Improvements in Nitrocellulose Processing:
Using Near Infrared Spectroscopy to Determine Total Volatiles Content

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Outline

- **Importance of Nitrocellulose**
- **Nitrocellulose Processing**
- **Methods Comparison of NC Total Volatiles**
- **Basics of Near Infrared (NIR) Spectroscopy**
- **Advantages of NIR Methods**
- **Impact to Production**
- **Future Work**

Radford Army Ammunition Plant: home of AES

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Importance of Nitrocellulose

An advanced weapon and space systems company

Nitrocellulose is essential to modern war fighting and produced as a major commodity (Mlbs/year)

Nitrocellulose is the elementary component of almost all gun propellants

- Can be used as the only energetic material (Single Base)
- Mixed with nitrate esters (NG, DEGDN, TEGDN) to form double-base propellants
- Double-base plus nitramines (NQ, RDX, HMX) to form triple-base propellants

RFAAP is sole manufacturer in North America

No replacement on horizon

Nitrocellulose Characterization is difficult

- Traditional Analysis is inefficient and of limited utility

An Essential but Neglected Material that Requires
New Analytical Techniques to Improve Product Quality

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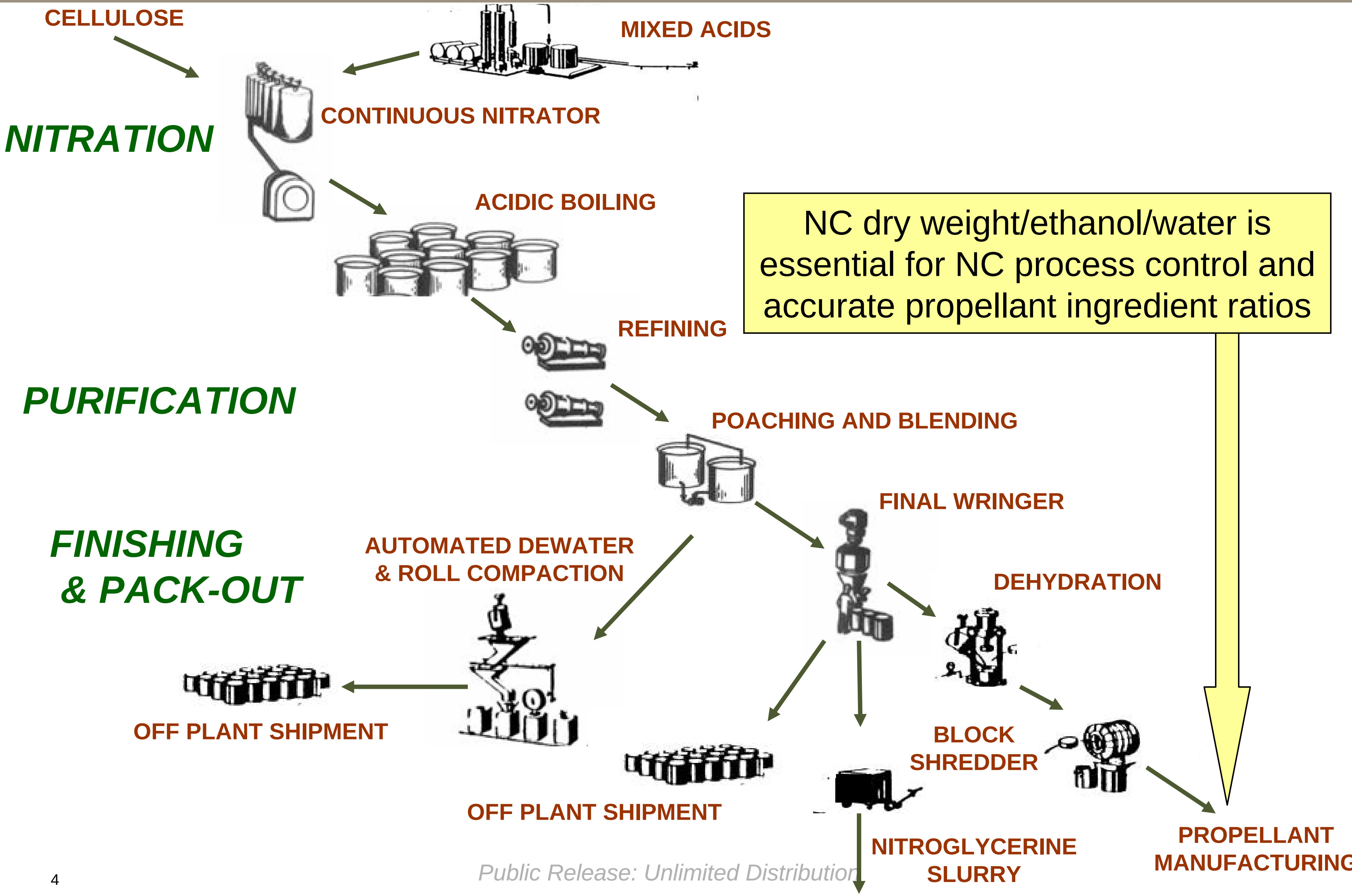
RFAAP Nitrocellulose Process



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Nitrocellulose Dehydration Process

All military grade nitrocellulose is processed and transported in water

- NC dehydration is a crucial step for propellant processing

Dehydration is a combination of pressing and water displacement by ethanol

The accurate determination of volatile levels (water & ethanol) is a limiting factor to process control

- Standard method is GC
  - Time consuming for in-process material, limited sample size, potential for volatiles loss

<u>NC Blending</u>
NC/water slurry

<u>NC Dehydration Press</u>
Exchanging water for EtOH

<u>Final Product</u>
Shredded EtOH wet NC

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NIR potential to replace GC

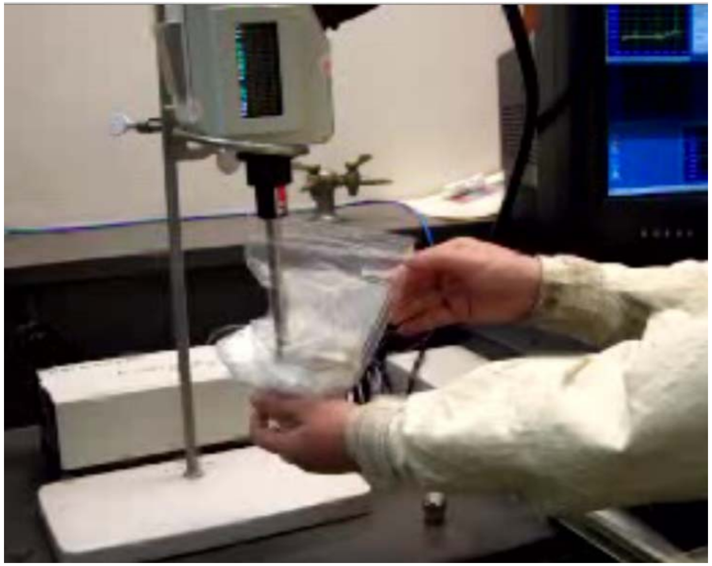
Near Infrared Spectroscopy uses response to electromagnetic radiation at wavelengths just beyond the visible spectrum (800-2500 nm)

First NIR commercial applications in the 1970s were for use in Agriculture (grains & cereal)

NIR energy penetrates into substances

allowing:

- testing of bulk samples (kg vs. mg)
- Increased speed (seconds vs. hours)
- Improved precision over alternative methods



NIR analysis of bulk NC samples

Current Instrumentation (AOTF-NIR)

- Acousto-Optic Tunable Filters to provide solid-state simplicity and reliability

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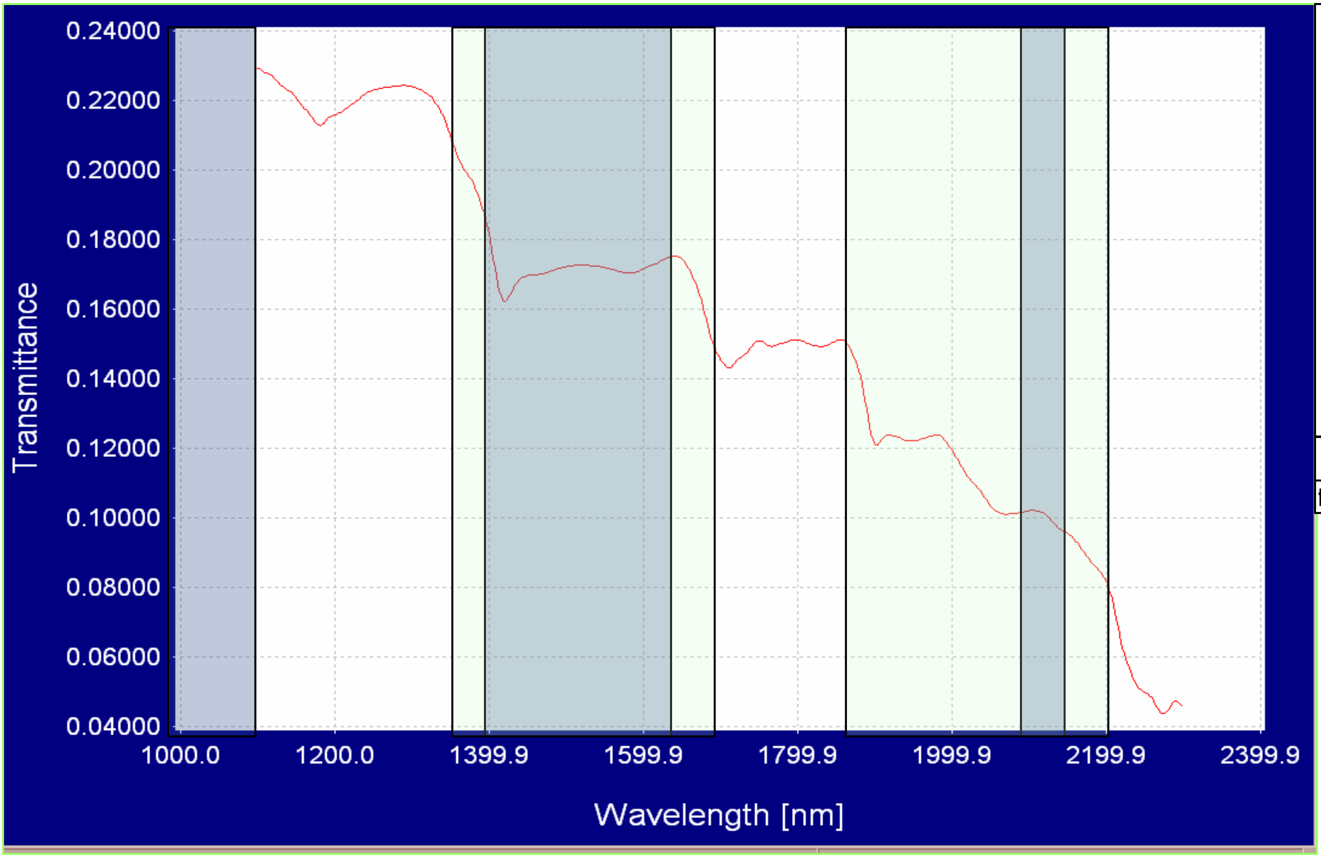
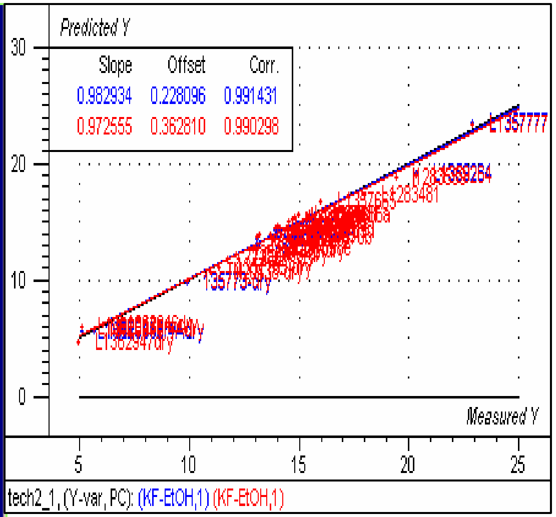
NIR Spectra – Dehy NC

NIR methods use statistical analysis of frequency response mapped to a known value to generate a calibrated model with which to quantify desired analytes.





Pooled Standard Deviations			
	% Water	**% Ethanol**	**% TV**
KF oven	0.03	0.35	0.36
GC TV	0.13	0.58	0.70

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Advantages/Challenges of NIR Method

Scale **– NIR samples are volume averaged over a bulk sample as opposed to pinpoint extracted samples for GC**

Speed **- 255 minutes (GC-MS) to 2 minutes (NIR) 120x faster to get an answer**

Precision **– Triplicate testing improves St. Dev.**

- 14% improvement in SD for Water analysis
- 24% improvement in SD for Ethanol analysis

Brinkmann/Metrohm KF Oven Instrumentation

Challenges

Calibration **- tied to Primary Method and Product**

Quality Control **– monitoring required using primary method**

- NIR method will always require representative samples for off-line analysis

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Nitrocellulose Analysis Comparison

"Was State" – >4hrs of production material at Risk

GC

Prepare Sample

Extraction - 2 hour

Instrumental analysis- 2 hours

Data Check and Entry

Clean Up

Total Analysis Time

4.25 hours

"Intermediate state"
used as primary standard for NIR

KF

Prepare Sample

Setup Instrument

Instrumental analysis - 12 minutes

Data Check and Entry

Clean Up

Total Analysis Time

30 minutes

"Is State"
Near Real Time

NIR

No Sample Preparation

- Open Sample Bag
- Insert NIR Probe into sample

Automated Data Transfer

Clean Up

- Wipe NC back into bag

Total Analysis Time

<2 minutes
(in triplicate)

No solvents and no waste

Method Progressively Improves

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Impact to Production

Effects on propellant processing

- **Improved yield**
- **Less reworked material**
- **Better final product quality**

Propellant Type	Nominal NG%	SD Reduction in NG Sublots	SD Reduction Extrusion Pressure	Density Variation Reduction	**Overall yield Improvement**
A	15	52%	6%	3%	**5.1%**
B	20	38%	7%	33%	**6.9%**
C	25	35%	-1%	31%	**2.2%**
D	35	3%	0%	19%	**1.0%**
E	40	28%	16%	27%	**3.8%**

Importance to final product

- **Greater consistency in delivered product**
- **Decreased round-round variation**
- **More capable weapons into the hands of the warfighters**

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Summary

- ***Transition*** made to rapid, highly accurate NC Total Volatiles analysis
- ***Reduced*** NC & Propellant production risk through near real time data
- ***Future Work -*** On-line assay of dehyed NC
  - Real time trending of in-process materials
  - Allows full Feed-Forward system control
  - Continuous improvement to provide the best possible products to soldiers on the front line

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