

Environmental Laboratory Accreditation Program Requirements for 8330B

*Environmental Data Quality
Workgroup*

December 2014

*Ed Corl
NAVSEA LQAO*

Background

- Environmental Data Quality Workgroup (EDQW)
 - Consensus org.-DoD Component Principals
 - Chair-Dr. Jordan Adelson (NAVSEA)
- Manage the DoD/DOE Environmental Laboratory Accreditation Program (ELAP)
- Quality Systems Manual (QSM- version 5)- Based upon ISO 17025, TNI, additional DoD requirements
- Purpose of presentation is to clarify QSM requirements for 8330B for ELAP organizations
 - Quality Control (QC) Criteria- Table 3
 - QSM LCS Data (Appendix C Tables)

Evolution of 8330 & EDQW implementation efforts

- EPA 8330- Published 1994
- EPA 8330A- Revision 1, Published February 2007
- EPA 8330B- Revision 2, Published October 2006
 - Appendix A- Collecting and Processing of Representative Samples For Energetic Residues In Solid Matrices From Military Training Ranges (Multi-Increment Sampling)
- **QSM Version 5 (July 2013)**
- **8330B training EMDQ (April 2014)**
- **Frequently Asked Questions (FAQ's) November 2014**
- **EDQW Web-based training (December 2014)**

Analytical Differences- 8330A to 8330B

➤ Differences:

- Additional analytes (Nitroglycerin, PETN, and 3,5-Dinitroaniline)
- Appendix A- Incremental Sampling Methodology (ISM)
- Subsample particle size and weight
- Final volume of extract
- Addition of shaker platform option
- Eliminate the addition of calcium chloride solution

EPA Method 8330B released in 2006
calls for drying and sieving
(10 mesh or 2 mm) entire sample →

**Drying and sieving should be performed
In the laboratory. NOT IN THE FIELD.**



← Entire portion < 2 mm subjected
to grinding, then subsampling is
conducted using a **IMS** technique in
the laboratory

Care for cross contamination

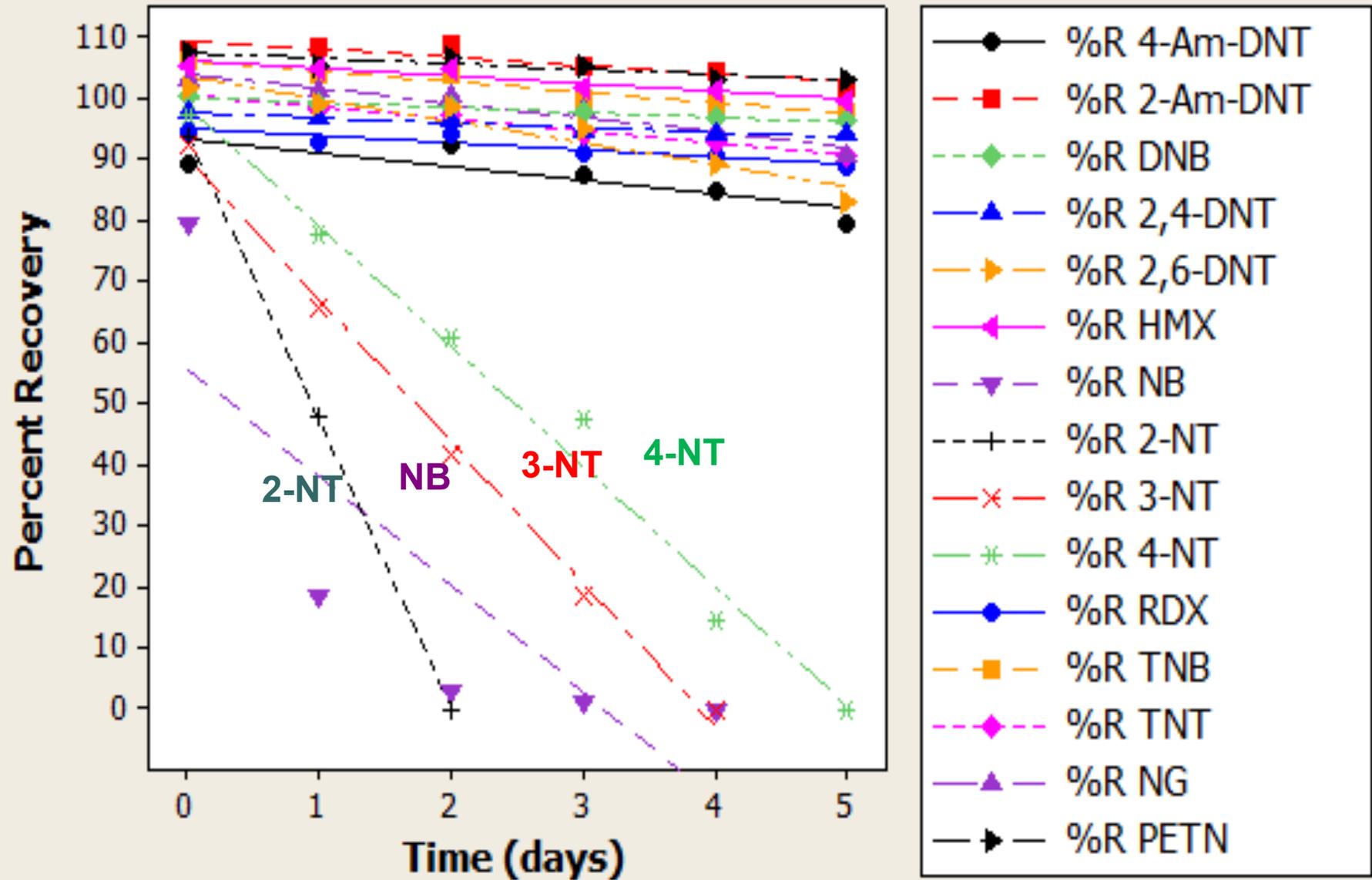
Soil Drying

- The Method Blank (MB) and samples
- The LCS is not required to be dried if the vendor of the solid matrix reference material used specifies for it not to be dried
- The matrix of the LCS and MB can be Ottawa sand, soil, or vendor supplied clean matrix

Drying and Grinding Studies

- After 5 days of drying at room temperature, nitrobenzene and the nitrotoluene isomers volatilized producing 0% recoveries; the 11 remaining explosives were negatively biased by an average of $\approx 10\%$
- Concentrations of the unground LCS decreased about 2% on the average after only 30 min of drying.
- Increasing the grinding time from 90 sec to 240 sec. (4 60-sec cycles) decreased the recoveries of the 15 compounds by an average of 4% - 5%.
- The Purchased Reference Material that is used as an LCS should not be air dried but processed (e.g., ground) with the environmental samples that have been air dried
- Excessive grinding of the LCS should be avoided as this will negatively bias the results

Percent Recoveries of 15 Explosives vs. Time



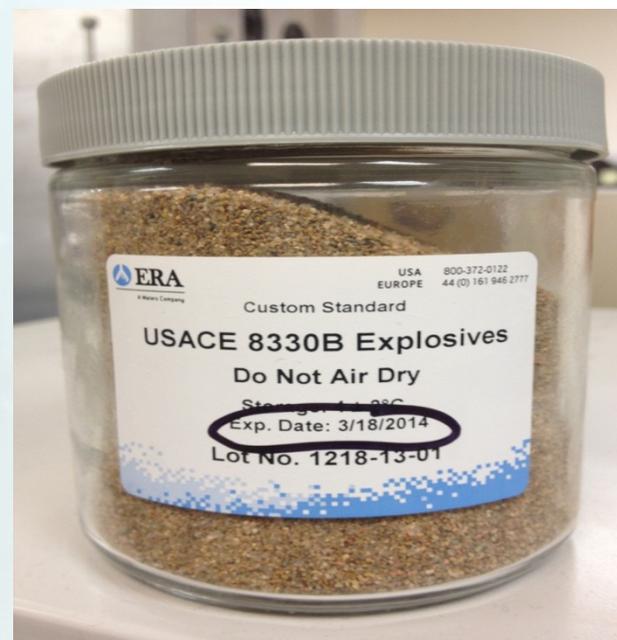
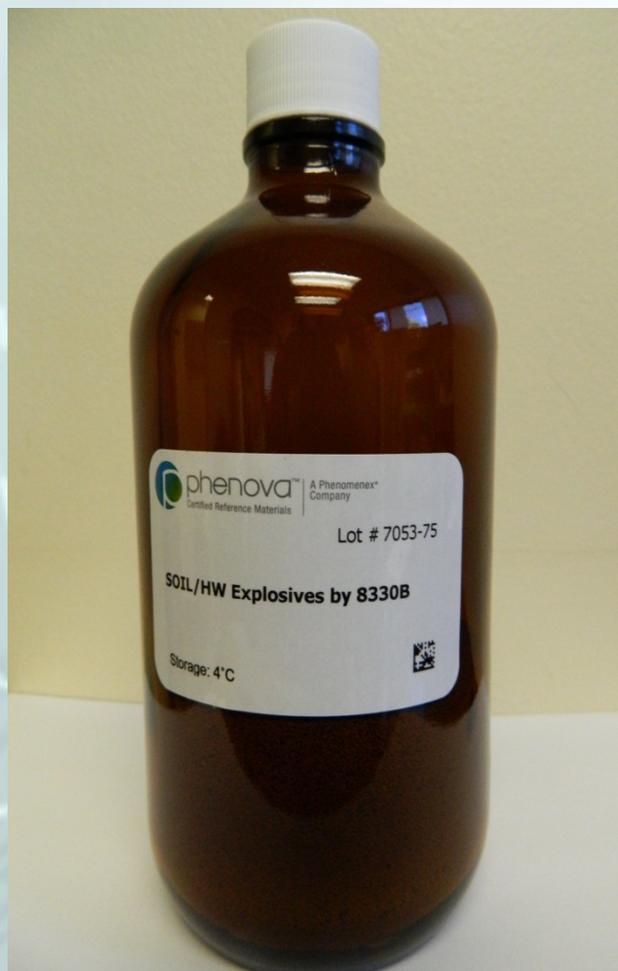
Soil Sieving

- Each Sample, LCS, MB
 - Weigh entire sample post drying
 - Sieve entire sample with a 2-mm nominal opening (US Standard 10) mesh sieve
 - Break up pieces of soil with gloved hands
 - Do not intentionally include vegetation unless project specifies
 - Collect and weigh the portion that does not pass through the sieve

There are standard reference materials available.

The material MUST be ground prior to extraction and analysis.

May not contain all project analytes which will have to be spiked.



Soil Grinding

What kind of grinder is required?

- Any mode of grinding that reduces the particle size to <75 μm consistently over the range of types of samples received (gravel, sand, peat, soil) while meeting QSM Version 5.0 QC criteria
 - **Puck Mill is preferred as it allows reference material sub-sampling options for the LCS**

Soil Grinding

➤ Grinding Blank

- Prepared and analyzed in exactly the same way as samples (such as same grinding intervals)
- Can be prepared and analyzed individually or as a composite
- No analytes detected $> \frac{1}{2}$ Limit of Quantitation (LOQ)

Soil Subsampling

- All samples and QC samples must be subsampled in same manner
 - Entire ground sample is mixed, spread out, and 30 or more randomly located increments are taken to total approximately 10 grams for each subsample
 - Added QC samples in this step are MS, MSD, and subsample triplicates

Aqueous Samples

- Solid phase extraction is the only extraction procedure allowed
- Uses a resin-based solid phase disk or cartridge
- Surrogates and matrix spikes are added to the original sample not SPE.

Analytical Instrument Options

- High Performance Liquid Chromatograph (HPLC)
 - Requires confirmation analysis
 - UV detection using a column with different retention time order from the primary column
 - UV diode array detector not permitted for confirmation analysis
- Liquid Chromatograph/Mass Spectrometer (LC/MS) or LC/MS/MS
 - No confirmation analysis required

QC Criteria

➤ LCS

- One per preparation batch
- Must use QSM Appendix C appropriate tables for batch control if project limits are not specified
 - Tables 33 and 34 for analysis by LC/MS or LC/MS/MS
 - Tables 36 and 37 for analysis by HPLC

Neutralization of Alkaline Hydrolysis Treated Soils for SW-8330B Analysis

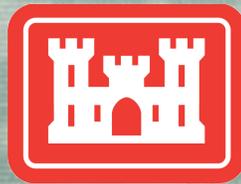
Presenters

Chuck Coyle, P.E. and Chung-Rei Mao, Ph.D.

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Resources

- Please see the DENIX website where all of the FAQs regarding 8330B are located

http://www.denix.osd.mil/edqw/upload/FAQS-2014_final.docx

- Any questions generated by this training session can be sent through DENIX for a response

<http://www.denix.osd.mil/tools/page-mgt.cfm?reqID=contactUs&pageid=34754>