ANALYSIS METHODS FOR EXPLOSIVE MATERIALS - I. POLYNITRO COMPOUNDS

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A high pressure liquid chromatography method was developed for rapid and quantitative chemical analysis of the following thermally stable explosive materials: DATB, DINA, DIPAM, DNT, DODECA, HNAB, HNBiB, HMX, HNBP, HNS, NONA, ONT, PA, PETN, PYX, RDX, TATB, TETRYL, TNA, TNB, TNN, TNS, TNT, and TPT (see glossary). Additional chemical characterization of the above materials was carried out by Nuclear Magnetic Resonance Spectroscopy and by Thin Layer Chromatography. Dimethylsulfoxide was used as the common solvent for all the compounds in this investigation.
FOREWORD

This report describes quantitative analytical techniques that can be used for several thermally stable explosives (e.g. TATB, HNS, ONT, NONA, DODECA, TNN, PYX, TPT, etc.) and related compounds. This work is currently being sponsored by the Lyndon B. Johnson Manned Spacecraft Center, Task NASAR12RB, and the Strategic Systems Projects Office, Task BO0035B001, R12GC. The identification of vendors and/or products implies neither endorsement nor criticism by the Naval Surface Weapons Center.

J. F. PROCTOR
By direction
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INTRODUCTION

The importance of heat resistant and insensitive high explosive compounds is increasing, since new weapons warheads are being sought that can withstand a higher degree of aerodynamic heating and that have a lower vulnerability to accidental initiation. A series of high temperature resistant explosives, most of which were first prepared or evaluated at NSWC, are shown in the glossary. Typical examples are: TATB, PYX, ONT, DATB, HNS, NONA, DODECA, AND TPT. Relatively few of the presently used common explosive materials can withstand temperatures above 200°C without melting and/or decomposition, whereas the compounds of this investigation are stable and can be used in the temperature range of 230°C-350°C.

In the past, there has not been any single chemical analysis method for all of these relatively insoluble heat-resistant explosives. However, this report describes a practical high performance liquid chromatographic (HPLC) analysis procedure that provides good accuracy and reproducibility. Additional information is provided on the chemical characterization of these explosive compounds by nuclear magnetic resonance spectroscopy (NMR) and by thin layer chromatography (TLC). The melting points of all of the materials are also reported.

The HPLC method described in this report uses the Waters Associates Radial Compression Separation System (RCSS). The RCSS consists of two components: a radial-Pak cartridge containing reverse phase C-18 column packing and a Model RCM-100 module that compresses the Radial-Pak cartridge. Using this system, a quantitative assay (±2%) is achieved for all of the twenty-four explosive compounds investigated.

Previously reported assay methods for the insensitive high explosive TATB include spectrophotometric determinations using ethylenediamine (EDA)¹, tetraethylammoniumhydroxide (TEAH)²⁻³,

dimethylsulfoxide (DMSO)\(^4\), and sulfuric acid\(^5\), as well as a liquid chromatography-spectrophotometric method\(^6\). Spectrophotometric methods of analysis\(^1,7\) have also been reported for HNS, DODECA, NONA, ONT, HNBP, TNN, TNB, HNBiB, TPT, PYX, and DNT as well as HPLC procedures\(^8-14\) for HNS, HNBiB, TNT, TNB, RDX, HMX, PA, TNA, DNT and TATB.

\(^4\) Selig, W., "Photometric Determination of 1,3,5-Triamino-2,4,6-Trinitrobenzene (TATB) in Dimethylsulfoxide (DMSO)," Lawrence Livermore Laboratory Report, UCID-17542, July 1977.


\(^12\) Sanford, Jr., T. B., "Determination of Tetryl and 2,3-,2,4-,2,5-,2,6-,3,4-, and 3,5-Dinitrotoluene Using High Performance Liquid Chromatography," Final Report, from Battelle Columbus Laboratories, Columbus, Ohio, submitted to the U.S. Army Research and Development Command, Washington, D.C., Contract No. DAMD-17-74-C-4123, 31 Jan 1977.


EXPERIMENTAL

MATERIALS AND SOLUTIONS. Most of the explosive compounds used in this investigation are not commercially available and were therefore synthesized in the Synthesis and Formulations Branch. The exceptions were PYX and DIPAM which were obtained from Los Alamos National Laboratory (LANL) and from Northrop Carolina Co., Inc. respectively.

The solubility of the less soluble compounds such as TATB, HNS, DODECA, NONA, PYX, TPT, and HMX was quantitatively determined in methanol, dimethylsulfoxide, dimethylformamide, and n-methylpyrrolidone. Although the dimethylformamide and n-methylpyrrolidone proved to have a greater solvent capacity for several of the explosive materials (TATB, HMX, and TPT), all of the compounds had sufficient solubility in DMSO at ambient temperature so that HPLC, NMR, and TLC data could be accurately obtained with this solvent. An HPLC trace of several neat DMSO samples indicated the presence of two small impurity peaks with a retention time of approximately 3.0-3.2 minutes (methanol: water, 70:30) which could have interfered with the peak height calculations of several of the explosives at the low concentration limits. Therefore all the DMSO used in this study was passed through a column of activated charcoal which removed over 95% of the impurities.

CALCULATIONS. The concentration of each explosive material was initially determined by peak area as well as peak height. Using the RCSS unit, the percentage accuracy of the area method was found to be equivalent to that of the peak height method, therefore for simplicity reasons, the latter method was used for all the analyses.

HPLC CONDITIONS. A high-performance liquid chromatograph (Waters Associates Model ALC 202) equipped with a 254 nm wavelength detector, a solvent delivery system (Model 6000), and a U6K high pressure loop injector was used with a Model RCM-100 module containing a reverse-phase C-18 Radial-Pak cartridge. Sample solutions were eluted isocratically at ambient temperature. Column flow was 2.0 ml/minute, with a mobile phase consisting of the following mixtures of HPLC grade methanol and distilled water: 40:60 (v/v), 50:50 (v/v), and 70:30 (v/v). The solvent mixtures were not degassed prior to use in the HPLC and sample injections of 2 to 10 microliters were used.

NMR CONDITIONS. Proton NMR spectra were obtained on a Varian XL-200 spectrometer. The chemical shift values (δ) were determined relative to the reference compound tetramethylsilane (TMS). The NMR solvent used was dimethylsulfoxide-d6 (99.5 atom % D), since it proved to be the best general solvent for all twenty-four compounds investigated.

TLC CONDITIONS. Thin layer chromatographic analyses of all the compounds reported used benzene as the developing solvent. In several cases where the samples did not chromatograph (e.g. HMX, PETN, TATB, TNN) methanol was also tried as the developing solvent.

The adsorbent used was Merck Silica Gel HF-254 coated on glass plates. A short wave UV lamp ($2537\text{A}$) was used for spot visualization.

RESULTS AND DISCUSSION

For the quantitative analysis of the explosive compounds by HPLC, DMSO solutions were used. Figure 1 shows the separation of a fourteen component synthetic mixture. Ten of the materials RDX, TATB, TNB, DATB, TETRYL, TTN, TNT, DNT, HNS, and DIPAM are completely resolved, two components HMX and PYX are partially resolved, and only TPT and HNBiB remain unresolved. Another HPLC chromatogram of a four component synthetic mixture containing three of the impurities$^9$-$^{11}$, $^{16}$ (TNB, TNT, HNBiB) found in several production grade samples of HNS-I is shown in Figure 2. The average retention time (minutes), average response factor (mm/mg), solution concentration (Molar), and approximate limit of detection (micrograms/ml) for all the explosive compounds studied are given in Tables 1, 2 and 3. From the peak height responses reported in Tables 1, 2 and 3, the detection limit for all the materials was calculated to be 15 mm on scale 0.005 absorbance units full scale ($3\times10^{-4}\text{AUFS}$). This limit was set assuming a signal to noise ratio of 5.

The $^1\text{H}$ NMR spectra of all the compounds were determined using DMSO-$d_6$ as the solvent. Deuterated benzene ($C_6D_6$) was also used as a sample solvent since peak overlap in the case of DATB, and H-D exchange of the OH in PA was noted in the DMSO. The pulse sequence was repeated four times and the signals time-averaged for all the compounds with the exception of TATB. In the case of TATB, the pulse sequence was repeated 5000 times, because of the low solubility, and the resulting signals time-averaged. The NMR data are reported in Table 4.

For the thin layer chromatographic analyses the glass plates were prepared according to the method of Hoffsommer$^{17}$ using Merck Silica Gel HF 254 as the adsorbent. This material contains a fluorescent indicator which allows location of the developed spots with 2540 light. The developing solvents used were benzene and methanol $R_f$ values were obtained for all the explosive compounds with the exception of HMX, TATB, PETN and TNN. The data are recorded in Table 5.

The melting points of most of the explosive compounds of this investigation were determined with a Thomas Hoover Capillary Melting Point Apparatus, with a heating rate of approximately 20°/minute. All temperatures measured are uncorrected. In addition, melting point data from the literature are cited. The melting points are listed in Table 6.


Solvent: methanol: water (50:50, v/v)
Flow rate: 2.0 ml/minute
Scale: 0.005 AUFS
Sample size: 2 µl
Chart speed: 0.5 cm/minute

FIGURE 1  HPLC CHROMATOGRAM OF A 14 COMPONENT MIXTURE
Solvent: methanol:water (50:50, v/v)
Flow rate: 2.0 ml/minute
Scale: 0.02 AUFS
Sample size: 5 µl
Chart speed: 0.5 cm/minute

**FIGURE 2** HPLC CHROMATOGRAM OF A FOUR COMPONENT MIXTURE
### TABLE 1  HPLC DATA OF EXPLOSIVE COMPOUNDS

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>AVERAGE RETENTION TIME (minutes)</th>
<th>AVERAGE RESPONSE FACTOR (peak height mm/mg)</th>
<th>DMSO SOLN Conc (moles/liter)</th>
<th>APPROX LIMIT OF DETECTIONa (micrograms/ml; ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DMSO</td>
<td>1.8</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>HMX</td>
<td>2.8</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PETN</td>
<td>21.2</td>
<td>4.5 x 10⁻³</td>
<td>10⁻² - 10⁻³</td>
<td>3332</td>
</tr>
<tr>
<td>PYX</td>
<td>9.6</td>
<td>1.1 x 10⁶</td>
<td>10⁴ - 10⁶</td>
<td>13.4</td>
</tr>
<tr>
<td>RDX</td>
<td>6.8</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>TATB</td>
<td>7.4</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>TPT</td>
<td>80.0</td>
<td>1.7 x 10⁶</td>
<td>10⁴ - 10⁶</td>
<td>87.5</td>
</tr>
</tbody>
</table>

**CONDITIONS:**

- Isocratic elution
- Detector wavelength, 254 nm
- Column: Radial-PAK A with the RCM-100 Radial Compression Module
- Flow rate: 2.0 ml/minute
- Mobile phase: 40% MeOH/60% H₂O by volume
- Chart speed: 0.6 cm/minute

a From the peak height responses given, the detection limit for all the explosive compounds was calculated to be 15 mm on scale 0.006 absorbance units full scale (3 x 10⁻⁴ AUFS). This limit was set assuming a signal/noise ratio of 5.
### TABLE 2  HPLC DATA OF EXPLOSIVE COMPOUNDS

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>AVERAGE RETENTION TIME (minutes)</th>
<th>AVERAGE RESPONSE FACTOR (peak height mm/mg)</th>
<th>DMSO SOLN CONC (moles/liter)</th>
<th>APPROX LIMIT OF DETECTION^8 (micrograms/ml; ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DATB</td>
<td>5.5</td>
<td>$2.77 \pm 0.01 \times 10^6$</td>
<td>$10^4 \cdot 10^5$</td>
<td>5.4</td>
</tr>
<tr>
<td>DINA</td>
<td>4.1</td>
<td>$1.82 \pm 0.04 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>8.2</td>
</tr>
<tr>
<td>DMSO</td>
<td>1.6</td>
<td>➥</td>
<td>➥</td>
<td>➥</td>
</tr>
<tr>
<td>DIPAM</td>
<td>16.1</td>
<td>$1.55 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>9.7</td>
</tr>
<tr>
<td>DNT</td>
<td>11.8</td>
<td>$4.37 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>3.4</td>
</tr>
<tr>
<td>HNAB</td>
<td>9.2</td>
<td>$9.5 \pm 0.06 \times 10^5$</td>
<td>$10^4 \cdot 10^6$</td>
<td>~15</td>
</tr>
<tr>
<td>HNBiB</td>
<td>22.8</td>
<td>$2.41 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>9.9</td>
</tr>
<tr>
<td>HNS</td>
<td>14.2</td>
<td>$2.31 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>6.5</td>
</tr>
<tr>
<td>HMX</td>
<td>2.0</td>
<td>$4.77 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>3.1</td>
</tr>
<tr>
<td>PA</td>
<td>0.8</td>
<td>$8.98 \pm 0.05 \times 10^7$</td>
<td>$10^4 \cdot 10^6$</td>
<td>0.2</td>
</tr>
<tr>
<td>PETN</td>
<td>12.0</td>
<td>$1.31 \pm 0.03 \times 10^4$</td>
<td>$10^2 \cdot 10^3$</td>
<td>1236</td>
</tr>
<tr>
<td>PYX</td>
<td>2.1</td>
<td>$4.04 \pm 0.04 \times 10^6$</td>
<td>$10^4 \cdot 10^5$</td>
<td>3.7</td>
</tr>
<tr>
<td>RDX</td>
<td>3.4</td>
<td>$4.35 \pm 0.04 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>3.5</td>
</tr>
<tr>
<td>TATB</td>
<td>4.0</td>
<td>$3.62 \pm 0.03 \times 10^6$</td>
<td>$10^6 \cdot 10^7$</td>
<td>4.1</td>
</tr>
<tr>
<td>Tetryl</td>
<td>5.8</td>
<td>$4.42 \times 10^6$</td>
<td>$10^4 \cdot 10^8$</td>
<td>3.4</td>
</tr>
<tr>
<td>TNA</td>
<td>6.8</td>
<td>$2.6 \pm 0.04 \times 10^5$</td>
<td>$10^4 \cdot 10^6$</td>
<td>5.8</td>
</tr>
<tr>
<td>TNB</td>
<td>5.3</td>
<td>$4.97 \times 10^6$</td>
<td>$10^4 \cdot 10^7$</td>
<td>3.0</td>
</tr>
<tr>
<td>TNN</td>
<td>7.0</td>
<td>$2.12 \pm 0.08 \times 10^6$</td>
<td>$10^3 \cdot 10^5$</td>
<td>7.3</td>
</tr>
<tr>
<td>TNT</td>
<td>7.8</td>
<td>$4.74 \times 10^6$</td>
<td>$10^4 \cdot 10^6$</td>
<td>3.1</td>
</tr>
<tr>
<td>TPT</td>
<td>22.9</td>
<td>$1.32 \pm 0.01 \times 10^6$</td>
<td>$10^4 \cdot 10^5$</td>
<td>11.4</td>
</tr>
</tbody>
</table>

**CONDITIONS:**

- Isocratic elution
- Detector wavelength, 254 nm
- Column: Radial-PAK A with the RCM-100 Radial Compression Module
- Flow rate: 2.0 ml/minute
- Mobile phase: 50% MeOH/50% H₂O by volume
- Chart speed: 0.5 cm/minute

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^8 From the peak height responses given, the detection limit for all the explosive compounds was calculated to be 15 mm on scale 0.005 absorbance units full scale (3 x 10⁴ AUFS). This limit was set assuming a signal/noise ratio of 5.
### TABLE 3  HPLC DATA OF EXPLOSIVE COMPOUNDS

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>AVERAGE RETENTION TIME (minutes)</th>
<th>AVERAGE RESPONSE FACTOR (peak height mm/mg)</th>
<th>DMSO SOLN CONC (moles/liter)</th>
<th>APPROX LIMIT OF DETECTION(^a) (micrograms/ml; ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DIPAM</td>
<td>3.3</td>
<td>7.36 ± 0.06 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>2.0</td>
</tr>
<tr>
<td>DMSO</td>
<td>1.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DNT</td>
<td>3.7</td>
<td>1.19 x 10^7</td>
<td>10^{-4} . 10^{-6}</td>
<td>1.3</td>
</tr>
<tr>
<td>DODECA</td>
<td>8.7</td>
<td>4.81 ± 0.03 x 10^6</td>
<td>10^{-6} . 10^{-7}</td>
<td>3.1</td>
</tr>
<tr>
<td>HNBIB</td>
<td>3.9</td>
<td>9.72 ± 0.03 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>1.9</td>
</tr>
<tr>
<td>HNBP</td>
<td>3.6</td>
<td>1.14 ± 0.02 x 10^7</td>
<td>10^{-4} . 10^{-7}</td>
<td>1.3</td>
</tr>
<tr>
<td>HNS</td>
<td>2.9</td>
<td>9.71 ± 0.03 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>2.1</td>
</tr>
<tr>
<td>NONA</td>
<td>4.7</td>
<td>8.03 ± 0.06 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>1.9</td>
</tr>
<tr>
<td>ONT</td>
<td>5.3</td>
<td>7.56 ± 0.04 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>2.0</td>
</tr>
<tr>
<td>PETN</td>
<td>3.3</td>
<td>5.37 x 10^4</td>
<td>10^{-2} . 10^{-3}</td>
<td>1009</td>
</tr>
<tr>
<td>TNN</td>
<td>2.3</td>
<td>6.32 ± 0.01 x 10^6</td>
<td>10^{-4} . 10^{-5}</td>
<td>2.4</td>
</tr>
<tr>
<td>TNS</td>
<td>5.2</td>
<td>5.48 ± 0.04 x 10^6</td>
<td>10^{-4} . 10^{-6}</td>
<td>2.7</td>
</tr>
<tr>
<td>TNT</td>
<td>2.9</td>
<td>1.07 x 10^7</td>
<td>10^{-4} . 10^{-6}</td>
<td>1.4</td>
</tr>
<tr>
<td>TPT</td>
<td>3.1</td>
<td>7.86 ± 0.07 x 10^6</td>
<td>10^{-4} . 10^{-7}</td>
<td>1.9</td>
</tr>
</tbody>
</table>

**CONDITIONS:**

- **Isocratic elution**
- **Detector wavelength, 254 nm**
- **Column:** Radial-PAK A with the RCM-100 Radial Compression Module
- **Flow rate:** 2.0 ml/minute
- **Mobile phase:** 70% MeOH/30% H2O by volume
- **Chart speed:** 0.5 cm/minute

\(^a\) From the peak height responses given, the detection limit for all the explosive compounds was calculated to be 15 mm on scale 0.006 absorbance units full scale (3 x 10^{-4} AUPS). This limit was set assuming a signal/noise ratio of 5.
<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>SOlVENT $^a$</th>
<th>NMR SPECTRUM $^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>DATB</td>
<td>DMSO-D$_6$</td>
<td>9.10 (s, Ar-H)</td>
</tr>
<tr>
<td>DATB</td>
<td>BENZEN' D$_6$</td>
<td>8.81 (s, Ar-H)</td>
</tr>
<tr>
<td>DINA</td>
<td>DMSO-D$_6$</td>
<td>4.76 (t, 2CH$_2$)</td>
</tr>
<tr>
<td>DIPAM</td>
<td></td>
<td>9.08 (s, 2Ar-H)</td>
</tr>
<tr>
<td>DNT</td>
<td></td>
<td>8.68 (s, 2NH$_2$)</td>
</tr>
<tr>
<td>DODECA</td>
<td></td>
<td>9.39 (s, 2Ar-H)</td>
</tr>
<tr>
<td>HNAB</td>
<td></td>
<td>10.19 (s, 4Ar-H)</td>
</tr>
<tr>
<td>HNBIB</td>
<td></td>
<td>9.05 (s, 4Ar-H)</td>
</tr>
<tr>
<td>HNB</td>
<td></td>
<td>3.39 (s, 2CH$_2$)</td>
</tr>
<tr>
<td>HMX</td>
<td></td>
<td>5.88 (s, 8R-H)</td>
</tr>
<tr>
<td>NONA</td>
<td></td>
<td>9.26 (s, 8Ar-H)</td>
</tr>
<tr>
<td>CNT</td>
<td></td>
<td>9.35 (s, 8Ar-H)</td>
</tr>
<tr>
<td>PA</td>
<td></td>
<td>8.56 (s, 2Ar-H)</td>
</tr>
<tr>
<td>PA</td>
<td>BENZENE-D$_6$</td>
<td>8.07 (s, 2Ar-H)</td>
</tr>
<tr>
<td>PETN</td>
<td>DMSO-D$_6$</td>
<td>4.65 (s, 4CH$_2$)</td>
</tr>
<tr>
<td>PYX</td>
<td></td>
<td>8.85 (s, 8Ar-H)</td>
</tr>
<tr>
<td>RDX</td>
<td></td>
<td>9.10 (s, 2NH)</td>
</tr>
<tr>
<td>TATB</td>
<td></td>
<td>6.06 (s, 6R-H)</td>
</tr>
<tr>
<td>TETRVL</td>
<td></td>
<td>10.00 (s, 3NH$_2$)</td>
</tr>
<tr>
<td>TNA</td>
<td></td>
<td>9.04 (s, 2Ar-H)</td>
</tr>
<tr>
<td>TNB</td>
<td></td>
<td>8.98 (s, NH$_2$)</td>
</tr>
<tr>
<td>TNN</td>
<td></td>
<td>9.13 (s, 3Ar-H)</td>
</tr>
<tr>
<td>TNS</td>
<td></td>
<td>8.80 (s, 4R-H)</td>
</tr>
<tr>
<td>TNT</td>
<td></td>
<td>9.14 (s, 2Ar-H)</td>
</tr>
<tr>
<td>TPT</td>
<td></td>
<td>8.98 (s, 2Ar-H)</td>
</tr>
<tr>
<td>DMSO-D$_6$ ( neat)</td>
<td></td>
<td>2.48 (m, 2CH$_2$)</td>
</tr>
<tr>
<td>BENZENE-D$_6$ ( neat)</td>
<td></td>
<td>7.18 (s, 8Ar-H)</td>
</tr>
</tbody>
</table>

$^a$ The solvents used were: benzene C$_6$H$_6$: 99.5 atom % D; dimethylsulfoxide DMSO-D$_6$: 99.6 atom % D. A water peak at 3.30 (s, 2H) was noted in the DMSO samples.

$^b$ s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, Ar = aromatic, R = ring protons. Chemical shifts are in ppm downfield from internal TMS with line multiplicity and relative intensity in parentheses. Spectra were determined on a Varian XL-200 Spectrometer. The pulse sequence was repeated 4 times and the signals time-averaged.

$^c$ The TATB pulse sequence was repeated 5000 times, and the resulting signals time-averaged.
### TABLE 5  TLC DATA OF EXPLOSIVE COMPOUNDS

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<td>DIPAM</td>
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<tr>
<td>DNT</td>
<td>0.78</td>
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<td>DODECA</td>
<td>0.63$^c$</td>
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<td>HNAB</td>
<td>0.67</td>
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<tr>
<td>HMX</td>
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<td>MeOH</td>
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<td>NONA</td>
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<td>ONT</td>
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<tr>
<td>PA</td>
<td>0.09$^c$</td>
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<tr>
<td>PA</td>
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<tr>
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<td>TETRYL</td>
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**a**  $R_f$ taken from leading edge of spot. A short wave UV lamp (2537 Å) was used for spot visualization. The adsorbent used was Merck Silica Gel HF-254 coated on glass plates.

**b**  The solvents used were: benzene ($C_6H_6$) and methanol (MeOH).

**c**  Streaking or tailing to origin.
### TABLE 6 MELTING POINT DATA OF EXPLOSIVE COMPOUNDS

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<td>DINA</td>
<td>52°</td>
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<tr>
<td>DIPAM</td>
<td>306 dec°, 304°19</td>
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<tr>
<td>DNT</td>
<td>71.5°, 71°18</td>
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<tr>
<td>DODECA</td>
<td>&gt; 428°</td>
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<tr>
<td>HNAB</td>
<td>220-221°20</td>
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<tr>
<td>HNBB</td>
<td>218°, 218-220°21</td>
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<tr>
<td>HNBP</td>
<td>239.3-240.5°</td>
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<tr>
<td>HNS-I</td>
<td>318 dec°</td>
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<td>318 dec°</td>
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<tr>
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<td>280 dec°, 273°18</td>
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<tr>
<td>NONA</td>
<td>440°19</td>
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<tr>
<td>ONT</td>
<td>&gt; 400°7</td>
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<tr>
<td>PA</td>
<td>121.8-122.4°</td>
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<td>141.3°20</td>
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<td>PVX</td>
<td>360°7</td>
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<tr>
<td>RDX</td>
<td>204 dec°, 204°20</td>
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<tr>
<td>TATB</td>
<td>&gt; 370°, &gt; 360°18, ~ 450 dec°19</td>
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<tr>
<td>Tetryl</td>
<td>130 dec°, 129.5°20</td>
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<tr>
<td>TNA</td>
<td>188°20</td>
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<tr>
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<td>121°, 121.3°20</td>
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<tr>
<td>TNN</td>
<td>450°19</td>
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<tr>
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<tr>
<td>TPT</td>
<td>362°7, 362-363°19, 22, 349-351°23</td>
</tr>
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*Melting points determined on a Thomas Hoover Capillary Melting Point Apparatus, using a heating rate of approximately 2°/minute. All measured temperatures are uncorrected.*

---

REFERENCES


4. Selig, W., "Photometric Determination of 1,3,5-Triamino-2,4,6-Trinitrobenzene (TATB) in Dimethylsulfoxide (DMSO)," Lawrence Livermore Laboratory Report, UCID-17542, July 1977.


REFERENCES (Cont.)


NOTE: \( X = \text{NO}_2 \)

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