



Assembled Chemical Weapons Assessment Program

Analytical Method Evaluation Neutralization/SCWO

Robert O'Neil
Arthur D. Little, Inc.

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Analytical Method Evaluation – Overview

- ◆ This overview of the analytical method evaluation effort will present the:
 - Goals of the method evaluation effort;
 - Method evaluation approach;
 - Methods evaluated; and
 - Method evaluation results

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Analytical Method Evaluation – Overview (Continued)

- ◆ Goals of the Method Evaluations
 - Identify simple, readily available, cost effective method modifications required to implement a standard analytical procedure.
 - Establish reproducible Method Detection Limits (MDL)
 - Establish baseline data for precision and accuracy

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Analytical Method Evaluation – Overview (Continued)

- ◆ Method Evaluation Approach
 - Obtain appropriate sample matrix
 - Develop method modifications to supplement published analytical method
 - Determine the Method Detection Limit for each target analyte in accordance with 40 CFR (Code of Federal Regulations) Part 136, Appendix B, July 1, 1999
 - Conduct a mid-level spike/recovery experiments to establish baseline accuracy and precision information

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Analytical Method Evaluation – Overview (Continued)

- ◆ The determination of a successful/unsuccessful method evaluation study was based upon several criteria:
 - Comparison to the precision and accuracy requirements stipulated in the PMACWA Quality Assurance Program Plan
 - Review of the spike recovery data and conduct follow-up discussions with the analyst
 - Use of professional judgement to determine if the analytical information could be effectively used to evaluate the technologies tested against the test objectives

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Analytical Method Evaluation – Overview (Concluded)

- ◆ The method evaluation effort conducted during the PMACWA Technology Test program included:
 - Evaluation of 138 analytical method/matrix combinations total; 33 applicable to neutralization/SCWO of HD and Tetrytol
 - Establishment of precision and accuracy criteria against which field sample analysis results can be compared
 - Identification of areas for improvement of the methods

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Analytical Method Modifications

- ◆ Modifications to the standard analytical method consisted of:
 - Sample dilution
 - Modified sample injection volume
 - pH adjustment during sample preparation
 - Use of centrifuge during sample preparation
 - Alternate separation columns
 - Modified liquid mobile phase, for Ion Chromatography methods
- ◆ All evaluated method modifications incorporate standard analytical instruments and techniques.

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Sample Matrices Evaluated – Neutralization/SCWO

- ◆ 3.8 wt% HD Hydrolysate - Hot water hydrolysis of HD followed by pH adjustment to 11 - 12
- ◆ 15 wt% % HD Hydrolysate - Hot water hydrolysis of HD followed by pH adjustment to 11 - 12
- ◆ 16.7 wt% Tetrytol Hydrolysate - Caustic (6% Sodium Hydroxide solution) Hydrolysis of Tetrytol (70% tetryl/30% TNT) followed by pH adjustment to neutral

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Analytical Methods Evaluated Prior to the Technology Tests – Neutralization/SCWO

- ◆ Alcohols by EPA SW-846/8015
- ◆ Aldehydes/Ketones by EPA SW-846/8315
- ◆ Anions by EPA SW-846/9056
- ◆ Chemical Oxygen Demand by EPA Method 410.4
- ◆ Cyanide by EPA SW-846/9010 Dioxins/Furans by EPA SW-846/8290
- ◆ Energetics by USCHPPM CAD 42.1 (isoamyl acetate extraction followed by analysis using Gas Chromatography w/Electron Capture Detector)
- ◆ HD by US Army HN-1/HN-2 (chloroform extraction followed by analysis using Gas Chromatograph/Mass Spectrometer)
- ◆ Mercury by EPA SW-846/7470

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Analytical Methods Evaluated Prior to the Technology Tests – Neutralization/SCWO (Concluded)

- ◆ Metals by EPA SW-846/6010
- ◆ Organic Acids/Inorganic Anions by Ion Chromatography (non-standard method)
- ◆ Schedule 2 Compounds by US Army ACT013
- ◆ Semi-volatile Organics by EPA SW-846/8270
- ◆ Thiodiglycol by US Army ACT016
- ◆ Total Kjeldahl Nitrogen by EPA Method 351.2
- ◆ Total Organic Carbon/Total Inorganic Carbon by EPA SW-846/9060
- ◆ Volatile Organics by EPA SW-846/8260

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Analytical Method Evaluation Results – Neutralization/SCWO

- ◆ All method evaluations were successful except for:
 - Total inorganic carbon, aldehydes/ketones, and semi-volatile organics in 15% HD Hydrolysate
 - Aldehydes/ketones in Tetrytol Hydrolysate
- ◆ HD and Tetrytol Hydrolysates are feed solutions to the secondary treatment SCWO unit not effluents.

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Additional Analytical Methods Implemented During the Technology Tests – Neutralization/SCWO

- ◆ Gas sample analysis methods evaluated through use during testing:
 - Acetic/Formic Acids by EPA Method 26
 - Aldehydes/Ketones by EPA SW -846/0011
 - Carbon Dioxide & Oxygen by EPA Method 3A
 - Carbon Monoxide by EPA Method 10
 - Energetics by US Army CHPPM Sampling Train for Energetic Materials (STEM Train)
 - HD by US Army Depot Area Air Monitoring System (DAAMS) Method
 - Hydrogen Chloride (HCl)/Hydrogen Cyanide (HCN)/ Ammonia (NH3) by EPA Method 26

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Additional Analytical Methods Implemented During the Technology Tests – Neutralization/SCWO (Continued)

- ◆ Gas sample analysis methods evaluated through use during testing (continued):
 - Hydrogen Sulfide (H₂S): sample collection by glass lined Summa Canister; analysis by GC
 - Metals/Mercury EPA SW -846/0060
 - Nitrogen Oxides by EPA Method 7E
 - Nitrous Oxide by NIOSH 6600
 - Particulate Matter by NIOSH Method 0500
 - Semi-volatile organics & dioxin/furans by EPA SW -846/0010/0023
 - Sulfur dioxide by EPA Method 6C
 - Volatile organics by EPA Method TO-15

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Results From Implementation of the Evaluated Analytical Methods – Neutralization/SCWO

- ◆ A quality review of the analytical data from the Demonstration I Test concluded:
 - 99% of the analytical data was deemed valid and usable for evaluation of the demonstrated technologies as compared to XX as specified in the PMACWA Quality Assurance Program Plan; and
 - Only 17% of the data was qualified as estimated
- ◆ Areas identified for improvement of the methods include:
 - Optimized dilution ratios
 - Additional sample clean-up steps
 - Introduction of routine instrument maintenance steps

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