Characterization of MTNP
(1-methyl-3,4,5-trinitro-1,2-pyrazole)

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# MTNP Overview

**Molecular Formula**: $C_4H_3N_5O_6$

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Molecular Weight</td>
<td>217</td>
</tr>
<tr>
<td>Melting Point</td>
<td>91°C</td>
</tr>
<tr>
<td>Exotherm</td>
<td>256°C</td>
</tr>
<tr>
<td>Density</td>
<td>1.839 g/cc</td>
</tr>
<tr>
<td>Heat of Formation</td>
<td>50.7 kJ/mol</td>
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</tbody>
</table>
SYNTHESESIZE AND PROVIDE MTNP FOR INITIAL EVALUATION EFFORTS

- UP TO DATE LITERATURE SEARCH AND ANALYSIS OF LITERATURE
- IDENTIFICATION OF SUITABLE ROUTES
- CONDUCT EXPERIMENTS TO DETERMINE THE SUITABILITY OF IDENTIFIED METHODS FOR LAB SCALE PREPARATION – DOWN SELECT THE RIGHT METHOD
- ANALYSIS, CHARACTIZATION AND PROPERTIES DETERMINATION
- DEVELOP PROCESSES AND METHODS FOR LAB SCALE SCALE-UP PROCESS
  - ENVIRONMENTALLY FRIENDLY
  - LEAST NUMBER OF STEPS
  - BETTER YIELDS
  - REPRODUCEABLE METHODS
  - LESS HAZARDOUS WASTE
- DEMONSTRATE THE VIABILITY OF THE DEVELOPED PROCESS BY PRODUCING SIGNIFICANT QUANTITY OF MTNP
- PROVIDE MTNP FOR PERFORMANCE EVALUATION
<table>
<thead>
<tr>
<th>Explosive</th>
<th>Formula</th>
<th>Density</th>
<th>DH&lt;sub&gt;f&lt;/sub&gt;</th>
<th>Det Vel</th>
<th>C-J P</th>
<th>Gurn Vel(3)</th>
<th>Gurn Vel(7)</th>
<th>OB</th>
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<tbody>
<tr>
<td>DNAN</td>
<td>C&lt;sub&gt;7&lt;/sub&gt;H&lt;sub&gt;6&lt;/sub&gt;N&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;</td>
<td>1.546</td>
<td>-186.5</td>
<td>6.14</td>
<td>14.8</td>
<td>1.88</td>
<td>2.10</td>
<td>-96.9</td>
</tr>
<tr>
<td>3,4 DNP</td>
<td>C&lt;sub&gt;3&lt;/sub&gt;H&lt;sub&gt;2&lt;/sub&gt;N&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>1.791</td>
<td>120.5</td>
<td>8.31</td>
<td>30.9</td>
<td>2.63</td>
<td>2.86</td>
<td>-30.4</td>
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<tr>
<td>MTNP</td>
<td>C&lt;sub&gt;4&lt;/sub&gt;H&lt;sub&gt;3&lt;/sub&gt;N&lt;sub&gt;5&lt;/sub&gt;O&lt;sub&gt;6&lt;/sub&gt;</td>
<td>1.82</td>
<td>4.53</td>
<td>8.36</td>
<td>31.1</td>
<td>2.59</td>
<td>2.82</td>
<td>-25.8</td>
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<td>PrNQ</td>
<td>C&lt;sub&gt;4&lt;/sub&gt;H&lt;sub&gt;10&lt;/sub&gt;N&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;2&lt;/sub&gt;</td>
<td>1.335</td>
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<td>6.45</td>
<td>14.4</td>
<td>1.95</td>
<td>2.10</td>
<td>-120</td>
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<td>TNT</td>
<td>C&lt;sub&gt;7&lt;/sub&gt;H&lt;sub&gt;5&lt;/sub&gt;N&lt;sub&gt;3&lt;/sub&gt;O&lt;sub&gt;6&lt;/sub&gt;</td>
<td>1.654</td>
<td>-63</td>
<td>6.89</td>
<td>19.8</td>
<td>2.20</td>
<td>2.43</td>
<td>-74.0</td>
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<td>RDX</td>
<td>C&lt;sub&gt;3&lt;/sub&gt;H&lt;sub&gt;6&lt;/sub&gt;N&lt;sub&gt;6&lt;/sub&gt;O&lt;sub&gt;6&lt;/sub&gt;</td>
<td>1.816</td>
<td>70</td>
<td>8.76</td>
<td>34.8</td>
<td>2.73</td>
<td>3.01</td>
<td>-21.6</td>
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<td>HMX</td>
<td>C&lt;sub&gt;4&lt;/sub&gt;H&lt;sub&gt;8&lt;/sub&gt;N&lt;sub&gt;8&lt;/sub&gt;O&lt;sub&gt;8&lt;/sub&gt;</td>
<td>1.905</td>
<td>75</td>
<td>9.09</td>
<td>38.7</td>
<td>2.76</td>
<td>3.04</td>
<td>-21.6</td>
</tr>
</tbody>
</table>
MTNP Starting from 3-NP

1. 3-NP reacts with HNO₃ and Ac₂O to form 1,3-DNP.
2. 1,3-DNP undergoes nitration with PhCN at 150-180°C to form 3,5-DNP.
3. 3,5-DNP undergoes methylation with DMS to form MDNP.
4. MDNP undergoes nitration to form MTNP.
Spectral analysis of MTNP

\[ ^{13}\text{C}-\text{NMR of 1-methyl-3,4,5-trinitropyrazole in DMSO-}d_6 \]
Spectral analysis of MTNP

**XRD of MTNP**
Properties of 1-methyl-3,4,5-trinitropyrazole (MTNP)

<table>
<thead>
<tr>
<th>Molecule</th>
<th>Impact (cm)</th>
<th>BAM Friction (N)</th>
<th>ABL ESD (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MTNP</td>
<td>54.1</td>
<td>No Reaction in 10 trials @ 360N</td>
<td>Reacted @ 0.063J, did not react in 20 trails @ 0.051J</td>
</tr>
<tr>
<td>RDX Class I Type II</td>
<td>18</td>
<td>Reacted @ 216N, did not react in 10 trials at 192N</td>
<td>Reacted @ 0.063J, did not react in 20 trails @ 0.051J</td>
</tr>
<tr>
<td>RDX Class V Type II</td>
<td>&gt;100</td>
<td>Reacted @ 324N, did not react in 10 trials at 288N</td>
<td>Reacted @ 0.051J, did not react in 20 trails @ 0.040J</td>
</tr>
<tr>
<td>DNP</td>
<td>&gt;100</td>
<td>No Reaction in 10 trials @ 360N</td>
<td>Reacted @ 0.063J, did not react in 20 trails @ 0.051J</td>
</tr>
<tr>
<td>TNT</td>
<td>88.3</td>
<td>Reacted @ 240N, did not react in 10 trials at 216N</td>
<td>Did not react in 20 trials @ 0.25J (Old Test Method)</td>
</tr>
</tbody>
</table>
SEM Images of MTNP
DSC of MTNP

Onset = 225.06 °C
End = 262.36 °C

Area = 299.056 mJ
ΔH = 144.19 mJ/g

Peak = 252.16 °C
Peak Height = 1.0452 mW

Peak X = 91.25 °C
• Performed compatibility testing in accordance to STANAG 4147 ED.2

• A DSC for each individual explosive, test material and mixture shall be run in duplicate

• Explosives and test materials are mixed in a 1:1 (w/w) ratio

• Samples are heated at a rate of 5°C/min from room temperature to 300°C or more for each sample

• The reactivity (compatibility) is then determined by comparing the decomposition profiles of the individual components to the mixture
• MTNP is compatible with Al.

• MTNP requires VTS testing with NTO, FOX-7, HMX and O-ring since DSC compatibility showed more than 10°C exotherm shift

• MTNP requires VTS testing with brass and 304/316 stainless steel to determine compatibility due to the appearance of a new exotherm

• VTS testing is also needed with copper and A2 Steel due to earlier onset of the decomposition (around 250°C)

• VTS testing with Copper: Pass
Conclusions

- Performed Literature Search and Analyzed Reported Methods
- Made attempts to synthesize MTNP in one pot process from Pyrazole
- Will Investigate Preparing MTNP Using Sequential Nitration Process
- Looking to Reduce the Amount of Required Reagents
- Characterized and Determined Small Scale Safety and Handling and Thermal Properties of MTNP
- Developed Scale up process

FUTURE WORK:

- Continue Investigating Alternate Nitrating Agents/Reagents