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Defining Homogeneity for Medium Caliber Ammunition and Small Grain Propellant Lots

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Background

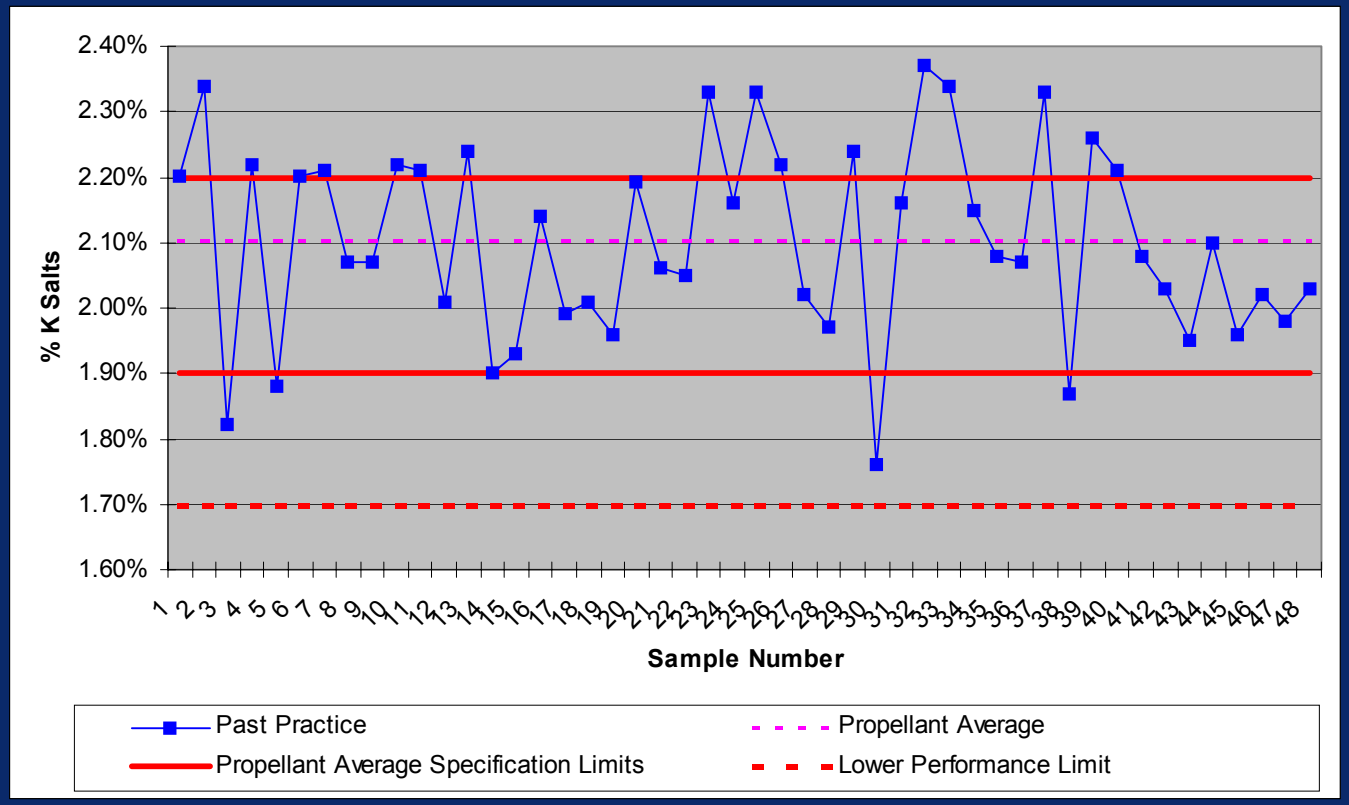
- **30mm GAU-8/A gun system requires potassium (K) salts to prevent secondary gun gas ignition (SGGI) at the muzzle**
  - 1.7% K salts minimum required in each cartridge (lower performance limit)

- **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

Past Practices

- **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

Proposed Definition of Homogeneity

- **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 = σ/avg.) ≤ 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

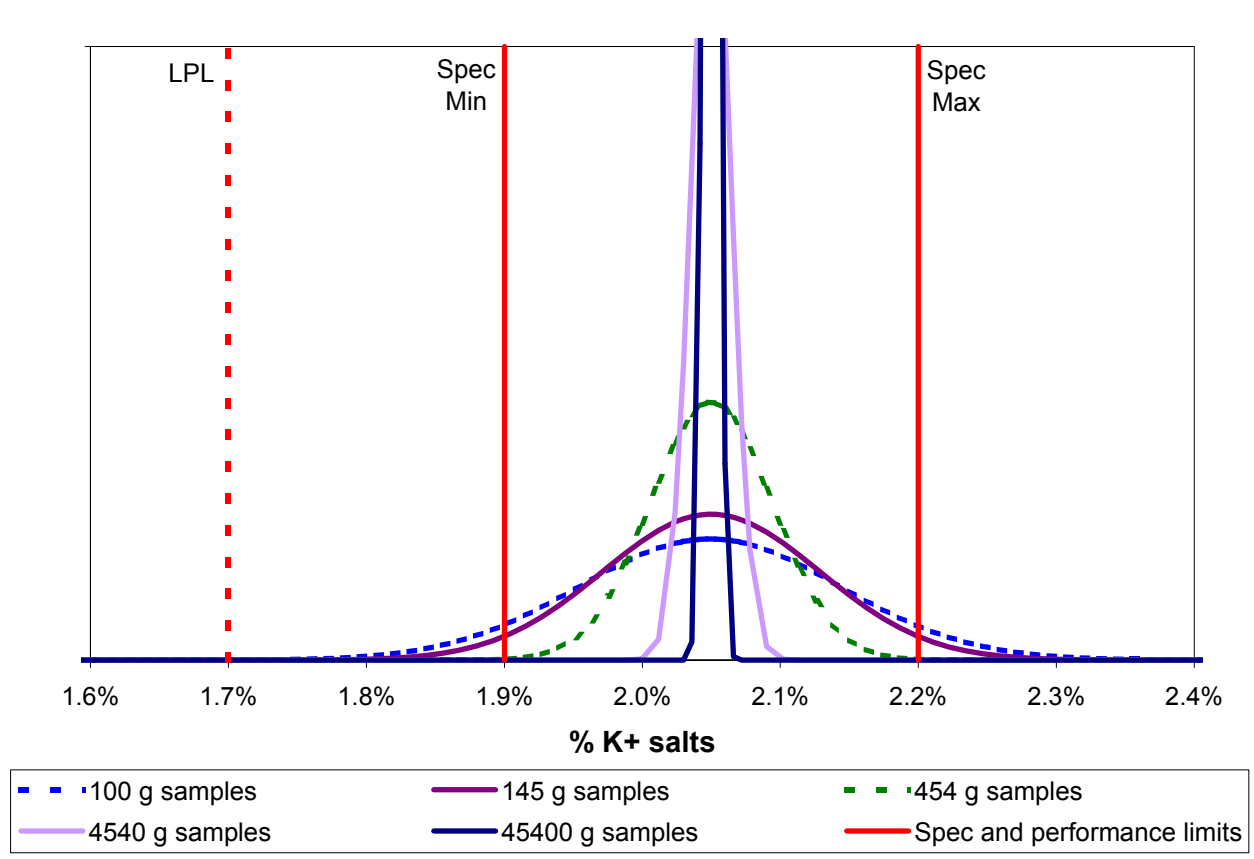
- **Sample size effects analytical results**
  - The smaller the sample size, the greater the effect of each FS grain

Total K Salts	Number of FS Grains in Sample (g)						
	10	50	100	145	454	4540	45400
2.25%	31.8	159.1	318.2	461.4	1444.6	14446.2	144461.6
2.20%	31.0	154.8	309.6	448.9	1405.6	14055.7	140557.3
2.05%	28.4	141.9	283.8	411.5	1288.4	12884.4	128844.2
1.90%	25.8	129.0	258.0	374.1	1171.3	11713.1	117131.1
1.85%	24.9	124.7	249.4	361.6	1132.3	11322.7	113226.7

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

Sample Size	FS Present	Number of Samples	Average (%)	Minimum (%)	Maximum (%)	SD
10 g	No	9	0.42	0.41	0.45	0.01
10 g	Yes	9	2.13	1.61	2.47	0.23
50 g	Yes	5	2.10	1.82	2.32	0.19

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

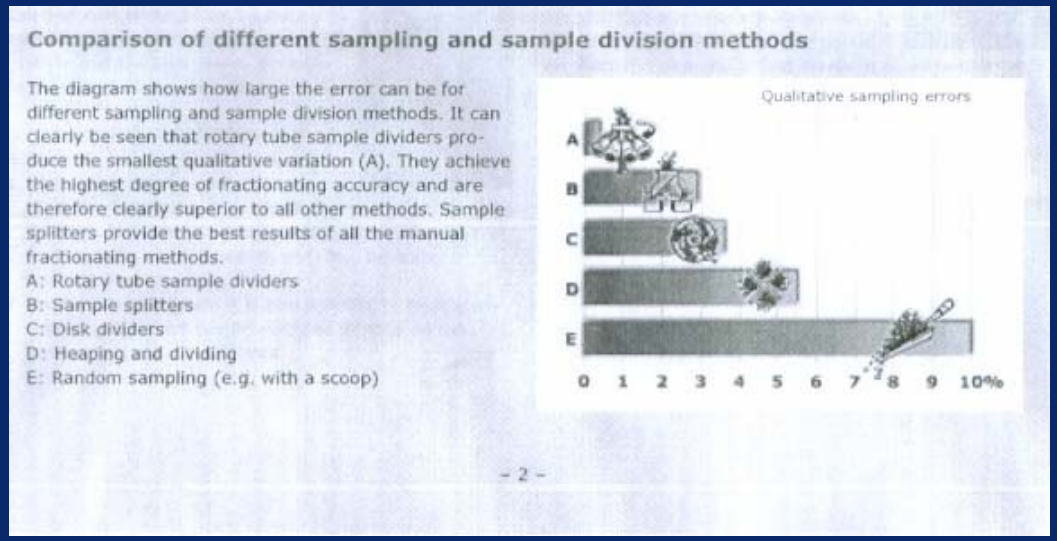
- **Desire a method to determine actual weight of components**
  - Solvent system of a density to separate FS grains from propellant grains
  - Components can be dried and weighed

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



Comparison of different sampling and sample division methods

The diagram shows how large the error can be for different sampling and sample division methods. It can clearly be seen that rotary tube sample dividers produce the smallest qualitative variation (A). They achieve the highest degree of fractionating accuracy and are therefore clearly superior to all other methods. Sample splitters provide the best results of all the manual fractionating methods.

A: Rotary tube sample dividers
B: Sample splitters
C: Disk dividers
D: Heaping and dividing
E: Random sampling (e.g. with a scoop)

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Proving Homogeneity Through Sampling

- **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

Final Parameters for Measuring Homogeneity

- **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

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

In the end, the requirements for homogeneity are tighter for propellant than those required by the FDA for the pharmaceutical industry!

Results of Homogeneity Improvements

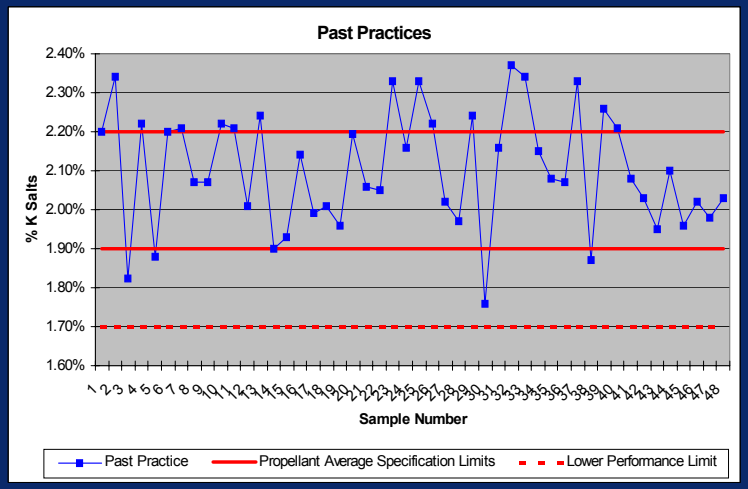
- **Implementation of process improvements have proven to be effective**
  - All propellant samples meet the specification
  - Customer confidence in propellant has increased
  - Muzzle SGGI has been reduced to desirable levels

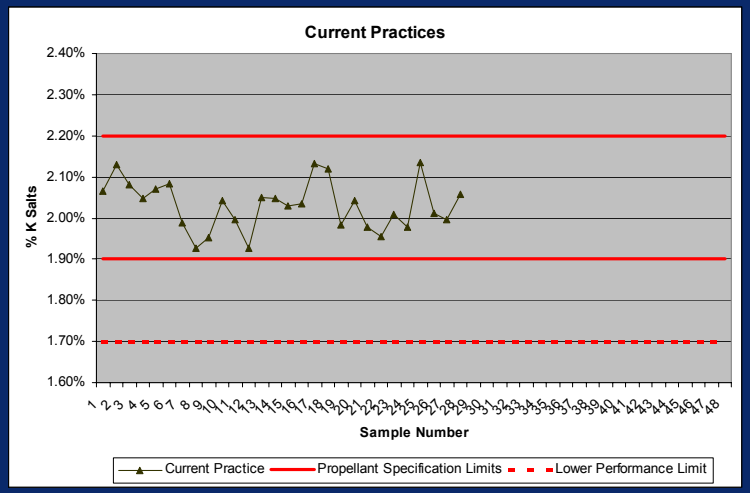
	Criteria / Parameters	Sample Size (g)	Range of Samples	Relative Standard Deviation	Maximum Population SD (%)	Population Limits for Lot x(bar) - 3SD	Population Limits for Lot x(bar) + 3SD	Number of Samples
Requirements	% K Salt	454	1.9 - 2.2%	4.0	0.08	>1.8%	<2.3%	Greater of 20 per lot or 1 per 5000 lb.
Results	Past Practice	30	1.76-2.37%	7.1	0.15	1.66%	2.55%	48
	Current Practice	454	1.93-2.13%	2.9	0.06	1.85%	2.21%	28

ATK has packed over 500,000 lb. of homogeneous propellant

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%

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