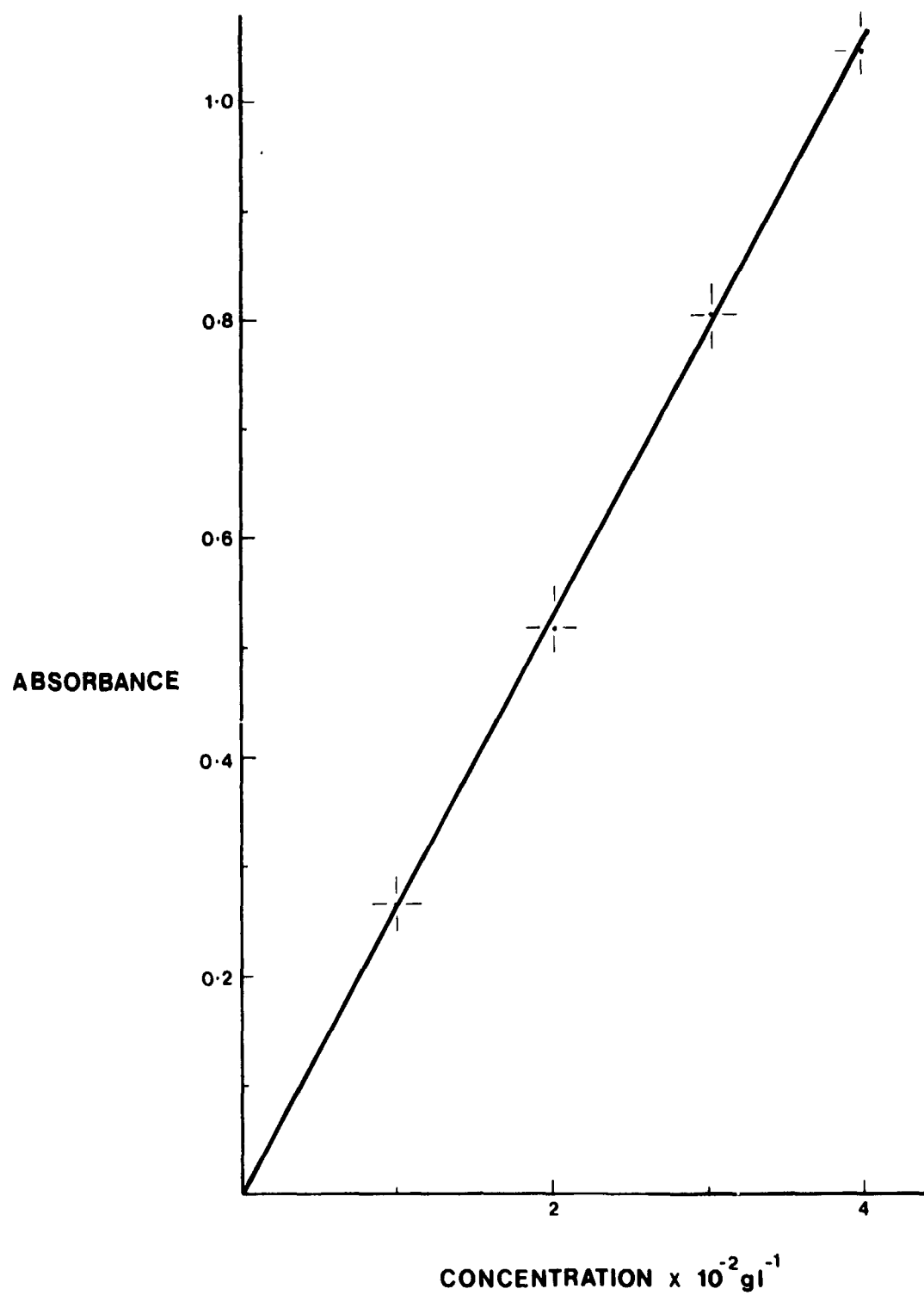


Fig. 1. UV ABSORBANCE AT 219 nm v. CONCENTRATION OF 5-AMINOTETRAZOLE.

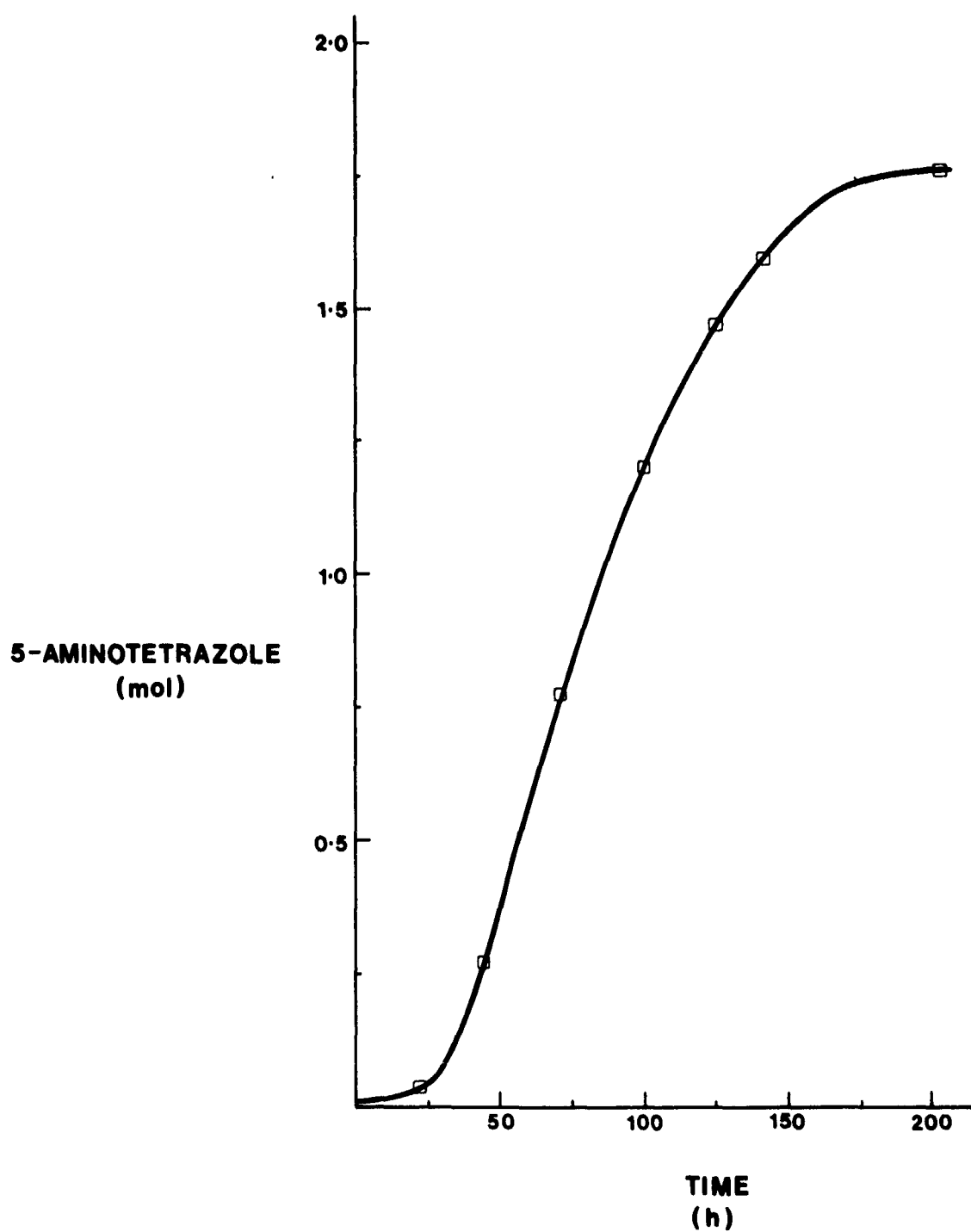


Fig. 2. RATE OF FORMATION OF 5-AMINOTETRAZOLE AT 90°C PER 1.0 mol OF TETRAZENE.

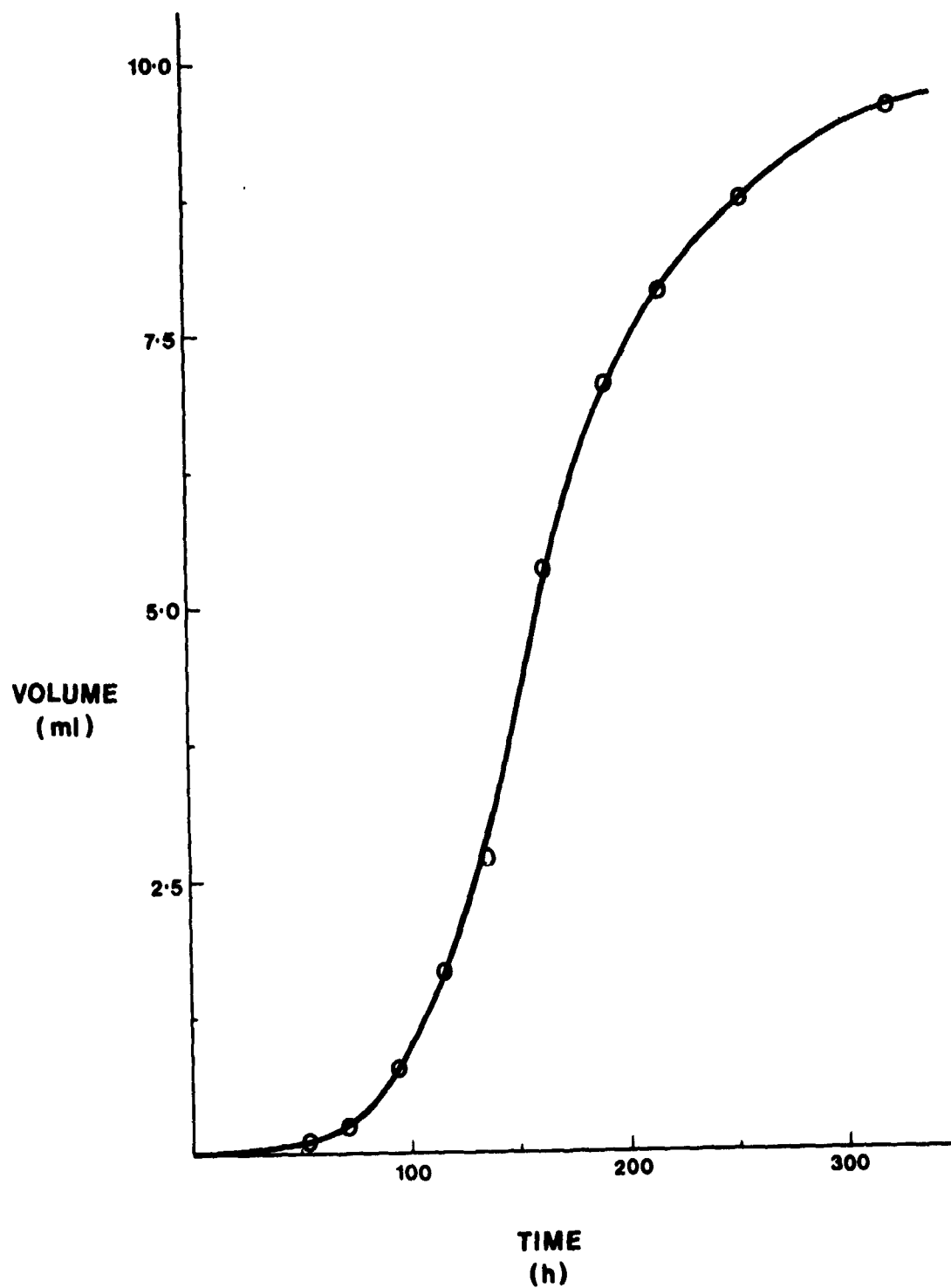
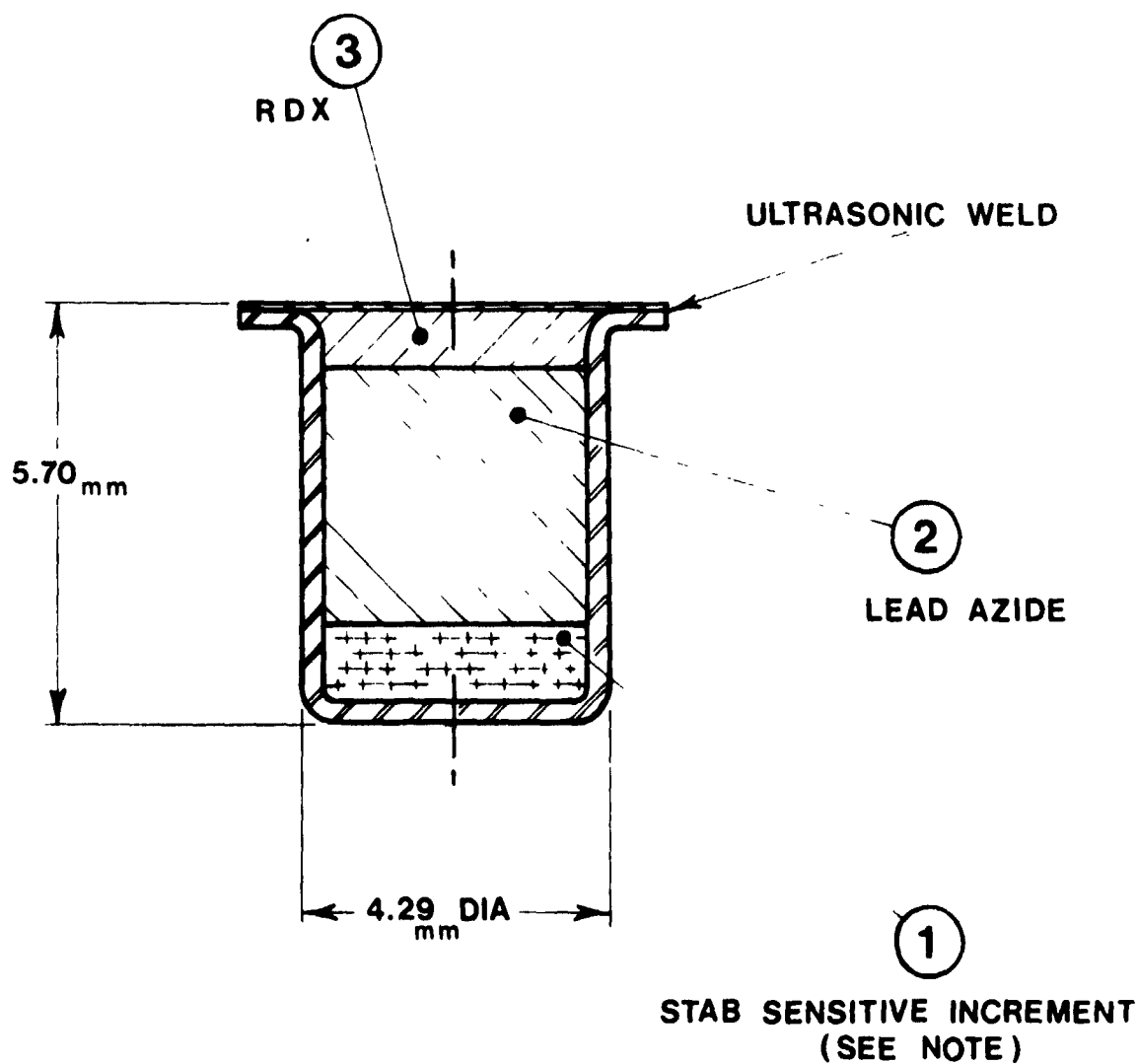


Fig. 3. RATE OF GAS EVOLUTION FROM 0.1g OF TETRAZENE AT 90°C.



NOTE:

STAB SENSITIVE INCREMENT 'NOL 130'

TETRAZENE	5 %
ANTIMONY SULPHIDE	15 %
BARIUM NITRATE	20 %
LEAD STYPHNATE	40 %
LEAD AZIDE	20 %

Fig. 4. EXPERIMENTAL SEALED DETONATOR

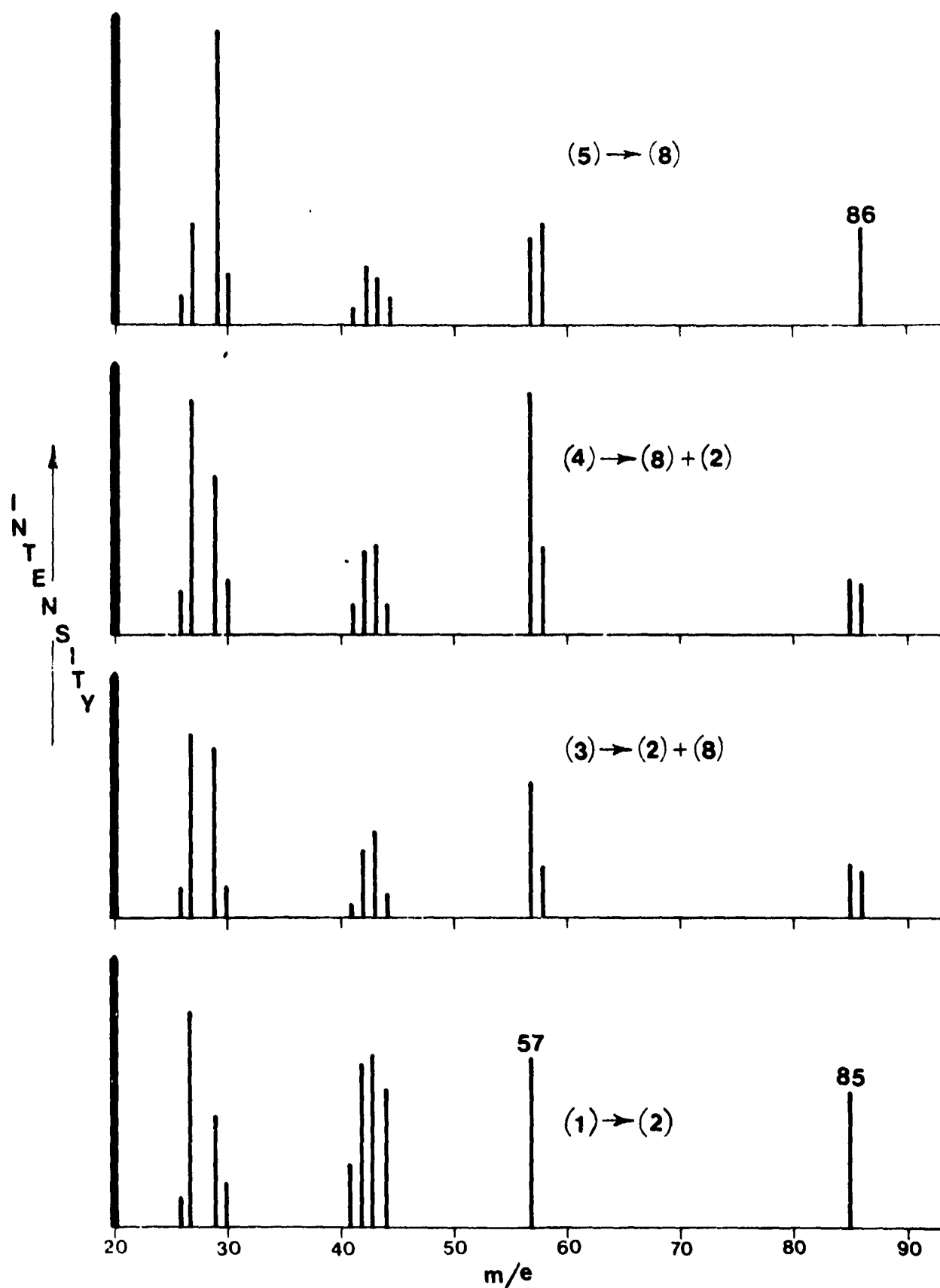


Fig. 5. MASS SPECTRA OF 5-AMINOTETRAZOLES DERIVED FROM THERMAL DECOMPOSITION OF TETRAZENES (1), (3), (4) AND (5).

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