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A CHARACTERIZATION STUDY OF SEVERAL HEAT RESISTANT EXPLOSIVES

BY

E.E. Kilmer

4 October 1974

NAVAL ORDNANCE LABORATORY
WHITE OAK, SILVER SPRING, MARYLAND 20910

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**Key Words:**
- New Explosives
- Thermally Stable Explosives
- Explosive Properties

**Abstract:**
Three heat resistant explosives were chosen as candidates for study of their physical and chemical characteristics. As a part of this study, the chemical synthesis and detonation properties were investigated to determine their suitability in applications to small explosive components. These secondary explosive candidates were subjected to temperatures approaching 315°C (600°F). The best candidate was selected for further study.
NOLTR 74-177

4 October 1974

A Characterization Study of Several Heat Resistant Explosives

This is a report on "A Characterization Study of Several Heat Resistant Explosives" work being conducted for the Lyndon B. Johnson Spacecraft Center at Houston, Texas, under Task NOL-0841/NASA-T-8917E, which covered the period 1 July 1973 - 30 June 1974.

This work is being carried out to characterize several new thermally stable explosives and to determine their usefulness in explosive components for possible application in the Space Shuttle Program. The latest findings on the thermal and explosive properties of these new materials are discussed.

The author wishes to acknowledge the explosive synthesis study by Dr. Thomas Hall and Mr. Michael Sitzmann, vapor pressure measurements by Mr. David Covell, chemical analysis by Dr. Donald Kubose, electrostatic sensitivity results by Mr. Louis Montesi, and vacuum thermal stability results by Mr. Herbert T. Simmons, Sr., of the Naval Ordnance Laboratory. The identification of commercial materials implies no criticism or endorsement by the Naval Ordnance Laboratory.

ROBERT WILLIAMSON II
Captain, USN
Commander

I. KABIK

By direction
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<td>4</td>
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<td>5</td>
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<td>17</td>
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</table>
1.0 BACKGROUND

1.1 This is the Annual Report for Fiscal 1974 on the "Space Shuttle Explosives" task being conducted for NASA, Lyndon B. Johnson Space Center at Houston, Texas. This work was carried out for NASA during the fiscal year under task NOL-0841/NASA. The work in part was accomplished in order to complete Phase I of the NASA work statement and to accelerate Phase II. The purpose of the work is to cover the development of a secondary explosive suitable for use in spacecraft hardware which is subjected to extremely high temperature and vacuum conditions. The design goal criterion shall be no significant degradation of the explosive after 600°F/1-hour exposure. It is anticipated that the total program will be accomplished over several phases of work. During the initial Phase I of the program several candidate explosives will be chemically and physically characterized. This characterization shall include as a minimum:

a. Melting Point Determination
b. Vacuum Thermal Stability
c. Shock Sensitivity
d. Electrostatic Sensitivity
e. Detonation Velocity

1.2 During Phase II the following is to be accomplished:

a. Completion of the characterization work on a selected candidate explosive,
b. Establish the compatibility of the explosive with materials to be defined,
c. Study explosive synthesis to insure feasibility of production and technical adequacy of the explosive product.

2.0 PROCUREMENT OF HEAT RESISTANT EXPLOSIVE CANDIDATES

The Naval Ordnance Laboratory (NOL) has begun to implement the above work statement in the following ways:

2.1 The selection of prime candidates which could possibly meet the high temperature requirement was followed by the partial characterization of three high explosive materials. Two of these materials were synthesized at NOL and the other was procured from the Los Alamos Scientific Laboratory (LASL).
2.2 The synthesis of two thermally stable explosives TPT (2,4,6-tripicryl-s-triazine) Figure 1 and ONT (2,2',4,4',6,6',6'-Octanitro-m-terphenyl) Figure 2 was accomplished at the NOL. The latest finding of a thermally stable explosive of interest is attributed to LASL. This material is named PYX (2,6-bis (Picrylamino)-3,5-dinitropyridine) Figure 3. According to LASL, this explosive has properties comparable to TPT. NOL procured between one and two pounds of this explosive for study. NOL had NASA concurrence (T. Graves, EP-4) on this matter.

2.3 The physical and chemical properties to be determined are:

a. Melting point range,
b. Bulk density,
c. Heat of combustion (refs. 4, 5),
d. Heat of vaporization (ref. 6),
e. Vacuum thermal stability (ref. 7),
f. Compatibility with metals and organic chemicals.

g. Chemical analysis according to typical specification WS5003, i.e., percent of surface moisture and volatile materials, percent of water insolubles, percent acetone insolubles (ref. 8).

2.4 The detonation properties will be determined by:

a. Impact Sensitivity (shock sensitivity)
b. Small Scale Detonation Velocity (ref. 9)
c. Small Scale Gap Test Sensitivity (various densities; ref. 10)
d. Electrostatic Sensitivity
e. Non-Reactive Hugoniot (ref. 11)

The data collected above should provide information for judgement of the candidate to best satisfy the design criterion of "no significant degradation of the explosive" and provide information for establishing a procurement specification.
CANDIDATE

2,4,6 TRIPICRYL-\(\text{H}\)-TRIAZINE

TPT

SYNTHESIS: REF 1

FIG. 1 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF TPT
CANDIDATE

\[
\begin{align*}
\text{ONO}_2 & \quad \text{ONO}_2 \\
\text{NO}_2 & \quad \text{NO}_2 \\
\text{NO}_2 & \quad \text{NO}_2 \\
\end{align*}
\]

2,2'',4,4'',6,6'',6''-OCTANITRO-\text{-m-TERPHENYL}

ONT

SYNTHESIS: REF 2

FIG. 2 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF ONT
FIG. 3 CHEMICAL STRUCTURE AND PHOTOMICROGRAPH OF PYX
3.0 EXPLOSIVE SYNTHESIS STUDY

3.1 In an effort to accelerate the synthesis program, the manpower was concentrated toward the preparation of single lots of material which could be studied for chemical purity and handling characteristics. This, in part, means a study of each chemical synthesis to determine how realistic and practical it is to prepare a pound of explosive and apply these techniques to "scale-up" procedures.

3.2 A considerable amount of time was required to recrystallize ONT (octanitro-m-terphenyl) to insure that the material had very high purity. After many recrystallizations, the results from the vacuum thermal stability tests (Table 1) were higher than those of earlier preparations at NOL. A limited study was made to determine the cause. Gas chromatograph-Mass Spectrometer (GC-MS) studies indicated the presence of nitrobenzene (the crystallization solvent) in the amount of approximately 0.04%. This was due to occluded solvent which was not removed by drying at 150°C under vacuum because of the very high boiling point (210°C) of nitrobenzene. The larger the particle size, the greater the amount of occlusion. Previous samples were of uniformly smaller particle size because they were prepared in smaller quantities (smaller volumes of nitrobenzene) and were cooled more rapidly. It is very difficult to rapidly cool large volumes of nitrobenzene at temperatures of approximately 200°C with conventional laboratory equipment.

3.3 The sample of PYX, 2,6-bis (Picrylamino-3, 5-dinitropyridine), prepared by LASL showed poor vacuum thermal stability. After reviewing the results of Table 1, LASL was contacted to verify the published data on the material. We were informed that the "purity" of the sample was questionable because it had not been recrystallized and had been dried only at 70°C. In an effort to repurify the sample, some of it was recrystallized from 90% nitric acid and dried under vacuum for 16 hours at 150°C.

3.4 Vacuum Thermal Stability tests have been made on PYX which has been purified by recrystallization from 90% nitric acid. The results are shown in Table 1. TPT was also run at the same time for comparison. Under the conditions of this test, PYX began to show some decomposition at 300°C (572°F) and definite decomposition at 315°C (600°F). By comparison TPT is thermally stable at 315°C.

4.0 VAPOR PRESSURE MEASUREMENTS OF TPT, ONT and PYX

4.1 A series of measurements has been completed, using a modified version of the apparatus described in references 6 and 15. The objective of these measurements was to determine vapor pressure as a function of temperature for TPT, ONT and PYX, and from the vapor pressure data, to calculate heats of sublimation for these explosives. A secondary objective was to obtain at least qualitative data in regard to the physical and chemical stability of these materials when exposed to temperatures up to 300°C.
TABLE 1  VACUUM THERMAL STABILITY TEST RESULTS

(SUMMARY)

<table>
<thead>
<tr>
<th>EXPLOSIVE</th>
<th>TEMPERATURE °C</th>
<th>20 MINUTE SURGE (cc/g)</th>
<th>TWO HOUR PERIOD (cc/g/hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPT</td>
<td>280</td>
<td>0.29</td>
<td>0.10</td>
</tr>
<tr>
<td>(NOL ID 1802)</td>
<td>300</td>
<td>0.37</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>315</td>
<td>0.50</td>
<td>1.20</td>
</tr>
<tr>
<td>PYX</td>
<td>280</td>
<td>3.21</td>
<td>0.46*</td>
</tr>
<tr>
<td>(NOL ID 1817)</td>
<td>280</td>
<td>1.27</td>
<td>0.64**</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>1.10</td>
<td>2.10***</td>
</tr>
<tr>
<td></td>
<td>315</td>
<td>1.41</td>
<td>7.06***</td>
</tr>
<tr>
<td>OXT</td>
<td>280</td>
<td>0.28</td>
<td>0.63</td>
</tr>
<tr>
<td>(NOL ID 1803)</td>
<td>300</td>
<td>0.88</td>
<td>3.94</td>
</tr>
</tbody>
</table>

*Material as received from LASL.
**After drying 16 hrs @ 150°C.
***After recrystallization from 90% nitric acid and dried 16 hrs @ 150°C.
4.2 Samples were prepared by packing portions of the material to be tested into receptacles which had been machined into stainless steel sample trays. The receptacles were 0.5 in. in diameter by 3/32 in. deep. For the packing, a hydraulic ram was used at a pressure of 14,000 lbs per sq. in. After weighing the samples, they were placed in a glass vacuum manifold and heated by passing vapors from a boiling, refluxed, organic solvent through a jacket around the vacuum manifold. A given sample was heated for a period of time, $\Delta t$, usually several hours in duration, under vacuum and then removed from the manifold and re-weighed. Vacuum, during the heating process was maintained in the range $2 \times 10^{-7}$ torr to $2 \times 10^{-6}$ torr.

4.3 Vapor pressures were computed from the observed weight loss. The expression used was:

$$P(\text{torr}) = 17.14 \frac{G}{T} \frac{1}{M^{1/2}}$$

where $G$ is the weight loss in grams/cm$^2$/sec.; $T$ is the absolute temperature, and $M$ is the molecular weight. The molar heat of sublimation, $\Delta H_s$ (kCal/mole), was calculated from the following relationship:

$$\log P_2/P_1 = \frac{\Delta H_s}{4.576} \left( \frac{T_2 - T_1}{T_1 T_2} \right)$$

where $P_1$, $P_2$, and $T_1$, $T_2$, represent the coordinates of two points on the curve relating vapor pressure and temperature. Table 2 shows the solvents and the corresponding temperatures used in this work. Benzyl Benzoate decomposed at its boiling point and could not be used for the experiment. A comparable material was not available.

4.4 Table 3 summarizes the data obtained by these measurements. All of the TPT samples withstood the heating very well, i.e., there was no marked change in the physical appearance of the samples as a result of the heating. All of the ONT samples, however, were broken up and granular in consistency; typically the upper layer of the sample was dislodged from the rest of the sample. No material appeared to be lost in the manifold by means other than evaporation. The break up of the sample caused large errors in the estimate of surface area shown in Table 2. The estimate of vapor pressure shown in Table 2 does not take into account this change in surface area. All of the PYX samples were badly broken up and material was physically strewn about the interior of the manifold. Under these conditions, no applicable determination of weight loss was possible, and no estimate of vapor pressure could be made. From direct observation, it appeared that this break-up phenomenon occurred during the first 15-20 minutes of heating.

4.5 TPT, PYX, and ONT after being subjected to the high temperatures were analyzed mass spectrometrically, and the maximum decomposition that took place was estimated to be less than 2.5%. This figure quickly dropped to insignificance as lower temperatures were used.
<table>
<thead>
<tr>
<th>SOLVENT</th>
<th>BOILING POINT (°C)</th>
<th>SAMPLE TRAY TEMP (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>METHYL ACETATE</td>
<td>57.4</td>
<td>56.9</td>
</tr>
<tr>
<td>WATER</td>
<td>100.0</td>
<td>98.9</td>
</tr>
<tr>
<td>ANISOLE</td>
<td>152.7</td>
<td>149.7</td>
</tr>
<tr>
<td>NITROBENZENE</td>
<td>210.3</td>
<td>206.9</td>
</tr>
<tr>
<td>NONANOIC ACID</td>
<td>253.0</td>
<td>247.6</td>
</tr>
<tr>
<td>HEXADECANE</td>
<td>287.5</td>
<td>277.8</td>
</tr>
<tr>
<td>BENZYL BENZOATE*</td>
<td>323.0</td>
<td>-</td>
</tr>
</tbody>
</table>

*Not used because the solvent tends to decompose badly at the boiling temperature.
TABLE 3  SUMMARY OF DATA OBTAINED AND VAPOR PRESSURES CALCULATED IN THE VAPOR PRESSURE STUDY

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>MOL. WT.</th>
<th>TEMP. (°C)</th>
<th>Δt (sec)</th>
<th>SURFACE AREA (cm²)</th>
<th>WT. LOSS (gm)</th>
<th>VAPOR PRESSURE (torr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPT</td>
<td>714</td>
<td>277.8</td>
<td>55,500</td>
<td>6.3</td>
<td>0.1268</td>
<td>4.77(10⁻⁶)</td>
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<tr>
<td></td>
<td></td>
<td>247.6</td>
<td>157,300</td>
<td>5.1</td>
<td>0.0227</td>
<td>4.26(10⁻⁷)</td>
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<tr>
<td></td>
<td></td>
<td>247.6</td>
<td>225,100</td>
<td>5.1</td>
<td>0.0303</td>
<td>3.70(10⁻⁷)</td>
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<td>206.9</td>
<td>239,400</td>
<td>5.1</td>
<td>0.0024</td>
<td>2.73(10⁻⁸)</td>
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<td>206.9</td>
<td>768,900</td>
<td>5.1</td>
<td>0.0041</td>
<td>1.48(10⁻⁸)</td>
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<td></td>
<td></td>
<td>98.9</td>
<td>149,040</td>
<td>5.1</td>
<td></td>
<td>INDETERMINATE</td>
</tr>
<tr>
<td>ONT</td>
<td>590</td>
<td>277.8</td>
<td>38,400</td>
<td>5.1</td>
<td>0.1760</td>
<td>1.50(10⁻⁵)</td>
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<tr>
<td></td>
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<td>247.6</td>
<td>56,220</td>
<td>6.3</td>
<td>0.0189</td>
<td>8.57(10⁻⁷)</td>
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<td>247.6</td>
<td>55,200</td>
<td>5.1</td>
<td>0.0185</td>
<td>1.07(10⁻⁶)</td>
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<td>247.6</td>
<td>190,920</td>
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<td>0.0341</td>
<td>5.67(10⁻⁷)</td>
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<td></td>
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<td>149.7</td>
<td>492,900</td>
<td>5.1</td>
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<tr>
<td>PYX</td>
<td>621</td>
<td>277.8</td>
<td>56,100</td>
<td>5.1</td>
<td>*</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>206.9</td>
<td>56,400</td>
<td>5.1</td>
<td>*</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>149.7</td>
<td>63,720</td>
<td>5.1</td>
<td>*</td>
<td></td>
</tr>
</tbody>
</table>

*Samples broken up and scattered around within the manifold.
4.6 Figures 4 and 5 show the vapor pressure versus temperature function for TPT and ONT respectively. The expected logarithmic function is clearly apparent in the TPT data, and from these data, the molar heat of sublimation was calculated to be 43.9 kCal/mole. The ONT data do not show the proper form, and this fact is believed to be due to sample break-up phenomenon described above which makes an estimate of surface area impossible. Since no meaningful surface area data were available, no attempt was made to calculate the heat of sublimation for ONT.

5.0 CHEMICAL ANALYSIS OF EXPLOSIVES AFTER PROLONGED HEAT TREATMENT UNDER VACUUM

5.1 In an attempt to measure the heats of sublimation of TPT, ONT and PYX, the explosives were heated at elevated temperatures under high vacuum (10^-6 to 10^-7 mm) in NOL's vapor pressure apparatus. To ascertain whether or not decomposition had occurred, chemical analysis of the explosive residue for each explosive was made.

5.2 Analyses of the residues were made by the ethylenediamine/dimethylsulfoxide spectrophotometric technique and compared to the starting materials as standards. In addition, the spectral envelope for each compound near the maximum absorption was measured and also compared to the standard.

5.3 The results obtained are as follows in Table 4. In all cases the spectral envelopes were the same as the standards.

6.0 DETONATION PROPERTIES

6.1 Detonation velocity measurement is an important tool in determining how an explosive will perform. For this task the explosive is being tested in column diameters of 0.1 and 0.2 in steel bodies 10 in O.D. The velocity is measured over a one inch length. The results of small scale detonation velocity measurements on PYX in the 0.1 column diameter are shown in Figure 6. Velocity measurements were made on ONT in 0.1 and 0.2 column diameters: on TPT in 0.1, 0.2, and 0.3 column diameters. The results are shown in Figures 7 and 8. All of these explosives appear to be performing as expected. The velocities are of the same magnitude as other heat-resistant explosives such as HNS. They are lower than RDX, for example, but this is to be expected since the detonation velocity and sensitivity are compromised for thermal resistance. Since these explosives are detonating in the 0.1 diameter column, it is conceivable that they will perform in detonating cord with a comparable diameter and very possibly at smaller diameters.

6.2 The shock sensitivity of TPT has been determined at various densities by the small scale gap test method. This test is an arbitrary configuration to study the shock sensitivity of small confined charges and thus the transfer of detonation between small-diameter confined charges. The results of the tests are shown in Table 5 (SSGT results are shown for ONT and PYX in Tables 6 and 7). By way of comparison, TPT has about the same shock sensitivity as HNS.
\[ \Delta H_s (\text{MOLAR}) = 43.9 \text{ kCal/mole} \]
\[ \Delta H_s (\text{SPECIFIC}) = 61.5 \text{ Cal/gm} \]

FIG. 4 VAPOUR PRESSURE OF TPT
FIG. 5 VAPOR PRESSURE OF ONT
### Table 4: Analysis of Explosives After Heat Treatment Under Vacuum

<table>
<thead>
<tr>
<th>Explosive</th>
<th>Temperature, °C</th>
<th>Time, Hours</th>
<th>% Explosive in Residue</th>
</tr>
</thead>
<tbody>
<tr>
<td>TPT</td>
<td>278</td>
<td>16</td>
<td>97.1</td>
</tr>
<tr>
<td>ONT</td>
<td>278</td>
<td>16</td>
<td>96.7</td>
</tr>
<tr>
<td>PYX</td>
<td>278</td>
<td>16</td>
<td>96.8</td>
</tr>
<tr>
<td>TPT</td>
<td>250</td>
<td>44</td>
<td>99.3</td>
</tr>
<tr>
<td>ONT</td>
<td>250</td>
<td>44</td>
<td>98.7</td>
</tr>
</tbody>
</table>
FIG. 6 THE SMALL SCALE DETONATION VELOCITY OF PYX AS A FUNCTION OF DENSITY AND COLUMN DIAMETER.
FIG. 7 THE SMALL SCALE DETONATION VELOCITY OF ONT AS A FUNCTION OF DENSITY AND COLUMN DIAMETER
FIG. 8  THE SMALL SCALE DETONATION VELOCITY OF TPT AS A FUNCTION OF DENSITY AND COLUMN DIAMETER.
EXPLOSIVE | TPT | L.O. | 1417
IMD | IMD | IMD | IMD

<table>
<thead>
<tr>
<th>LOADING PRESSURE (KPSI)</th>
<th>DENSITY (GM/CM3)</th>
<th>% IMD</th>
<th>SENSITIVITY (DBG)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AVG.</td>
<td>S</td>
<td>AVG.</td>
<td>S</td>
</tr>
<tr>
<td>4</td>
<td>1.193</td>
<td>0.0557</td>
<td>4,800</td>
</tr>
<tr>
<td>8</td>
<td>1.242</td>
<td>0.0214</td>
<td>4,753</td>
</tr>
<tr>
<td>16</td>
<td>1.325</td>
<td>0.0496</td>
<td>5059</td>
</tr>
<tr>
<td>32</td>
<td>1.444</td>
<td>0.0268</td>
<td>5,423</td>
</tr>
<tr>
<td>64</td>
<td>1.658</td>
<td>0.0111</td>
<td>7,217</td>
</tr>
</tbody>
</table>

SENSITIVITY PARAMETERS ARE GIVEN IN REFERENCE 14

TABLE 5 SMALL SCALE GAP TEST (SSGT) DATA FOR TPT
(2, 4, 6 - TRIPICRYL 1,5 - TRIAZINE)
<table>
<thead>
<tr>
<th>EXPLOSIVE</th>
<th>ONT</th>
<th>L.O. NO.</th>
<th>1416</th>
</tr>
</thead>
<tbody>
<tr>
<td>TMD</td>
<td></td>
<td>I.D. NO.</td>
<td>1803</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LOADING PRESSURE (KPSI)</th>
<th>DENSITY (GM/CM³)</th>
<th>% TMD</th>
<th>SENSITIVITY (DBG)¹</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AVG.</td>
<td>s</td>
<td>AVG.</td>
</tr>
<tr>
<td>4</td>
<td>1.396</td>
<td>0.0113</td>
<td>4.567</td>
</tr>
<tr>
<td>8</td>
<td>1.487</td>
<td>0.0082</td>
<td>4.752</td>
</tr>
<tr>
<td>16</td>
<td>1.595</td>
<td>0.0068</td>
<td>5.213</td>
</tr>
<tr>
<td>32</td>
<td>1.6775</td>
<td>0.0045</td>
<td>5.950</td>
</tr>
<tr>
<td>64</td>
<td>1.7688</td>
<td>0.0038</td>
<td>6.936</td>
</tr>
</tbody>
</table>

¹ SENSITIVITY PARAMETERS ARE GIVEN IN REFERENCE 14

---

**TABLE 6**

SMALL SCALE GAP TEST (SSGT) DATA FOR ONT

(2, 2', 4, 4', 4'', 6, 6', 6'' - OCTANITRO - m - TERPHENYL)
### Table 7: Small Scale Gap Test (SSGT) Data for PYX (2, 6 - Bis (Picrylamino) - 3, 5 - Dinitropyridine)

<table>
<thead>
<tr>
<th>LOADING PRESSURE (KPSI)</th>
<th>DENSITY (G/M² CM²) AVG.</th>
<th>% TMD</th>
<th>SENSITIVITY (DBG)</th>
<th>AVG.</th>
<th>g</th>
<th>f_m</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1.0788</td>
<td>0.0336</td>
<td>4.758</td>
<td>0.0502</td>
<td>0.0312</td>
<td>21</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1.2298</td>
<td>0.0119</td>
<td>5.122</td>
<td>0.0251</td>
<td>0.0190</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>1.3691</td>
<td>0.0140</td>
<td>5.410</td>
<td>0.0567</td>
<td>0.0338</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>32</td>
<td>1.5154</td>
<td>0.0082</td>
<td>5.734</td>
<td>0.0498</td>
<td>0.0283</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>64</td>
<td>1.6754</td>
<td>0.0042</td>
<td>6.599</td>
<td>0.0268</td>
<td>0.0208</td>
<td>20</td>
<td></td>
</tr>
</tbody>
</table>

1 Sensitivity parameters are given in Reference 14.
The results of impact tests are often used as a guide to the handling sensitivity of explosives. The results of tests on TPT and PYX are shown in Table 8 along with the values for TNT and tetryl. The tests were made on standard Type 12 tools, sandpaper and 2.5 Kg weight. As can be seen, the heat resistant explosives are somewhat less sensitive than the common booster explosive tetryl but more sensitive than main charge explosives like TNT.

7.0 EXPLOSIVE/METAL AND EXPLOSIVE/ORGANIC MATERIAL COMPATIBILITY TESTS

7.1 The thermal stability of explosive materials at elevated temperatures is greatly affected by the materials in contact with them. Rosen and Simmons have pointed out incompatibilities of high explosives with lead azide when heated to an elevated temperature range of 200-260°C. The decomposition was evidenced by evolution of gas and the formation of a black insoluble product which did not melt at 475°C. The reaction was interpreted as an oxidation of the azide ion by the nitro groups and must be considered as a limitation on the use of azides in intimate contact with typical organic explosives at elevated temperatures.

7.2 Since the explosives being considered will be used at temperatures exceeding 260°C, it is imperative to consider the materials that contact them at various temperatures to ascertain whether a compatibility problem would exist.

7.3 TPT, ONT, and PYX are being considered for first application in detonating cords, i.e., mild detonating cords and flexible linear shaped charges. The current materials used for the sheathing of these cords are silver, aluminum, and lead/antimony alloy. In view of this, explosive/metal mixtures 50/50 by weight were prepared and submitted to the NOL vacuum thermal stability/compatibility test. Results are shown in Table 9.

7.4 The approach used for the selection of the explosive/organic material contact was to reduce the number of combinations for test by considering possible applications. If the explosive was to be stored in a non-metallic container at temperatures approaching 600°F, then most plastics would be eliminated. Sealing materials such as epoxy resins would be incompatible at elevated temperatures (particularly those which are amine cured).

7.5 A high temperature polymer pioneered at the NOL is being evaluated in an effort to improve the performance of reinforced plastic heat shields for advanced reentry vehicles. This cast polymer is polyphenylquinoxaline (PPQ). It has a potential application as a potting compound and may be used in impact extrusions to fabricate containers. The potential of PPQ lies in its high thermal stability (800°F compared to 400°F for phenolics) and its increased toughness over brittle phenolics.
TABLE 8  IMPACT SENSITIVITY TEST RESULTS

<table>
<thead>
<tr>
<th>EXPLOSIVE</th>
<th>50% HEIGHT (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ONT (NOL X-559) M. P. ABOVE 400°C</td>
<td>64</td>
</tr>
<tr>
<td>TPT (NOL ID 1802) M. P. 352°C</td>
<td>74</td>
</tr>
<tr>
<td>PYX (NOL ID 1817) M. P. 360°C</td>
<td>94</td>
</tr>
<tr>
<td>Tetryl**</td>
<td>40</td>
</tr>
<tr>
<td>TNT**</td>
<td>160</td>
</tr>
</tbody>
</table>

*Type 12 tools, sandpaper, 2.5 Kg wt.

**Established in earlier test runs.
7.6 The results of compatibility tests using a TPT/PPQ 90/10 mixture of powders are encouraging between 300°C and 315°C. The test results for the explosive/polymer mixtures are very good as shown in Table 9.

7.7 This study will be expanded to cover various stages of polymerization of the PPQ during which times excess solvent materials such as meta-cresol will be present and will be in contact with the explosive. This might be considered to be the environment for the explosive during a potting application.

8.0 ELECTROSTATIC SPARK SENSITIVITY OF EXPLOSIVE CANDIDATES

8.1 The electrostatic spark sensitivity test is one of the tests used to characterize and order the initiation sensitivity of explosives. Individuals and structural materials will accumulate static charges which when discharged might initiate sensitive explosives. The chance initiation by electrostatic spark discharge is therefore a real hazard in the handling and use of explosives.

8.2 TPT, ONT, and P were tested by two methods. The first method was in accordance with the procedures described in reference 13. The results are given in Table 11. The second method involved the use of the Wyatt apparatus. Four capacitors (1.0, 0.1, 0.01, and 0.001 mfd) were used with metal/metal (m/m) and metal/rubber (m/r) electrodes and test voltages of 7500, 5500, 4000, and 250 volts. The maximum no-fire test level was determined using a maximum of 20 trials at any one of the above capacitor-voltage test level. The criterion used for ignition was any evidence of smoke, flame, sparks, and/or burning of the explosive sample. The results of the test (Table 11) are indicative of explosives which are not sensitive to electrostatic spark discharge.

8.3 For comparison, a table of results from previous tests on primary and more sensitive high explosives is shown also in Table 11.

9.0 DETONATING CORDS

9.1 The first application of heat resistant explosives to be considered will be in small detonating cords. Detonation velocity results show that TPT, PYX, and ONT will perform in small diameters (0.2 explosive column) and hopefully will perform in low core load detonating cords. All three of the candidate explosives have been fabricated into 2-3 gr/ft and 10-15 gr/ft. MDC using silver and aluminum sheaths. These cords will be subjected to elevated temperatures in subsequent tests.
<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>STAB COMPO 20 MIN GAS SURGE (cc/6)</th>
<th>2 hrs mI/g/hr</th>
<th>2 hrs mI/g/hr</th>
</tr>
</thead>
<tbody>
<tr>
<td>ONT (P-513-12-2) 1D 185G</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PYX (ASR-2D) UNDER VACUUM 150C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TFF ID 1802</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TFF/Pyrex (51°C)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TFF/Al (51°C)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TFF/Al + Sb 94/6 #55880</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ONT/Al + Sb</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ONT/Al + Sb STAINLESS STEEL</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PYX/Recy/Al Sb STAINLESS STEEL</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

TABLE 9: EXPLOSIVE PROPERTY MEASUREMENTS (VACUUM THERMAL STABILITY AND COMPATIBILITY TEST)
TABLE 10  ELECTROSTATIC SENSITIVITY TEST RESULTS  
(AS SPECIFIED BY NAVORD OD 44811) 

TEST CONDITIONS:  
POTENTIAL - 10K VOLTS  
CAPACITANCE - 0.01 MFD 

PASSED*  |  PYX  | ID 1817  
         |  TPT  | ID 1802  
         |  ONT  | ID 1803  

* OBSERVATION OF 20 CONSECUTIVE SHOTS WITH NO SMOKE,  
BURNING, AND/OR EXPLOSION.
### TABLE 11  ELECTROSTATIC SPARK SENSITIVITY TEST RESULTS

<table>
<thead>
<tr>
<th>CAP. (mfd)</th>
<th>HNS-TEFLON 7C 90/10 (ID 1494)</th>
<th>PYX (ID 1817)</th>
<th>TPT (ID 1802)</th>
<th>ONT (ID 1803)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>m/m</td>
<td>m/r</td>
<td>m/m</td>
<td>m/r</td>
</tr>
<tr>
<td>1.0</td>
<td>2500</td>
<td>-</td>
<td>2000</td>
<td>-</td>
</tr>
<tr>
<td>0.1</td>
<td>4000</td>
<td>4000</td>
<td>3000</td>
<td>2500</td>
</tr>
<tr>
<td>0.01</td>
<td>5500</td>
<td>4500</td>
<td>6000</td>
<td>4000</td>
</tr>
<tr>
<td>0.001</td>
<td>7500*</td>
<td>7500*</td>
<td>7500*</td>
<td>7500*</td>
</tr>
</tbody>
</table>

NOTE:

1. Unless otherwise specified, values given are maximum no fire values for Capacitance/Voltage level given (20 shot data)

2. Maximum test voltage, no fires observed at this level.
10.0 CONCLUSIONS AND RECOMMENDATIONS

10.1 Considering the synthesis of both ONT and PYX, the results of the vacuum thermal stability tests are indicative of occluded solvents in the crystals and would result in poor performance of these materials at 300°F and above. TPT should be stable for 2 hours at 315°C, and longer.

10.2 Vapor pressure measurement was attempted on TPT, ONT, and PYX. PYX began to break up in the equipment, as did ONT, resulting in indeterminate values of surface area. At the limit of the test, (with temperature and pressure) TPT performed the best of the three candidates and would be expected to show little decomposition at 315°C.

10.3 TPT, ONT, and PYX perform satisfactorily in small column diameters of 0.02 and 0.01. These materials should perform well in small detonating cords of 3 - 15 gr/ft.

10.4 The results of the small scale gap test are indicative of relatively insensitive explosive performance and show that these materials are in the booster category for shock sensitivity. They have about the same sensitivity as Hexanitrostilbene (HNS).

10.5 All of these materials are safe to handle from the viewpoint of electrostatic sensitivity. They pass the Navy OD 44811 tests for acceptability in fuze applications and show no indication of burning in the Wyatt apparatus.

10.6 Compatibility at elevated temperatures is always of concern, but TPT, ONT, and PYX are compatible with silver, aluminum, lead alloy, and stainless steel at temperatures comparable to normal decomposition of the individual explosive. TPT is also compatible with a high temperature polymer, PFQ.

10.7 It can be concluded from the results of tests to date, that TPT is the most likely of the three explosive candidates to survive the high temperature requirement goal of 315°C (= 600°F) and perform satisfactorily after exposure for 2 hours.

10.8 However, it is recommended that since the expected application will be in detonating cords, the screening of all three candidates continue. Detonating cords manufactured with these materials should be exposed to elevated temperatures to determine their thermal limitations of performance.
11.0 REFERENCES


8. WS5003, Purchase Description, HNS Explosive, Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland


10. Ayres, J. N., "Standardization of the Small Scale Gap Test Used to Measure the Sensitivity of Explosives," NAVWEPS Rpt 7342, 16 Jan 1961


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