Welcome to the CLU-IN Internet Seminar

Site Characterization for Munitions Constituents

Sponsored by: EPA Federal Facilities Forum & USACE

Delivered: October 10, 2012, 1:00 PM - 3:00 PM, EDT (17:00-19:00 GMT)

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Although I'm sure that some of you have these rules memorized from previous CLU-IN events, let's run through them quickly for our new participants.

Please mute your phone lines during the seminar to minimize disruption and background noise. If you do not have a mute button, press *6 to mute #6 to unmute your lines at anytime. Also, please do NOT put this call on hold as this may bring delightful, but unwanted background music over the lines and interupt the seminar.

You should note that throughout the seminar, we will ask for your feedback. You do not need to wait for Q&A breaks to ask questions or provide comments. To submit comments/questions and report technical problems, please use the ? Icon at the top of your screen. You can move forward/ backward in the slides by using the single arrow buttons (left moves back 1 slide, right moves advances 1 slide). The double arrowed buttons will take you to 1st and last slides respectively. You may also advance to any slide using the numbered links that appear on the left side of your screen. The button with a house icon will take you back to main seminar page which displays our agenda, speaker information, links to the slides and additional resources. Lastly, the button with a computer disc can be used to download and save today's presentation materials.

With that, please move to slide 3.

Site Characterization for Munitions Constituents

Web-Based Document at:
http://www.epa.gov/fedfac/

Site Characterization for Munitions Constituents
(EPA-505-S-11-001, January 2012)

Sponsored by: US EPA Federal Facilities Forum
Hosted by: US EPA Clean Up Information Network
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USEPA Disclaimer

- This training is based on the EPA Federal Facilities Forum Issue Paper - Site Characterization for Munitions Constituents. The US Army Corps of Engineers trainers are providing technical expertise for munitions constituents sampling and analysis and are not presenting or discussing DoD sampling policy.

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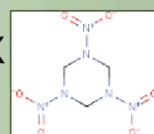
Energetics Background: Active Range Findings

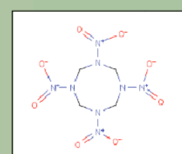
High Explosive Solid Residues in Impact Areas

Comp B (RDX/TNT)

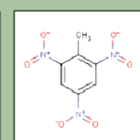
- Nitramines: RDX, HMX
- Nitroaromatics: TNT



RDX



HMX



TNT

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Residues at Active Demolition Training Ranges

Nuggets of C4 (RDX)

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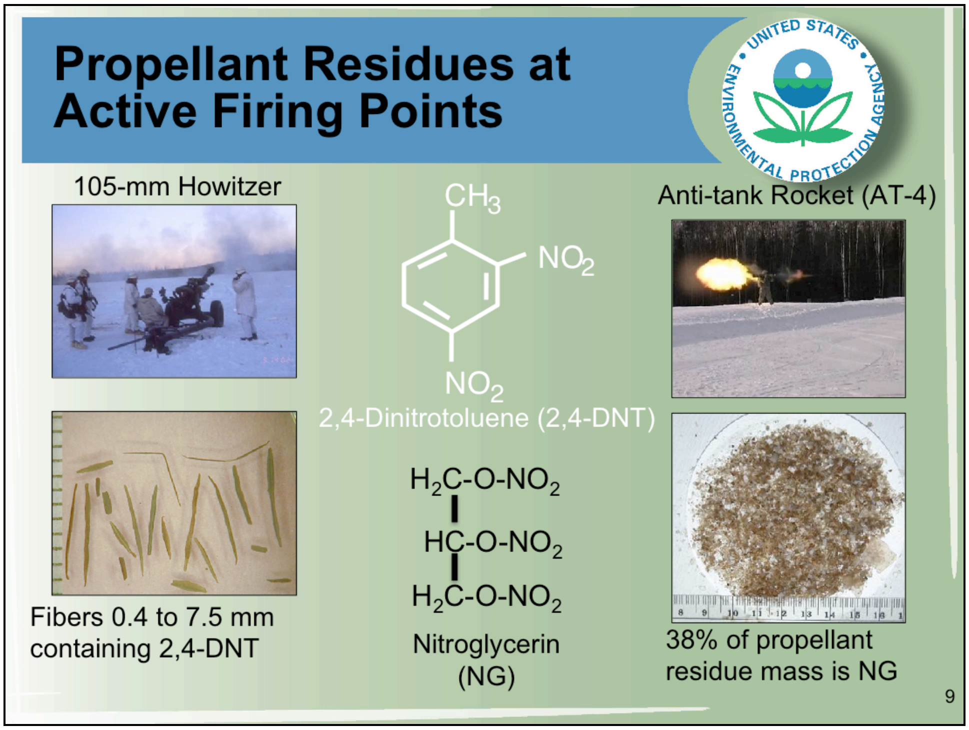
Propellant Residues at Active Firing Points
105-mm Howitzer
Fibers 0.4 to 7.5 mm containing 2,4-DNT
CH3
NO2
NO2
2,4-Dinitrotoluene (2,4-DNT)
H2C-O-NO2
HC-O-NO2
H2C-O-NO2
Nitroglycerin (NG)
Anti-tank Rocket (AT-4)
38% of propellant residue mass is NG
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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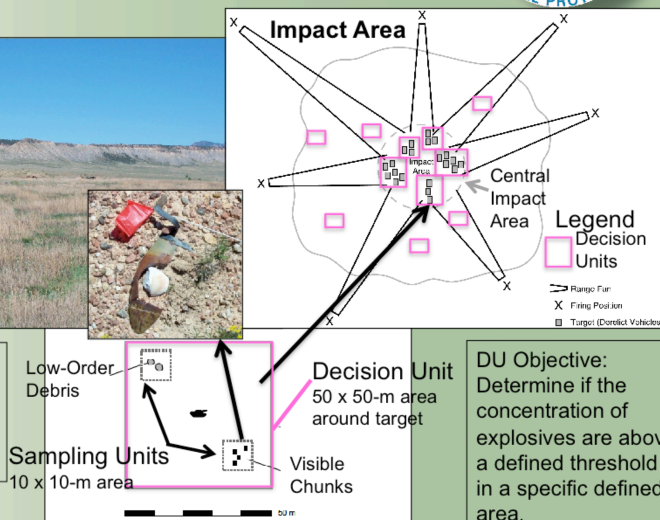
Terminology

- Incremental Sampling Methodology (ISM) – ISM is a structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of mean contaminant concentrations in volume of soil targeted for sampling.
- Decision Unit (DU) – the smallest volume of soil (or other media) for which a decision will be made based upon ISM sampling. A DU may consist of one or more sampling units (SUs).
- Sampling Unit (SU) – user-defined volume of soil (or other media) from which increments are collected to determine an estimate of the mean concentration for that volume of soil (or other media).
- Increment – a portion of the sampling unit that is collected with a single operation of a sampling device and combined with other increments to form an incremental sample.

For more details see, ITRC. 2012. *Incremental Sampling Methodology*. ISM-1. Interstate Technology and Regulatory Council, Incremental Sampling Methodology Team. Washington, DC, http://www.itrcweb.org/ISM-1/

Example of an Impact Area Decision Unit



SU Objective: Assess the concentration of residue around specific items of interest.

DU Objective: Determine if the concentration of explosives are above a defined threshold in a specific defined area.

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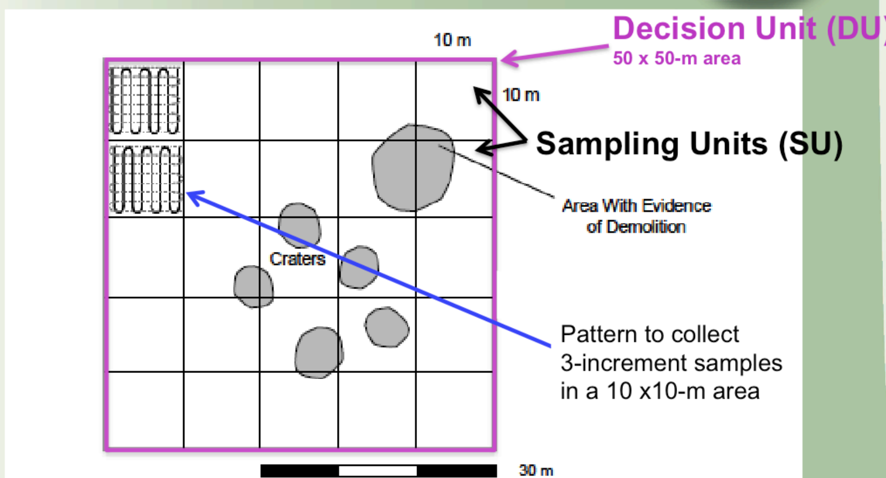
Example of a Demolition Area Decision Unit



DU Objective: Determine if concentrations are above a threshold concentration.
SU Objective: Assess the concentration of residue on the scale of individual craters.

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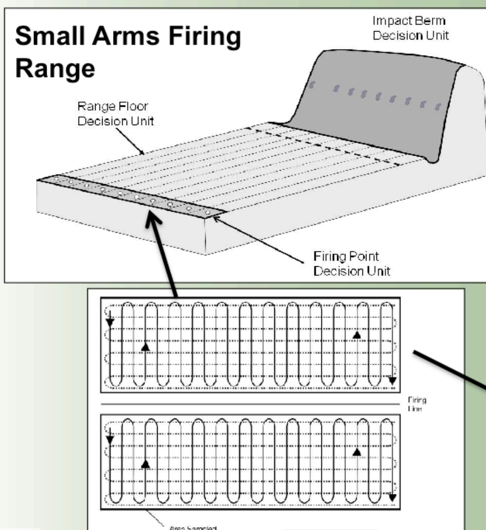
Example of a Small Arms Range Decision Unit



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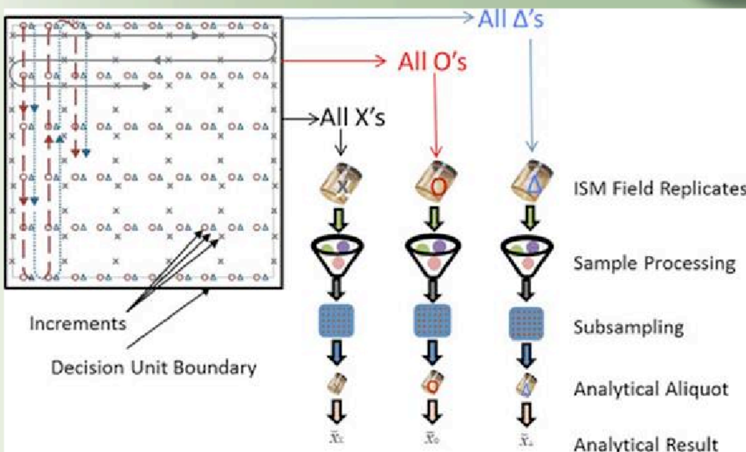
DU Objective: Determine the concentration of metals and propellants in the area of interest.

Incremental Sampling Methodology (ISM)



Detailed discussion of ISM in ITRC. 2012. *Incremental Sampling Methodology*. ISM-1. Interstate Technology and Regulatory Council, Incremental Sampling Methodology Team. Washington, DC
http://www.itrcweb.org/ISM-1/

Adequate Sample Mass and Number of Increments

- Soil samples collected within areas of extreme spatial heterogeneity, i.e. DU
- Samples must have sufficient mass to include all constituents in the same proportions as the soil surface (CRREL recommends including vegetation)
- Multi-increment samples composed of a minimum of 30 soil increments “aliquots”. CRREL recommends collection of 50-100 increments.
- Energetic chunks not collected (weighed only by EOD).

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CRREL Recommended Sampling Depth for Active Ranges

- Firing point residues are associated with NC propellants and remain at surface
- Impact area residues are result of low-order detonations and ruptured rounds and the bulk remains at the surface
- For most ranges a sampling depth of 2.5 to 5 cm is recommended
- There are exceptions
  - Demolition and hand grenade ranges
  - Ranges with water-saturated soils/ sediments

Portion of Core	Depth (cm)	DNT (µg/g)
Surface	0 - 1	7.8
Root Zone	1 - 3	1.2
Mineral Soil	3 - 10	ND

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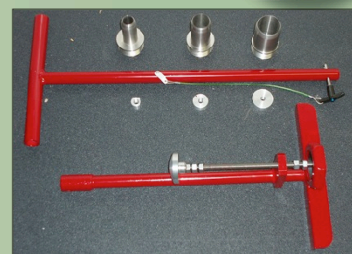
ISM Tools



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Sampling Recommendations for Training Ranges

- Stratify ranges into functional areas, based on past use if known
- Divide functional areas into DUs based on sampling objectives
- Collect multi-increment samples from within each DU using a systematic-random pattern
- Collect field replicate samples (3) to provide estimate of uncertainty
- No chunk sampling! If needed use Expray or Chemometric Test Kits

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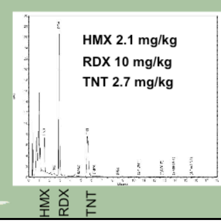
Laboratory Processing

- Air-dry
- Sieve using 2-mm mesh
- Particle size reduction
  - Grind for the appropriate amount of time without heating the sample
- Subsample
  - Many increments (>30) through the thickness of the sample
- Solvent extraction with acetonitrile
- Analysis
  - HPLC-UV or HPLC-MS



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Particle Size Reduction by Machine Grinding

- The multi-increment sample contains very few energetic particles or propellant fibers compared to the total soil matrix.
- Grinding reduces particle size of course soil to texture of flour (<75 µm) and vastly increases number of particles.

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Milling Precision

Subsample	RDX Conc. (µg/g) Not Ground	RDX Conc. (µg/g) Ground	TNT Conc. (µg/g) Not Ground	TNT Conc. (µg/g) Ground
1	1.68	4.75	0.25	2.03
2	1.77	4.71	1.81	2.04
3	1.46	4.80	0.37	2.00
4	3.80	4.73	1.48	2.03
5	7.83	4.67	7.93	1.97
6	1.81	4.66	0.56	2.00
7	2.35	4.62	0.35	1.90
8	2.51	4.62	0.75	2.02
9	2.08	4.64	0.56	1.97
10	1.98	4.69	0.35	1.98
11	1.68	4.66	0.62	1.90
12	13.0	4.60	5.62	1.91

*50-g subsamples.

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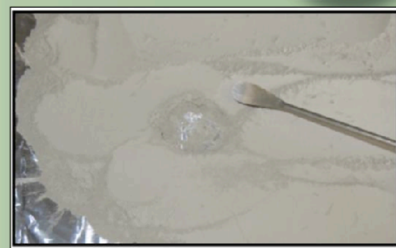
Analytical Subsampling



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Can a 10-g Subsample Represent a >1-kg Field Sample?

Location	Sample	Mass (g)	Concentration (mg/kg)			
			HMX	RDX	TNT	2,4-DNT
Demolition Range	**Subsample 1**	10.0	1.98	11.7		4.58
	Subsample 2	10.0	2.00	11.6		4.92
	Subsample 3	10.0	1.98	11.8		5.22
	Rest of Sample	1766	**2.02**	**11.9**		**4.81**
	Relative Percent Difference		**1.5%**	**1.7%**		**2.1%**
Artillery Impact Area	**Subsample 1**	10.0	2.72	14.1	1.60	
	Subsample 2	10.0	2.72	14.1	1.60	
	Subsample 3	10.0	2.60	13.9	1.63	
	Rest of Sample	1278	**2.76**	**14.3**	**1.56**	
	Relative Percent Difference		**3.1%**	**2.0%**	**3.2%**	

Yes, if the sample is processed correctly!

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Solvent Extraction

- Standard Method: Sonic bath (cooled) for 18 hours

- Alternative: Shaker table for 18 hours
  - ambient temperature
  - larger batches
  - less noise (!)

Analytical Methods

- **Method 8330B:** Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)
  - UV Detector or MS
  - Detection limits around 0.05 mg/kg

- **Method 8095:** Nitroaromatics and Nitramines by Gas Chromatograph(GC)
  - Electron Capture Detector
  - Detection limits less than 0.010 mg/kg for TNT and RDX

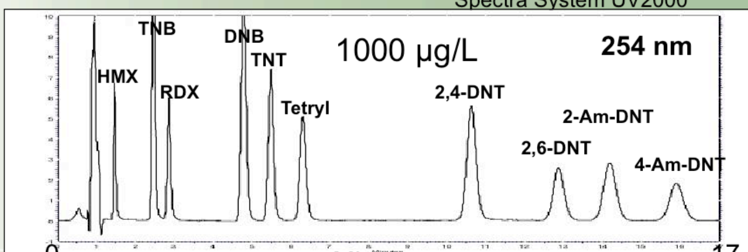
Sample processing is the same for both methods.

HPLC-UV Instrumentation

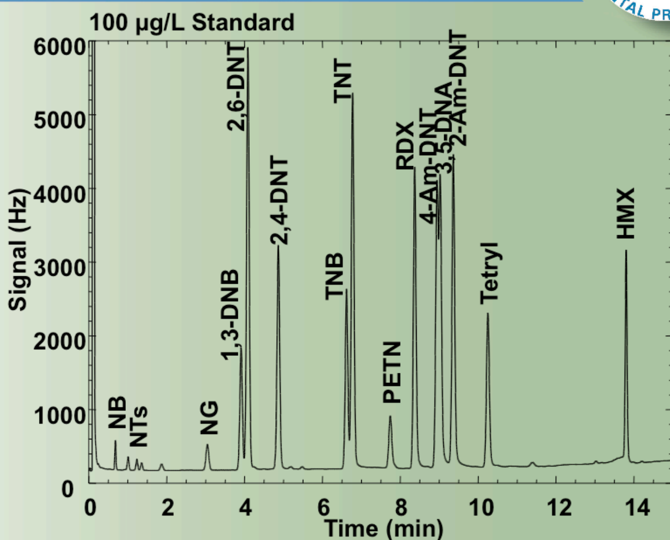
Column:
Waters Nova-Pak C8
60Å 4µm
3.9 X 150mm

Eluent:
1.4 mL/min
15:85 2-Propanol:Water
28°C

Detector:
Thermo Separations Products
Spectra System UV2000



Chromatogram (GC-µECD) Calibration Standard



Determination: HPLC or GC?

HPLC

- More rugged
- Better reproducibility (smaller analytical error)

GC

- Lower detection limits for most analytes
- Determination of nitramines and nitrate esters requires constant injector maintenance
- Appropriate for soils with explosives concentrations ranging from 1 to 100 µg/kg
- Good method for confirmation

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Important Sample Preparation Considerations

- The entire sample received by an analytical lab must be processed (not just a small portion off the top). Contracts must incorporate this requirement.
- Attention given to analytical error should also be directed to field sampling and laboratory subsampling error.
- Appropriate analytical method must be used. Standard methods for semi-volatiles **ARE NOT** appropriate for explosives and propellants.
- All determinations MUST BE CONFIRMED either by secondary column or another detector.

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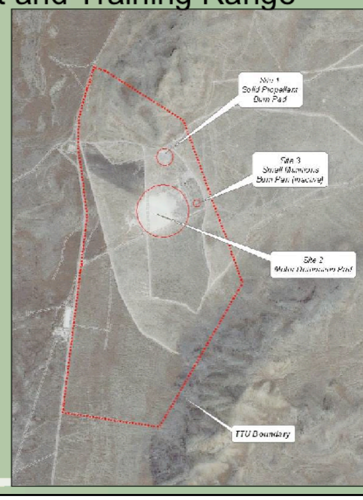
Case Studies: Open Burn Open Detonation

Repeated Multi-Incremental Explosive Residue Characterization at the Utah Test and Training Range

- Approximately 1500 acres
- RCRA permitted treatment facility
  - ➢ Operated for past 30+ years
  - ➢ Large motor detonations since early 90's
  - ➢ RCRA permit finalized in 2003
- Only facility in US where detonations of large missile motors greater than 10,000 lbs NEW are permitted



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Rocket Motor Thermal Treatment

Titan Booster Segment Disposal

Trident C4 Motor Disposal

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Low Order Versus High Order Detonation

Titan Booster Segment Disposal

Trident C4 Motor Disposal

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RCRA Sampling Requirements

- Part B permit requires annual soil sampling to support human health and ecological risk assessments and ensure that risk thresholds are not exceeded.
- Discrete sampling events conducted in 1989, 1991, 2002, and 2004
- Multi-Incremental sampling conducted in 2005 and 2006
- GOAL: Collect accurate, repeatable data that reflects actual site conditions

- 40 sample areas, 31 operational areas, 6 non-operational areas, 3 "background"
- 100 x 100m grids
- Surface samples - top 1-2 cm
- Dedicated field equipment no decon
- Field triplicates to evaluate reproducibility
- One sample analyzed in triplicate before and after milling to evaluate metal contamination
- Analysis for metals and perchlorate (STL-Denver) and explosives (STL and CRREL)

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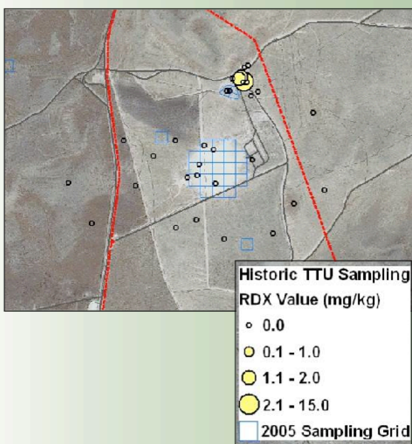
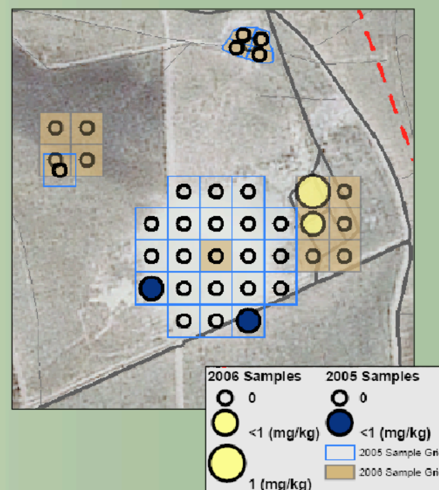
RDX Results: Discrete Versus Incremental Sampling Methodology

Discrete



ISM



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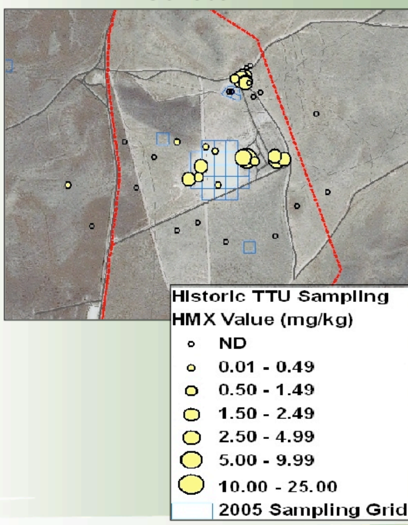
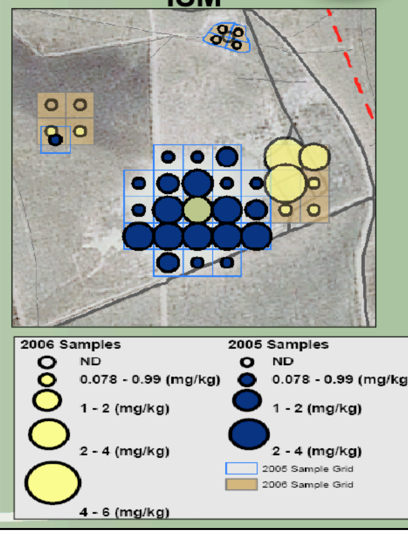
HMX Results

Discrete



ISM



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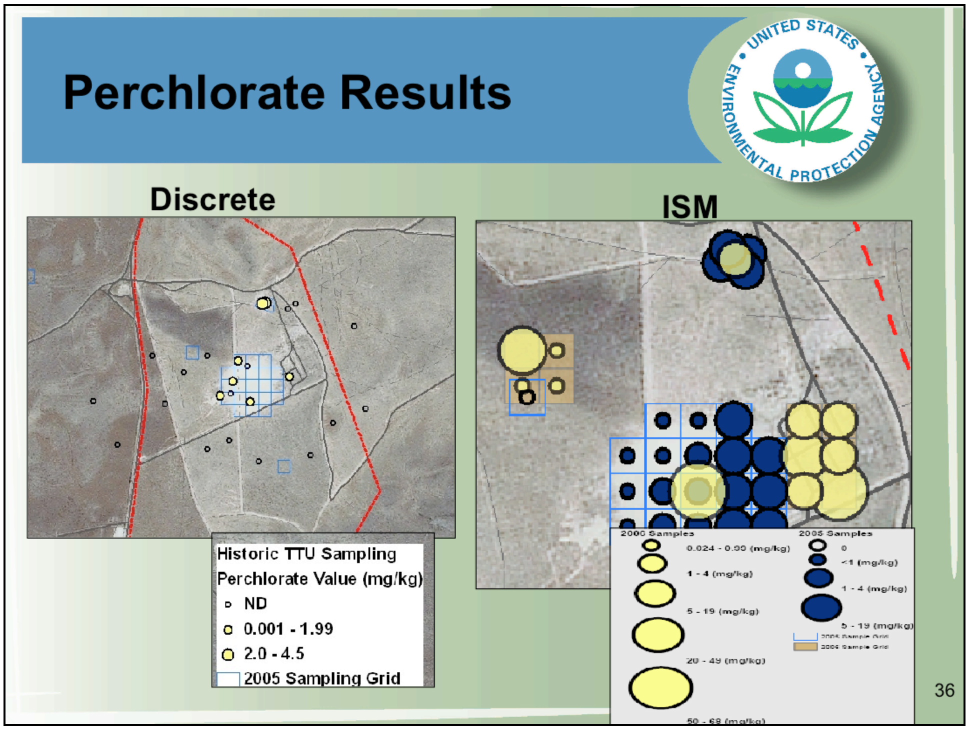
Perchlorate Results
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Discrete
ISM
Historic TTU Sampling
Perchlorate Value (mg/kg)
ND
0.001 - 1.99
2.0 - 4.5
2005 Sampling Grid
2000 Samples
0.024 - 0.99 (mg/kg)
1 - 4 (mg/kg)
5 - 19 (mg/kg)
20 - 49 (mg/kg)
50 - 68 (mg/kg)
2005 Samples
0
<1 (mg/kg)
1 - 4 (mg/kg)
5 - 19 (mg/kg)
2005 Sample Grid
2006 Sample Grid
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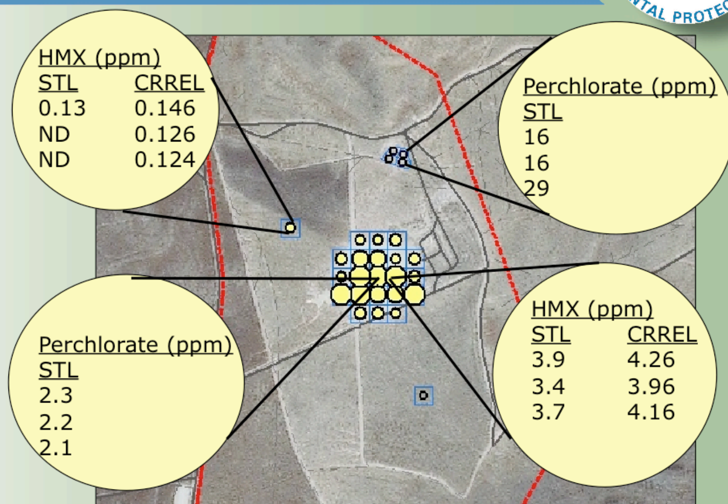
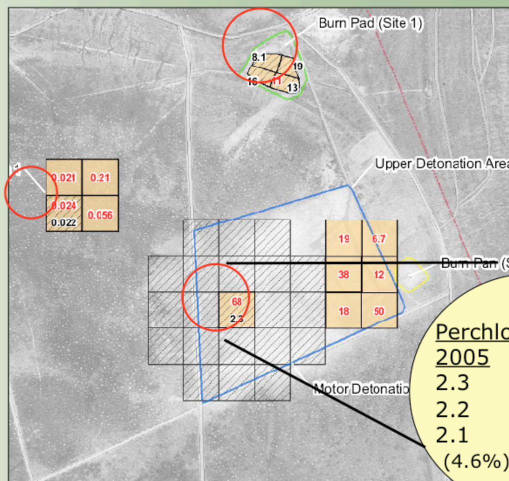
Replicate Sample Results 2005



2005 vs 2006: Perchlorate



Perchlorate (ppm)

2005	2006
2.3	68
2.2	62
2.1	86
(4.6%)	(17.4%)

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Conclusions

- Multi-Incremental sampling was successful in defining the spatial distribution and level of residual energetics in support of the site risk assessments
  - Within year repetition was excellent
  - Year to year repetition was excellent
  - May allow for relaxation of sampling frequency
- Should reconsider the determination of Exposure Point Concentrations

Case Studies: Firing Points

- Propellants residues are produced during military live-fire training:
  - firing of weapon systems
  - disposal of excess propellant charges
- Firing points are relatively small in size and repeatedly used.

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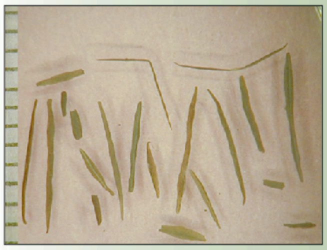
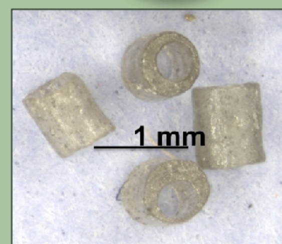
Physical Form of Propellant Residue



From 105-mm howitzer (FP Nieber 2002)

From 120-mm mortar (FP Lower Fox 2006)



Mk19 40-mm Grenade Machine Gun (40-90 Range JBER 2008)

Propellant residue from howitzers, mortars, and machine guns consists of discrete fibers or fragments of partially consumed propellant.

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Propellant Residues at Firing Points

Objective of soil monitoring:

- Estimate the mean concentration of 2,4-DNT and NG in the surface soil at mortar and howitzer firing points.
- Determine if 2,4-DNT and NG are accumulating at firing points.

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Firing Point Residue

Month - Year	Increments per sample	Mean Field Sample Mass (kg) <2mm	2,4-DNT (mg/kg) in Replicate Field Samples	Mean 2,4-DNT (mg/kg)	RSD (%)	95% UCL (mg/kg)
July 2003	49	3.0	0.43, 0.49, 0.62, 0.70, 0.73	0.59	22	0.73
June 2005	64	1.7	0.81, 0.95, 1.19, 1.23, 1.53, 1.63, 1.93, 2.20	1.43	33	1.8
July 2007	64	2.8	0.77, 1.00, 1.02	0.93	15	1.2
Sept 2007	64	3.0	1.44, 1.62, 1.85	1.64	13	2.0
May 2008	64	1.6	1.12, 1.47, 1.89	1.49	26	2.2
Sept 2008	56	1.1	0.52, 0.74, 1.13	0.80	39	1.3
July 2009	110	3.0	0.55, 0.64, 1.37	0.85	53	1.6

No evidence of accumulation at current rate of use.

Concentration of Nitroglycerin in Firing Point Soils

40-mm Machine Gun
580 to 5,700 mg/kg

AT-4 rocket firing points in Canada
1000s mg/kg behind the firing line
(Thiboutot *et al.* 2003, 2004, 2010).

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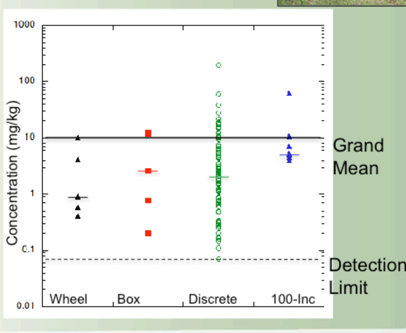
Mortar Firing Point Sampling Strategy Comparisons

Total Mass of Soil Collected:
21 kg

Grand Mean:
10 mg/kg



NG Concentration (mg/kg)				
	Wheel (n=5)	Box (n=5)	Discrete (n=100)	100-inc (n=10)
Minimum	0.41	0.20	<0.07	4.0
Maximum	10	13	200	63
Median	**0.90**	**2.6**	**1.8**	**5.0**
Mean	3.2	5.6	7.3	11

Case Studies: Impact Area and Demolition Training Range

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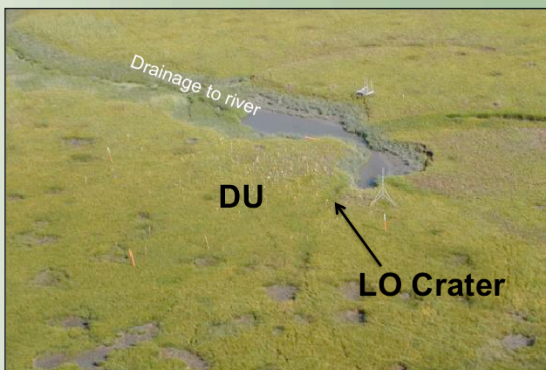
Impact Area Decision Unit

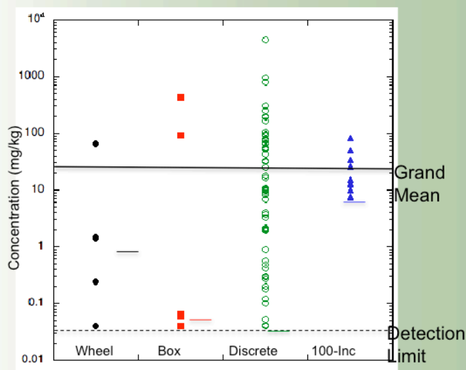


Areal oblique view of the 20 x 20-m DU from which 100-increment systematic-random sediment samples were collected. The DU was centered on and included the scatter area of the pieces of high explosives.

Incremental Sampling Methodology

- Each orange flag marks the position of a deposit of solid Comp B.
- A DU was centered on the Comp B scatter area for comparison of sampling methodologies.
- Five Wheel and Box samples collected in 2007.
- 200 discrete samples collected in 2007.
- Ten 100-increment systematic-random samples in 2007.
- Subsurface samples collected (2.5 - 5 cm, 5 to 15 cm and 15 to 25 cm) for one 100-increment sample in 2008.

Impact Area Sampling Strategy Comparison



Total Mass of Soil Collected:
38 kg

Grand Mean:
49 mg/kg

RDX Concentration (mg/kg)				
	Wheel (n=5)	Box (n=5)	Discrete (n=200)	100-inc (n=10)
Minimum	<0.04	<0.04	<0.04	7.3
Maximum	65	430	4,500	82
Median	**1.4**	**0.07**	**<0.04**	**14**
Mean	14	110	50	26

Sampling of Demolition Training Range

- A 30 x 30-m DU was centered on an area with visible C4 residue to compare sampling methods.
- Five Wheel and Box samples
- 100 discrete samples
- Ten 100-increment systematic-random samples

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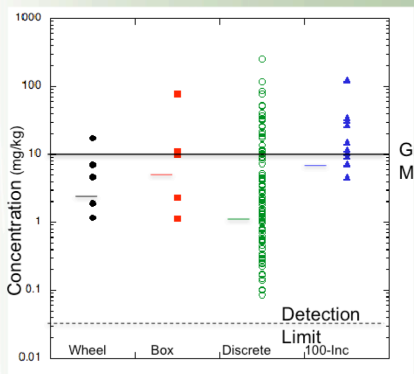
Demolition Range Sampling Strategies Comparison



Total Mass of Soil Collected:
30 kg

Grand Mean:
21 mg/kg

RDX Concentration (mg/kg)				
	Wheel (n=5)	Box (n=5)	Discrete (n=100)	100-inc (n=10)
Minimum	1.2	1.1	0.08	4.7
Maximum	17	77	250	130
Median	**4.6**	**10**	**2.0**	**14**
Mean	6.3	20	12	28

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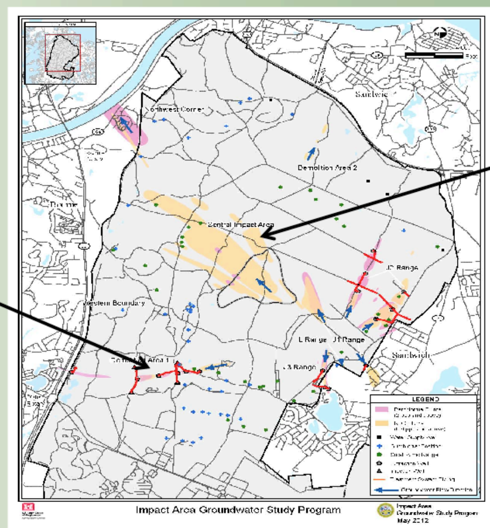
Groundwater Plumes at MMR

Impact Area
RDX Plume

Demolition Area
RDX and
Perchlorate
Plume



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Universal Findings: Artillery Range Impact Areas

- Most of the impact area is not contaminated with energetics.
- **Chunks of explosives are highly localized with elevated surface soil concentrations near low-order (partial) detonations.**
- Explosives-residues can be located anywhere within the Impact Area and not necessarily near targets.
- The energetics present in soil are RDX, HMX, TNT and in some cases a-DNTs (transformation products of TNT).
- Perchlorate originating from spotting charges may be present although not typically in soil (dissolves rapidly and migrates).
- White Phosphorus particles can be persistent in wetlands, i.e. anoxic (no oxygen) environment.

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Universal Findings: Demolition Ranges

- RDX residues from C4 (demolition explosive). Demolition explosives with RDX were developed during World War II.
- Residues can be deeper in soil profile than at other ranges because of continued use of demolition craters
- Propellant grains frequently found
- Demolition range at Massachusetts Military Reservation is largest identified source of RDX, HMX, TNT, and perchlorate in ground water

Metallic Residues at Small Arms Ranges

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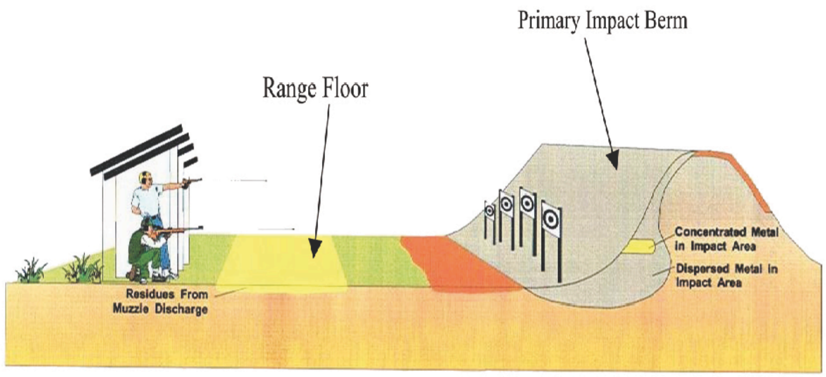
Generic Small Arms Layout



(modified from ITRC, 2003)

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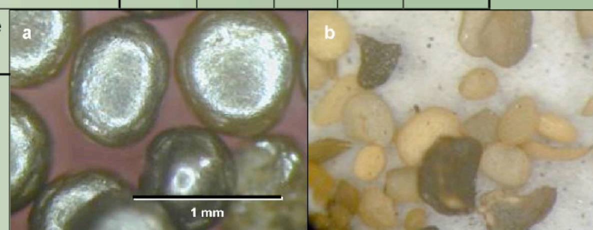
Small Arms Propellant Composition

Munition	Propellant	Composition (%)				
		NC	NG	DNT	DB*	DP**
9-mm Pistol	WPR289	79.1	12.5			0.9
M-16 Rifle						
5.56 mm (ball)	WC844	66.9	9.9		6.0	1.5
5.56 mm (tracer)	WC844	69.4	10.1		4.8	
M-14 Rifle (7.62 mm)	WC846	80.5	10.0	0.1	5.2	1.1
50 cal Machine gun	WC860	78.9	9.7		8.0	1.1
50 cal Machine gun	WC857	68.5	10.8		5.9	1.2

* DB – Dibutyl phthalate
**DP – Diphenyl amine



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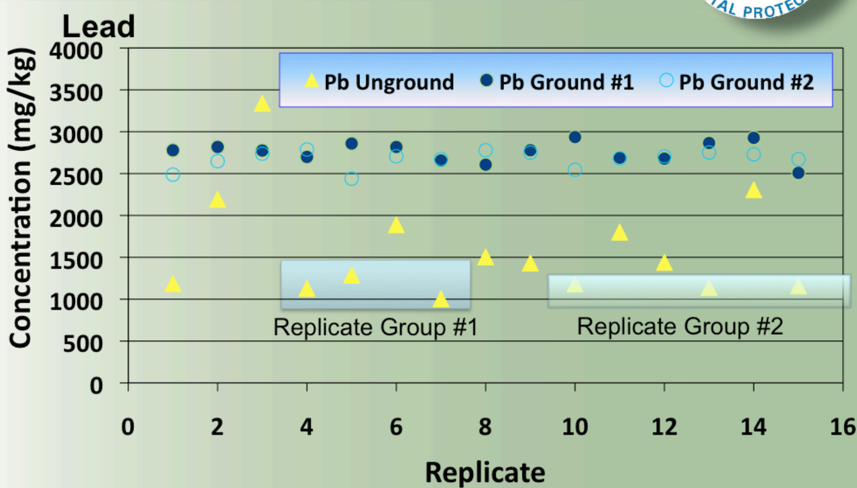
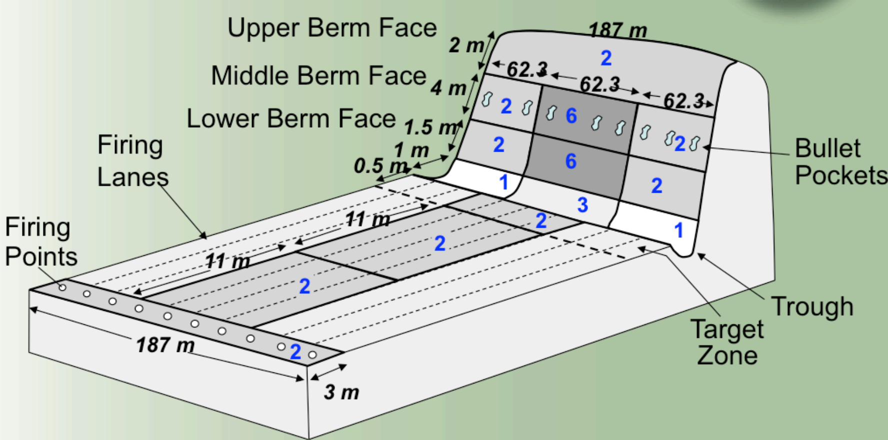
Impact of Milling on Precision



Case Study: Small Arms Training Ranges

Camp Edwards, MA
Small Arms Bravo Range



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Residues at Small Arms Range Firing Points

- Nitroglycerin (NG) and dinitrotoluene (DNT) present at small arms range firing points, initially
- Concentrations in surface soils at firing points
  - 9-mm pistol ranges (80 to 120 ppm at 29 Palms)
  - 5.56-mm rifle ranges (280 to 520 ppm at Ft. Lewis)
  - Machine gun ranges
  (10 to ppm µg/g FRA Oates)
- Most residue within 10-m of firing line

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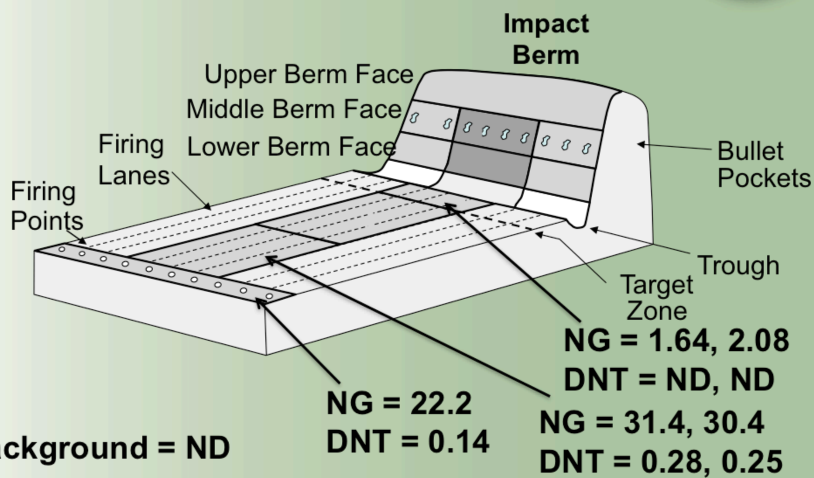
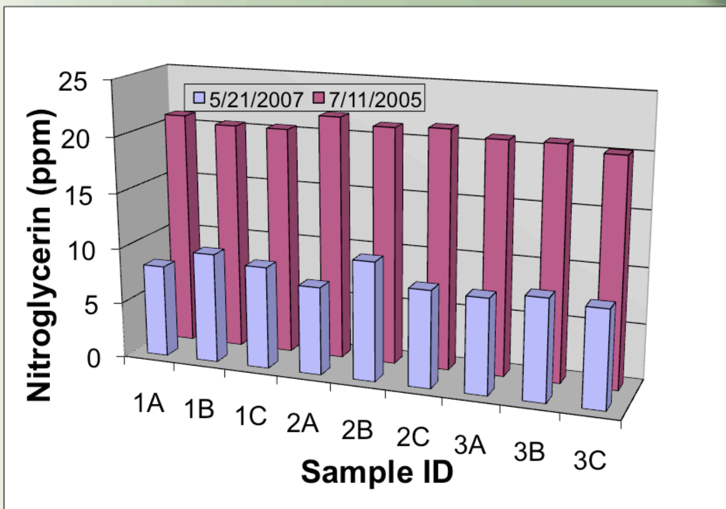
Nitroglycerin and Dinitrotoluene (mg/kg) Surface Soil



Background = ND

ISM Replicate Precision



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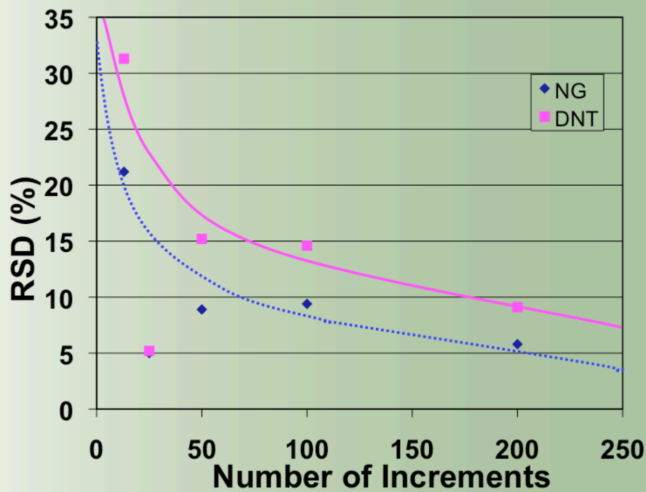
Sample Precision



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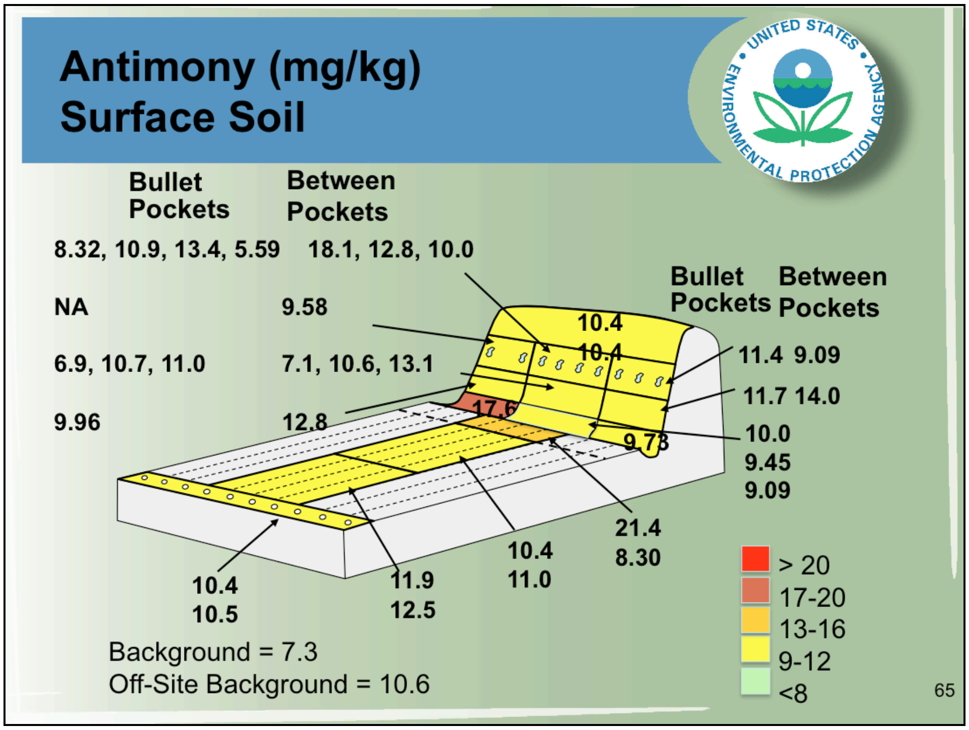
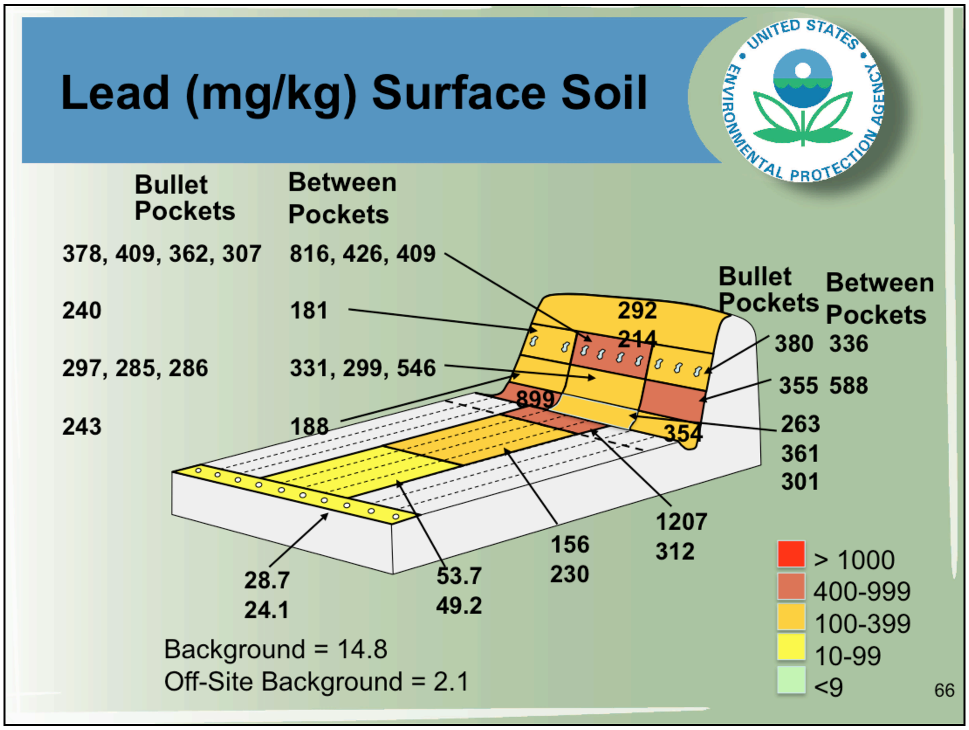
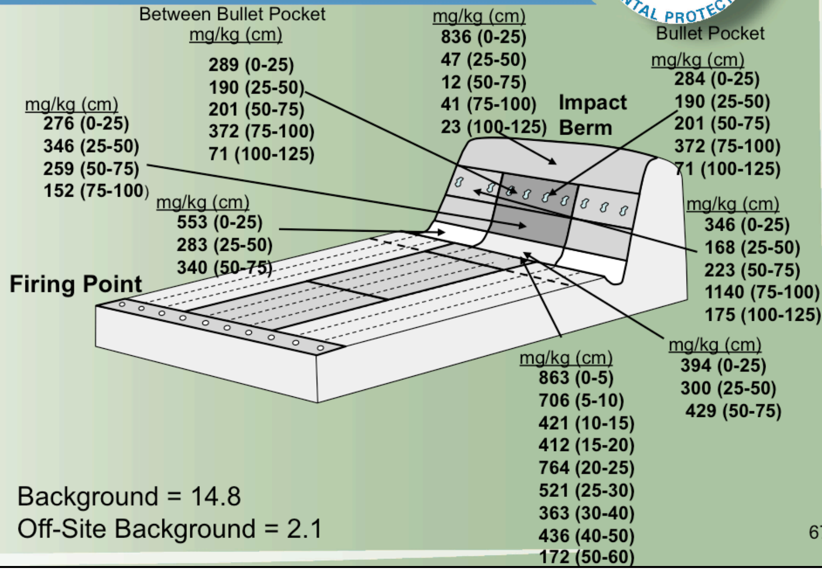


Antimony (mg/kg)
Surface Soil
Bullet Pockets
Between Pockets
8.32, 10.9, 13.4, 5.59
18.1, 12.8, 10.0
NA
9.58
6.9, 10.7, 11.0
7.1, 10.6, 13.1
9.96
12.8
10.4
10.4
17.6
9.73
Bullet Pockets
Between Pockets
11.4 9.09
11.7 14.0
10.0
9.45
9.09
21.4
8.30
10.4
11.0
11.9
12.5
10.4
10.5
Background = 7.3
Off-Site Background = 10.6
> 20
17-20
13-16
9-12
<8
65



Lead (mg/kg) Surface Soil
Bullet Pockets
Between Pockets
378, 409, 362, 307
816, 426, 409
240
181
297, 285, 286
331, 299, 546
243
188
292
214
899
354
Bullet Pockets
Between Pockets
380 336
355 588
263
361
301
1207
312
156
230
28.7
24.1
53.7
49.2
Background = 14.8
Off-Site Background = 2.1
> 1000
400-999
100-399
10-99
<9
66

Lead (mg/kg) by Soil Depth (cm)



Background = 14.8
Off-Site Background = 2.1

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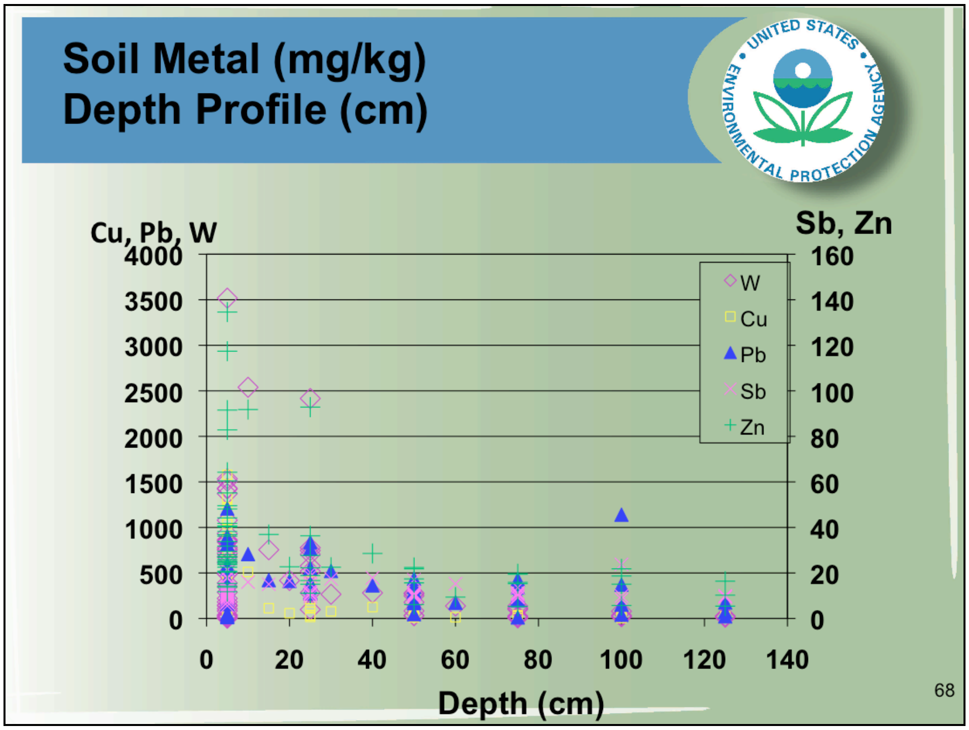
Soil Metal (mg/kg)
Depth Profile (cm)
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Cu, Pb, W
Sb, Zn
4000
3500
3000
2500
2000
1500
1000
500
0
160
140
120
100
80
60
40
20
0
W
Cu
Pb
Sb
Zn
0
20
40
60
80
100
120
140
Depth (cm)
68

Successful SAR Characterization Needs

Activity	Yes	No
ISM, 30+ increments	√	
Grab Sampling		√
Field Splitting		√
Sieving	√	
Milling necessity	√	
Increased digestion mass		√
Increased digestion time		√
Subsampling	√	

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CONCLUSIONS

- Ranges and OB/OD units have unique patterns of contamination due to the type of environmental releases that have occurred.
- Significant heterogeneity occurs as a result of detonation and combustion effects, and the particulate chemical characteristics of energetic materials and metals on ranges and OB/OD units.
- Demonstrated sampling and analytical procedures can be utilized to increase sample representativeness and control heterogeneity effects.

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SUMMARY

- Use of the Technical Project Planning (TPP) or Systematic Project Planning (SPP) process is important to gather all historical use and disposal activities at ranges and OB/OD units.
- Historical records of use and disposal and will still likely be incomplete.
- A well developed Conceptual Site Model (CSM) should be utilized to design the site Sampling Units (SU) and Decision Units (DU).

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SUMMARY (CONT.)

- If correctly applied, the ISM sampling methodology can control the accuracy, bias, and precision of sampling results within the SU/DU.
- Representativeness of sampling results is based on application of *all components* of the ISM methodology
- Elimination of steps in the ISM methodology (“ISM Lite”) will degrade data quality, and compromise accuracy gained from other steps in the process.
- *All* steps in the ISM process are important!

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Questions

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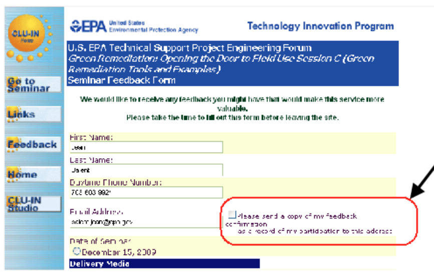
Resources & Feedback

- To view a complete list of resources for this seminar, please visit the **Additional Resources**
- Please complete the **Feedback Form** to help ensure events like this are offered in the future



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