Advances in Propellant Stability Screening

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Funded under DAAE30-01-9-0800 TOSA 102

2007 Global Demilitarization Symposium & Exhibition
Grand Sierra Resort, Reno, NV, May 14-17, 2007
Integrated Research & Engineering Division

Background

Propellant stability screening in the field

- Real-time, non-destructive operation
- Operated by field personnel
- Eleven instruments
- Applicable to eleven propellant types:
  - M1-MP, M1-SP, M6-MP, M6+2, M8-S, M9-F, M10-SP, M14, M38, WC*, SPD*

Deployed in Multiple locations

- McAlester Army Ammunition Plant
- Kuwait – Needed real time stability results
  - Deployed to CFLCC
- Tooele Army Depot – WC propellant
  - Very limited OB/OD
- RDECOM-ARDEC
Objective

Evaluate New Propellant Stability Analysis Technologies

- Want the ability to transfer calibration curves electronically
  - Must calibrate all NIR spectrometers individually in the lab
  - Time consuming
  - Expensive
- Smaller sample size
- Ease of Operation
- Ease of transport

Must demonstrate electronic calibration transfer with SECV ≤ 0.07
## Technology / Instrument Comparison

<table>
<thead>
<tr>
<th></th>
<th>FOSS 5000</th>
<th>FOSS XDS</th>
<th>Buchi NIRFlex N-500</th>
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</thead>
<tbody>
<tr>
<td>TECHNOLOGY</td>
<td>NIR</td>
<td>NIR</td>
<td>Polarization FT-NIR</td>
</tr>
<tr>
<td>AGE</td>
<td>12</td>
<td>3</td>
<td>2</td>
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<td>AVAILABILITY</td>
<td>2 Years</td>
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<tr>
<td>SOFTWARE</td>
<td>Vision</td>
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<td>Internally Developed</td>
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<tr>
<td>CALIBRATION TRANSFER</td>
<td>No</td>
<td>Claimed</td>
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<td>EASE OF USE</td>
<td>Medium</td>
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<td>Transport Cell</td>
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<td>SOLID SAMPLES</td>
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<td>REQUIRED ACCESSORIES</td>
<td>Transport cells,</td>
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<td>Beakers</td>
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<td>external standards</td>
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### Evaluated
- Foss 5000
- Foss XDS
- Buchi NIRFlex N-500 FT-NIR
- Zeiss
- Perkin Elmer
Buchi NIRFlex N-500 – Key Points

- FT-NIR Polarizing Interferometer
- State of the Art, patented technology
- Fast - All frequencies are measured simultaneously and strike the detector at the same moment
  - high optical throughput
  - improved signal- to-noise ratio
- High Precision and Reproducibility
  - HeNe laser
  - Assures electronic calibration transfer
- Improved ruggedness
  - No moving gratings, etc.
- Sensitive in spectral range of interest
  - Better accuracy and precision
- Solids Module
  - Beaker, Petri dish, Bags
- Software
  - Operates equipment, records data
  - Calibration
    - Expert Wizard
- All internal standards

Technical Specifications

- Resolution (minimum) 8 cm⁻¹
- Lamp lifetime min 12’000h (2*6000h)
- Baseline drift < 0.5 %
- Wavelength Accuracy ± 0.2 cm⁻¹
- S/N p2p 1/10000
- Detector InGAs thermostated
- Laser HeNe

Interferometer

- real-world implementation

HeNe laser
wedge drive (voice coil)
laser oscillations for wedge speed control
scanning wedge
polarising beamsplitters
lamp unit
Buchi Spectrometer System

- Battery Backup
- Computer
- Spectrometer
- Sample Beaker

- Top, Front Mounted Rotating Plate
  - Rotates solid samples
  - Petri dishes, beakers, vials, bags
  - Relatively small sample size
  - Simple and easy operation
**Initial Calibration Studies - WC Propellant**

**WC propellant**: nitrocellulose, nitroglycerine, diphenylamine, calcium carbonate, sodium sulfate, potassium nitrate, dibutyl pthalate, and graphite

Propellant types in the WC calibration curve include: 814, 818, 819, 842, 844, 945, 846, 870, 872, 890

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42 Spectra, 21 samples  
Double scan  
About 1400 to 2500 nm  
Second derivative math pre-treatment

**Figure 3. Second derivative spectra of calibration samples after second derivative math treatment (15 point segment, 15 point gap)**
First calibration model met initial criterion with SECV ≤ 0.07

WC calibration model

- “Leave one out” calibration model
- PLS
- R=0.96
- SECV = 0.066
Calibration model from spectrometer 1 electronically transferred to spectrometer 2.

Spectra on spectrometer 1 nearly indistinguishable from spectra of same sample on spectrometer 2.

Figure 2. Sample 49519 as measured in the initial study compared to re-measurement on second N-500 system.
WC Sample %RES prediction on spectrometer 2 using calibration model from spectrometer 1

NIR-Lab ≤ 2*SECV; ≤ 0.14
### Results

- Demonstrated electronic calibration transfer
  - SECV ≤ 0.07
- Small sample size
- Rugged – few moving parts
  - Safer transport
- Fewer shipping cases
  - Easier to transport

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Plan

- Instrument and process validation
  - Establish precision, accuracy, and repeatability
  - Calibration model for all propellant types
  - Determine SECV for all propellant types
  - Confirm electronic calibration transfer
- Field trials
- PSSB approval
- Safety approval
- SOP and operation manual
- Training documentation

Propellant Types

- M1_MP
- M1_SP
- M6_MP
- M6+2
- M8_S
- M9_F
- M10_SP
- M14_MP
- M38
- SPDX
- WCXXX

SPDX
- SPD
- SPDB
- SPDN
- SPDW
- SPWF

WCXXX
- 814
- 818
- 819
- 842
- 844
- 845
- 846
- 847
- 870
- 872
- 890
Presenter Information

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