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# HIGH-TEMPERATURE VACUUM THERMAL

### STABILITY TESTS OF EXPLOSIVES

#### by

### John F. Baytos

### ABSTRACT

Twenty-six explosive compounds, candidates for high-temperature applications, were subjected to vacuum thermal stability tests at 200, 175, and 150 °C for periods up to 90 days. RDX, HMX. and TNT were also run at the lower temperatures for comparison. The test results are given in tabular form, grouped by temperature, and in graphical form by compound for the three temperatures.

# I. INTRODUCTION

Among several tests used to determine the resistance to decomposition of explosives at higher than normal test temperatures, the vacuum thermal stability (VTS) test at constant pressure and constant temperature is a fairly reliable one for screening candidates for high-temperature applications. Twenty-six explosive compounds secured from Group WX-2 were subjected to the VTS tests at temperatures of 200, 175. and 150 °C for periods of time up to 90 days or until 15 cm<sup>3</sup>/g at STP total gas was evolved. This report gives only the results of the tests performed at Group WX-3, without any attempt at screening.

#### **II. EXPLOSIVES TESTED**

The explosives in this test series are given below, along with their acronyms. The acronyms are used throughout the rest of the report. In addition, the common explosives HMX, RDX, and TNT used in this laboratory have been tested at the lower temperatures to serve as comparisons. Some of the compounds are used as main charge explosives: second booster explosives, or EBW detonator explosives. Some of the samples were synthesized at Group WX-2, others were secured from various AEC installations, and some were obtained commercially. The explosives were dried in a vacuum oven overnight at 60°C and no further treatment was given before the test, except as noted for PADP-I.

#### III. RESULTS

The results of the total gas evolution of the various explosives tested at 200 °C are given in Table I. When the total gas evolved was greater than 15 cm<sup>3</sup>/g, the test was terminated and appropriate foot notes were posted. Where the test went the full 90 days, no comment was made. As a ground rule, those explosives that evolved 2 cm<sup>3</sup>/g or less of total gas were not run at the next lower temperatures.

The results of the tests at 175°C are given in Table II. and the same rules were followed. RDX. HMX. and TNT were run at this temperature. and they decomposed quickly.



## NOMENCLATURE OF EXPLOSIVES TESTED FOR

#### HIGH TEMPERATURE RESISTANCE

ABH	Azobishexanitrobiphenyl
BTX	Dinitropicrylbenzotriazole
bis-HNAB	bis-Hexanitroazobenzene
DATB	Diaminotrinitrobenzene
DIPAM	Dipicramide
DODECA	Dodecanitroquaterphenyl
DPBT	Dipicrylbenzobistriazoledione
DPPM	Dipicrylpyromellitide
HNAB	Hexanitroazobenzene
HNBP	Hexanitrobiphenyl
HNDS	Hexanitrodiphenylsulfone
HNS	Hexanitrostilbene
KHND	Potassium salt of hexanitrodiphenylamine
NONA	Nonanitroterphenyl
ONT	Octanitroterphenyl
PADP-I	Bis(picrylazo)dinitropyridine
PATO	Picrylaminotriazole
PENCO	Pentanitrobenzophenone
PYX	Bis(picrylamino)dinitropyridine
TATB	Triaminotrinitrobenzene
TNN	Tetranitronaphthalene
TPB	Tripicrylbenzene (Windmill)
TPM	Tripicrylmelamine
TPT	Tripicryltriazine
T-TACOT	Tetranitrobenzotriazolo(1,2-a)benzotriazole
Z-TACOT	Tetranitrobenzotriazolo(2, l-a)benzotriazole

The results of the tests at 150°C are given in Table III. RDX, HMX, and TNT did a little better at this temperature, but this temperature is still too severe for these compounds.

The data for each explosive were plotted for each of the three temperatures of test. The solid curve represents the data at 200°C. the closely broken curve represents the next lower temperature, 175°C, and the widely spaced curve represents the 150°C test in Figs. 1-30. As expected, the curves generally became less steep with the lower test temperature.

The exceptions to this observation were the compounds TPB and Z-TACOT, which behaved eratically at 200°C. Repeat tests showed the same pattern. DIPAM and HNAB at 175°C exhibited the same type of reaction also.

PADP-I at 200°C evolved gas immediately and blew mercury out of the manometer into a safety catch basin in less than two days. At 175°C, PADP-I evolved gas not as rapidly and the gases reacted with the mercury in the manometer. At 150°C, PADP-I went the full 90 days, but it reacted with mercury also. After the initial high amount of gassing, it stabilized to evolve very little more gas. When the PADP-I sample was dried 24 hours at 150°C in a vacuum oven, the sample showed very little gassing initially, which suggests that the impurities left in the sample had been removed.

Except for these anomalies, the rest of the compounds exhibited decreasing activity as the test temperature was lowered.

The post-test examination of the manometer tube. its contents, and other residuals was made after the tube was removed from the bath and allowed to cool to room temperature. Each sample tube was examined for sublimate of the explosive above the sample in the tube where it just leaves the hot oil and for quantity of condensate on the cold member. Some evolved gases also reacted with the mercury interface of the manometer and this was noted. On opening the sample bulb to the atmosphere, the odor of oxides of nitrogen was noticed. The residue was compared to the original material before it was shipped to WX-2 for microscopic and x-ray diffraction examination and evaluation.

The results of the post-test examination are given in Table IV.

## IV. APPARATUS

The apparatus used is similar to that described in "Military Explosives" <sup>1</sup> with the following modifications. The thermostated and stirred bath used DC-550 heating oil to withstand the higher test temperatures. Thermocouples to a calibrated recorder monitored the temperatures and showed that the baths were being controlled to  $\pm 1$  °C. An NBS calibrated platinum resistance thermometer was used to calibrate the temperature of the bath and the controllers periodically.

The sample bulb was a standard taper 12/30 male jointed. 110 mm long by 7 mm o.d. by 5 mm i.d., and was coupled to the manometer with springs over glass hooks to form a gas-tight seal. A standard tapered Teflon sleeve inserted between the male and female taper made the vacuum seal more secure. The sample bulb was inserted in the bath to the level of the lower glass hooks. The sample bulb was calibrated with mercury to determine absolute volume. Calibration of several sample bulbs gave a percent standard deviation of 2.6.

The manometer was a U-tube positioned on a meter stick with clamps, with the left opening a standard taper 12/30 female joint and the other end open to the atmosphere. To this end a short piece of vinyl tubing was loosely fitted which led into a catch basin to contain the mercury if it overflowed. The mercury levels on both sides were read from the attached meter stick, once for mercury height at the sample side and once for mercury height at the atmospheric side. The calibration of the absolute manometer volume was determined by weighing with mercury. From this procedure the volume per unit length was also determined. The percent standard deviation of several manometer callbrations is 1.9.

Racks for holding seven manometers in a secure position, and overflow catch basins for catching mercury in case of overgassing complete the apparatus.

### V. EXPERIMENTAL

All samples of explosives were routinely dried overnight in a vacuum oven at 60°C. A sample, 0.3 g, weighed to the nearest milligram, was inserted into a test bulb. The test bulb was attached to the U-tube manometer and secured with Teflon sleeves and springs over the glass hooks. This assembly was fitted into the vacuum pumping and mercury loading system. The system was pumped down to 30 mm Hg, before the mercury was transferred to the manometer to seal the sample in a partial vacuum. The vacuum pump was disconnected from the system by valves at the atmospheric end of the manometer. Atmospheric air was bled into the manometer and the mercury rose to its equilibrium level. The assembly was removed from the pumping system and transferred to the test bath. The bulb end of the manometer was then lowered into the thermostated oil bath as the measuring end was attached to the holding rack. After 30 minutes of equilibration in the test bath, the manometer was read and the data recorded. The original volume was calculated by applying the STP gas laws to the calibrated measurements of the volume of the manometers. The manometer was read daily for the first week. and then weekly up to 13 weeks when the test was terminated. The increase in volume at STP over the original volume was calculated and normalized to a unit mass basis for each weekly reading and plotted. The measurements were made until 15  $cm^3/g$  at STP total gas evolved or until 90 days had elapsed. The tubes were removed from the bath and were inspected for

condensate on the cold part of the tube, sublimate above the hot bulb, and for reaction of the evolved gases with the mercury interface. A check for odor of oxides of nitrogen, if any, was made on opening the tube, and the state of the residue and change in color was noted. The solid residue was then packaged and sent to WX-2 for examination by x-ray diffraction to get powder patterns to find what is left of the explosive.

The VTS tests in this experiment were compared with the standard method of test as described in Analytical Instructions<sup>2</sup> on DATB and TATB. Both systems generated the same gas evolution curve as a function of time and temperature, and results determined by either method were considered interchangeable. Since this was a long-term test, the experiment was isolated from the routine

operations in the blowout bay facility of the WX-3 laboratory, TA-16-460.

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### ACKNOWLEDGMENTS

This work originally started as a Plowshare experiment, but evolved into a general development program for high temperature resistant explosives evaluation. The materials support and samples were provided by L. C. Smith and M. D. Coburn, WX-2.

#### REFERENCES

- "Military Explosives" Technical Manual 1. No. 9-1300-214, Technical Order No. 11A-1-34, Department of the Army and the Air Force, Washington, DC, 28 Nov. 1967.
- WX-3 Analytical Instruction 7.22.1, Vacuum 2. Stability Tests, October 1969, Internal Document, Group WX-3, LASL.

200°C TEMPERATURE VACUUM STABILITY TESTS														
Material	2	7	14 Total	21 gas	Tim 28 evolved	e of e 35 (cm <sup>3</sup> /	42	49	ys) 56 Average	63 of tw	70 o samp	77 les.)	84	91
АВН •	.9	2.4	4.7	7.9	12.9	19.5								
BTX	4	. 9	1.5	2.0	2.4	3.1	3.9	5.0	6.2	7.7	9.9	12.0	14.6	17.6
bis-HNAE DATB <sup>o</sup>	1 2 8	18.4	, -		<i>(</i> )									
DIPAM <sup>d</sup>	2.8 2.5	3.6 3.3	4.5 4.2	5.5	6.2	7.4	8.4	9.5	12.7	11.8	13.2	14.7	16.4	
DODECA	2.5	1.4	4.2	5.0 2.4	5.9 2.9	6.9 3.5	7.9	9.4	10.4	11.7	12.8	14.2	15.6	
DPBT•	.8	2.2	4.4	6.8	9.0	12.3	4.2	6.3	7.1	7.7	8.0	8.4	9.0	9.7
DPPM	.7	1.5	2.0	2.7	4.0	5.4	6.6	7.7	9.2	9.6	10.3	10.8	11.6	12.8
HNAB	.4	2.0	9.8	27.8	4.0	5.4	0.0		9.2	9.0	10.5	10.0	11.0	14.0
HNBP	. 6	ī.5	2.2	3.0	3.6	4.4	4.8	6.8	7.6	8.4	9.1	9.8	10.9	11.8
HNDS	2.4	15.0	-;-	5.00	5.0	<b>.</b>	4.0	•••	7.0	0.4	<b>J</b> .1	2.0	10.9	11.0
HNS	.4	.7	1.0	1.2	1.4	1.6	1.7	1.8	2.0	2.3	2.5	2.6	2.8	3.0
K H N D <sup>a</sup>	.7	1.3	3.7	8.2	18.0									
NONA	.4	.8	1.1	1.6	2.0	2.3	2.8	3.2	3.6	3.9	4.3	4.7	5.1	5.4
ONT	.9	1.3	1.4	1.5	1.6	1.7	1.9	1.9	2.0	2.1	2.3	2.4	2.5	2.6
PADP-11	15.0													
PATO	1.0	1.9	2.8	3.4	4.0	4.6	5.2			6.5	7.2	7.7	8.7	10.7
PENCO	.1	. 2	.3	.4	. 6	.6	.6	. 7		.9	1.0	1.1	1.2	1.4
PYX	.1	.1	. 2	. 2	2	3		. 4	.4	.4	.5	.6	.6	.7
TAT B <sup>j</sup> TAT B <sup>k</sup>	.3 1.0	,• <u>9</u>	$2.1 \\ 12.5$	4.1	7.2	11.1	15.8							
TNN	.3	4.7	.6	20.0	.9	1.0	1.2	1 1		1 (		1 0		
TPB	.1	.1	.2	.3	.3	.3	.3			1.6	1.7	$1.8 \\ 3.2$	2.0	2.2
TPM	:8	4.2	7.7	11.7	15.6	e	• •	• 4	• 4	.4	. 5	3.2	7.0	9.5
TPT	.2	.2	.4	.4		.4	.5	. 5	.6	. 7	.9	1.0	1.0	1.1
T-TACOT	.ī	.5	و	1.3	1.6	1.9	2.1	.5 2.5	3.Ŏ	3.5	3.9	4.3	4.8	5.3
Z-TACOT	.4	. 6	. 7	. 8	ī.ŏ	<b>4.1</b>	4.9	7.1	8.6	10.8	11.2	11.4	11.5	11.5
ABH te	rmina	ted af	ter 35	davs	_									
ABH te bis-HN	AB ab	orted	after	5 days	3.									
°DATB t	ermin	ated a	fter 83	2 days	в.									
d DIPAM	termi	nated	after	86 da	ys.									

	•	FABLE I		
200°C	TEMPERATURE	VACUUM	STABILITY	TESTS

DPBT terminated after 42 days. HNAB terminated after 18 days.

HNDS aborted after 5 days, evolved gas blew mercury into catch basin between 3 and 5 days. KHND terminated after 30 days.

PADP-I aborted at 2 days, evolved gas blew mercury into catch basin in less than 2 days. TATB terminated after 42 days.

TATE (recrystallized from DMSO) aborted at 20 days, DMSO odor strong on opening of tube. <sup>1</sup>TPM terminated after 27 days.

# TABLE II

## 175°C TEMPERATURE VACUUM STABILITY TESTS

Not ond ol	2	7	14	21	Time 28	35	xposu: 42	49	ys) 56	63	70	77	84	91
Material			Total	gas e	volved	(cm <sup>3</sup> /	<u>g at </u>	STP.	Average	of tw	<u>ro samp</u>	1es.)		
ABH	.4	.7	1.0	1.3	1.6	2.0	2.4	2.7	3.0	3.4	3.7	4.0	4.3	4.7
BTX	. 2	.3	.4	.4	.5	.6	.7	. 8		1.1	1.1	1.2	1.2	1.3
bis-HNAB		4.2	8.2	12.2	17.1									
DATB	1.4	1.8	2.1	2.3	2.5	2.7	2.8	2.9		3.2	3.4	3.6	3.7	3.8
DIPAM	.4	. 8	1.3	1.4	4.6	8.2	9.6	9.9	10.2	10.3	10.4	10.5	10.6	10.8
DODECA	. 5	.7	.8	.9	.9	.9	1.0	1.0		1.1	1.2	1.3	1.4	1.4
DPBT	. 9	1.8	2.5	3.2	4.0	4.8	5.6	6.4	7.1	8.1	9.0	9.8	10.8	11.9
DPPM	.7	1.3	1.8	2.1	2.3	2.5	2.7	2.8	3.0	3.1	3.2	3.3	3.3	3.4
нмхъ	3.1	31.2												
HNAB	.1	. 3	.6	. 8	.9	1.1	1.2	8.5	9.2	9.7	10.6	11.4	12.5	13.4
HNBP	.4	.6	. 8	1.1	1.2	1.4	1.5	1.7	1.8	2.0	2.2	2.4	2.6	2.8
HNDS	.3 .2	• 7	2.4	7.1	20.5									
HNS	• 2	.4	. 5	.6	.7	.8	.8	. 9		1.1	1.1	1.2	1.2	1.2
KHND	.1	. 4	.5	.7	.8	1.0	1.1	1.3		1.6	1.8	2.0	2.2	2.4
NONA	.1	.1	. 2	. 2	.3	.4	.5	. 6	.8	.9	1.0	1.1	1.2	1.3
ONT	.2 8.2	.6	.9	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	1.9	2.0	2.0
PADP-1ª		9.0	11.7	14.2	19.7									
PATO	.1	.4	.6	.7	.8	.9	1.1	1.2	1.3	1.4	1.4	1.4	1.4	1.5
RDX	4.2	15.1												
TATE	. 3	.4	.4	.5	.6	.7	. 8	. 9	1.0	1.1	1.2	1.4	1.5	1.6
TNT	8.0	45.0												
TPB	.1	.1	.1	.1	.1	.1	. 2	. 2	.2	. 2	. 2	. 2	.3	.3
TPM	1.5	2.0	2.3	2.6	2.9	3.3	3.7	4.1		4.9	5.2	5.5	5.9	6.7
T-TACOT	.1	• 3	• 5	.6	• 8	. 9	1.1	1.3	1.5	1.6	1.7	1.8	1.9	2.0
Z-TACOT	.4	. 5	.6	.7	. 7	.7	.7	.7	.8	.9	1.0	1.1	1.4	1.7

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<sup>a</sup>bis-HNAB terminated after 28 days.
<sup>b</sup>HMX aborted after 7 days, evolved gas blew mercury out of tube into catch basin.
<sup>d</sup>PADP-I started gassing immediately on heating, gases reacted with mercury to form white deposit. Test terminated after 27 days.
<sup>e</sup> RDX aborted after 6 days, evolved gas blew mercury out of tube into catch basin.
<sup>f</sup> TNT aborted after 8 days, evolved gas blew mercury out of tube into catch basin.

## TABLE III

150°C TEMPERATURE VACUUM STABILITY TESTS

	2	7	14	21	Time 28	35	42	re (day 49	ys) 56	63	70	77	84	91
Material			Total	gas e	volved	(cm3/g	<u>at</u>	STP.	Average		samp	les.)		
ABH bis-HNAB	.2	1.3	.4	<u>، 5</u>	. 6	.7	. 8		9	1.0	1.0	1.1	1.2	1.2
DATB	.6	$1.2 \\ 1.1$	$1.8 \\ 1.5$	2.5 1.7	2.9 1.7	3.6 1.7	4.2		5.3 1.9	6.0 1.9	6.7 2.0	7.4	8.1 2.0	8.8 2.1
DIPAM DPBT	. 2	.4	. 5	. 5	.6	.7	.7	. 8	. 8	. 9	.9	1.0	1.0	1.0
DPPM	.4 .3	. 7	.9 1.0	$1.0 \\ 1.2$	$1.3 \\ 1.4$	1.5 1.5	$1.7 \\ 1.5$	$1.9 \\ 1.6$	$2.1 \\ 1.7$	2.2 1.8	2.3 1.9	2.4 2.0	2.5 2.1	2.7
HMX≞ HNAB	1.2	8.9	32.0	.3	.3			. 5	.5					
HNBP	.1	. 3	. 3	.4	. 4	.4 .5	.4	.5	.6	.6 .6	.6 .7	:7	.7 .8	.8
HNDS KHND	.1	· 2 · 2	.2	.3	.3	• 3 • 5	.4	.5	.5	.6	.7	• 9	1.1	1.3
ONT	.1	. 2	. 2	. 3	.4	.4	.5	. 6	.6	. 7	. 8	.8	.9	.8 .9
PADP-1° PADP-1°	5.8	4.9	4.3	4.1	3.9	4.1	4.2	4.2	4.3	4.4	4.5	4.7	4.9	5.0
RDX <sup>d</sup>	3.2	.5	1.3	3.5	16.8									
TATB TNT⊖	.2	.3	.3	.4	.4	.4 18.1	.4	.4	.5	. 5	• 5	. 5	.6	.6
TPM	1.0	1.1	1.1	1.2	1.2	1.2	1.2	1.3	1.3	1.3	1.3	1.4	1.4	1.5

<sup>a</sup> HMX aborted after 13 days.
<sup>b</sup> PADP-I started gassing immediately on heating, gases reacted with mercury to form white deposit.

white deposit. • PADP-I sample was dried in a vacuum oven for 24 hours at 150°C at WX-2 before testing. This treatment seems to eliminate the excessive gassing noted on the first test. <sup>d</sup> RDX aborted after 28 days. • TNT aborted after 34 days.

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## TABLE IV

## POST TEST EXAMINATION OF SAMPLES AND MANOMETERS

			00°C			11	75°C					
Explosive	Subl <sup>1</sup>	Cond <sup>2</sup>	Odor <sup>3</sup>	Residue	Subl <sup>1</sup>	Cond <sup>2</sup>	Odor <sup>3</sup>	Residue	Subl	Cond <sup>2</sup>	Odor <sup>3</sup>	Residue
АВН	0	3,5	8	12	0	2	8	12	0	0	9	11
BTX	3	4,5	8	12	0	2	8	12	-	-	7	-
bis-HNAB	3	4	8	13	2	3	8	12	0	2	9	12
DATB	3	3	8	13	2	2	7	12	ž	2	7	11
DIPAM	õ	3,5	7	13	õ	2	8	12	õ	0	7	11
DODECA	õ	1.5	7	12	õ	2	8	11	-	-	-	-
DPBT	õ	3	8	12	õ	1	8	12	0	0	8	11
DPPM	2	3,5	8	12	2	2	6	11	õ	1	6	11
HMX	-	-	-	-	ō	4,5	8	12	2	4	8	12
HNAB	3	4	8	12	ō	3,5	8	12	ō	2	9	11
HNBP	0	ĩ	7	12	0	2,5	6	12	0	0	7	12
HNDS	0	2	8	13	2	2	8	12	0	2,5	8	12
HNS	3	ō	6	12	ō	ī	8	11	_		-	-
KHND	0	3.5	8	12	0	i	7	11	0	0	7	11
NONA	0	4	8	12	0	4	8	11	0	-	-	-
ONT	0	1	8	12	0	2	8	11	0	0	7	11
PADP-I	2	4,5	8	13	2	4,5	8	12	2	3,5	8	11
PADP-I (dried)	-	-	-	-	-	-	-	-	0	0	7	11
PATO	3	3	8	12	3	4	8	11	-	-	-	-
PENCO	0	2,5	8	11	-	-	-	-	-	-	-	-
PYX	0	2,5	6	11	-	-	-	-	-	-	-	-
RDX	-	-	-	-	0	4.5	8	12	0	4	8	12
TATB	3	3	8	13	0	2	8	12	0	1	7	11
TATB (DMSO												
crystallized)	3	3	10	13	-	-	-	-	-	-	-	-
TNN	0	1	7	12	-	-	-	-	-	-	-	-
TNT	-	-	-	-	2	4	8	13	2	4	8	12
TPB	4	3,5	8	12	2	2,5	8	11	-	-	-	-
TPM	3	3,5	8	12	0	2,5	7	12	0	0	9	11
TPT	0	2,5	7	11	-	-	-	-	-	-	-	-
T-TACOT	0	1	8	12	0	1	8	11	-	-	-	-
Z-TACOT	0	2,5	7	12	0	1	8	11	-	-	-	

<sup>1</sup>Sublimate: Detection of amount of sublimed sample above hot portion of sample bulb.

0 not detectable visually

1 slight, barely visible or detectable

2 some amount visible without having to examine closely

3 moderate amount visible spread evenly over area

4 heavy amount of condensate in large drops, very noticeable

<sup>a</sup>Condensate: Detection of reaction products condensed on cold member of manometer.

0 through 4 same order as for sublimate

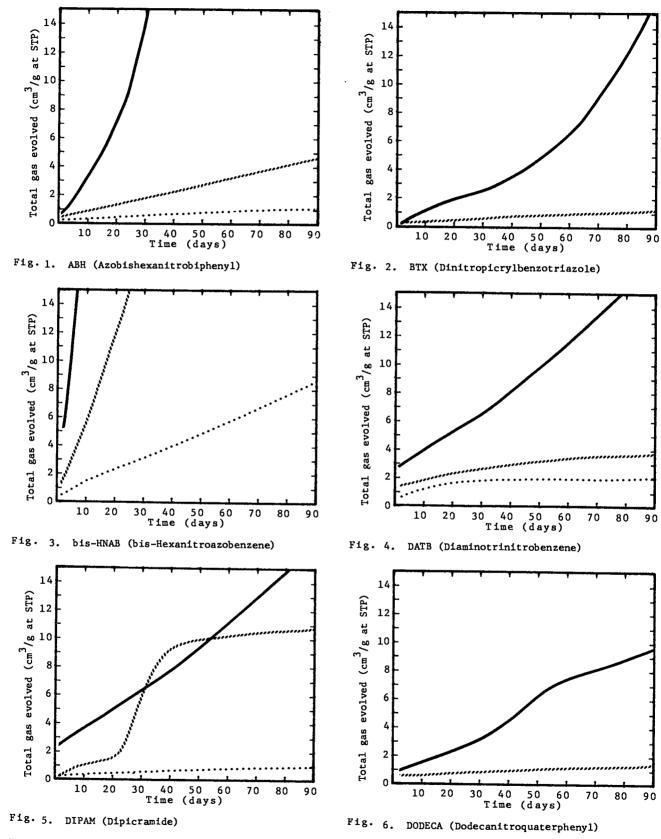
5 white deposit shows from reaction with gas at mercury interface of manometer

<sup>3</sup>Odor: On dismantling of bulb from manometer, odors were noticeable.

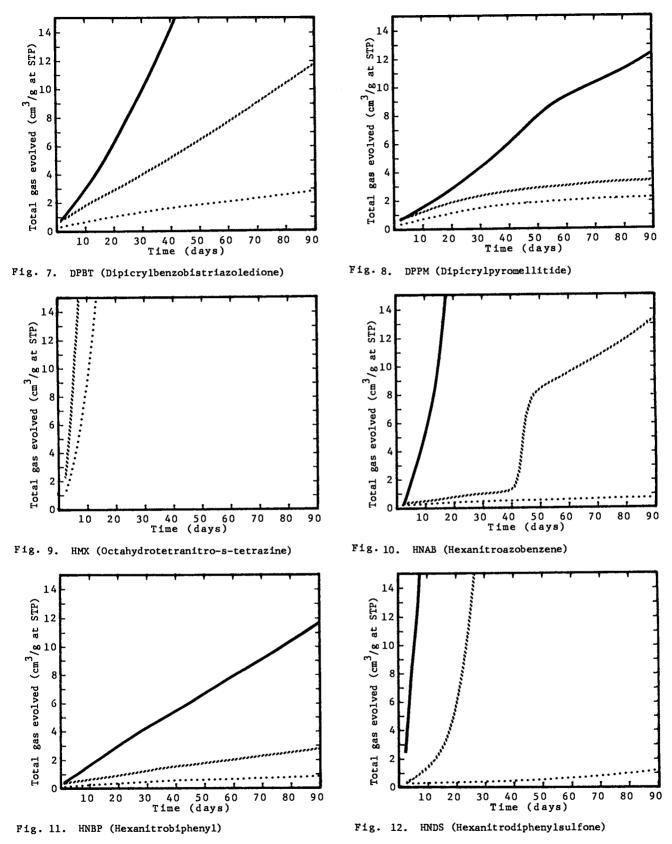
- 6 no odor was noticed
- 7 only slight odor of nitrogen oxides was noticed
- 8 strong odor of nitrogen oxides was noticed
- 9 odor more acetic acid than nitrogen oxides
- 10 odor of DMSO strong on opening

<sup>4</sup>Residue: Examination of residue for change in color or darkening compared to original.

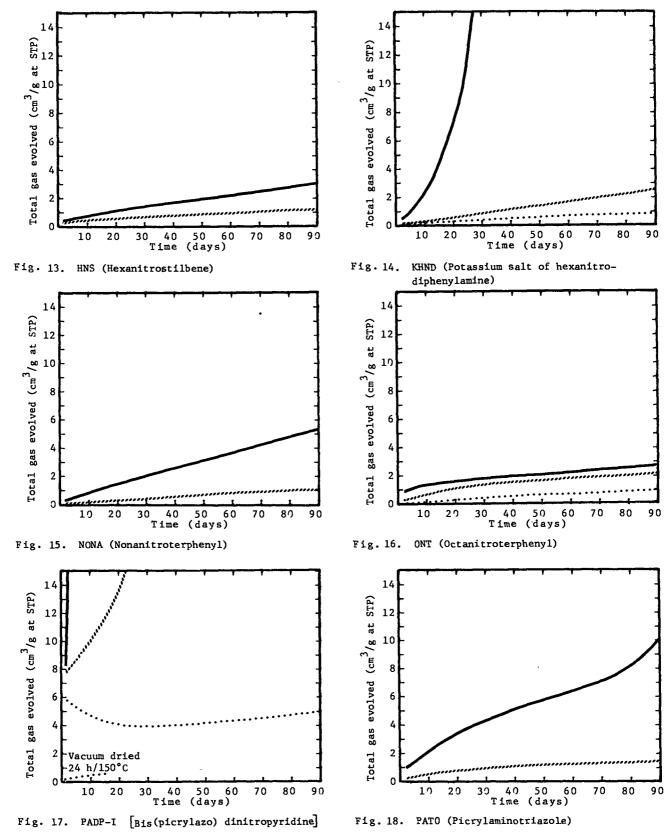
- 11 not much change from original
- 12 darker than original material
- 13 not recognizable as original material



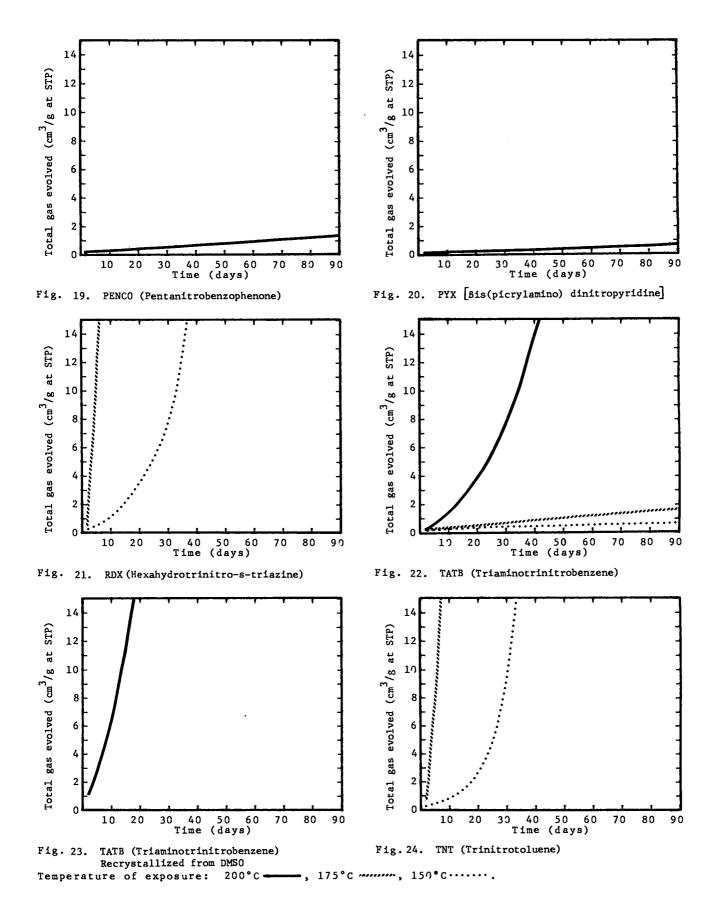
Temperature of exposure: 200°C ------, 175°C ......, 150°C ......

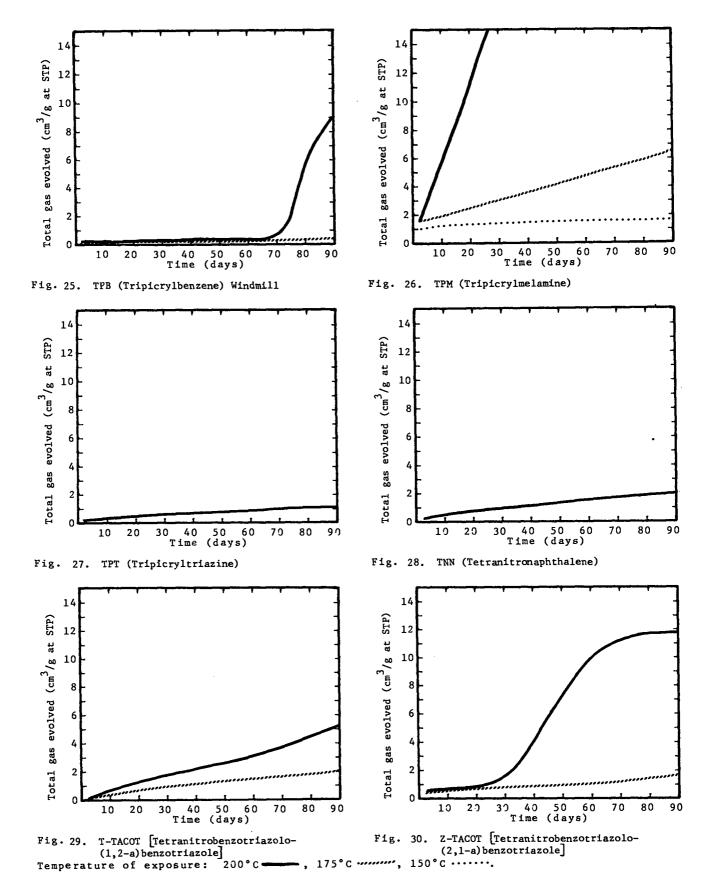


Temperature of exposure: 200°C ------, 175°C ......, 150°C .......



Temperature of exposure: 200°C ------, 175°C ------, 150°C ------.





50:151(35)

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