

# ***Catching Crooks with Chemistry:***



## ***NIST Research, Current and Future Perspectives on Trace Forensic Chemical Analysis***

**Bill MacCrehan**

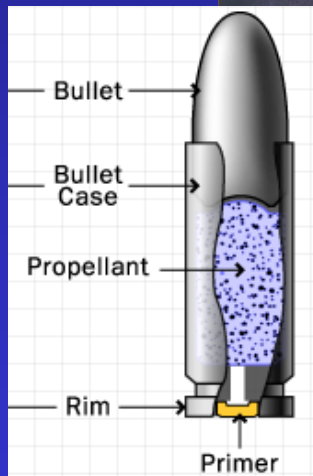
**Analytical Chemistry Division**

**National Institute of Standards and Technology (NIST)**

**Gaithersburg, MD**



# *NIST Research: Gunshot Residue Analysis*



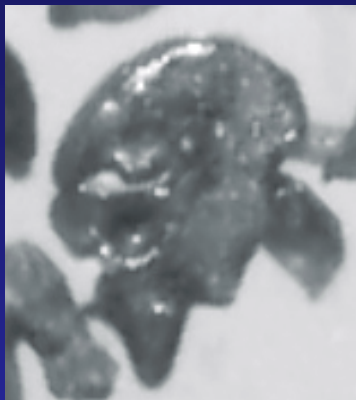
**National Institute of Justice**

The Research, Development, and Evaluation Agency of the U.S. Department of Justice



**NIST Office of Law Enforcement Standards**

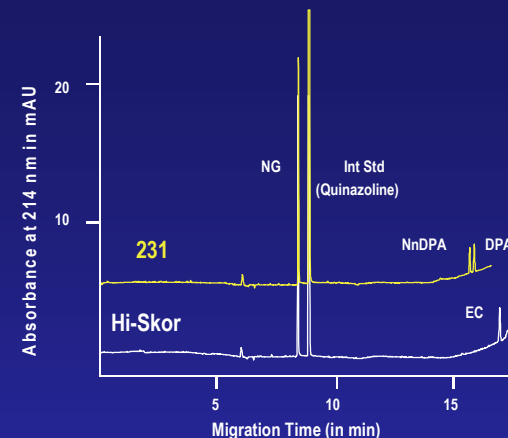
# Organic Gunshot Residue (OGSR) Analysis



OGSR Particle



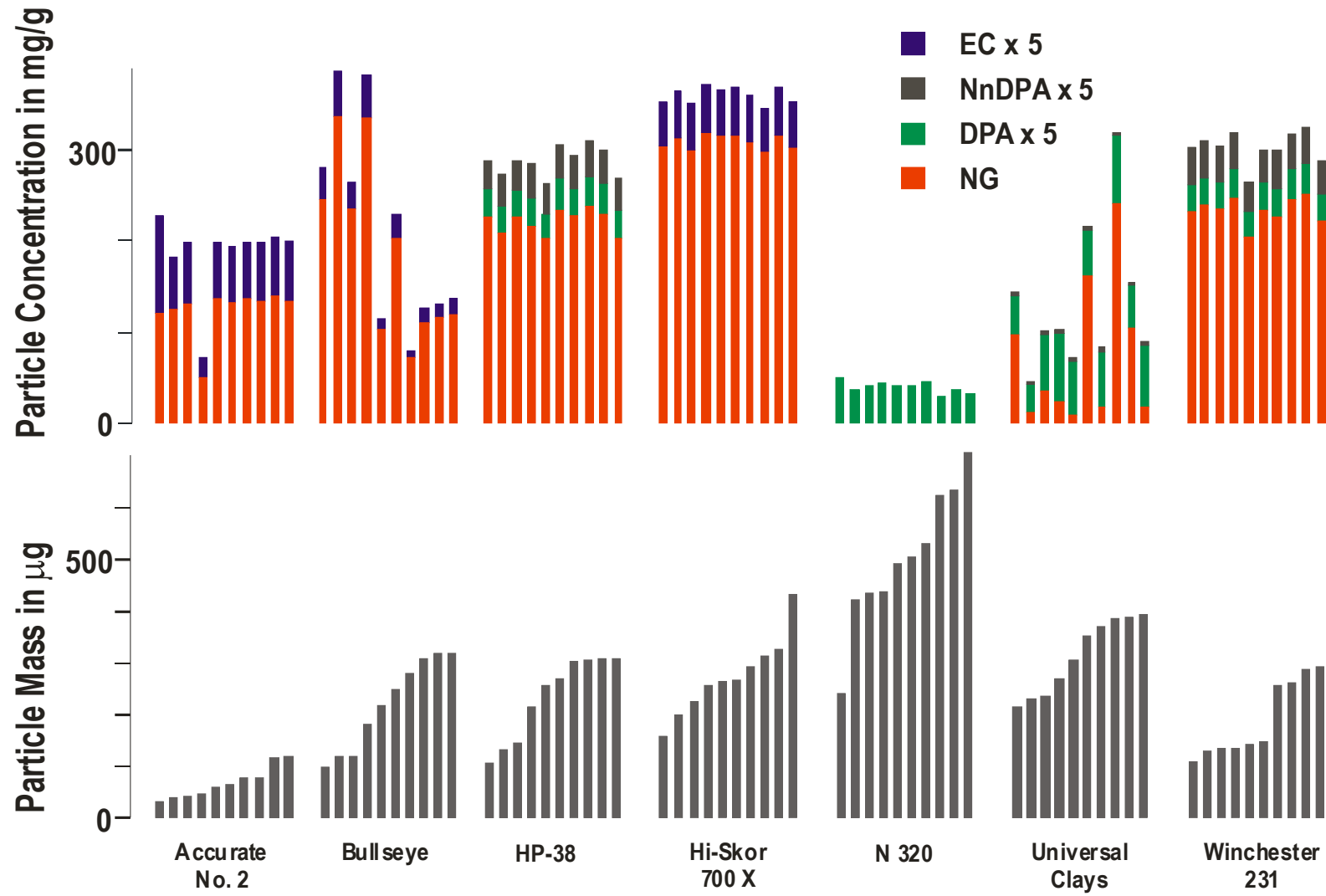
CE Analyzer (<\$50K)



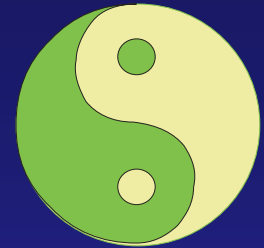
OGSR Component Analysis

- ➡ Detection based on organic propellant (nitroglycerin-NG) and stabilizers (diphenylamine-DPA and ethyl centralite-EC)
- ➡ May be collected with masking tape or combing
- ➡ OGSR tests:
  - relatively rapid (< 1 hour); require less expensive equipment
- ➡ Can often associate residues and unfired ammunition
- ➡ No evidence of occupational exposure to COGC

# Chemical Composition of Single Smokeless Powder Particles



# ***Some of the OGSR Experiments***



## **Before and After Experiment:**

- Can unfired powders and OGSR be linked?

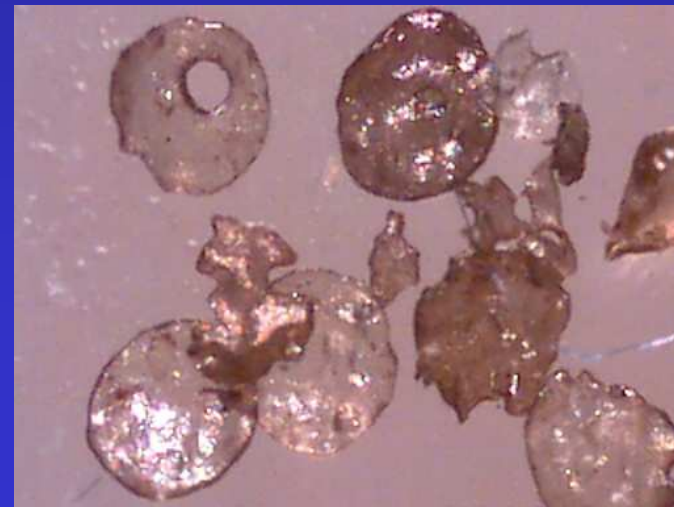
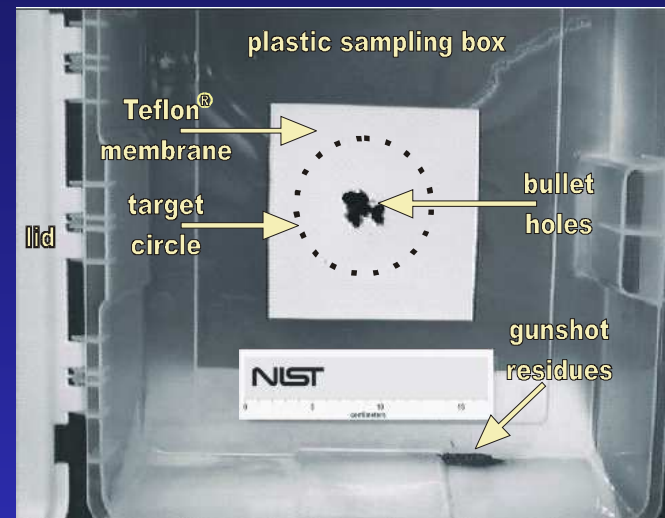
## **Ammochange Experiment:**

- Does the composition of residues change when the composition of the ammunition powder is changed?

## **Blind Residue Study:**

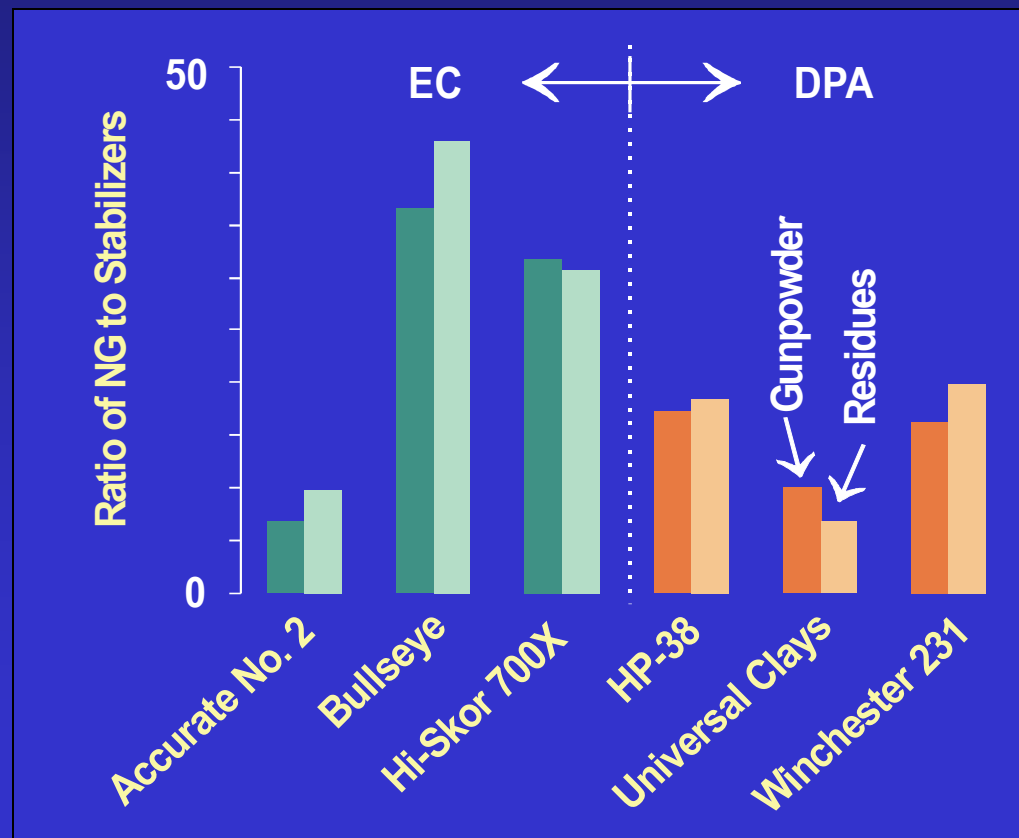
- Given 7 randomly coded boxes of ammunition, how many can be reliably distinguished?

# *Test Setup for OGSR Experiments*

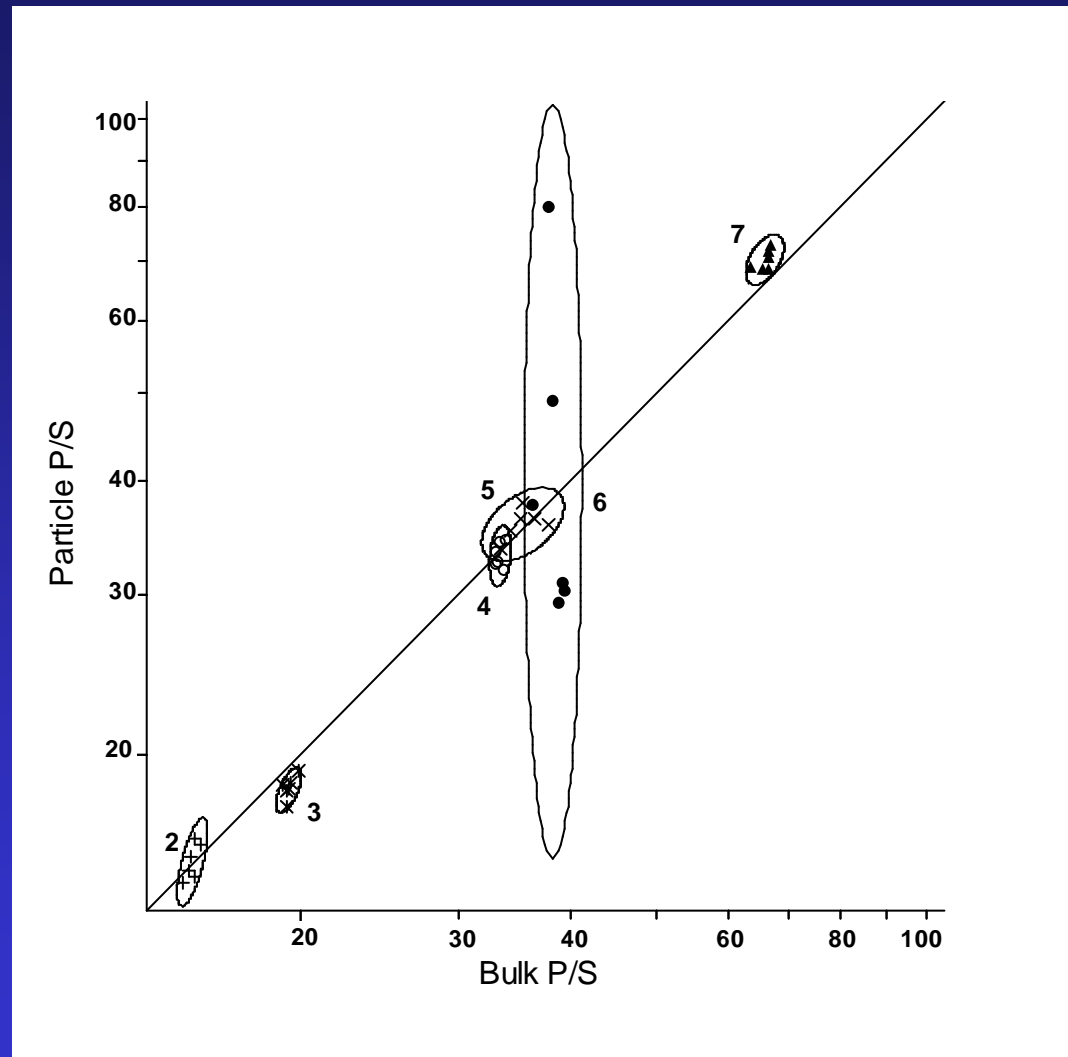


# Evaluating OGSR Composition

$$E/S = \frac{\text{Energetic [NG]}}{\text{Stabilizer [DPA + NnDPA] or [EC]}}$$



# *Graphically Displaying Certainty of Data*





# *Improvised Explosive Devices (IEDs)*



***Centennial Olympic Park Bombing  
Summer Olympics, July 27, 1996***

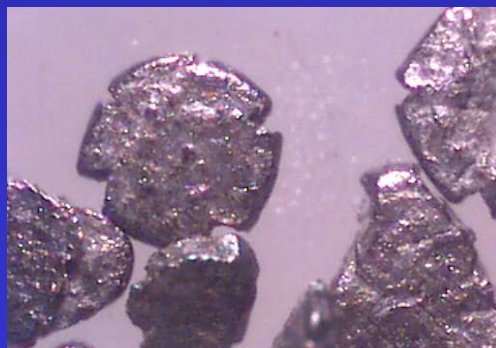
# ***Interlaboratory Study of Smokeless Powder Measurements***

## ***Goals:***

- Evaluate the practice of SP measurements
- Learn about measurement needs

## ***Study:***

- Two test samples prepared (one EC, one DPA)
- Make reliable NIST measurements
- Samples sent to 19 participant laboratories



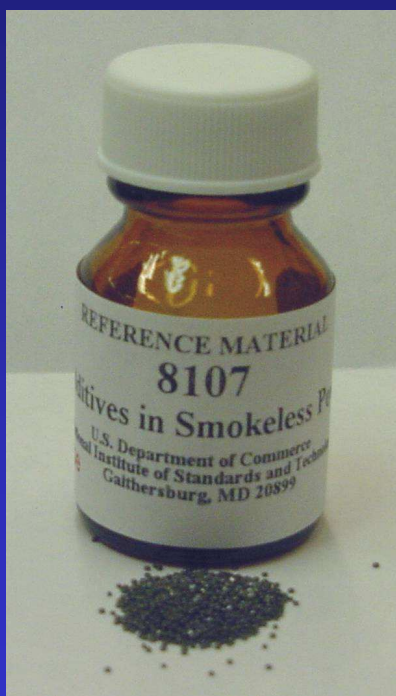
# Interlaboratory Measurement Comparison Results

HiSkor 700X

Win 231

	Powder 1									
Lab ID	NG	DPA	NnDPA	2-NDPA	4-NDPA	EC	MEC	4-NEC	2,4-DNT	DBP
NIST	298 (5)					9.6 (0.3)				
1	328.8 (0.1)					10.3 (0.1)		0.1 (0.1)		
2	260 (30)					11.5 (2.2)				
3	274.8 (8.2)					9.5 (0.2)				
4a	297.52 (3.43)					10.23 (0.03)				
4b	298.18 (3.28)					10.26 (0.05)				
5										
6										
7										
8										
9										
10										
11										
12										
13			*			*				
14										
15										
16		**				**				
17										
18										
19			***							
	Powder 2									
Lab ID	NG	DPA	NnDPA	2-NDPA	4-NDPA	EC	MEC	4-NEC	2,4-DNT	DBP
NIST	205 (3)	5.0 (0.1)	6.1 (0.3)							
1	233.8 (0.1)	5.0 (0.1)	7.0 (0.1)	0.3 (0.1)	0.2 (0.1)					
2	231 (19)	7.4 (0.9)	8.8 (1.1)							7.9 (3)
3	190.8 (5.7)	5.4 (0.1)	6.7 (0.2)	0.3 (0.01)	0.3 (0.01)					
4a	186.24 (2.51)	5.26 (0.03)	7.08 (0.05)	0.61 (0.32)						
4b	180.68 (1.32)	4.85 (0.04)	6.92 (0.06)	0.77 (0.37)						
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# ***NIST RM 8107 Additives in Smokeless Powder***



***Now Available***

## Reference Concentrations mg/g

Nitroglycerin	129.1 $\pm$ 2.1
Diphenylamine	7.80 $\pm$ 0.18
N-nitrosodiphenylamine	3.05 $\pm$ 0.09
Ethyl Centralite	36.4 $\pm$ 1.3

# ***Standards For Trace Explosives Detectors***



***Challenge: to reliably detect trace high explosives with a variety of detectors***



# ***Three-pronged Approach to Address Trace Explosives Standards Needs***

***⇒ SRM 2905 Trace Particulate Explosives***

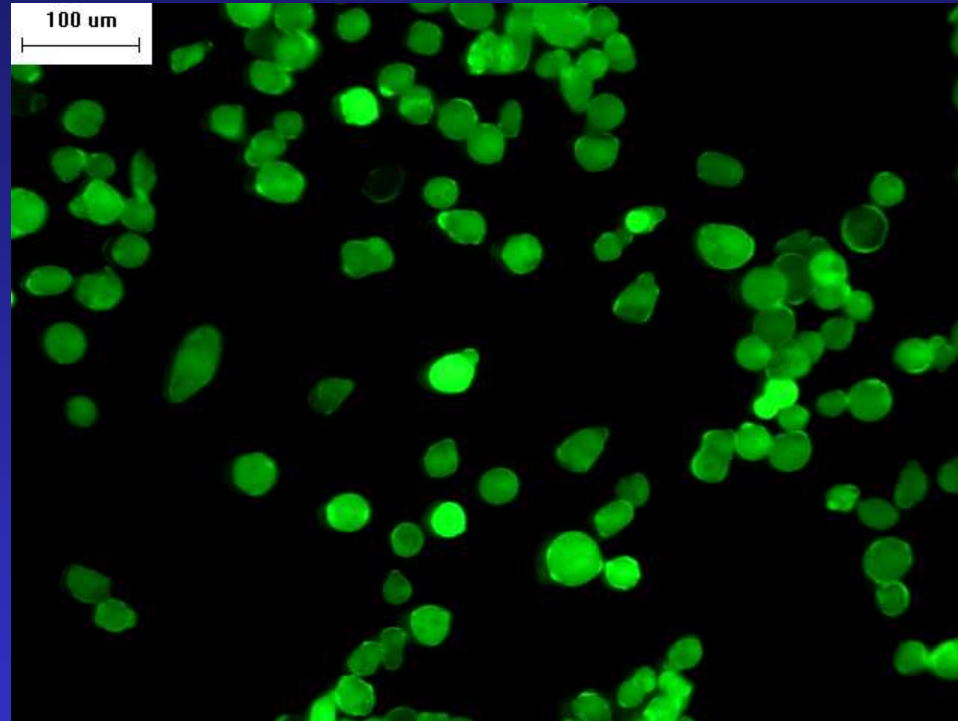
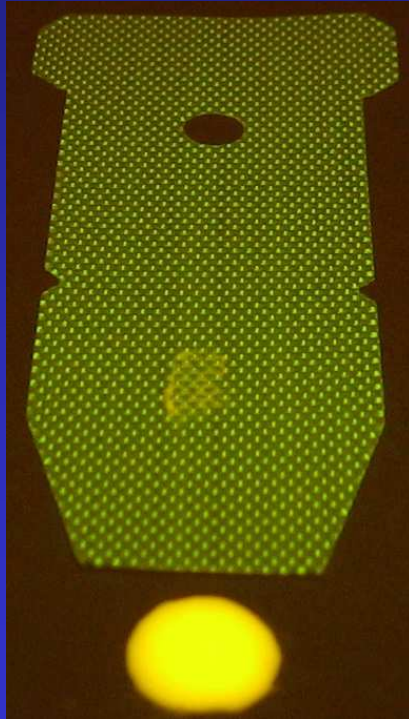
***⇒ ASTM Standard Practice 2520-07***

***E54.01 Homeland Security Applications: CBRNE  
Sensors and Detectors***

***⇒ SRM 2906 Trace Solution Explosives***

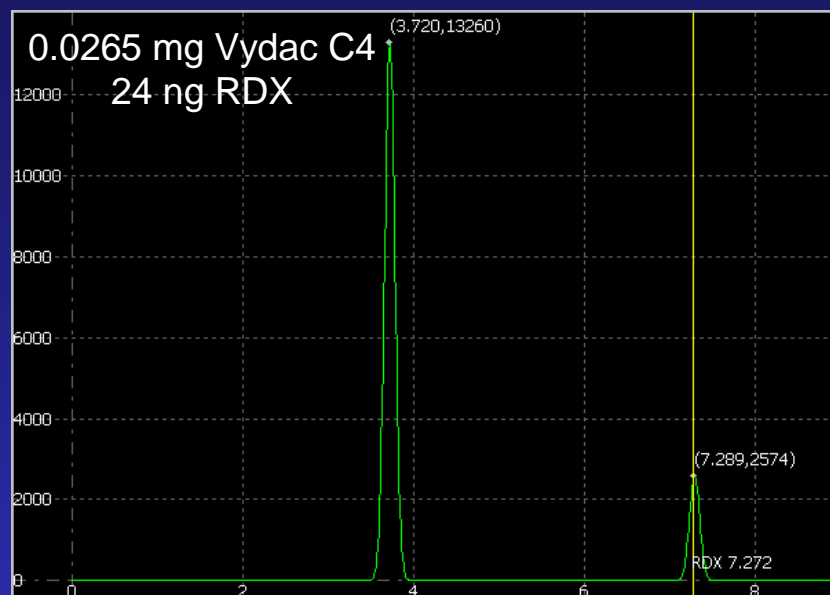


# ***Testing SRM 2905 on Field Explosives Detector (IMS)***



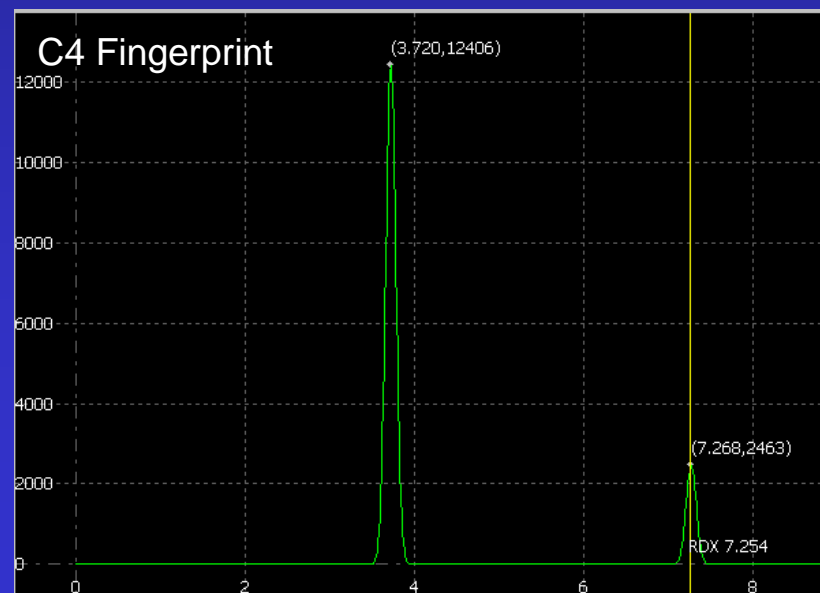
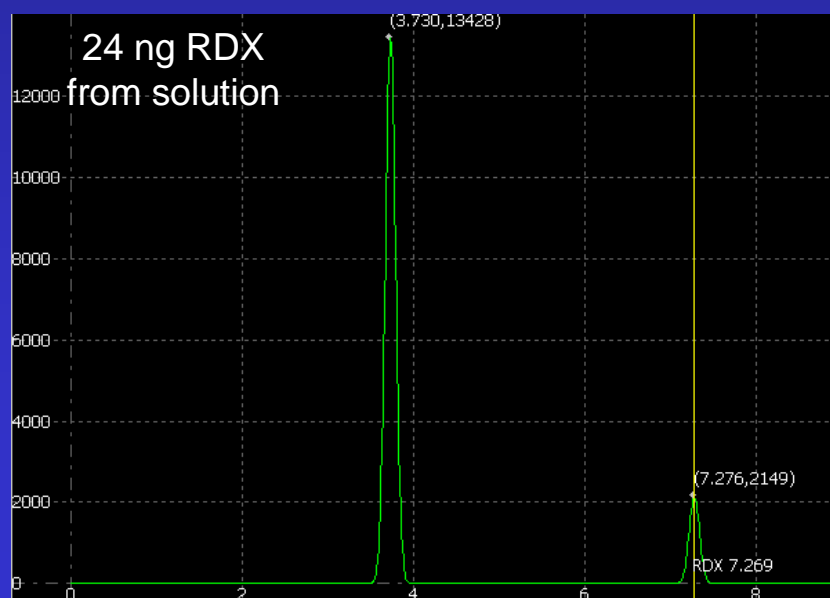
0.01% and 0.1% TNT and Comp C4  
coated on 20 – 30  $\mu\text{m}$  inert silica particles

# IMS Experiments



## Conditions:

- PDMS membrane
- $^{63}\text{Ni}$  ionization
- $\text{CH}_2\text{Cl}_2$  dopant
- desorber 210 °C
- detector 162 °C
- 7 s sampling





# *Testing C4 SRM on Portal Explosives Detector*



Data courtesy of  
Wayne Smith, NIST



# ASTM E 2520-07 Standard Practice for Verifying the Minimum Acceptable Performance of Trace Explosives Detectors

## Tests successful alarm signal for low level solutions of RDX, PETN, and TNT



X XXXX

Instrument Manufacturer and Model Number \_\_\_\_\_

Serial Number \_\_\_\_\_

Test Kit Identification Number | \_\_\_\_\_

Instruction	Evaluation 1		Evaluation 2	
	pass	fail	pass	fail
1. Power on as described in 6.1.				
2. Preparation of work area is completed (5.1).				
3. Preparation of test swipes have been completed and are ready for evaluation as described in 5.2.				
4. Analyze instrument blank swipe as described in 6.3. To pass this test, there should be no explosives alarm signal.				
5. Analyze process blank swipe as described in 6.4. To pass this test, there should be no explosives alarm signal.				
6. Analyze test swipes as described in 6.5. To pass this test, the correct alarm signal should be observed for each corresponding test swipe (that is, a TNT alarm was observed for a TNT-dosed test swipe).				
• TNT				
• RDX				
• PETN				
7. Analyze instrument blank swipe as described in 6.6. To pass this test, there should be no explosives alarm signal.				
• Following TNT test swipe				
• Following RDX test swipe				
• Following PETN test swipe				

Test Operator Name (Print) \_\_\_\_\_

Test Operator Signature \_\_\_\_\_ Date \_\_\_\_\_

Test Operator Comments \_\_\_\_\_

FIG 1 Explosives Detector Performance Checklist

# ***Quality Assurance in Forensic Science***

- ✓ ASTM Committee E30 Forensic Sciences
- ✓ ASCLD-LAB Certification of agency
- ✓ Collaborative Testing Services
- ✓ ABC Certification of forensic scientists
- ✓ TWG/SWGs
- ✓ NIST Standard Reference Materials  
(alcohol, DNA, drugs-of-abuse, arson)



**Issues and opportunities in technology development and implementation, education, and quality assurance – or - Can we put *Science* in U.S. Forensic Science?**

# ***Issues***

## **Technology Development:**

- Research labs at National Forensic Laboratories (FBI, BATFE) are small or non-existent (also true for TSA, C&BP), fugetaboutit at state and local level
- Problems at the labs: caseloads are extremely high, no funding for basic research, lack of strong. enduring relationships with Universities, formal education largely undervalued, internal “guild” system in place for training and advancement
- American Academy of Forensic Sciences is a generous misnomer

## **Implementation:**

- Serious lack of funding at the state and local lab level (equipment, personnel)
- Limited budgetary opportunity for participation in training and interfacial activities (attending ForSci meetings, TWG/SWG meetings, ASTM meetings)
- Only limited database information on trace evidence is communally gathered and shared

# *Issues*

## *Education:*

- Funding sources: ~~NSF~~, NIJ (issues: yearly congressional agenda, relationship with FBI, staffing, limited funding)
- Limited number of comprehensive and stellar academic programs for advanced degrees in specific areas (forensic chemistry, biology, pathology, etc.)
- Few links (in the US) between labs and universities

## *Quality Assurance:*

- Lack of continued reliable funding for TWG/SWGs
- ASTM E30 strongest in arson
- ASCLD/LAB Certification is a double-edged sword limiting advancement of technology

# *Where do we go from here?*



- Increase funding for personnel and equipment for state and local crime labs to address case loads – particularly for drug analysis (regional centers with advanced equipment/technology/training?).
- Establish a national grant system for lab personnel to attend scientific, training, and quality assurance meetings.
- Establish a grant system to subsidize the purchase of CRMs.
- Whose on first (for ForSci funding), NSF or NIJ? Establish a RANN2 that values research applied to national *forensic* needs.
- Establish National Databases (analogous to DNA) for trace evidence.
- Live in the world post-Daubert: Forensic Science school for judges.
- Encourage forensic laboratory participation in homeland security needs...hmmm...