Nitrocellulose Specification:
In need of Analytical Resuscitation

A. Faburada, Armtec Defense
N. Campesi, ARDEC
N. Eldredge, ARDEC
L. Fishbein. ARDEC
L. Lopez, ARDEC
B. Mrazek, St. Marks
K. Murphy, ARDEC
H. Raines, St. Marks
J. Rutkowski, ARDEC
M. Paquet, Expro TEC
T. Williams, ATK RFAAP
N. Zink, ARDEC
C. Zisette, ATK RFAAP

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- Introduction
  - Presentation goals
  - Contents of MIL-DTL-244
  - What is Nitrocellulose
  - Why change the specification
- Progress of the Integrated Product Team
  - Options available for specification change
  - Current test methods
  - Critique of analytical techniques
  - Proposed specification changes
  - Validation process
- Summary

MIL-DTL-244B WAS a great document that focused on NC stability/testing and not on propellant performance

Resuscitating Nitrocellulose Specification

- Inform the DoD community of the IPT's intent
  - Analytical upgrade of the NC military specification
- Enhance the community-wide understanding of nitrocellulose and its influence on end-item performance

Program will serve as a model for future modifications to military specifications

What is MIL-DTL-244?

- MIL-DTL-244 originated 1940's
  - Analytical knowledge from late 19th & early 20th century
- Describes the NC requirements
- Primarily a Performance Specification
  - Sole manufacturing section
    - Stabilization of NC
  - Analysis section details test methods and desired chemical/physical properties

Nitrocellulose: Long and Storied Existence

- Discovered in the mid-1800's
  - First synthesized by Schönbein, highly unstable
  - Abel perfected the purification process allowing "safe" manufacture
    - First Application: Black powder replacement
    - Other: Early photographic film
- Primary component in 1st commercially available thermoplastic
  - Celluloid – 70% NC & 30% Camphor
    - Applications: Billiard and table tennis balls, toys, fountain pens, knife handles
- Current Applications
  - Lacquers, Nail polish, Filter Media, Energetic products across entire DoD agency

Updating the analytical methods in MIL-DTL-244 will enhance all energetics programs

- Preserve community knowledge
  - Corporate/Government wisdom of NC properties and processability needs to be retained
- Enhance technical understanding of nitrocellulose
  - Link between NC and propellant processability/performance
  - Use analytical data to drive the NC process
- Improved analytical reliability
  - Better characterization techniques results in improved laboratory precision and efficiencies

Several options for updating a military specification were considered

- Specification rewrite options available
  - Performance Specification
    - Too general, Limited control
  - Army Specific Specification
    - Time consuming process, No association with MIL-DTL-244B
  - Amendment
    - Extensive coordination effort
  - Interim Amendment
    - Army applications, Linked to MIL-DTL-244, Less time extensive

IPT Best Option: Interim Amendment

Analytical methods in MIL-DTL-244

Test Method	Analytical Significance
Nitrogen Content	Measure degree of nitrate ester substitution
Ether-Alcohol Solubility	Establish low N_2 NC content and possible contamination
Acetone Insoluble	Purity test
Ash	Accounts for non-combustible impurities
Viscosity	Indirect measure of degree of polymerization
Fineness	Indirect measure of fiber length
134.5°C Heat Test	Direct of measure of NC stability, Acidic species
65.5°C Heat Test	Direct of measure of NC stability, Neutralization salts
H_20 and Alcohol Content	Quantification of total moisture and volatiles

Analytical methods built on turn-of-the-century technology

Test Method	Strength of Analytical Method	Weakness of Analytical Method
Nitrogen Content	▪ Simple, reliable semi-automated titration	▪ Stability concerns of titrant ▪ Test variability reduces manufacturing options
Ether-Alcohol Solubility	▪ Simple analysis ▪ Good indicator of manufacturing issue	▪ Low precision ▪ Does not truly reveal the processability of the NC blend ▪ Requires specialized glassware
Viscosity	▪ Simple analysis	▪ Not "true" dilute solution characterization
Fineness	▪ Simple and relatively quick analysis ▪ Best method available at the time	▪ No quantification of fiber length distribution ▪ Outcome does not quantify processability of NC
H_20 and Alcohol Content	▪ Simple gravimetric technique	▪ Time intensive ▪ Unable to differentiate H_20 from alcohol content

Analytical methods will improve understanding and guide manufacturing process

Test Method	Proposed New Method	Benefits
Nitrogen Content	▪ Investigate applicable titration technologies ▪ Improve experimental control	▪ Improved electrode response ▪ Reduction in experimental variability
Ether-Alcohol Solubility	▪ Gravimetrically determine insoluble fraction ▪ Develop rate of dissolution test	▪ Eliminate need for specialized glassware ▪ Understand relationship between NC and propellant manufacturing
Viscosity	▪ Implement dilute solution characterization techniques	▪ True molecular weight characterization
Fineness	▪ Computer controlled optical and image processing unit ▪ Quantify fiber dimensions	▪ Directly measure fiber properties ▪ Correlate with new solubility information
H_20 and Alcohol Content	▪ Implement moisture analyzing technology	▪ Real-time data ▪ Differentiate alcohol and water content

Validation will occur through extensive R&R studies, side-by-side analyses, and laboratory round robins

The purpose of this program is not to eliminate or simplify the specification

- MIL-DTL-244 should be made more specific and provide more guidelines
  - Implementing improved analytical tools are an important improvement
- The purpose of the analytical tools are:
  - Confirm compliance
  - Guide manufacturing process
  - Understand and predict performance