Determination of Lead in Dust Wipes using Field Analytical Technology

Presented by

U.S. Environmental Protection Agency's (EPA) Office of Superfund Remediation and Technology Innovation (OSRTI) and

Office of Research and Development (ORD)

and the Department of Energy's (DOE)
Oak Ridge National Laboratory (ORNL)

1

Background
Environmental Technology Verification Program

- **Early 1990s - Need for environmental technology verification identified**
  - Slow rate of innovation; poor U.S. markets
  - Lack of credibility of new technologies
  - Inertia of system, risk aversion of purchasers and permitters
  - Burgeoning international market
- **EPA initiates ETV in October, 1995**

ETV Objectives

- Provide credible **performance data** for **commercial environmental technologies** to aid
  - **vendors in selling innovative technologies,**
  - **purchasers in making decisions to purchase innovative technologies, and**
  - **regulators in making permitting decisions regarding environmental technologies.**

ETV Successes

- **240 Verifications, 78 protocols** to date
- Vendor demand continues – over 100 technologies in testing/evaluation, over 100 applications pending
- Increasing funding from vendors and others
- 805 Stakeholders in 21 groups
- Commendations from EPA science and policy advisory boards
- Supports regulatory and voluntary Agency, other Federal and state programs
- Growing international interest
- New role in homeland security verifications

ETV Verifies only

- ***Definition:* *Verify* is to determine performance under test plan defined conditions**
  - **No** winners or losers
  - **No** approvals
  - **No** certification
  - **No** pass or fail
  - **No** guarantees
- **Responsibility rests with the technology user to correctly choose and apply technologies**

Stakeholder Roles

- Help set verification priorities
- Review protocols and operating procedures
- Review other important documents
- Assist in designing and conducting outreach activities
- Serve as information conduits to their constituencies

ETV Centers

- **ETV Air Pollution Control Technology Center**
  - *Research Triangle Institute*
- **ETV Drinking Water Systems Center**
  - *NSF International*
- **ETV Greenhouse Gas Technology Center**
  - *Southern Research Institute*
- **ETV Advanced Monitoring Systems Center**
  - *Battelle*
- **ETV Water Quality Protection Center**
  - *NSF International*
- **ETV- Building Decontamination Center**
  - *Battelle*
- **ETV P2 Coatings and Coating Equipment Pilot**
  - *Concurrent Technologies Corporation*

46 Verifications in 2003

- AMS: 5 Arsenic Detection; 5 Mercury CEMs; 1 Onboard Mobile Emission Monitor; 1 Portable Multi-Gas Emission Monitor; 2 Multi-Parameter Water Probes; 6 Cyanide Detection Kits
- SCMT: 1 Lead in Dust; 2 Groundwater Sampling Devices
- APCT: 3 Mobile Source Devices
- GHG: 1 Fuel Cell; 2 Micro-turbine CHP; 1 Vehicle Axle Lubricant; 1 Natural Gas Dehydration
- DWS: 2 Filtration Technologies
- WQP: 5 Residential Nutrient Reduction Systems; 1 Animal Waste Treatment (Solids Separator); 3 UV Disinfection
- CCEP: 1 Liquid Paint; 1 UV Curable Coating; 1 High Transfer Efficiency Paint Spray Gun
- P2-MF: 1 Sludge Reduction

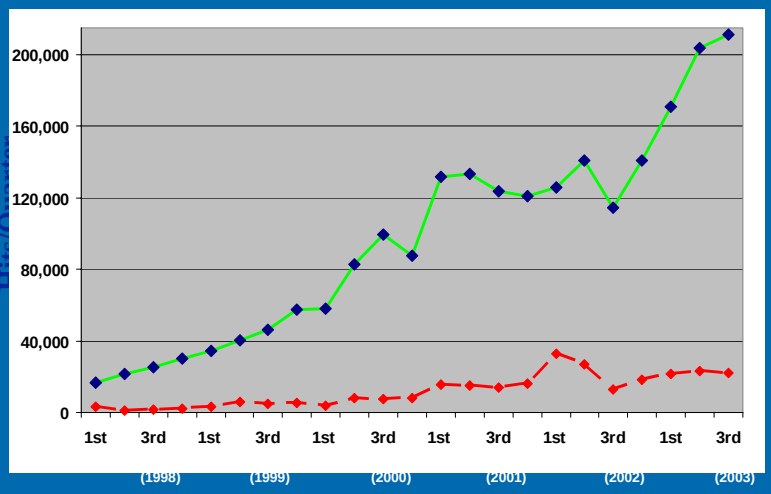
Projections for 2004

- **Over 80 verifications**
  - **half in base ETV**
  - **half in homeland security technologies**

ETV is partnering with ..

- US National Oceanic and Atmospheric Administration
  - Multi-parameter water probes
- US Coast Guard
  - Ballast water treatment
- US Dept of Energy, State of Massachusetts
  - Continuous emission mercury monitors
- US Dept of Defense
  - Monitors for explosives; PCBs in soils; dust suppressants
- States of Alaska, Pennsylvania
  - Drinking water arsenic treatment
- States/counties in Georgia, Kentucky, Michigan
  - Storm water treatment
- States of New York, Colorado
  - Waste to energy
- USDA
  - Ambient ammonia monitors

www.epa.gov/etv

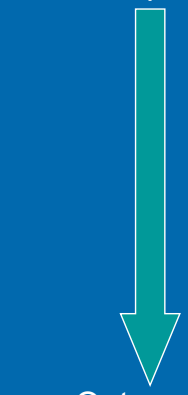


Note: There were 76,588 total hits and 7,075 international hits in September 2003.

Getting to ETV Outcomes

Measuring outputs to outcomes

Outputs



Outcomes

- Number of protocols and verifications
- **Value placed on ETV by vendors in selling and innovating technology**
- **Value to potential purchasers; influence of ETV on purchase decisions**
- **Use of better technologies; reduced emissions because of ETV**
- Reduced exposure; reduced risk because of ETV
- Improved health/environmental quality because of ETV

12

Overview of Environmental Technology Verification Process

Statisticians

Project Officers

Developers

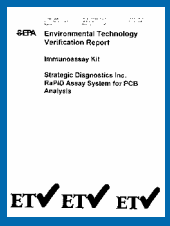
Chemists

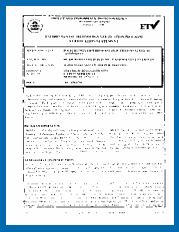
Stakeholders

Experimental Plan

Samples are collected, homogenized, labeled, and assembled for distribution.

Technology developers analyze randomized samples under field conditions.





Product is report and verification statement.

13

Lead in Dust:
Rationale for Performance Verification

"Childhood lead poisoning remains a major preventable environmental health problem in the United States."

- Centers for Disease Control and Prevention

"Children are most frequently lead poisoned by household lead paint dust."

- Massachusetts Dept of Public Health

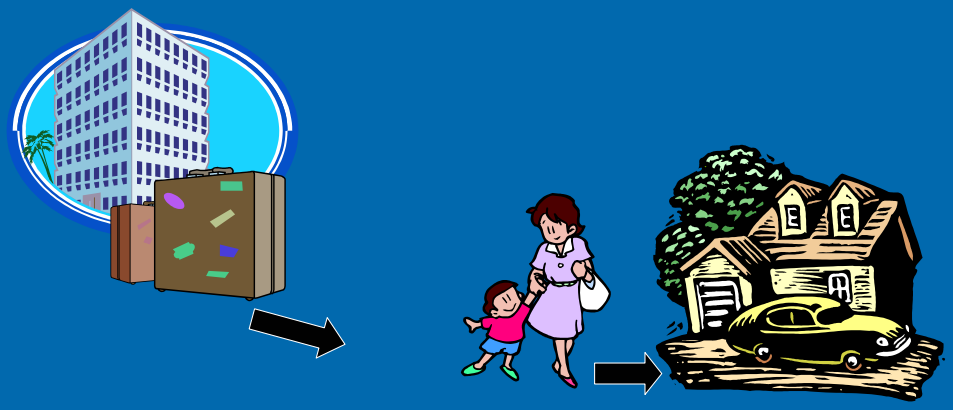
Selection of the Most Appropriate Material to Test

Technical panel prioritized current industry needs for evaluation of field technologies for detection of lead as:

- DUST
- PAINT
- SOIL

Fundamental Issue:

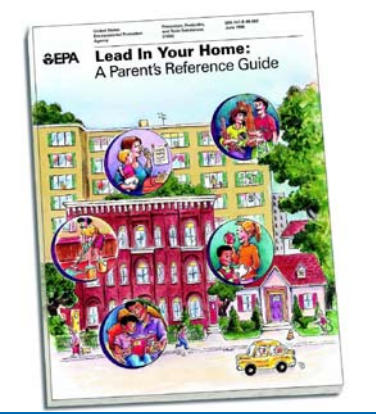
Can Field Analytical Technology be Used to Facilitate Home Reuse Following Remediation?



16

Why “dust wipes” versus “bulk dust”?

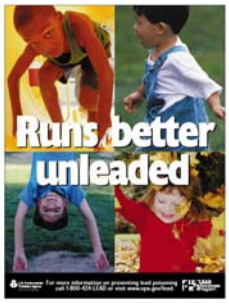
- Wipe sampling estimates surface lead loading
  - μg of lead per unit area
- Risk-based dust-lead loading standards established based on dust wipe sampling
- Testing under the NLLAP is restricted to dust wipes.
- Readily available ELPAT samples with certified concentrations
  - “Real-world” samples of known content



17

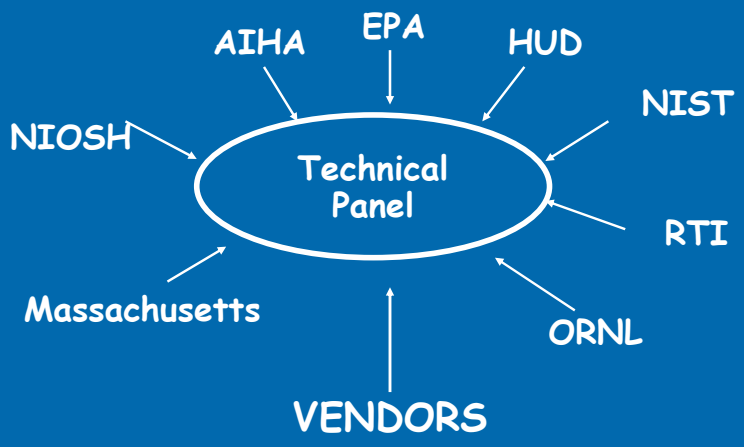
What were the regulatory drivers for this dust wipe testing?

- ETV tests provide information on potential applicability of field technologies for clearance testing.
- Relevancy to clearance levels†
  - 40 µg/ft^2 floors
  - 250 µg/ft^2 window sills
  - 400 µg/ft^2 window troughs
- Applications
  - Clearance testing
  - Risk assessment



† *Identification of dangerous levels of lead, Final Rule, 1/5/01, 40 CFR 745.65*

How did we arrive at this experimental design?

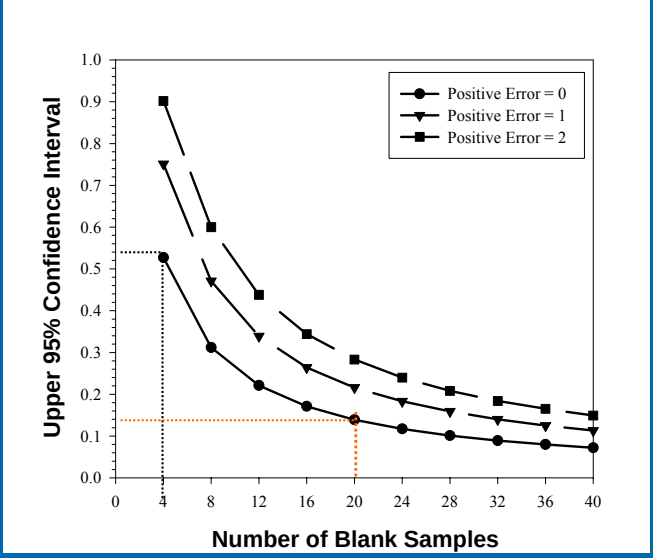


19

How did we arrive at 160 samples?

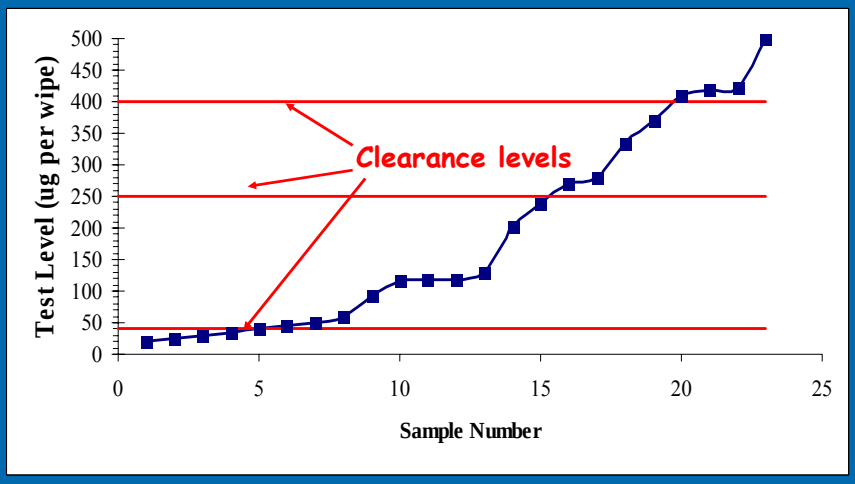
- Looked at all of the archived ELPAT samples; selections based on concentration and number of samples available
- Requested newly-prepared samples to focus on particular clearances levels (40, 250, 400 μg)
- Implemented statistically-balanced design of four replicates

Determining the Number of Blank Samples to Evaluate False Positive

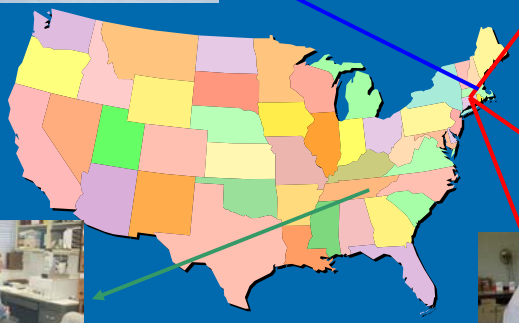


Confidence in the estimate of the false positive error rate increases as more blank samples are evaluated.

Attention to Clearance Levels



Testing Venues Focused on Where the Interest Lies



23

Two Very Different Analytical Techniques Verified

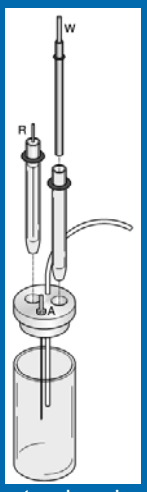
- Portable X-ray fluorescence
- Portable anodic stripping voltammetry

Vendors That Participated in the Lead in Dust ETV Tests

- Niton Corporation (3 XRF systems)
- Monitoring Technologies International (ASV)
- Palintest (ASV)
- Key Master Technologies/EDAX (XRF)

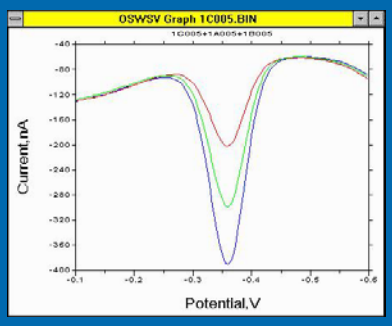
25

Anodic Stripping Voltammetry for Determination of Lead



Electrochemical cell uses a working (W), reference (R), and auxillary (A) electrodes in cylindrical tube with teflon cap.

Pb(II) is reduced to Pb(0) by holding potential at cathodic value for brief period; Pb quantified with anodic potential sweep, measuring current for oxidizing Pb(0) to Pb(II) and stripping it from solid electrode.



Anodic stripping voltammograms for the sample and two standard additions of 50 ppb Pb(II). Deposition potential = -600 mV; deposition time = 1 min.; quiet time = 10 sec. S.W. frequency = 15 Hz; step potential = 4 mV;

S.W. amplitude = 25 mV

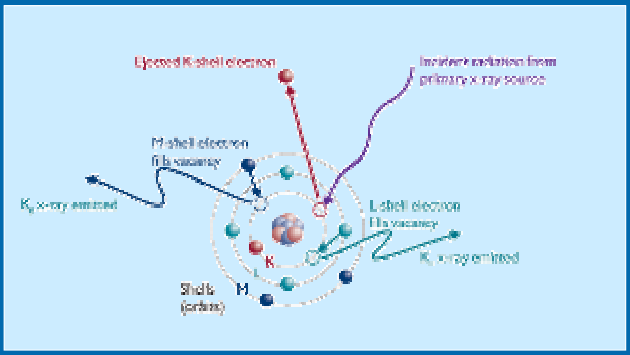
26

Anodic Stripping Voltammetry

- Advantages
  - Low capital cost
  - Disposable material
  - Very high sample throughput
- Disadvantages
  - Generates small amounts of chemical waste

X-Ray Fluorescence

Exposing metallic materials to high energy x-rays stimulates ejection of electrons the energies of which provide information concerning the identity of the metal in question.



28

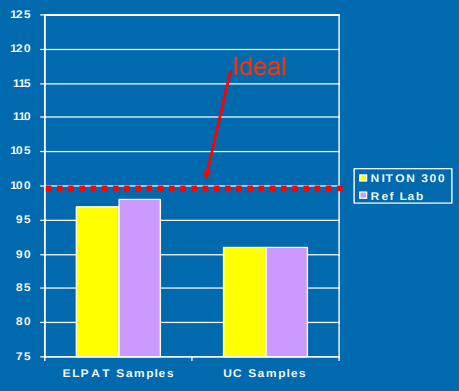
X-Ray Fluorescence

- Advantages
  - Non-destructive analysis
  - Produces no chemical waste
  - Good sample throughput
- Disadvantages
  - High capital cost
  - May need radiation source license

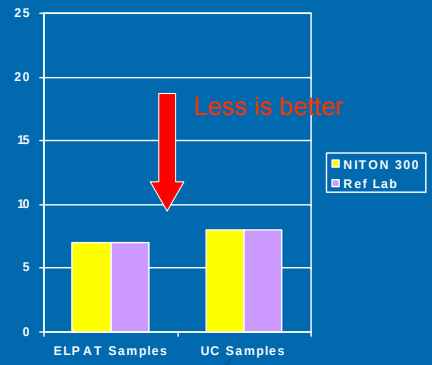
29

NITON XL300

Accuracy



Precision



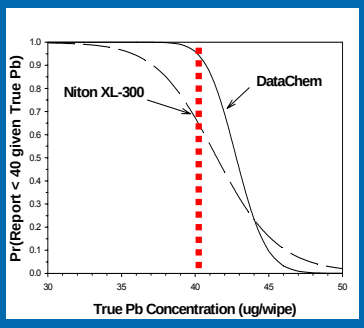
30

NITON XL300

Reported Concentrations at Clearance Levels

Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	39	42
250	224	213
400	346	303

Probabilities of False Negatives



31

NITON XL300

Comparability: R = 0.999 (ELPAT samples); R = 0.999 (UC samples)

False positive results (relative to clearance levels): 0% (0 of 12 ELPAT Samples); 0% (0 of 30 UC samples)

False negative results (relative to clearance levels): 54% (15 of 38 ELPAT); 70% (21 of 30 UC samples) [25% and 77% for Reference Laboratory]

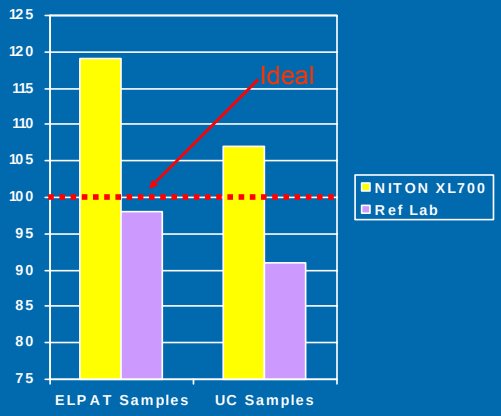
Reporting limit: 15 µg/wipe

Throughput (1 analysts): 40 samples/12 hr day

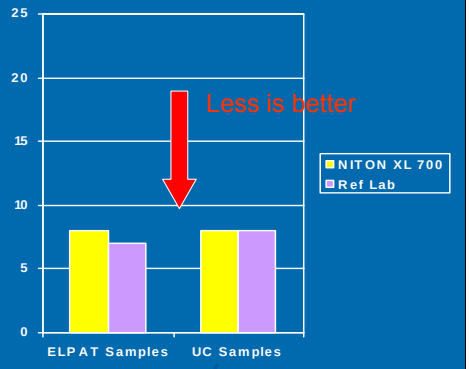
Statistically significant negative bias ("penalty" for high precision) but within acceptable bias range.

NITON XL700

Accuracy



Precision



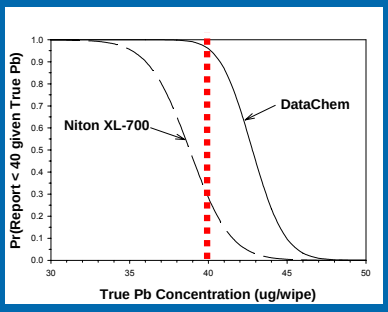
33

NITON XL700

Reported Concentrations at Clearance Levels

Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	42	49
250	276	272
400	431	372

Probabilities of False Negatives



NITON XL700

Comparability: R = 0.999 (ELPAT samples); R = 0.999 (UC samples)

False positive results (relative to clearance levels): 50% (6 of 12 ELPAT Samples); 62% (21 of 34 UC samples)

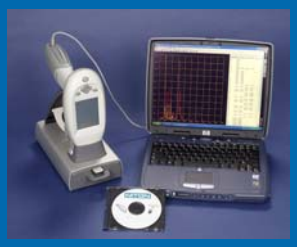
False negative results (relative to clearance levels): 7% (2 of 28 ELPAT); 8% (2 of 26 UC samples) [25% and 77% for Reference Laboratory]

Reporting limit: 15 µg/wipe

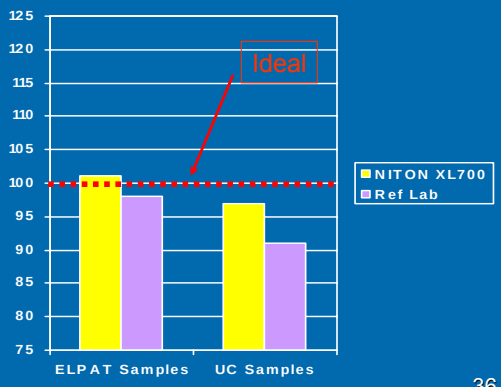
Throughput (1 analyst): 30 - 60 samples/12 hr day

Statistically significant positive bias ("penalty" for high precision) but within acceptable bias range.

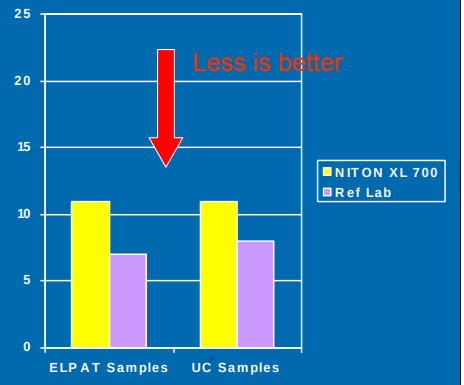
NITON XLt 700



Accuracy



Precision



36

NITON XLt 700

Comparability: R = 0.999 (ELPAT samples); R = 0.999 (UC samples)

False positive results (relative to clearance levels): 8% (1 of 12 ELPAT Samples); 22% (8 of 37 UC samples)

False negative results (relative to clearance levels): 29% (8 of 28 ELPAT); 43% (10 of 23 UC samples) [25% and 77% for Reference Laboratory]

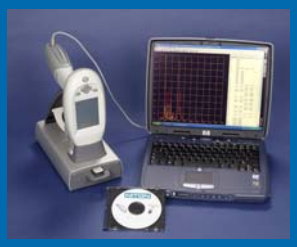
Reporting limit: 10 µg/wipe

Throughput (2 analysts): 45 - 50 samples/10 hr day

Statistically significant negative bias ("penalty" for high precision) but within acceptable bias range.

37

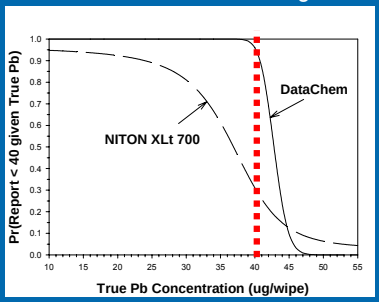
NITON XLt 700



Reported Concentrations at Clearance Levels

Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	42	42
250	232	234
400	371	361

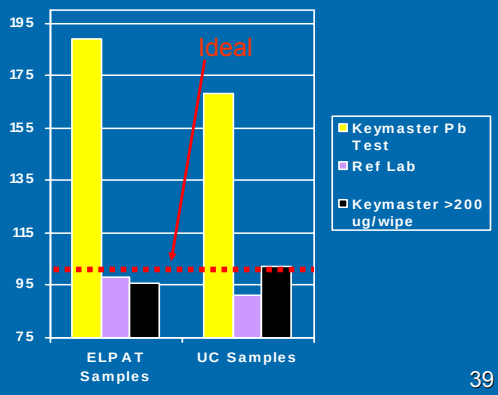
Probabilities of False Negatives



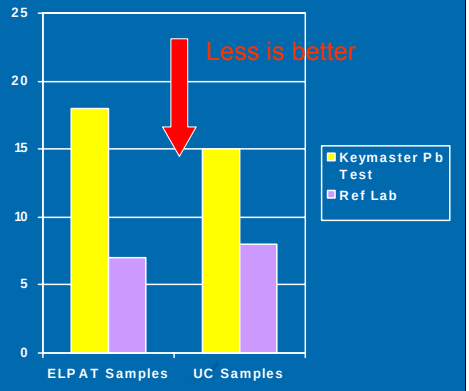
38

Keymaster Pb-Test XRF

Accuracy



Precision



39

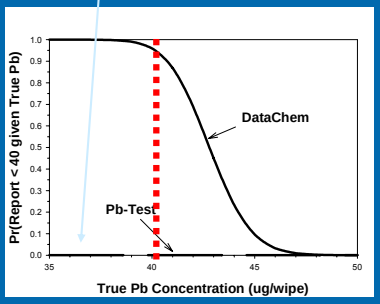
Keymaster Pb Test XRF

Due to the positive bias at low lead levels, there was no chance of a false negative Response at the 40 µg/wipe level

Reported Concentrations at Clearance Levels

Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	118	99
250	275	254
400	365	248

Probabilities of False Negatives



40

Keymaster Pb Test XRF

Comparability: R = 0.967 (for samples ≤ 200 µg/wipe); R = 0.989 (for samples > 200 µg/wipe);

False positive results (relative to clearance levels): 50% (6 of 12 ELPAT Samples); 53% (20 of 38 UC samples)

False negative results (relative to clearance levels): 29% (8 of 28 ELPAT); 32% (7 of 22 UC samples) [25% and 77% for Reference Laboratory]

Reporting limit: None provided

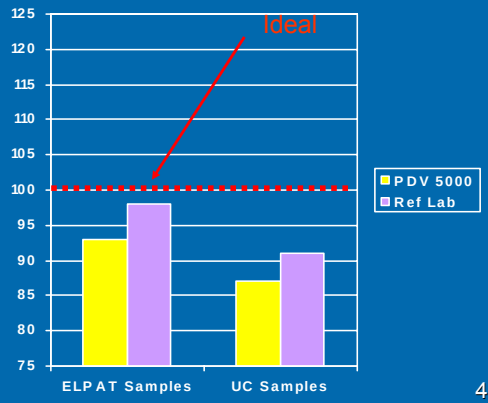
Throughput (2 analysts and 2 instruments): 80 samples/10 hr day

Statistically significant positive bias for samples ≤ 200 µg/wipe; unbiased for samples above 200 µg/wipe; acceptable precision.

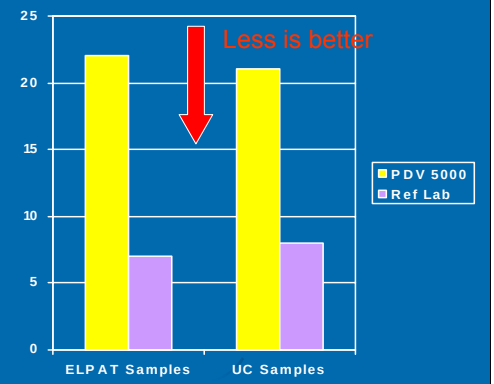
41

MTI PDV 5000

Accuracy



Precision



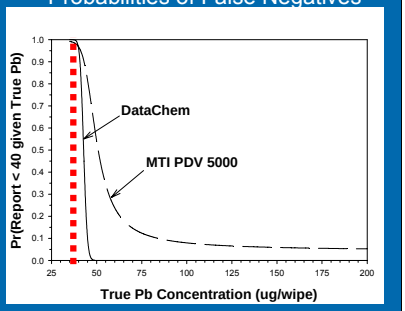
42

MTI PDV 5000

Reported Concentrations at Clearance Levels

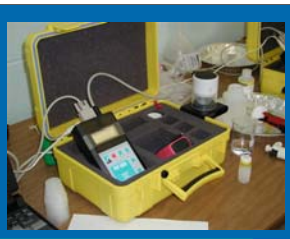
Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	29	44
250	240	213
400	375	258

Probabilities of False Negatives



43

MTI PDV 5000



Comparability: R = 0.999 (for UC samples); R = 0.988 (for ELPAT samples);

False positive results (relative to clearance levels): 25% (3 of 12 ELPAT Samples); 14% (4 of 29 UC samples)

False negative results (relative to clearance levels): 43% (12 of 28 ELPAT); 59% (17 of 29 UC samples) [25% and 77% for Reference Laboratory]

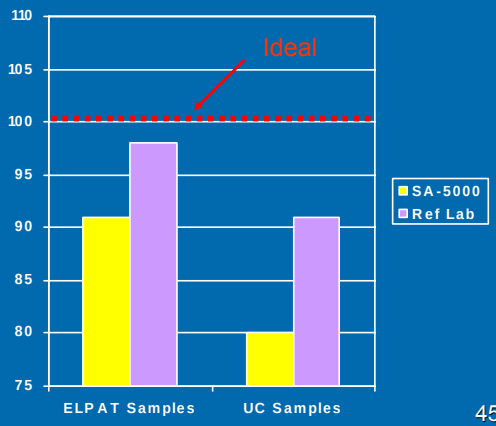
Reporting limit: < 20 µg/wipe

Throughput (2 analysts and 1 instrument): 80 samples/10 hr day

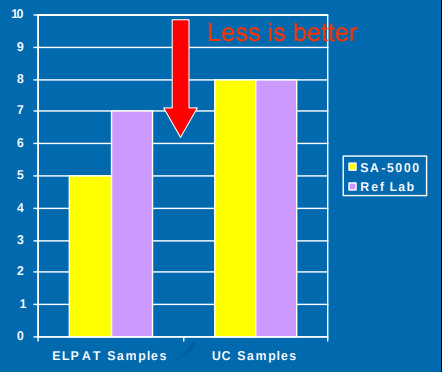
Statistically significant negative bias; less precise than typically acceptable levels; strong linear relationship between PDV 5000 response and that of comparable lab method.

Palintest Scanning Analyzer SA-5000

Accuracy



Precision



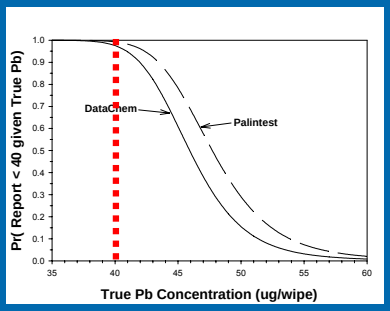
45

Palintest
Scanning Analyzer SA-5000

Reported Concentrations at Clearance Levels

Clearance Level µg/wipe	UC Samples, µg/wipe	ELPAT Samples, µg/wipe
40	35	36
250	189	221
400	308	372

Probabilities of False Negatives



46

Palintest
Scanning Analyzer SA-5000

Comparability: R = 1.00 (for UC samples); R = 0.995 (for ELPAT samples);

False positive results (relative to clearance levels): 0% (0 of 12 ELPAT Samples); 0% (0 of 38 UC samples)

False negative results (relative to clearance levels): 61% (17 of 28 ELPAT); 100% (22 of 22 UC samples) [25% and 77% for Reference Laboratory]

Reporting limit: < 25 µg/wipe

Throughput (1 analyst and 1 instrument): 80 samples/10 hr day

Statistically significant negative bias; very precise; strong linear relationship between SA-5000 response and that of comparable lab method; no false positives, high number of false negatives.

47

ETV Program does NOT make Head to Head comparisons of technologies, because there are needs for a variety of tools in the environmental technology toolbox

48

Asking: “What is the Best Technology?” is Like Asking “What is the Best Vehicle to Purchase?”

It depends on what you need!

Sports car vs. MiniVan

PS: Your mileage may vary

Upcoming Technology Verifications by the Advance Monitoring Systems Center

- More rounds of arsenic test kits for water
- Multi-parameter water monitors
- Ambient ammonia monitors for animal feed operations
- Ammonia continuous emission monitors
- Immunoassay kits for anthrax, botulinum toxin, & ricin
- PCR kits for anthrax, plague, Tularemia, Brucellosis

Thank You

After viewing the links to additional resources, please complete our online feedback form.

Links to Additional Resources

51