Defining Homogeneity for Medium Caliber Ammunition and Small Grain Propellant Lots

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Background

• 30mm GAU-8/A gun system requires potassium (K) salts to prevent secondary gun gas ignition (SGGI) at the muzzle
  – 1.7% K salts minimum required in each cartridge (lower performance limit)

• Propellant requirements
  – 1.90% to 2.20% K salts
  – Specification allowed averaging of samples to determine final value
  – Homogeneity requirement poorly defined

• Use distribution of flash suppressant (FS) grains to determine homogeneity
  – Propellant contains ~3% by weight of FS grains to adjust K salts
• **Baseline determined prior to beginning investigation**
  
  – 3 x 30 g samples pulled every 5000 lb.
  – Analyzed by Atomic Absorption Spectroscopy (AAS)
  – Average = 2.10% K salts (SD = 0.15%)
  – Relative SD = 7.1%
Historical Ambiguity of Homogeneity

- Webster’s Dictionary
  - “Of the same or a similar kind.”

- MIL-STD-1168B for Ammunition Lot Numbering and Ammunition Data Card
  - “When all units of product in an ammunition lot have been produced by one manufacturer, in one unchanged process, under stable conditions of production, in accordance with the same drawings, the same specifications and any revisions thereto, a ‘state of homogeneity’ shall exist.”

- 30mm propellant specification DS 8560
  - “The procedure utilized for the blending of propellant, sublots and/or batches to form a lot shall result in a homogeneous mixture.”

A new quantitative and qualitative definition of homogeneity is needed
• ATK and Hill Air Force Base (HAFB) teamed to write a new definition
  – “The procedure utilized for the blending of propellant sublots and/or batches to
    form a lot shall be such that random samples have the same characteristics as
    the entire lot. “

• With the qualitative definition completed, unanswered questions remained
  for the quantitative definition
  – What is the ideal sample size?
  – What analytical method would be used?
  – How would the sample be obtained?
  – How many samples will be needed?

Find a model to measure homogeneity
Basis for Homogeneity Parameters

- **ATK retained the services of Jenike & Johanson (J & J)**
  - A bulk solids flow engineering consulting firm
  - Extensive background in blending
  - Pharmaceutical industry experience
  - Involved in Federal Court case to define homogeneity

- **FDA definition utilized as starting point**
  - FDA: Finished product at USP I stage testing
    - Sample 30 units and test 10 (N = 10)
    - Limits
      - Mean within 90-110%
      - Relative standard deviation (RSD = $\sigma$/avg.) $\leq$ 6%
      - All individual results are within range of 85 to 115% of label claim

**FDA requirement is tighter than past practice**
Determining a Sufficient Sample Size

- Sample size effects analytical results
  - The smaller the sample size, the greater the effect of each FS grain

<table>
<thead>
<tr>
<th>Total K Salts</th>
<th>Number of FS Grains in Sample (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
</tr>
<tr>
<td>2.25%</td>
<td>31.8</td>
</tr>
<tr>
<td>2.20%</td>
<td>31.0</td>
</tr>
<tr>
<td>2.05%</td>
<td>28.4</td>
</tr>
<tr>
<td>1.90%</td>
<td>25.8</td>
</tr>
<tr>
<td>1.85%</td>
<td>24.9</td>
</tr>
</tbody>
</table>

Need to determine the ideal sample size that meets the requirements
Ideal Sample Size

- Theoretical calculation to predict the distribution of K salts for a statistically homogeneous mixture

A 1 lb. sample meets the requirements
Quantitative Analysis of Propellant Samples

• Past practice was to use AAS for potassium salt analysis
  – Two issues identified
    1. Presence of FS grains increased SD
    2. Sample size is limited – 50 gram max

<table>
<thead>
<tr>
<th>Sample Size</th>
<th>FS Present</th>
<th>Number of Samples</th>
<th>Average (%)</th>
<th>Minimum (%)</th>
<th>Maximum (%)</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 g</td>
<td>No</td>
<td>9</td>
<td>0.42</td>
<td>0.41</td>
<td>0.45</td>
<td>0.01</td>
</tr>
<tr>
<td>10 g</td>
<td>Yes</td>
<td>9</td>
<td>2.13</td>
<td>1.61</td>
<td>2.47</td>
<td>0.23</td>
</tr>
<tr>
<td>50 g</td>
<td>Yes</td>
<td>5</td>
<td>2.10</td>
<td>1.82</td>
<td>2.32</td>
<td>0.19</td>
</tr>
</tbody>
</table>

An improved analytical method is needed
Low-Tech Can be Better than High-Tech

• FS grains contain potassium – an alkali metal
  – FS grains "light up" when exposed to x-ray
  – Positives
    - High reliability and repeatability – 99%
    - Sample size up to 150 g
  – Negatives
    - FS weight is estimated during calculations
    - Spreading sample into a mono-layer

• Desire a method to determine actual weight of components
  – Solvent system of a density to separate FS grains from propellant grains
  – Components can be dried and weighed

Density separation analytical method is most desirable
Sampling is as Important as the Analysis

- J & J stressed the importance sampling on the final results
  - Past practice was to sample using a scoop (highest error)

- Now all samples are obtained with a sample splitter and rotary riffler
  - Sample splitter can handle drum-size quantities
  - Rotary riffler divides sample into 8 sub-samples
• Past practice was to randomly select 5 drums from the propellant lot for acceptance testing per specification
  – A propellant lot could consist of up to 1,500 drums
  – 50 drums of propellant are packed through each final blend cycle

• Studies were conducted to prove the propellant is homogeneous within a blend cycle
  – Randomly select a drum from each final blend cycle
  – Each drum sampled for analysis

The number of samples shall be the greater of 20 samples per lot or 1 sample per 5,000 lb. of propellant


• The quantitative definition of homogeneity
  – All acceptance samples to be obtained using a sampling device
  – Analysis to be conducted using x-ray or density separation analysis
  – Population limits added to ensure propellant is above 1.7% K salts

<table>
<thead>
<tr>
<th>Criteria Parameters</th>
<th>Sample Size (g)</th>
<th>Specification Range per Sample</th>
<th>Relative Standard Deviation</th>
<th>Maximum Population SD (%)</th>
<th>Population Limits for Lot x(bar) - 3SD</th>
<th>x(bar) + 3SD</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>% K Salt</td>
<td>454</td>
<td>1.9 - 2.2%</td>
<td>4.0</td>
<td>0.08</td>
<td>&gt;1.8%</td>
<td>&lt;2.3%</td>
<td>Greater of 20 per lot or 1 per 5000 lb.</td>
</tr>
</tbody>
</table>

In the end, the requirements for homogeneity are tighter for propellant than those required by the FDA for the pharmaceutical industry!
• Implementation of process improvements have proven to be effective
  – All propellant samples meet the specification
  – Customer confidence in propellant has increased
  – Muzzle SGGI has been reduced to desirable levels

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<th>Range of Samples</th>
<th>Relative Standard Deviation</th>
<th>Maximum Population SD (%)</th>
<th>Population Limits for Lot</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Requirements</td>
<td>% K Salt</td>
<td>454</td>
<td>1.9 - 2.2%</td>
<td>4.0</td>
<td>&gt;1.8%</td>
<td>&gt;2.3%</td>
</tr>
<tr>
<td>Results</td>
<td>Past Practice</td>
<td>30</td>
<td>1.76-2.37%</td>
<td>7.1</td>
<td>1.66%</td>
<td>2.55%</td>
</tr>
<tr>
<td></td>
<td>Current Practice</td>
<td>454</td>
<td>1.93-2.13%</td>
<td>2.9</td>
<td>1.85%</td>
<td>2.21%</td>
</tr>
</tbody>
</table>
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Current Practices

• An example of a current production lot
  – 1 lb. sample pulled every 5000 lb.
  – Analyzed by x-ray
  – Average = 2.03% K salts (SD = 0.06%)

Variation reduced by more than 50%
Acknowledgements

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