A New Small-Scale DDT Test for "Flash Compositions"

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**Supplementary Notes:**

See also ADM002313. Department of Defense Explosives Safety Board Seminar (34th) held in Portland, Oregon on 13-15 July 2010, The original document contains color images.
What Are Flash Compositions?

Flash Compositions represent a unique class of chemical compositions which:

- Are uniform powders which pass through a 40 mesh screen typically containing more than 40 percent by weight of certain finely divided oxidizing salts and more than 15 percent by weight of certain metallic particles, of which 100 percent are less than 53μ(microns)

- Those “oxidizing salts” include potassium perchlorate, potassium nitrate, barium nitrate and strontium nitrate.

- Those metallic particles include aluminum, magnesium and a 50/50 alloy called “Magnalium”.

- GIVE A POSITIVE RESULT IN THE PROPOSED “SMALL-SCALE DDT TEST”
<table>
<thead>
<tr>
<th>US Screen Size</th>
<th>Approximate Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 Mesh</td>
<td>400 microns (μ)</td>
</tr>
<tr>
<td>100 Mesh</td>
<td>150 &quot;</td>
</tr>
<tr>
<td>200 Mesh</td>
<td>75 &quot;</td>
</tr>
<tr>
<td>270 Mesh</td>
<td>53-54 &quot;</td>
</tr>
<tr>
<td>325 Mesh</td>
<td>44-45 &quot;</td>
</tr>
<tr>
<td>400 Mesh</td>
<td>37-38 &quot;</td>
</tr>
</tbody>
</table>
# Heats of Reaction For Selected Oxidizers and Metallic Fuels at Ideal Weight Ratios

<table>
<thead>
<tr>
<th>Oxidizer/Fuel</th>
<th>Weight Ratio</th>
<th>Kcal/gram</th>
</tr>
</thead>
<tbody>
<tr>
<td>KClO₄/Mg</td>
<td>59/41</td>
<td>2.44</td>
</tr>
<tr>
<td>KClO₄/Al</td>
<td>66/34</td>
<td>2.54</td>
</tr>
<tr>
<td>KNO₃/Mg</td>
<td>63/37</td>
<td>1.76</td>
</tr>
<tr>
<td>KNO₃/Al</td>
<td>69/31</td>
<td>1.77</td>
</tr>
</tbody>
</table>

**BUT OPTIMUM ENERGETIC CONTENTS DON’T ALWAYS PRODUCE OPTIMUM KINETIC EFFECTS!**
Flash Compositions Have Unique Explosive Properties

- They will rapidly ignite and quickly transition to a detonation velocity of 1-3 Km/second, depending on diameter; and

- Either as unconfined substances or when incorporated into articles, they potentially present a “high order” explosive hazard whether initiated by a detonator or igniter.
How to Develop a Test Method For Flash Compositions?

- **First Attempt** – the UK Health and Safety Executive Branch modified the UN Test 2 C(i) Test – A Time-Pressure Test which measures the rate of pressure rise from 0 to 2070 psi of 0.5 grams of substance in an autoclave with a piezo gauge coupled to a transient event recorder for the first 10-15 milliseconds of the reaction. Adopted by the UN Committee of Experts in 2008.

- **Limitations** – High standards of deviation in the 0-8 millisecond range. Method does not distinguish between ultra-fine black powders and typical flash compositions.
A New Approach to Characterizing Flash Compositions -- Use Deflagration-to-Detonation (DDT) Properties!

- Modify the UN Test Method 5a Set-up and Criteria for Measuring Damage Results to a 1mm thick steel witness plate beneath a pint size cardboard container of sample ...

- Replace the detonator with an standard electric match igniter inserted in the top-center of the test powder

- Reduce the sample size grams by decreasing the diameter and/or height of the test sample container.

- Provide additional confinement by using a convolute paper tube and surrounding that with a heavy steel confinement of slightly larger diameter.
QUESTIONS TO BE ADDRESSED BY THIS STUDY

• Is the proposed test method reproducible?
• Is it practical?
• What role does oxidizer-fuel ratio play in making a flash composition?
• What particle size range of metallic fuels make them more or less likely to produce a flash composition?
• Are there similar particle size effects in the oxidizer salts?
• Are there “non-metallic” fueled flash compositions?
After Trial & Error With 70 gram and 35 gram quantities with little or no confinement...

- Sample weight was reduced to 25 grams.
- Sample Tube Cardboard Wall Thickness increased from 0.06” to 0.15” so that the outer diameter was 38 mm (1.3 inches)
- Sample Diameter reduced to 25 mm (1 inch) and sample height increased to approx. 75 mm (3 inches)
- Steel Confinement Sleeve reduced from 3.25 inches I.D. to 1.5 inches I.D. and Collar Height matched to Sample Tube.
- Steel Confinement Sleeve Wall Thickness increased to 1.25 inches (strictly for wear and tear and flight resistance) –
“Second Generation” DDT Test Apparatus
“Second Generation” Test Fixture Results – 25 Gram Samples

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Quantity</th>
<th>Composition</th>
<th>Additional Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>164</td>
<td>25 grams</td>
<td>70% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /30% Flake Aluminum “A” (92% &lt; 148 μ and 34% &lt; 44 μ)</td>
<td>(+) Perforated plate (See Photo)</td>
</tr>
<tr>
<td>165</td>
<td>25 grams</td>
<td>65% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /35% Flake Aluminum “A” (92% &lt; 148 μ and 34% &lt; 44 μ)</td>
<td>(+) Perforated plate</td>
</tr>
<tr>
<td>166</td>
<td>25 grams</td>
<td>70% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /30% Flake Aluminum “B” (100% &lt; 105 μ and 39% &lt; 53 μ)</td>
<td>(+) Perforated plate</td>
</tr>
<tr>
<td>167</td>
<td>25 grams</td>
<td>65% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /35% Flake Aluminum “B” (92% &lt; 105 μ and 39% &lt; 53 μ)</td>
<td>(+) Perforated plate</td>
</tr>
<tr>
<td>168</td>
<td>25 grams</td>
<td>70% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /30% Flake Aluminum “C” (99% &lt; 74 μ and 40% &lt; 44 μ)</td>
<td>(+) Perforated plate</td>
</tr>
<tr>
<td>169</td>
<td>25 grams</td>
<td>65% Ultra-fine Potassium Perchlorate (100% &lt;37 μ) /35% Flake Aluminum “C” (99% &lt; 74 μ and 40% &lt; 44 μ)</td>
<td>(+) Perforated plate</td>
</tr>
</tbody>
</table>

* “As received” Potassium Nitrates and Potassium Perchlorates contains 2% fumed silica and was measured to have an approximate particle size distribution of 97% < 74 μ and 37% < 37μ.
Witness Plates for Tests 164-169 – 25 Gram Samples
“Third Generation” Flash Composition Test Fixture Development

- Would the results be influenced by additional confinement on the top of the steel sleeve?
- Wanted to make the test fixture somewhat similar the UN Test Series 5B Deflagration To Detonation (DDT) Tests (i) or (ii); BUT
- Wanted to keep the low sample size (25 grams) and the inner cardboard sample container; AND
- Wanted to keep the 1 mm thick witness plate and same “pass-fail” criteria as UN Test Series 5A
- Wanted to be able to use the Test Fixture repeatedly!
“Third Generation” Flash Composition Test Fixture

- 25-mm Paper Plug
- Steel Sleeve, 38-mm i.d., 100-mm o.d., 165-mm long, bored from solid billet
- Electric Ignitor
- Substance to be tested, about 75-mm deep
- Tube, Convoluted Kraft Paper, 25-mm i.d., 3.8-mm wall, 150-mm long
- 25-mm Paper Plug
- Groove in Sleeve for Ignitor wires
- Witness Plate, Steel, 160-mm x 160-mm x 1.0-mm
- Steel Ring, 93.5-mm i.d., 100-mm o.d., 50-mm long
- Steel Plate, 152-mm x 152-mm, 25-mm thick
“Third Generation” Flash Composition Test Fixture
“Third Generation” Test Fixture Results

- **Test 1**: Goex Black powder 5Fa “Unglazed”
- **Test 2**: A Typical Non-Metallic “Breaking” or “Bursting” Charge consisting of:
  
  35% Potassium Nitrate (100% < 37 µ); 31% Potassium Perchlorate (100% < 37 µ) 13.5% Potassium Benzoate (fine powder); 10% Sulfur (fine powder) and 10.5% Lampblack (nano-material).

- **Test 3**: A Typical “White Sparkling” Composition consisting of:

  70% Potassium Perchlorate -- (100% < 37 µ)

  30% “Semi-coarse” Magnesium powder -- (297µ<25%>149µ; 148µ<58%>53µ; 52µ<5%>44µ; 12%<43µ)
# 1 Slight Indentations – Negative Result (Pass)

#2 Slight Indentations – Negative Result – 3 trials (Pass)

#3 Tear Perforation – (Fail)
“Third Generation” Test Fixture Results

• **Test 4**: An Mg – PP Flash Composition “A” consisting of:
  - 65% Potassium Perchlorate (100% < 44µ)
  - 35% Magnesium (105µ <15%>74µ; 73µ < 39%>44µ; 46%<43µ)

• **Test 5**: An Mg – PP Flash Composition “B” consisting of:
  - 65% Potassium Perchlorate (100% < 44µ)
  - 35% “Ground” Magnesium (100% <43µ)

• **Test 6**: An Al-PP Flash Composition consisting of:
  - 70% Potassium Perchlorate -- (100% < 37 µ)
  - 30% “Atomized” Aluminum powder -- (74µ<2.4%>53µ; 52µ<2.9%>44µ; 94.7%<44µ)
#4 Perforation Positive Result – 2 of 3 (Fail)

#5 Perforation Positive Result-1 Trial (Fail)

#6 Perforation Positive Result-1 Trial (Fail)
“Third Generation” Test Fixture Results

- **Test 7**: An Al – PP Flash Composition consisting of:
  
  65% Potassium Perchlorate (100% < 44µ)
  
  35% “Flake” Aluminum “A” (105µ <72%>53µ; 52µ <17%>44µ; 11.5%<43µ)

- **Test 8**: An Al – PP Flash Composition consisting of:
  
  65% Potassium Perchlorate (100% < 44µ)
  
  35% “Flake ” Aluminum “B” (74µ<39% >53µ; 52µ<22%>44µ; 40%<43µ)

- **Test 9**: An Mg/Al-PP Flash Composition consisting of:
  
  70% Potassium Perchlorate -- (100% < 37 µ)
  
  30% “Ground” Magnalium powder --
  (74µ<37%>53µ; 52µ<11%>44µ; 52%<44µ)

- **Test 10**: An Barium Nitrate-Al/S Composition consisting of:
  
  68% Barium Nitrate -- (105µ < 10% > 74 µ;
  
  73 µ<12%>44 µ; 43 µ < 24%>37 µ; 53%<37 µ)
  
  23% “Dark Flake” Aluminum (100%< 73 µ)
  
  9% Sulfur (fine powder)
#7 Perforation (Positive) – 1 Trial (Fail)

#8 Vertical Tear (Positive) – 1 Trial (Fail)

#9 Deep indent with perforation at bottom (Positive) – 1 Trial

#10 Deep Indents (Negative) – 3 Trials (Pass)
### SUMMARY TABLE OF EXAMPLES

<table>
<thead>
<tr>
<th></th>
<th>Example Description</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Goex Black powder 5Fa “Unglazed”</td>
<td>( - )</td>
</tr>
<tr>
<td>2</td>
<td>“Burst Charge” of 35% Potassium Nitrate (100% &lt; 37 µ)/ 31% Potassium Perchlorate (100% &lt; 37 µ) /13.5% Potassium Benzoate (fine powder)/ 10% Sulfur (fine powder)/10.5% Lampblack (nano-material).</td>
<td>( - )</td>
</tr>
<tr>
<td>3</td>
<td>70% Potassium Perchlorate -- (100% &lt; 37 µ) / 30% “Semi-coarse” Magnesium powder -- (297µ&lt;25%&gt;149µ; 148µ&lt;58%&gt;53µ; 52µ&lt;5%&gt;44µ; 12%&lt;43µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>4</td>
<td>65% Potassium Perchlorate (100% &lt; 44µ)/ 35% Magnesium (105µ 5%&gt;74µ; 73µ &lt;39%&gt;44µ; 46%&lt;43µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>5</td>
<td>65% Potassium Perchlorate (100% &lt; 44µ)/ 35% “Ground” Magnesium (100% &lt;43µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>6</td>
<td>70% Potassium Perchlorate -- (100% &lt; 37 µ)/ 30% “Atomized” Aluminum powder (74µ&lt;2.4%&gt;53µ; 52µ&lt;2.9%&gt;44µ; 4.7%&lt;44µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>7</td>
<td>65% Potassium Perchlorate (100% &lt; 44µ)/ 35% “Flake” Aluminum “A” (105µ &lt;72%&gt;53µ; 52µ &lt;17%&gt;44µ; 11.5%&lt;43µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>8</td>
<td>65% Potassium Perchlorate (100% &lt; 44µ)/35% “Flake ” Aluminum “B” (74µ&lt;39% &gt;53µ; 52µ&lt;22%&gt;44µ; 40%&lt;43µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>9</td>
<td>70% Potassium Perchlorate -- (100% &lt; 37 µ)/ 30% “Ground” Magnalium powder --(74µ&lt;37%&gt;53µ; 52µ&lt;11%&gt;44µ; 52%&lt;44µ)</td>
<td>( + )</td>
</tr>
<tr>
<td>10</td>
<td>68% Barium Nitrate (105µ &lt; 10% &gt; 74 µ; 73 µ&lt;12%&gt;44 µ; 43 µ &lt; 24%&gt;37 µ; 53%&lt;37 µ)/23% “Dark Flake” Aluminum (100%&lt; 73 µ)/9% Sulfur (fine powder)</td>
<td>( - )</td>
</tr>
</tbody>
</table>
Future Work:

• Last month, a UN Working Paper presented findings of the Modified UN 5B DDT Test for Flash Compositions to UN Subcommittee of Experts and requesting review and inviting international round-robin tests.