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Integrated Research & Engineering Division

Advances in Propellant Stability Screening

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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

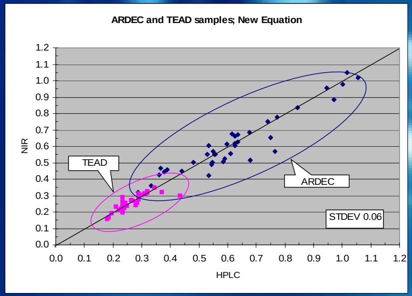
- McAlester Army Ammunition Plant
- Kuwait – Needed real time stability results
  - Deployed to CFLCC
- Tooele Army Depot – WC propellant
  - Very limited OB/OD
- RDECOM-ARDEC

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

Evaluated

- Foss 5000
- Foss XDS
- Buchi NIRFlex N-500 FT-NIR
- Zeiss
- Perkin Elmer

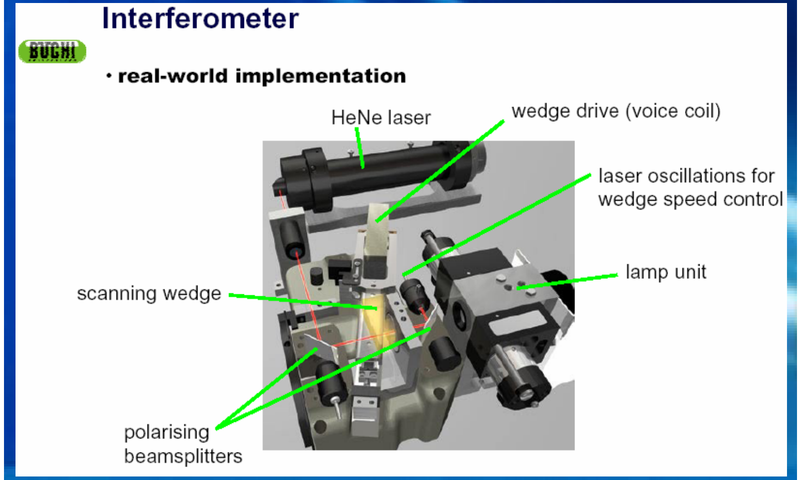
	FOSS 5000	FOSS XDS	Buchi NIRFlex N-500
TECHNOLOGY	NIR	NIR	Polarization FT-NIR
AGE	12	3	2
AVAILABILITY	2 Years	Yes	Yes
SOFTWARE	Vision	Vision	Internally Developed
CALIBRATION TRANSFER	No	Claimed	Claimed
EASE OF USE	Medium	Medium	High
SAMPLE SIZE	200 to 300 grams	200 to 300 grams	< 50 grams
COMPUTER	Standard Laptop	Standard Laptop	Standard Laptop
EQUIPMENT SIZE	Largest	Largest	Smallest
RUGGEDNESS	Medium	Medium	High
SAMPLING SYSTEM	Transport Cell	Rotating Cell	Rotating Cell
SOLID SAMPLES	Yes	Yes	Yes
REQUIRED ACCESSORIES	Transport cells, external standards	Transport cells, external standards	Beakers

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)	8cm-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



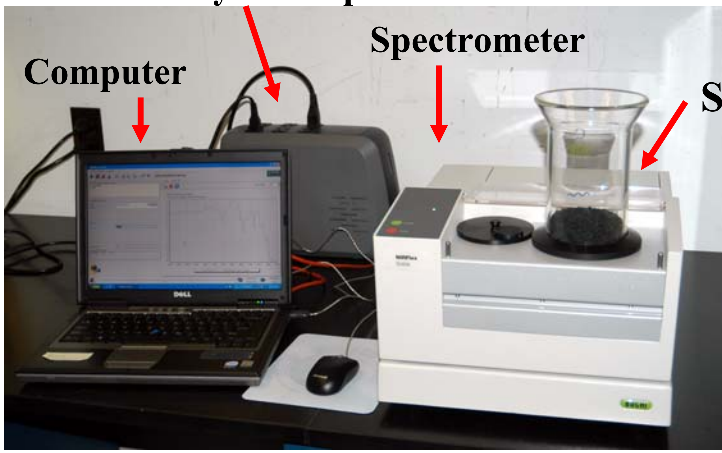
Buchi Spectrometer System

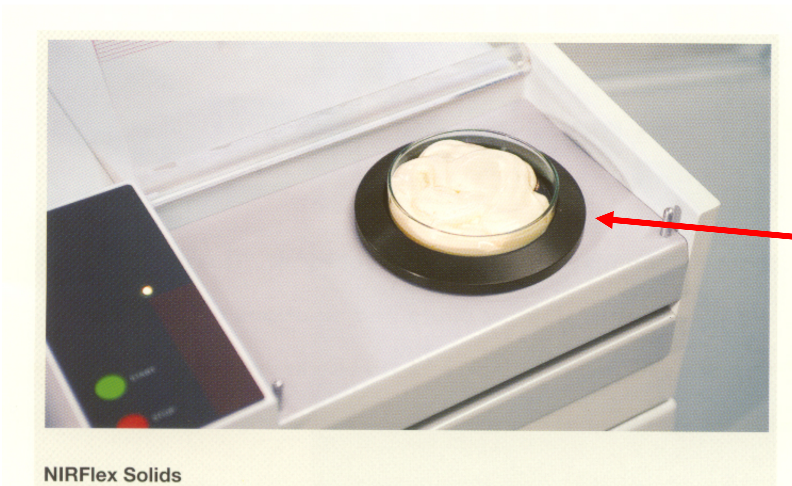
Battery Backup

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

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

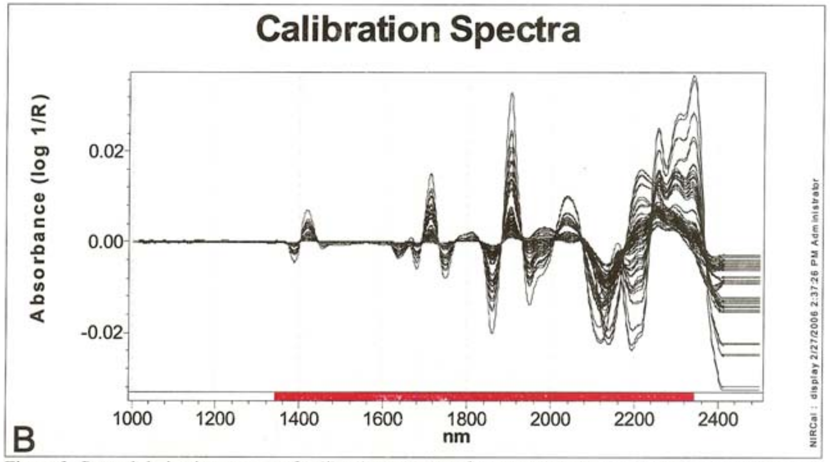
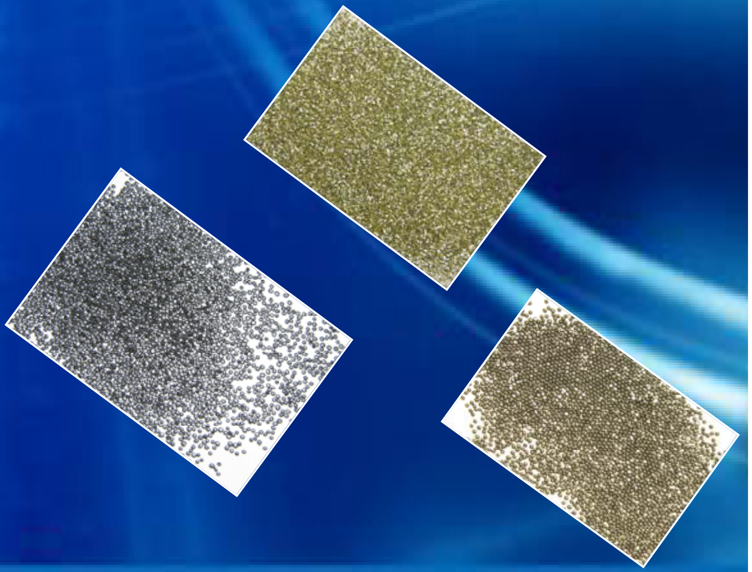


Figure 3. Second derivative spectra of calibration samples after second derivative math treatment (15 point segment, 15 point gap)

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

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



First WC Calibration Model – Spectrometer 1

Leave-One-Out Results

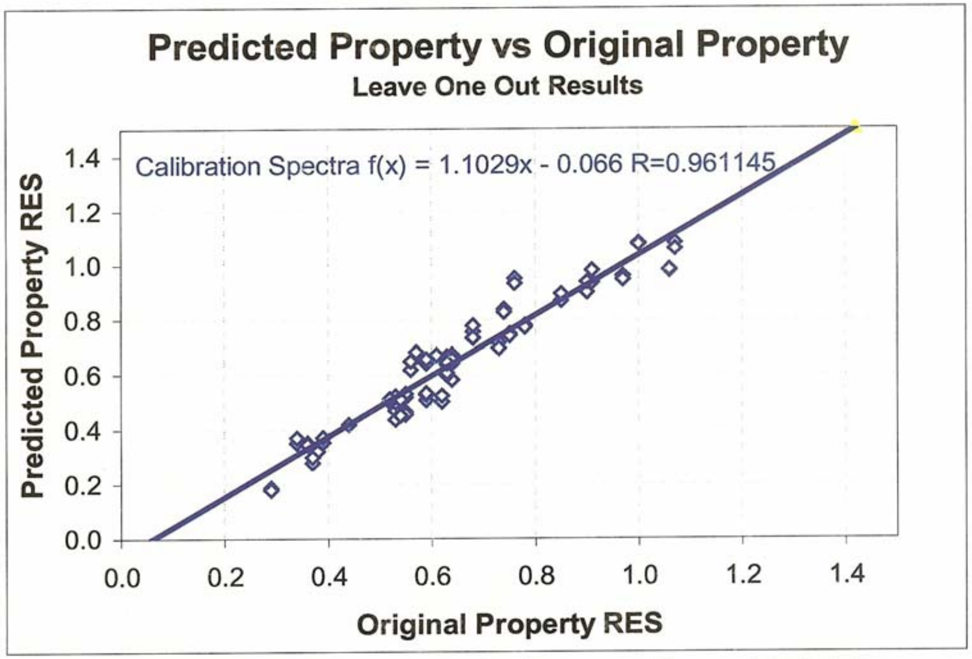


Figure 6. Predicted RES value from each sample when removed from model calculation

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

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Calibration Transfer

Calibration model from spectrometer 1 electronically transferred to spectrometer 2

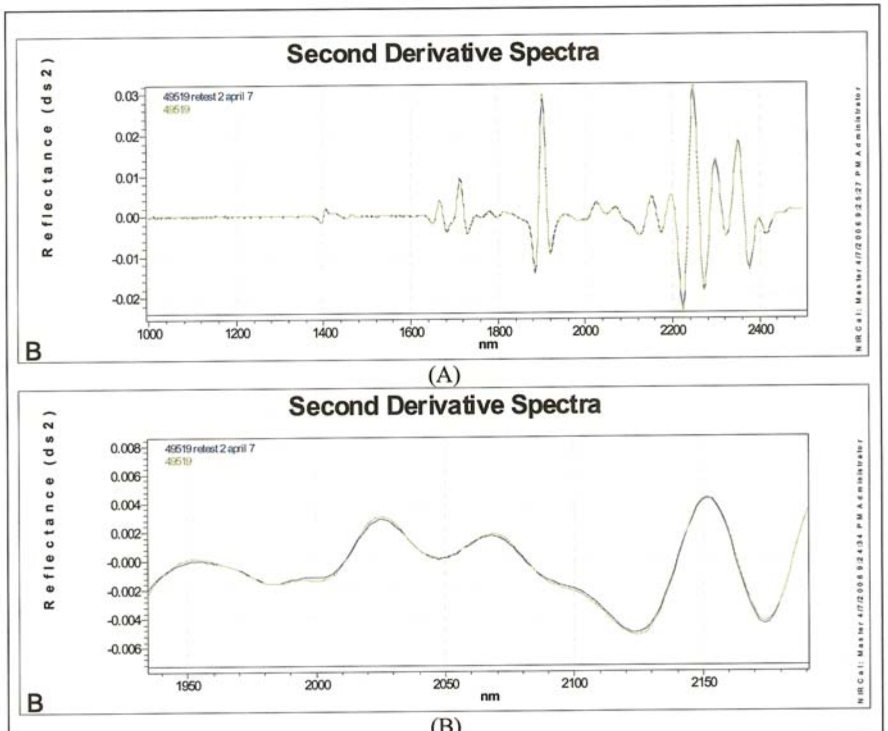

Figure 2. Sample 49519 as measured in the initial study compared to re-measurement on second N-500 system.

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

WC Sample Analysis

WC sample %RES prediction on spectrometer 2 using calibration model from spectrometer 1

NIR-Lab ≤ 2*SECV; ≤ 0.14

Sample Name	RES by Lab	NIR Predicted	(NIR-Lab)
47776	0.37	0.47	0.10
47776	0.37	0.48	0.11
289	0.44	0.48	0.04
289	0.44	0.48	0.04
49602	0.55	0.60	0.05
49602	0.55	0.61	0.06
49519	0.64	0.69	0.05
49519	0.64	0.71	0.06
48778	0.73	0.78	0.05
48778	0.73	0.81	0.08
49842	0.90	0.86	-0.04
49842	0.90	0.89	-0.01
709	0.97	0.99	0.02
709	0.97	1.06	0.09
80005	1.00	0.99	-0.01
80005	1.00	1.01	0.01
47782	0.36	0.40	0.04
47782	0.36	0.40	0.04
47444	0.29	0.39	0.10
47444	0.29	0.41	0.12
Bias			0.05
SEP(no bias correction)			0.06

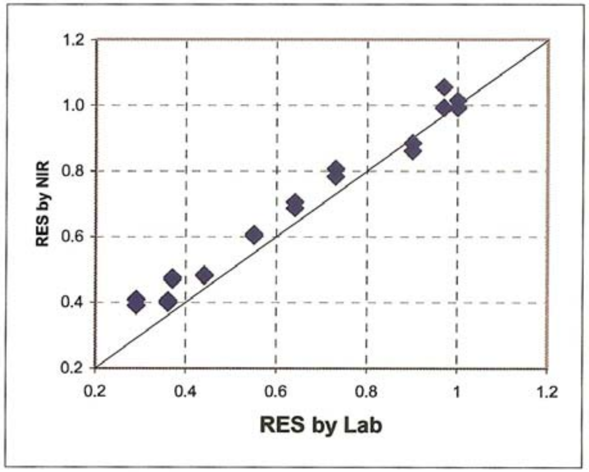

Table 3. Prediction of WC samples using equation developed from spectra measured on different spectrometer.

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Results

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

Easier to operate and transport, with the potential for future reduced downtime and operating costs

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SOLID SAMPLES	Yes	Yes	Yes
REQUIRED ACCESSORIES	Transport cells, external standards	Transport cells, external standards	Beakers
SECV	0.07	Failed	0.065

Plan

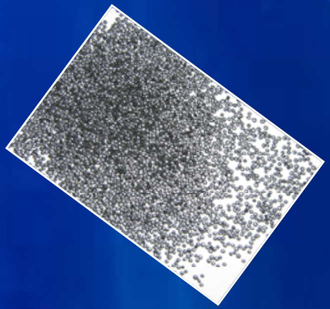
- Instrument and process validation
  - Establish precision, accuracy, and repeatability
  - Calibration model for all propellant types
  - Determine SECV for all propellant types
  - Confirm electronic calibration transfer
- Field trials
- PSSB approval
- Safety approval
- SOP and operation manual
- 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
- 870
- 872
- 890



Presenter Information

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