



*Field-Portable Thin Layer Chromatography  
Propellant Stability Test Kit*

*Moving Forward*

PIKA – Pelatron Consortium

**Presenters:**

**Harvey Kim, Pelatron TLC Program Manager**

**Wendy Church, PIKA TLC Program Manager**



## Agenda

- ❖ TLC Background and History Harvey Kim
- ❖ TLC Implementation/Training Wendy Church
- ❖ Technology Transfer Harvey Kim



## What?

- ❖ Field-portable thin layered chromatography (TLC) system developed by Lawrence Livermore National Laboratory's Forensic Science Center
- ❖ Developed under contract by the Defense Ammunition Center to detect the level of stabilizers in aging propellant stock
- ❖ Reduced sample size (100 mg per sample), reduced waste, reduced per sample cost
- ❖ On-site quantitative and qualitative results within hours



## Why?

- ❖ Aging propellants needing stabilization testing
- ❖ Increasing incidents of self-ignition endangering life, property, and environment.
- ❖ Another tool to screen propellant stabilization in the field – identify strongly stabilized propellant lots, only send suspect propellants to laboratory



## Components



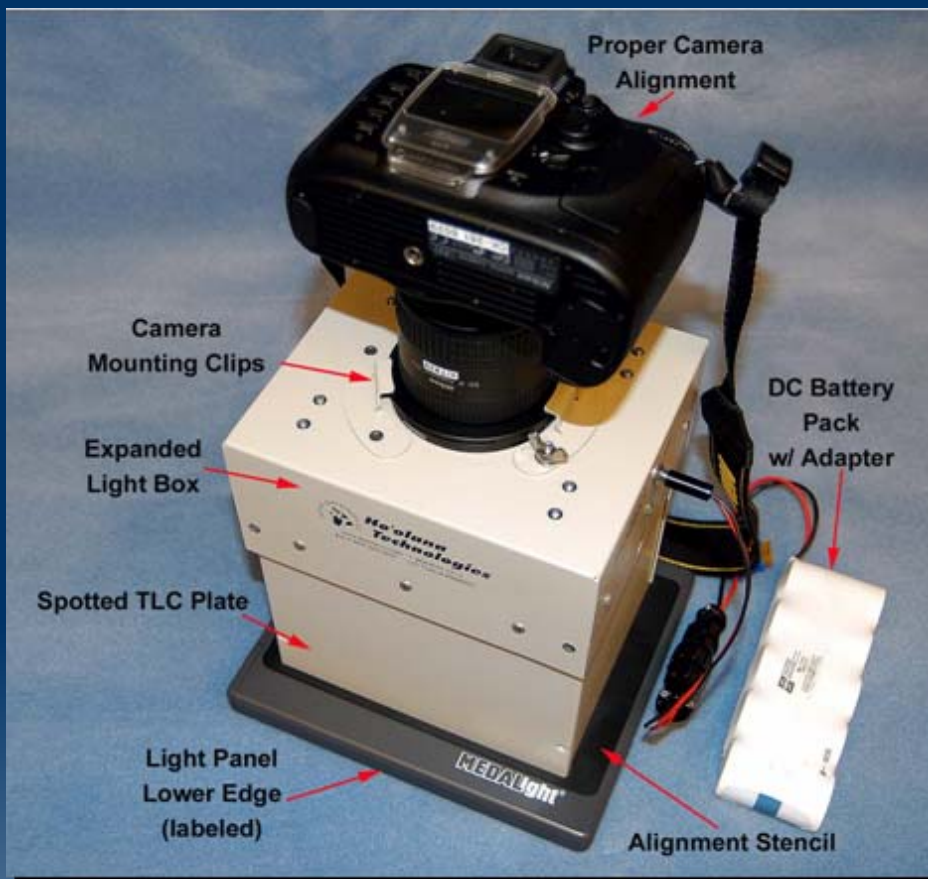
Propellant crusher, vial rack, dip basket with plate



Camera on light box, computer, balance, process tank, developing tank, heating plate



## Digital Imaging System



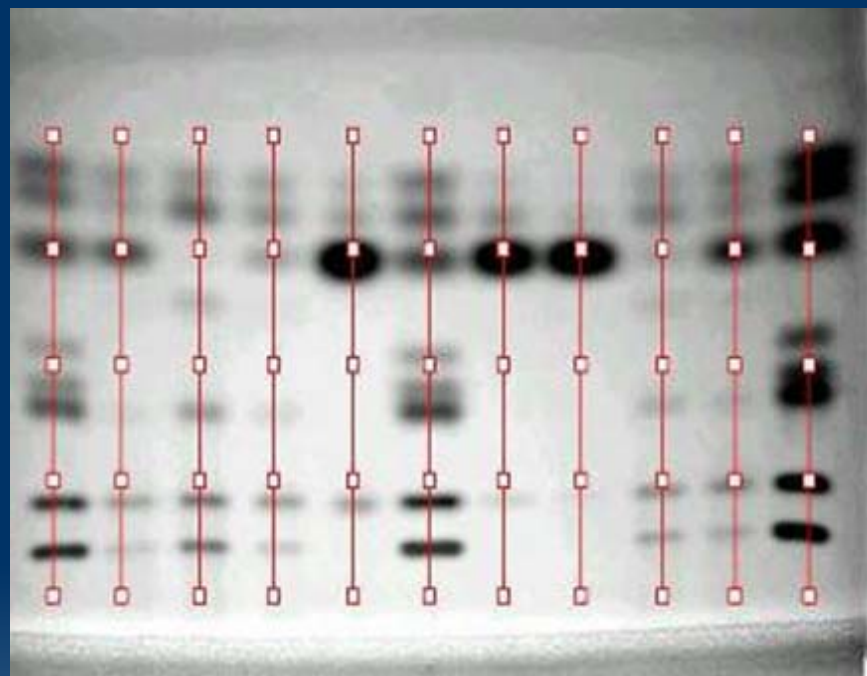
- ❖ Digital Camera
  - ✓ Nikon D100
- ❖ Light Box
  - ✓ internal UV light and filter
- ❖ Light Panel w/temp
  - ✓ source for white light
- ❖ Battery Pack



# Digital Imaging Analysis



Developed TLC plate



Digital image with lane identification



# TLC Process

Computer Analysis



Sample Prep



Documentation



Imaging



Plate Spotting



Coloring



Plate Development







## Propellant Stabilizer Analysis

- ❖ Determination of % total remaining 'effective' stabilizer (%RES)
- ❖ High throughput screening
- ❖ Simple
- ❖ Cost effective
- ❖ Field portable
- ❖ Minimal waste





## TLC Project History

- ❖ 1997 - Initial funding – DAC contracted LLNL to develop system.
- ❖ 1999 - LLNL developed a TLC system to determine the percent remaining effective stabilizer (%RES) in propellant samples.
- ❖ 2003 – 50 TLC kits manufactured by Ho`olana LLC in Hilo, HI.
- ❖ Nov 2004 - PS-TLC system validated by DAC and endorsed by the Army Propellant Surveillance Laboratory (APSL).
- ❖ Jul 2006 – PS-TLC system validated by the PSSB (joint services)
- ❖ Aug 2006 – First training class, Tooele, UT
- ❖ Jan 2007 – Second training class, Hawthorne, NV
- ❖ April 2007 – Third training class, Aberdeen, MD



## Certified and Validated

- ❖ 2004 - Certified for use by the Army
- ❖ July 2006 – Certification and Validation by the Propellant Surveillance Safety Board, a subgroup of the Joint Ordnance Commander's Group





## Technology Transfer Team

**PIKA**  
INTERNATIONAL, INC.



**PELATRON**  
INC.

- ❖ Cliff Ancelet
- ❖ Kate Anthony
- ❖ Wendy Church

- ❖ Rich Whipple
- ❖ Greg Klunder

- ❖ Kaleo Elia
- ❖ Helene Elia
- ❖ Eric Kim
- ❖ U`ilani Peralta
- ❖ Harvey Kim



## TLC Trainings

- ❖ Three trainings conducted since last August
  - ✓ Tooele Army Depot (TEAD)
  - ✓ Hawthorne Army Depot (HWAD)
  - ✓ Aberdeen Proving Grounds (APG)
- ❖ Two-weeks (80 hours)
- ❖ TLC Kits signed over to each facility for continued use



TLC Training at TEAD



TLC Training at HWAD



TLC Training at APG



## TLC Training Objectives

- ❖ Provide JMC installations with capability to perform real-time, on-site propellant stability testing
- ❖ Transfer TLC Kit from LLNL to Pelatron and PIKA for future development and distribution



# Training Curriculum



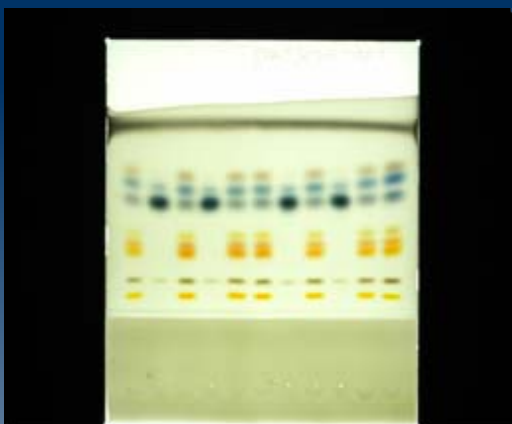
1) Propellant grains are cut and weighed



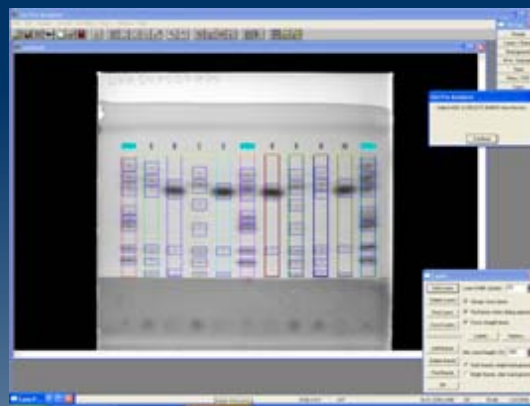
2) Dissolved propellants are spotted onto TLC plate



3) TLC plate is developed and colored



4) TLC plate image is captured



5) Digital image is analyzed by GelPro application

Propellant Stability Analysis Report

Analyst: ESB (201507) Analysis Date: 02/07 (201507)  
Tutor's ID: 254260001007 Analyst Title: SAsA (201507)  
Plate ID: 254260001007 Plate Name: ESB25426001007\_0207\_0207 (25426001007)  
Prepared: 02/07/2015 10:17 AM Director: 254260001007 (25426001007)  
Method: DPLA

Plate Label	Propellant Type	Area Number	Weight (mg)	Total (mg)	Residue (%)	Res. Code	Res. Cnt	3,988	Category
1	MS	72216	100	5	5	0.2			A
2	MS	72216	100	5	5	0.2			A
3	MS	72216	100	5	5	0.2			A
4	MS	72216	100	5	5	0.2			A
5	MS	72216	100	5	5	0.2			A
6	MS	72216	100	5	5	0.2			A
7	MS	72216	100	5	5	0.2			A
8	MS	72216	100	5	5	0.2			A
9	MS	72216	100	5	5	0.2			A
10	MS	72216	100	5	5	0.2			A
11	MS	72216	100	5	5	0.2			A

Calibration curve (spiked Standard 1)

Experimental Conditions: Temperature 25.0, Humidity 20, Solvent: MCHACRODENDRUS, TLC Plate: GPCF 60F, Plate ID: 25426001007, Plate Name: ESB25426001007\_0207\_0207 (25426001007), Director: 254260001007 (25426001007)

Check Standard Used for Normal Factor: 1.04 ± 0.2, GPCF Value: 0.20

6) %RES and Propellant Stability Analysis Report



# TEAD Training August 14 – August 24, 2006

- ❖ Eleven Students
- ❖ Fourteen Propellant Samples
- ❖ Average Temperature > 90 degrees
- ❖ Average Relative Humidity < 20 percent
- ❖ Students from TEAD and the PIKA-Pelatron Team
- ❖ Training led by LLNL with Pelatron and PIKA acting as assistant instructors and students







## HWAD Training January 22 – February 1, 2007

- ❖ Eleven Students
- ❖ Eighteen Propellant Samples
- ❖ Average Temperature = 70 degrees
- ❖ Average Relative Humidity = 20 percent
- ❖ Students from Day & Zimmerman, APSL, and HWAD
- ❖ Training led by Pelatron and PIKA with LLNL acting as assistant instructors





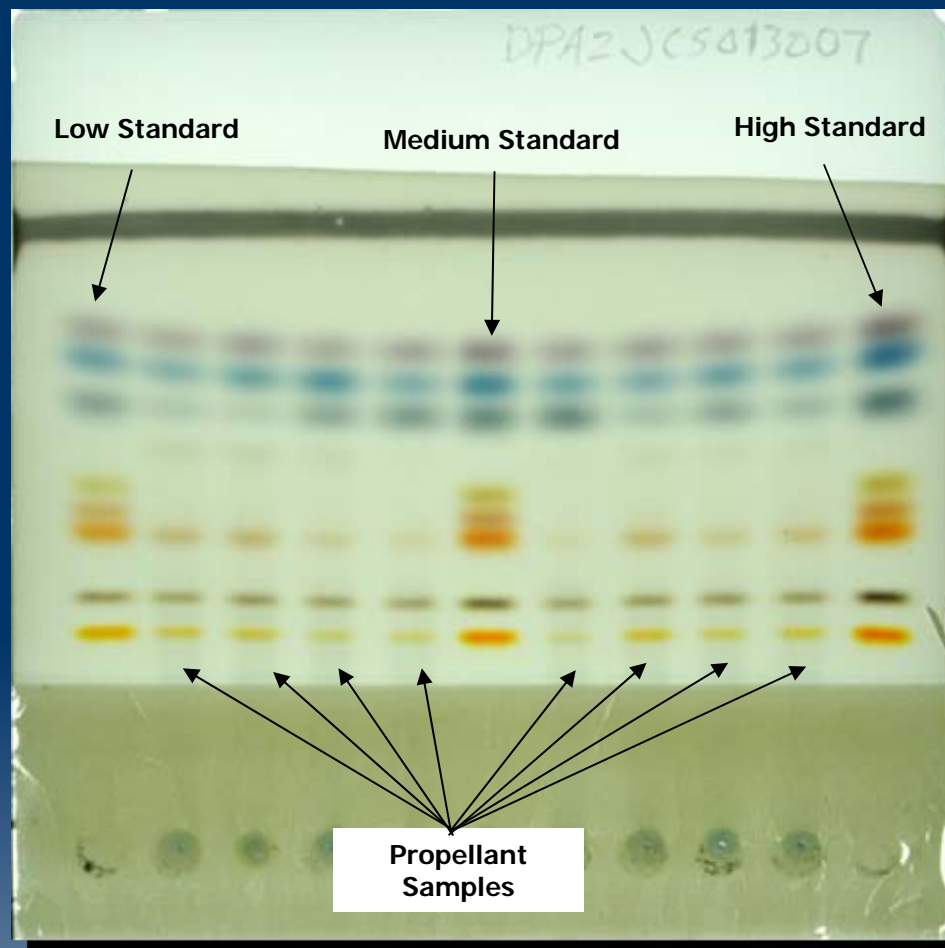
## APG Training April 23 – May 4, 2007

- ❖ Eight Students
- ❖ Nine Propellant Samples
- ❖ Temperature Ranged from 60 to 80 degrees
- ❖ Relative Humidity Ranged from 30 to 50 percent
- ❖ Students from APG and YPG
- ❖ Training led by Pelatron and PIKA with LLNL acting as observers and technical advisors





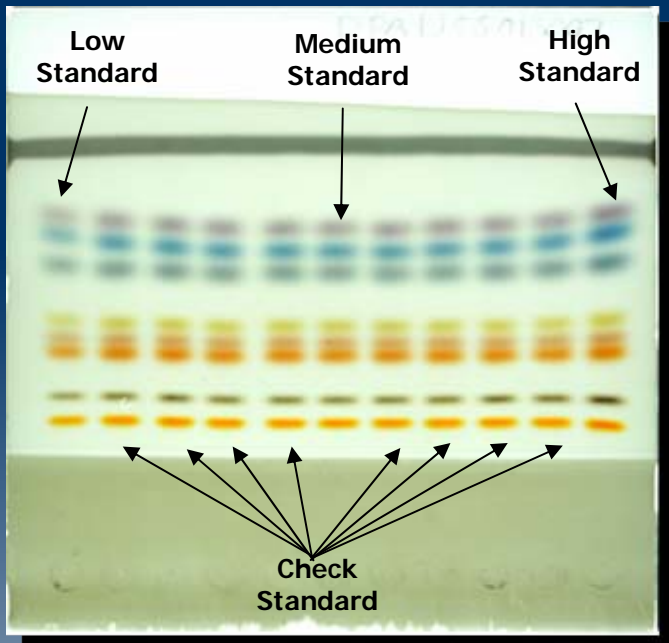
# Propellant Stability Analysis



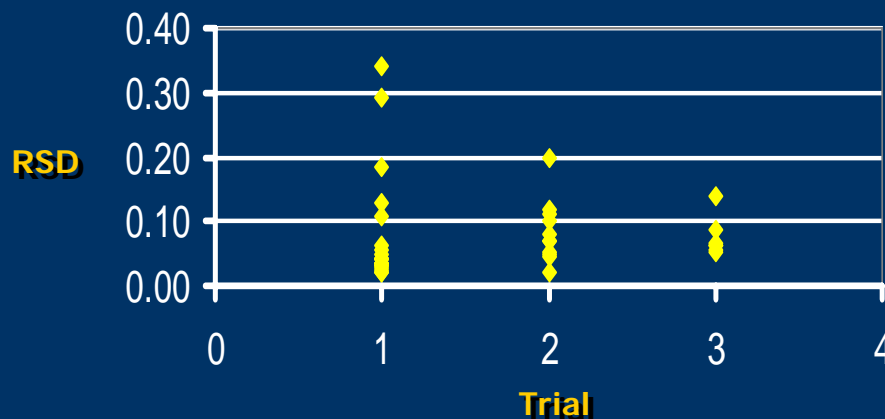


# Reproducibility Results

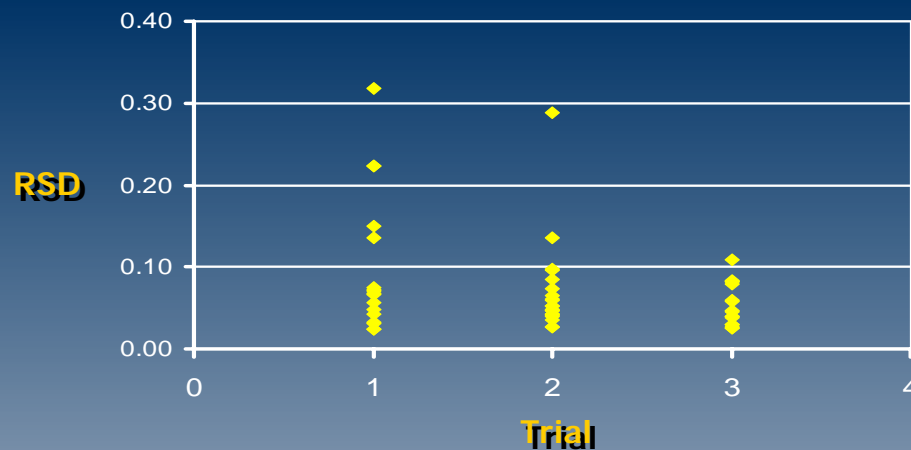
- ❖ Analyze check standard in all sample lanes
- ❖ Measure of ability of the TLC procedure



## Tooele Reproducibility Results



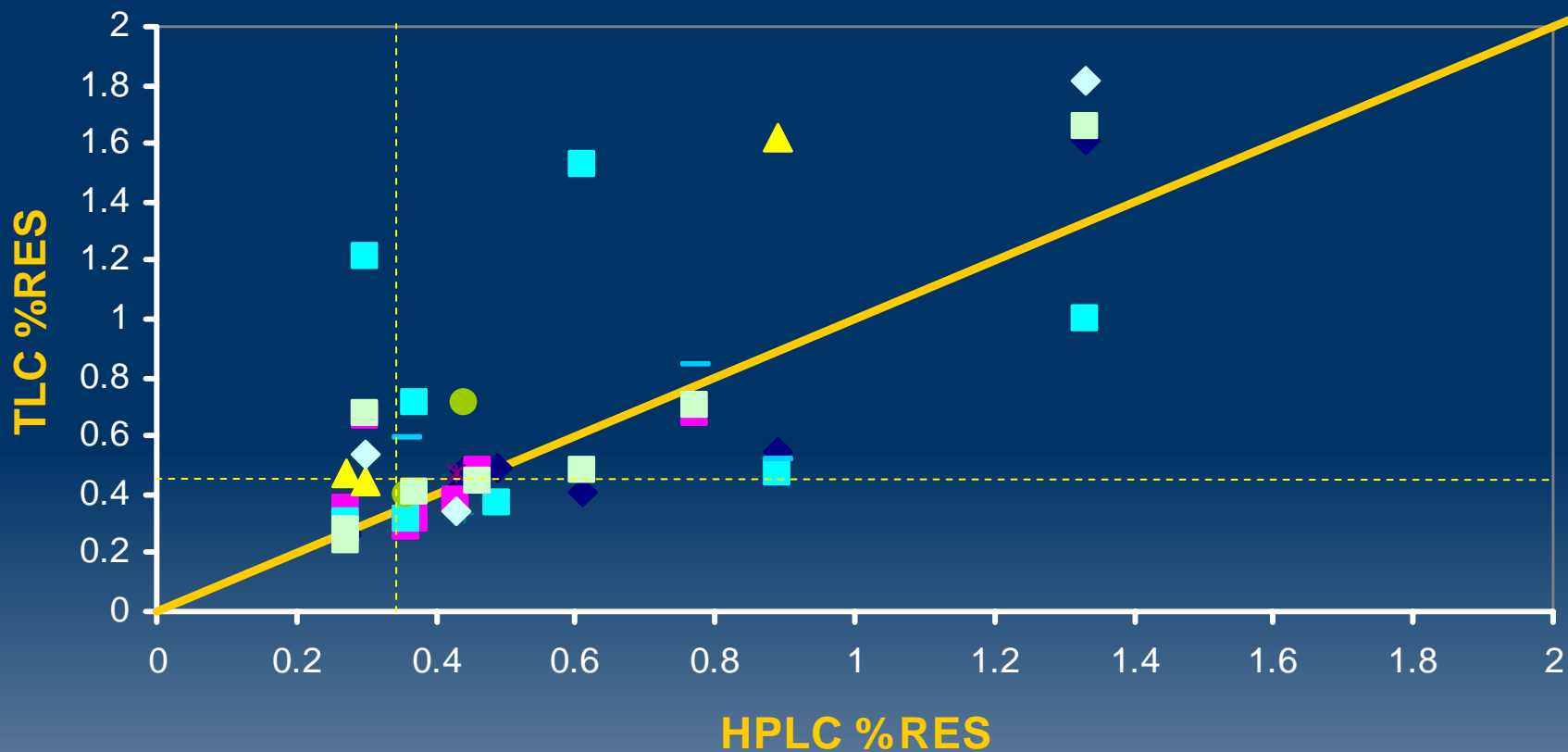
## Hawthorne Reproducibility Results





# Analysis Results

## HWAD Analysis





## TLC Training Summary

- ❖ Completion of Technology Transfer
- ❖ Field results correlate well with lab results
- ❖ TEAD, HWAD, APG, and YPG now have capability to perform real-time, onsite analysis of propellant stability



## Technology Transfer

- ❖ Three two-week fieldings to train Army technicians to use the TLC kits
  - ✓ Tooele Army Depot, UT
  - ✓ Hawthorne Ammunition Depot, NV
  - ✓ Aberdeen Proving Grounds, MD
- ❖ Mobile Analysis Teams to perform TLC analysis as needed by the military
- ❖ End-user service and support
- ❖ Data collection and analysis for kit effectiveness
- ❖ Supply chain system for replacement parts, consumable supplies, manufacturing kits, etc.



## Technology Transfer

- ❖ Maintain integrity of TLC system
  - ✓ Quality Assurance/Quality Control (QA/QC) of TLC process and results
  - ✓ Certified TLC Analysts and Operators
  - ✓ Re-certification and training
- ❖ Continuing education
- ❖ Strategic implementation





## Certification Levels

### ❖ Level 1 Certified PS-TLC Operator

- ✓ Able to perform analysis of propellant stabilizers under supervision of a Level 2 Certified PS-TLC Analyst
- ✓ All TLC analysis results must review and certified by a Level 2 Certified PS-TLC Analyst.
- ✓ Level 1 Operators require additional experience and education to become Level 2 Analysts.

### ❖ Level 2 Certified PS-TLC Analyst

- ✓ Able to perform independent analysis of propellant stabilizers and certify the results.
- ✓ Able to assist and mentor Level 1 Certified PS-TLC Operators.
- ✓ Able to review and certify reports and plates performed by Level 1 Certified PS-TLC Operators.
- ✓ Able to submit official results

### ❖ Certification Criteria

- ✓ Points awarded based on practical exercise, written exam, and an oral analysis



## End User Support

- ❖ TLC Sales and Support – Pelatron primary point-of-contact
  - ✓ Website: [www.pelatron.com/tlc](http://www.pelatron.com/tlc) - best practices, FAQ's (frequently asked questions), updates and patches, online continuing education, shopping cart
  - ✓ Phone: toll-free (866) 460-1356
  - ✓ Fax: (801) 660-4297
  - ✓ Email: [TLC@pelatron.com](mailto:TLC@pelatron.com)



## Hawaiian Jade

- ❖ Unknown propellants washing up on Hawaiian shores – Jan/Feb 07
  - ✓ People collecting and making necklaces of Hawaiian Jade
  - ✓ Most collected by Army and destroyed
- ❖ PIKA-Pelatron team analyzed propellant grains for stability





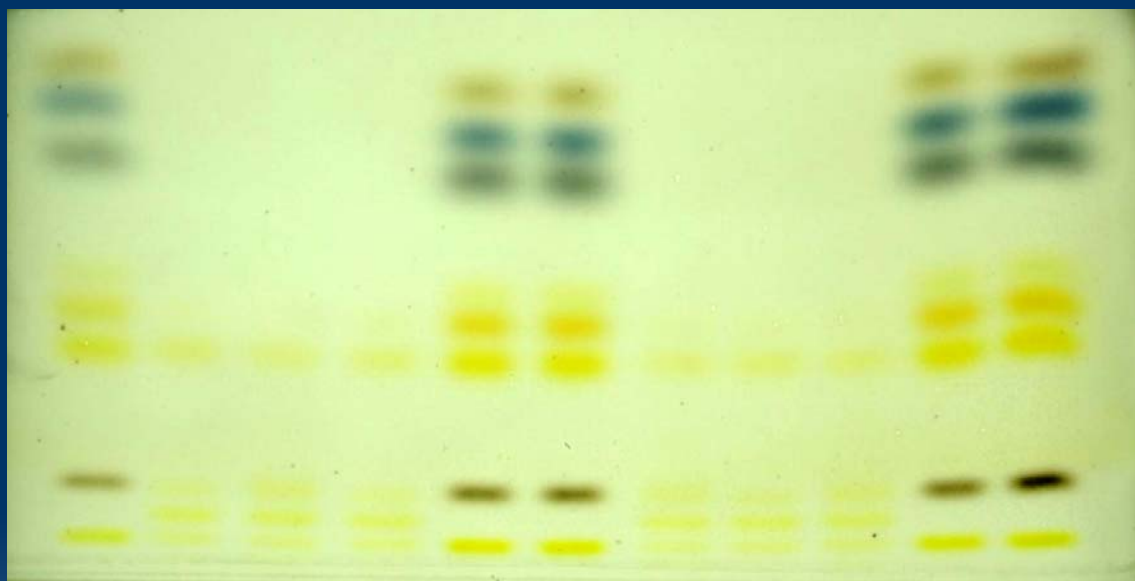
## Hawaiian Jade

- ❖ Tested for DPA, EC/AK, 2NDPA stabilization
- ❖ Identified
  - ✓ DPA stabilized
- ❖ Results
  - ✓ very poor stabilization
  - ✓ .12% %RES





## Hawaiian Jade



2-NDPA  
NNO-DPA  
DPA

2,4-DNDPA  
2,2'-DNDPA  
2,4'-DNDPA

4-NDPA  
4,4'-DNDPA



## Acknowledgement

The PIKA-Pelatron Consortium greatly appreciates the Defense Ammunition Center and the scientists at the Lawrence Livermore National Laboratory Forensic Science Center for the TLC technology, their guidance, their expertise, and their un-ending momentum to enable the TLC technology transfer process.

As we express thanks in Hawaii,

**Mahalo Nui Loa!**



Questions?