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TECHNICAL REPORT 4203

IMPROVED METHOD
OF
MANUFACTURE OF A BALLISTIC MODIFIER
FOR
SMOKELESS ROCKET PROPELLANTS (U)

DANIEL R. SATRIANA
ARTHUR J. BRACUTI

JULY 1971

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DOVER, NEW JERSEY

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AN IMPROVED METHOD OF MANUFACTURE OF A BALLISTIC MODIFIER FOR SMOKELESS ROCKET PROPELLANTS (U)

by

Daniel R. Satriana
Arthur J. Bracuti

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(C) ABSTRACT

Several improved processes for the preparation of a ballistic modifier derived in this laboratory under the name lead stannate-TDI (tolylene diisocyanate) reduced (LSTR) have been developed. Presented are the parameters of time, temperature, and concentration of reactants, the X-ray diffraction patterns of several modifiers, and the effects of catalysis on the combustion of an experimental nitramine plastisol formulation.

Also included in the report is a comparison of the burning rate characteristics of crosslinked and uncrosslinked compositions containing various lead-based modifiers.

(C) CONCLUSION

Several novel methods have been developed to improve the preparation of a ballistic modifier that is highly effective in producing near plateau and mesa ballistics in a high energy smokeless nitramine plastisol propellant system.

(C) RECOMMENDATIONS

It is recommended that the manufacture of the combustion catalyst described in this report be adopted for use in high energy nitramine double-base propellants in which smokelessness and combustion reliability are essential.

It also is recommended that the following investigations be conducted:

1. A study of the pyrolysis step in the process to include a properly controlled inert atmosphere as well as the use of reactive gases, namely, ammonia and hydrogen.

2. The use of differential thermal analysis as a technique for monitoring combustion catalysis.
3. A study of the combustion products of modified and unmodified propellants by gas chromatography to properly understand the mechanism by which catalytic activity may be recognized.
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(C) INTRODUCTION

The combustion catalyst, lead stannate-TDI reduced (LSTR) (Ref 1, 2), which was developed a few years ago at Picatinny Arsenal was extremely effective in reducing variations in burning to feasible temperatures (160°F to -40°F) and pressures over practical pressure ranges when used in an experimental high energy nitramine plastisol propellant. Thus far, efforts to improve the burning rate behavior of these high energy propellants with ballistic modifiers ordinarily employed in double-base systems met with only limited success (Ref 4, 5). There is no basic rule for determining the effect a certain catalyst will have on the burning characteristics of a given propellant system.

With the advent of the LSTR modifier, an investigation was undertaken to determine the ingredient responsible for ballistic modification.

(C) RESULTS AND DISCUSSION

In an attempt to attain a reasonable degree of understanding of combustion catalysis, an improved method of manufacture of the catalyst, lead stannate-TDI reduced (LSTR, was developed.

In the more recent past, a process had been devised which was based on the assumption that a specific "complex" between lead stannate and 2, 4-tolylene diisocyanate was essential in the preparation of the LSTR modifier. The process was not without certain drawbacks since it required accurate control of the organic component of the "complex" to insure a successful product. However, since the stoichiometry of a complex compound is firmly established, it was logical to conclude that the organic portion also was an established quantity. With this in mind, it was believed that what was actually pyrolyzed possibly was a mixture of lead stannate and mixed ureas emanating from the reaction of TDI and water present in the system, rather than the complex of lead stannate-TDI.
To investigate this possibility, solid ureas were prepared by simply reacting TDI with an excess of aqueous acetone solution. The white solid obtained, which does not melt below 300° C, probably is a mixture of ureas and the corresponding biurets that can be expected from a difunctional isocyanate. This material, which for convenience will be referred to as Solid I, was pyrolyzed with lead stannate under varying conditions of temperature and concentration relative to the metal oxide. Each of the products was formulated in an experimental nitramine propellant as shown in Table 1. The cured grains were tested in the strand burner apparatus to determine their burning characteristics over a wide pressure range. The results of this study are presented in Tables 2, 3, and 4. The burning rate curves of the various modified compositions are shown in Figures 1 through 14.

Examination of the data reveals unequivocally that Solid I when pyrolyzed with lead stannate yields a product possessing extremely effective ballistic properties in the formulation studied. Equally important, however, is the fact that a surprisingly wide range of experimental conditions in the preparation of the pyrolyzed product can be permitted without significantly altering the combustion characteristics of the propellant. The results are particularly encouraging since the LSTR catalyst prepared by the original method necessitates, as said above, rather precise control in its fabrication. As little as 5% of Solid I is effective in producing a catalyst that will lower the pressure exponent significantly in the 800 to 1600 psi region from 0.81 to 0.10 as well as increase the burning rate from 0.21 to 0.39 in./sec at 1000 psi. Higher concentrations of Solid I improve these characteristics only slightly. A wide range of pyrolysis temperatures, i.e., 350° C to 550° C (Table 2), as well as pyrolysis times, i.e., 30 minutes to 120 minutes (Table 3), can be used to produce effective catalysts.

Representative X-ray diffraction patterns of the modifiers are shown in Figures 15 through 19. They reveal without exception the presence of stannic oxide, lead oxide, and lead in varying amounts, depending on experimental conditions. Elemental lead was identified also by differential thermal analysis. A typical thermogram of a modifier is shown by Figure 20. On the basis of the analytical data, it is conjectured that the reduction of lead stannate to probably finely divided lead may be the key toward eventually understanding ballistic modification of lead compounds.
To further extend this process to encompass other materials, a series of mixed oxides of lead and tin was substituted for lead stannate. Mixtures of yellow lead oxide (PbO) and stannic oxide (SnO₂), in concentrations ranging from 10% to 90% PbO, were pyrolyzed with Solid I at 350° C to 400° C and at 500° C to 550° C. The results of this study are presented in Table 5. Burning rate curves of the various modified propellants are graphically represented in Figures 21 through 36. Observation of the data clearly indicates that there is some improvement of the pressure index in 800 to 1600 psi region. However, products derived from the pyrolysis of mixed oxides are by far inferior to the products obtained from the pyrolysis of lead stannate. The presence of elemental lead was detected in all cases also by X-ray diffraction. At the higher pyrolysis temperatures, formation of metallic globules of lead, with high concentrations of lead oxide, was evident, while agglomeration did not occur at the lower pyrolysis temperatures.

Attempts were made to investigate other combustible materials in order to simplify the process further. However, only slight improvements in the burning characteristics of the propellant composition were observed when using modifiers derived from the pyrolysis of lead stannate with graphite, charcoal, or urea instead of Solid I.

The method of preparation of the LSTR catalyst, which consists essentially of a pyrolyzed mixture of lead stannate and mixed ureas formed by the reaction of TDI and water (Solid I), suggested the use of analogous compounds. Consequently, the following ureas were tried: Urea per se, phenyl urea, diphenyl urea, and diethyl diphenyl urea (commonly known as ethyl centralite). Each pyrolysis product which subsequently will be designated as the catalyst—then was formulated into an experimental nitramine plastisol propellant composition (Table 1).

The results of the investigation presented in Table 6 reveal that urea compounds, in general, produce excellent modifiers if pyrolyzed with lead stannate. With the exception of urea itself, all of the compounds studied give good plateau ballistics in the 800 to 1600 psi region. It also is evident that catalytic activity increases as the degree of substitution on the nitrogen atom rises. The burning rate curves of the propellants containing the various modifiers are shown in Figures 37 through 41.
In order to establish whether or not the isocyanate function also is effective in producing good catalysts on direct pyrolysis with lead stannate, several commercially available diisocyanates were examined. Each pyrolysis product was formulated into the same experimental nitramine propellant composition (Table 1). The cured grains were tested in the strand burner to determine the burning rate and pressure index from 200 to 3000 psi. The results of this investigation are shown in Table 7. The burning rate curves of the propellant compositions containing the various modifiers are illustrated in Figures 42 through 46.

The data clearly indicates that effective catalysts also can be produced by direct pyrolysis of diisocyanates with lead stannate. In all cases, excellent plateau burning results were realized in the 800 to 1600 psi region, in which the pressure index ranged from 0.08 to 0.19. The burning rate at 1000 psi was 0.36 in./sec to 0.43 in./sec compared with 0.29 in./sec for the propellant composition containing unpyrolyzed lead stannate as the modifier.

In view of the preceding study, it is conceivable that the presence of TDI as a crosslinking agent might improve the burning characteristics of the propellant if it is used in compositions containing modifiers of the lead oxide type. Therefore, a series of experiments was carried out, in which a nitramine plastisol propellant composition (Table 8), modified with various lead oxides, was formulated with and without TDI. The combustion properties of each composition, as derived from strand burner data, are summarized in Table 9.

In general, the presence of the crosslinker has an erratic effect on the pressure exponent. This phenomenon was not observed in a previous investigation with mixed oxides of lead and tin (Ref 3). In the case of the formulation with lead stannate modifier, the pressure exponent in the pressure region of 800-1400 psi was significantly higher with the crosslinker present. Since a pyrolyzed mixture of TDI and lead stannate (LSTR modifier) produces an excellent ballistic modification, one would expect the addition of previously unpyrolyzed TDI and lead stannate to give similar modification effects. However, the combustion properties of propellant in the formulation of which unpyrolyzed components are present results in unsatisfactory modification. In the case of lead oxide (PbO), lead sesquioxide (Pb₂O₃), and lead tetraoxide (Pb₃O₄), the opposite effect is observed. Lower burning rate slopes are obtained with the crosslinked formulations in the
800-1400 psi region. The crosslinked propellant containing lead di-
oxide (PbO$_2$), on the other hand, gives unique burning characteristics
over the whole pressure range. A comparison of the burning rate
curve of the crosslinked and uncrosslinked propellant is shown in
Figure 47.

Formulations with the LSTR catalyst give mesa pressure slopes
without the crosslinking agent, and only plateau ballistics with the
crosslinker present over the 800-1600 psi region. A comparison
of the burning rate curves with and without crosslinker present is shown
in Figure 48.

With propellant containing the mixed oxide modifier, lead sesqui-
oxide-stannic oxide (30/70), there are slightly less favorable burning
properties of the crosslinked propellant system evident. The burning
rate curves of crosslinked and uncrosslinked formulations are shown
graphically in Figure 49.

(C) EXPERIMENTAL

General

The propellants were prepared in fifty-gram batches in polyethylene
cups. They were thoroughly mixed by hand with wooden applica-
tors, cast into appropriate molds, and cured overnight in the oven at
60°C.

The cured strands were tested in the Crawford strand burner to
determine the burning rate over a wide pressure range.

Preparation of the Catalyst by Pyrolysis

A given weight ratio of metal oxide to organic material is mi-
thorously and then transferred to an aluminum container (1 1/2'x
3 1/2" dia), which is covered with aluminum foil. The foil is
slit in 5 to 6 places, and the container is placed in the muffler furna-
ace with the door ajar and the temperature between 500°C to 550°C.
When the initial reaction is completed (i.e., smoke is no longer
evolved) the container is removed from the oven and quickly covered
with aluminum foil (DO NOT EXPOSE TO AIR UNNECESSARILY).
The sealed container is replaced into the furnace. After heating at
500°C to 550°C for one hour, the container is again removed and the contents are allowed to cool overnight without exposure to air. Finally, the pyrolyzed mixture is sifted through a No. 325 mesh screen.

**Preparation of TDI-Urea Compound (Solid I)**

To a three liter beaker containing 350 ml of water and 500 ml of acetone is added 112 grams of 2,4-tolylene diisocyanate. The mixture is stirred slowly during the addition of the diisocyanate to prevent excessive foaming. After standing for one-half hour with occasional stirring, the mixture is filtered. The solid is washed several times with acetone and then air-dried. Finally, it is dried in the oven at 100°C to constant weight, which is approximately 95 grams.

(U) **REFERENCES**


**TABLE 1 (C)**

Experimental nitramine plastisol formulation (U)

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Percentage</th>
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<tr>
<td>Ball powder (19800)</td>
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<tr>
<td>Ball powder (Quick Gel-2161)</td>
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</tr>
<tr>
<td>HMX (705-screened)</td>
<td>30.00%</td>
</tr>
<tr>
<td>Casting solvent X</td>
<td>46.50%</td>
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<tr>
<td>Modifier</td>
<td>4.00%</td>
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</table>
TABLE 2 (C)

The effect of temperature on the pyrolysis of PbSnO$_3$ and Solid I (C)

<table>
<thead>
<tr>
<th>Run</th>
<th>Solid I %</th>
<th>Pyrolysis Temperature $^\circ$C</th>
<th>Pyrolysis Time (min)</th>
<th>Pressure Exponent at $70^\circ$F (800-1600 psi)</th>
<th>Burning Rate at 100 psi (in./sec)</th>
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<tr>
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<tr>
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<td>0.65$^b$</td>
<td>0.29</td>
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</table>

$^a$Control plastisol propellant without modifier

$^b$Control plastisol propellant with PbSnO$_3$ as modifier
# TABLE 3 (C)

Effect of time of pyrolysis on the reaction of PbSnO$_3$ and Solid I (C)

<table>
<thead>
<tr>
<th>Run</th>
<th>Solid I %</th>
<th>Pyrolysis Temperature °C</th>
<th>Pyrolysis Time (min)</th>
<th>Pressure Exponent at 70°F</th>
<th>Burning Rate at 100 psi (in./sec)</th>
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<tr>
<td>1</td>
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<td>0.81$^a$</td>
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<td>0.65$^b$</td>
<td>0.29</td>
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</table>

$^a$ Control plastisol propellant without modifier

$^b$ Control plastisol propellant with PbSnO$_3$ as modifier
TABLE 4 (C)
The effect of concentration of Solid I compound on the pyrolysis of PbSnO$_3$ (C)

<table>
<thead>
<tr>
<th>Run</th>
<th>Solid I %</th>
<th>Pyrolysis Temperature °C</th>
<th>Pyrolysis Time (min)</th>
<th>Pressure Exponent at 70°F (800-1600 psi)</th>
<th>Burning Rate at 1000 psi (in./sec)</th>
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<td>0.05</td>
<td>0.42</td>
</tr>
<tr>
<td>3</td>
<td>15.0</td>
<td>550° ± 25°</td>
<td>120</td>
<td>0.04</td>
<td>0.43</td>
</tr>
<tr>
<td>4</td>
<td>20.0</td>
<td>550° ± 25°</td>
<td>120</td>
<td>0.06</td>
<td>0.45</td>
</tr>
<tr>
<td>5</td>
<td>25.0</td>
<td>550° ± 25°</td>
<td>120</td>
<td>0.07</td>
<td>0.45</td>
</tr>
<tr>
<td>6</td>
<td>30.0</td>
<td>550° ± 25°</td>
<td>120</td>
<td>0.05</td>
<td>0.45</td>
</tr>
</tbody>
</table>

\[a\] Control plastisol propellant without modifier

\[b\] Control plastisol propellant with PbSnO$_3$ as modifier
### TABLE 5 (C)

Properties of nitramine plastisol propellant based on pyrolyzed Pb/SnO₂ modifiers (C)

<table>
<thead>
<tr>
<th>Run</th>
<th>PbO/SnO₂ Ratio</th>
<th>Pyrolysis Temperature (°C)</th>
<th>Pressure Exponent (200-600 psi)</th>
<th>Exponent (800-1600 psi)</th>
<th>Burning Rate at 1000 psi (in./sec)</th>
<th>SnO₂</th>
<th>PbO</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10/90</td>
<td>375</td>
<td>0.52</td>
<td>0.44</td>
<td>0.29</td>
<td>56</td>
<td>1</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>20/80</td>
<td>375</td>
<td>0.44</td>
<td>0.52</td>
<td>0.30</td>
<td>60</td>
<td>3</td>
<td>16</td>
</tr>
<tr>
<td>3</td>
<td>30/70</td>
<td>375</td>
<td>0.42</td>
<td>0.51</td>
<td>0.30</td>
<td>38</td>
<td>7</td>
<td>11</td>
</tr>
<tr>
<td>4</td>
<td>40/60</td>
<td>375</td>
<td>0.35</td>
<td>0.49</td>
<td>0.29</td>
<td>43</td>
<td>8</td>
<td>19</td>
</tr>
<tr>
<td>5</td>
<td>50/50</td>
<td>375</td>
<td>0.31</td>
<td>0.45</td>
<td>0.30</td>
<td>39</td>
<td>10</td>
<td>22</td>
</tr>
<tr>
<td>6</td>
<td>60/40</td>
<td>375</td>
<td>0.28</td>
<td>0.40</td>
<td>0.26</td>
<td>26</td>
<td>21</td>
<td>31</td>
</tr>
<tr>
<td>7</td>
<td>70/30</td>
<td>375</td>
<td>0.26</td>
<td>0.55</td>
<td>0.26</td>
<td>21</td>
<td>19</td>
<td>62</td>
</tr>
<tr>
<td>8</td>
<td>80/20</td>
<td>375</td>
<td>0.26</td>
<td>0.63</td>
<td>0.25</td>
<td>57</td>
<td>--</td>
<td>12</td>
</tr>
<tr>
<td>9</td>
<td>20/80</td>
<td>525</td>
<td>0.54</td>
<td>0.67</td>
<td>0.27</td>
<td>54</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>10</td>
<td>30/70</td>
<td>525</td>
<td>0.40</td>
<td>0.64</td>
<td>0.31</td>
<td>62</td>
<td>3</td>
<td>38</td>
</tr>
<tr>
<td>11</td>
<td>40/60</td>
<td>525</td>
<td>0.34</td>
<td>0.34</td>
<td>0.31</td>
<td>46</td>
<td>3</td>
<td>37</td>
</tr>
<tr>
<td>12</td>
<td>50/50</td>
<td>525</td>
<td>0.32</td>
<td>0.36</td>
<td>0.29</td>
<td>32</td>
<td>7</td>
<td>35</td>
</tr>
<tr>
<td>13</td>
<td>60/40</td>
<td>526</td>
<td>0.26</td>
<td>0.45</td>
<td>0.26</td>
<td>24</td>
<td>2</td>
<td>52</td>
</tr>
<tr>
<td>14</td>
<td>70/30</td>
<td>525</td>
<td>0.31</td>
<td>0.49</td>
<td>0.25</td>
<td>18</td>
<td>5</td>
<td>59</td>
</tr>
<tr>
<td>15</td>
<td>80/20</td>
<td>525</td>
<td>0.32</td>
<td>0.54</td>
<td>0.25</td>
<td>8</td>
<td>9</td>
<td>40</td>
</tr>
<tr>
<td>16</td>
<td>90/10</td>
<td>525</td>
<td>0.37</td>
<td>0.81</td>
<td>0.24</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*a Runs in which globules of lead were visible*
TABLE 6 (C)

Burning properties of the catalysts produced from various ureas (U)

<table>
<thead>
<tr>
<th>Run</th>
<th>Organic Compound</th>
<th>Amount of Urea Added %</th>
<th>Pyrolysis Temperature (°C)</th>
<th>Pyrolysis Time (min)</th>
<th>Pressure index at 70°F (800-1600 psi)</th>
<th>Burning Rate at 1000 psi in./sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Urea</td>
<td>25.0</td>
<td>525-550</td>
<td>60</td>
<td>0.40</td>
<td>0.34</td>
</tr>
<tr>
<td>2</td>
<td>Phenyl Urea</td>
<td>20.0</td>
<td>525-550</td>
<td>60</td>
<td>0.21</td>
<td>0.44</td>
</tr>
<tr>
<td>3</td>
<td>Diphenyl Urea</td>
<td>20.0</td>
<td>525-550</td>
<td>60</td>
<td>0.17</td>
<td>0.37</td>
</tr>
<tr>
<td>4</td>
<td>Ethyl Centralite</td>
<td>20.0</td>
<td>525-550</td>
<td>&lt;0.02</td>
<td></td>
<td>0.35</td>
</tr>
<tr>
<td>5</td>
<td>TDI-Urea</td>
<td>20.0</td>
<td>525-550</td>
<td>&lt;0.02</td>
<td>0.81&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.21</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.65&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.29</td>
</tr>
</tbody>
</table>

<sup>a</sup> Control plastisol propellant without modifier

<sup>b</sup> Control plastisol propellant with lead stannate as modifier
### TABLE 7 (C)

<table>
<thead>
<tr>
<th>Run</th>
<th>Diisocyanate Compound</th>
<th>Diisocyanate Added %</th>
<th>Pyrolysis Temp. (°C)</th>
<th>Pyrolysis Time (Min)</th>
<th>Pressure Index at 70°F (600-1600 psi)</th>
<th>Burning Rate at 1000 psi (in. /sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hexamethylene</td>
<td>20.0</td>
<td>500-550</td>
<td>60.0</td>
<td>0.06</td>
<td>0.36</td>
</tr>
<tr>
<td>2</td>
<td>Methylene-di-P-Phenylene</td>
<td>20.0</td>
<td>500-550</td>
<td>60.0</td>
<td>0.19</td>
<td>0.36</td>
</tr>
<tr>
<td>3</td>
<td>2,4-Tolylene</td>
<td>10.0</td>
<td>500-550</td>
<td>60.0</td>
<td>0.16</td>
<td>0.41</td>
</tr>
<tr>
<td>4</td>
<td>2,4-Tolyene</td>
<td>15.0</td>
<td>500-550</td>
<td>60.0</td>
<td>0.10</td>
<td>0.43</td>
</tr>
<tr>
<td>5</td>
<td>2,4-Tolyene</td>
<td>20.0</td>
<td>500-550</td>
<td>60.0</td>
<td>0.08</td>
<td>0.40</td>
</tr>
</tbody>
</table>

---

*Control plastisol propellant without modifier

*Control plastisol propellant with lead stannate as modifier
<table>
<thead>
<tr>
<th></th>
<th>Uncrosslinked Wt, gms</th>
<th>Crosslinked Wt, gms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ball Powder Lot 19800</td>
<td>11.90</td>
<td>11.90</td>
</tr>
<tr>
<td>HMX (2)</td>
<td>40.00</td>
<td>40.60</td>
</tr>
<tr>
<td>Casting Solvent &quot;X&quot;&lt;sup&gt;a&lt;/sup&gt;</td>
<td>44.10</td>
<td>44.10</td>
</tr>
<tr>
<td>Ballistic Modifier</td>
<td>4.00</td>
<td>4.00</td>
</tr>
<tr>
<td>2,4-Tolylene diisocyanate (added)</td>
<td>----</td>
<td>1.50</td>
</tr>
<tr>
<td>Resorcinol (added)</td>
<td>----</td>
<td>0.50</td>
</tr>
</tbody>
</table>

<sup>a</sup>Composition of casting solvent

Ethylene glycol dinitrate, 65%
Butanetriol trinitrate, 34%
2-Nitrophenylamine, 1%
### Table 9 (C)

Burning characteristics of crosslinked and uncrosslinked HMX (40%) plastisol propellants containing various lead oxide modifiers (U)

<table>
<thead>
<tr>
<th>Metal Oxide</th>
<th>Pressure Exponent at 70°F</th>
<th>Burning Rate at 1000 psi (70°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>200-600 psi</td>
<td>800-1400 psi</td>
</tr>
<tr>
<td>Lead stannate</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.47</td>
<td>0.38</td>
</tr>
<tr>
<td>Crosslinked</td>
<td>0.54</td>
<td>0.55</td>
</tr>
<tr>
<td>Lead sesquioxide</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.63</td>
<td>0.71</td>
</tr>
<tr>
<td>Crosslinked</td>
<td>0.40</td>
<td>0.49</td>
</tr>
<tr>
<td>Lead dioxide</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.58</td>
<td>0.41&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Crosslinked</td>
<td>0.47</td>
<td>0.91</td>
</tr>
<tr>
<td>Lead oxide (yellow)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.71</td>
<td>0.96</td>
</tr>
<tr>
<td>Crosslinked</td>
<td>0.39</td>
<td>0.57</td>
</tr>
<tr>
<td>Lead tetraoxide</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.64</td>
<td>0.83</td>
</tr>
<tr>
<td>Crosslinked</td>
<td>0.46</td>
<td>0.63</td>
</tr>
<tr>
<td>Lead sesquioxide-stannic oxide (30/70)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.36</td>
<td>0.19</td>
</tr>
<tr>
<td>Crosslinked&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.50</td>
<td>0.30</td>
</tr>
<tr>
<td>Lead stannate-TDI red (LSTR)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Uncrosslinked</td>
<td>0.41</td>
<td>-0.10</td>
</tr>
<tr>
<td>Crosslinked&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.38</td>
<td>0.19</td>
</tr>
</tbody>
</table>

<sup>a</sup> (600-800 psi)

<sup>b</sup> 0.75% TDI added (others 1.50%)
Fig 1 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$ / Solid I modifier (Table 2, Run 1) (C)
Fig 2 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$ / Solid I modifier (Table 2, Run 2) (C)
Fig 3 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/Solid I modifier (Table 2, Run 3) (C)
Fig 4 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO\textsubscript{3} Solid I modifier (Table 2, Run 4) (C)
Fig 5 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO₃/ 
Solid I modifier (Table 3, Run 1) (C) 3
Fig 6 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO₃, Solid 1,2,4-diiodo-6-methylphenol (Table 3, Run 2) (C)
Fig 7 (II) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO₃ with Solid I modifier (Table 3, Run 3) (C)
Fig 8 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_2$/Solid I modifier (Table 3, Run 4) (C)
Fig 9  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PLSnO$_3$/ Solid I modifier (Table 4, Run 1) (C)
Fig 10 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/Solid I modifier (Table 4, Run 2) (C)
Fig 11 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$ Solid I modifier (Table 4, Run 3) (C)
Fig 12  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/Solid I modifier (Table 4, Run 4) (C)
Fig 13 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO₃ / Solid I modifier (Table 4, Run 5) (C)
Fig 14  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO₃/ Solid I modifier (Table 4, Run 6) (C)
Diffraction Angle, $2\theta$

Fig 15 (U) Diffraction pattern of catalyst from pyrolysis of lead stannate with 5% Solid I (C)
Diffraction Angle, $2\theta^\circ$

Fig 16  (U) Diffraction pattern of catalyst from pyrolysis of lead stannate with 15% Solid I (C)
Diffraction Angle, 2θ°

Fig 17  (U) Diffraction pattern of catalyst from pyrolysis of lead stannate with 30% Solid I  (C)

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Fig 16 (U) Diffraction pattern of catalysts-lead stannate/15%
Solid I-pyrolyzed in 0.5 hours and 1.5 hours (C)
Fig 20 (U) Thermogram of a catalyst produced by pyrolysis of lead stannate and Solid I (C)
Fig 21 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/ SnO₂ modifier (Table 5, Run 1) (C)
Fig 22 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 2) (C)
Fig 23 (U) Burning rate curve of HMX (20% plastic) propellant containing pyrolyzed PbO/SnO$_2$ modifier (Table 5, Run 3) (C)
Fig 24 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO$_2$ modifier (Table 5, Run 4) (C)
Fig 25 (U) Burning rate curve of HMX (30%) plasticized propellant containing pyrolyzed PbO/SnO$_2$ modifier (Table 5, Run 5) (C)
Fig 26 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 6) (C)
Fig 27 (U) Burning rate curve of HMX (30\% I plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 7) (C)
Fig 28 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 8) (C)
Fig 29 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO$_2$ modifier (Table 5, Run 9) (C)
Fig 30 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/ShO₂ modifier (Table 5, Run 10) (C)
Fig 31 (U) Burning rate curve of HMX (36%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 11) (C)
Fig 32  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 12) (C)

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Fig 33 (U) Burning rate curve of HMX (30\%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 13) (C)
Fig 34 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO$_2$ modifier (Table 5, Run 14) (C)
Fig 35 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/ SnO₂ modifier (Table 5, Run 15) (C)
Fig 36  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbO/SnO₂ modifier (Table 5, Run 16) (C)
Fig 37 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$ and urea modifier (Table 6, Run 1) (C)

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Fig 38  (U) Burning rate curve of HMX (30\%) plastisol propellant containing pyrolyzed PbSnO$_3$ / phenyl urea modifier (Table 6, Run 2) \(^3\) (C)
Fig 39 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO3/ diphenyl urea modifier (Table 6, Run 3) (C)
Fig 40 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/ethyl centralite modifier (Table 6, Run 4) (C)
Fig 41 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/TDI-urea modifier (Table 6, Run 5) (C)
Fig 42 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/hexamethylene modifier (Table 7, Run 1) (C)
Fig 43 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/methylene-di-$p$-phenylene modifier (Table 7, Run 2) (C)
Fig 44 (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/10% TDI modifier (Table 7, Run 3) (C)
Fig 45 (U) Burning rate curve of HMX (16.5%) plastic propellant containing pyrolyzed PbSnO$_3$/15% TDI modifier (Table 7, Run 1) (C)
Fig 46  (U) Burning rate curve of HMX (30%) plastisol propellant containing pyrolyzed PbSnO$_3$/20% TDI modifier (Table 7, Run 5) (C)
Fig. 4: (U) Burning rate curve of HMX (40%) plastisol propellant with lead dioxide modifier (C)
Fig 49 (U) Burning rate curve of HMX (40%) plastisol propellant with lead sesquisoxide-stannic oxide (30/70) modifier (C)

1. Crosslinked
2. Uncrosslinked

pHg, sec

Burning Rate, in/sec

Pressure, psi (10^-2)

1.00
0.80
0.60
0.40
0.20
0.00
-0.20
-0.40
-0.60
-0.80
-1.00
-1.20
-1.40
-1.60
-1.80
-2.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
26.00
28.00
30.00
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Andrews Air Force Base, Maryland 20331  

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AN IMPROVED METHOD OF MANUFACTURE OF A BALLISTIC MODIFIER FOR SMOKELESS ROCKET PROPELLANTS (U)

(C) Several improved processes for the preparation of a ballistic modifier derived in this laboratory under the name lead stannate-TDI (tolylene diisocyanate reduced (LSTR) have been developed. Presented are the parameters of time, temperature, and concentration of reactants, the X-ray diffraction patterns of several modifiers, and the effects of catalysis on the combustion of an experimental nitramine plastisol formulation.

(U) Also included in the report is a comparison of the burning rate characteristics of crosslinked and uncrosslinked compositions containing various modifiers.
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<th>KEY WORDS</th>
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<td>Urea, phenyl urea, diphenyl urea, diethyl diphenyl urea (ethyl centralite)</td>
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