

2005/2006 Report Carlsbad Environmental Monitoring & Research Center



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Table of Contents

| | |
|--|------|
| Table of Contents | 2 |
| List of Tables | 3 |
| List of Figures | 5 |
| Acronyms and Abbreviations | 10 |
| Foreward | 13 |
| Overview | 14 |
| Chapter 1 Ambient Aerosol Studies for the WIPP-EM..... | 1-1 |
| Chapter 2 Radionuclides and Inorganics in WIPP Exhaust Air | 2-1 |
| Chapter 3 Surface Soil Radionuclides | 3-1 |
| Chapter 4 The Identification and Quantification of Gnome-Derived Radionuclides in WIPP Environmental Samples..... | 4-1 |
| Chapter 5 Vertical Distribution of ¹³⁷ Cs, ²⁴¹ Am and ^{239,240} Pu in the Vicinity of the Waste Isolation Pilot Plant..... | 5-1 |
| Chapter 6 Radionuclides and Inorganics in Selected Water Sources..... | 6-1 |
| Chapter 7 Occurrence of Radionuclides in Residents of the Carlsbad, New Mexico Area | 7-1 |
| Chapter 8 Analysis of Volatile Organic Compounds | 8-1 |
| Chapter 9 The RACER Project..... | 9-1 |
| Chapter 10 First Responder Radiological Response Training..... | 10-1 |
| Chapter 11 <i>In Situ</i> Aerosol Probe Occlusion Tests at Station A | 11-1 |
| Appendices | |
| A: Brief History of CEMRC | A-1 |
| B: Students/Visiting Scientists at CEMRC..... | A-3 |
| C: Publications during 2005/2006 | A-4 |
| D: Tours, Public Presentations and Other Outreach | A-6 |
| E: Performance Tests and Audits..... | A-7 |
| References..... | A-27 |

List of Tables

| | | |
|-----------|---|------|
| Table 1 | CEMRC Budget Changes Since FY2002 | 17 |
| Table 2 | Listing of CEMRC Staff as of September 30, 2006 | 18 |
| Table 1-1 | Summary Statistics for Aerosol Mass Loadings and Actinide Activities in High-Volume Aerosol Samples | 1-4 |
| Table 1-2 | Aerosol Sampling Status for WIPP EM (January 2007) | 1-5 |
| Table 2-1 | Summary Statistics for Gross Alpha Analyses of Daily FAS Filters | 2-6 |
| Table 2-2 | Summary Statistics for Gross Beta Analyses of Daily FAS Filters..... | 2-7 |
| Table 2-3 | Summary Statistics for Aerosol Mass Loadings (micrograms per filter) on FAS Filters..... | 2-8 |
| Table 3-1 | RPDs and RERs for Soil Gamma Duplicate Measurements | 3-3 |
| Table 3-2 | Summary Statistics for Radionuclides in Soil Samples Collected in 2005..... | 3-3 |
| Table 3-3 | Concentrations of Radionuclides in Soil by Grid Node for 2005 in Comparison to the Means of Concentrations from Previous Years..... | 3-6 |
| Table 4-1 | Mean Concentrations in Bq kg ⁻¹ of Radionuclides for Each of Nine Gnome Sample Locations and Across-sample Statistics in Comparison to the Concentrations in Surface Soils Collected from Two Areas Near the WIPP Site..... | 4-8 |
| Table 4-2 | Ratios of ¹³⁷ Cs and ²³⁸ Pu to ^{239,240} Pu in Gnome Samples in Comparison to Mean Values for WIPP Soils | 4-9 |
| Table 4-3 | Relative Percent Differences Between Sieved and Unsieved Soils Paired by Sample | 4-10 |
| Table 4-4 | Ratio of Concentration of Metals in Sieved Gnome Soils to Those in WIPP Soils..... | 4-12 |
| Table 4-5 | Relative Percent Differences Between the Gnome and WIPP Normalized Analyte Concentration | 4-13 |
| Table 4-6 | ²⁴⁰ Pu/ ²³⁹ Pu Isotopic Ratios and Activity Concentrations for Gnome and WIPP Samples | 4-14 |

| | | |
|------------|---|-------|
| Table 5-1 | Specific Conductivity and pH Data as a Function of Depth for the Four Soil Profiles..... | 5-8 |
| Table 5-2 | Distribution of Clay, ¹³⁷ Cs and ^{239,240} Pu as a Function of Depth in the Four Soil Profiles | 5-9 |
| Table 5-3 | Profile Inventories for ¹³⁷ Cs and ^{239,240} Pu | 5-9 |
| Table 6-1 | Range of Activity Concentrations for Uranium Isotopes Measured in Drinking Water During 2005 and Comparisons with 1998/1999 Baseline..... | 6-7 |
| Table 6-2 | Measured Concentration of Selected Inorganic Analytes in Drinking Water from 1998 to 2005 at Five Locations | 6-11 |
| Table 6-3 | Range of Concentrations for Baseline Inorganic Constituents in Surface Water Collected During 1999 to 2005 from Three Regional Reservoirs | 6-16 |
| Table 6-4 | Comparison of Inorganic Analyte Concentrations in Shallow vs. Deep Surface Water from 1998 to 2005..... | 6-19 |
| Table 7-1 | Demographic Characteristics of the “Lie Down and Be Counted” Population Sample through December 31, 2006 | 7-4 |
| Table 7-2 | Minimum Detectable Activities..... | 7-5 |
| Table 7-3 | “Lie Down and Be Counted” Results through December 31, 2006 | 7-7 |
| Table 8-1 | Compounds of Interest for WIPP Confirmatory Volatile Organic Compounds Monitoring Program | 8-6 |
| Table 8-2 | CEMRC Procedures for Confirmatory Volatile Organic Compounds Monitoring Program..... | 8-6 |
| Table 11-1 | Comparisons for the Probe Occlusion Tests..... | 11-15 |
| Table 11-2 | Comparison of Gravimetric Data: 1/3 Occluded XO Probe vs. Sampler of Record (SOR) | 11-16 |
| Table 11-3 | Comparison of Gravimetric Data: 2/3 Occluded XO Probe vs. Sampler of Record (SOR) | 11-16 |
| Table 11-4 | Comparison of Gravimetric Data: 3/3 Occluded XO Probe vs. Sampler of Record (SOR) | 11-16 |

| | | |
|-------------|--|-------|
| Table 11-5 | Comparison of Gross Alpha and Beta Activities: 1/3 Occluded XO Probe vs. Sampler of Record (SOR)..... | 11-17 |
| Table 11-6 | Comparison of Gross Alpha and Beta Activities: 2/3 Occluded XO Probe vs. Sampler of Record (SOR)..... | 11-17 |
| Table 11-7 | Comparison of Gross Alpha and Beta Activities: 3/3 Occluded XO Probe vs. Sampler of Record (SOR)..... | 11-18 |
| Table 11-8 | Comparison of Trace Element Data: Experimental/Occluded Probe (XO) vs. Sampler of Record (SOR)..... | 11-18 |
| Table 11-9 | Summary Statistics and t-test Results for a Comparison of Aerosol Mass Concentrations on the Sampler of Record vs. Back-Up Filters.. | 11-19 |
| Table 11-10 | Summary Statistics and t-test Results for a Comparison of Gross Alpha Activities on the Sampler of Record vs. Back-Up Filters..... | 11-19 |
| Table 11-11 | Summary Statistics and t-test Results for a Comparison of Gross Beta Activities on the Sampler of Record vs. Back-Up Filters | 11-19 |
| Table 11-12 | Trace Element Concentrations in Filter Samples (mg per filter) from Station A | 11-20 |
| Table E-1 | Internal Audit Summaries (# of Findings)..... | A-9 |
| Table E-2 | Blind Check Study for Internal Dosimetry 2006 by the ORNL Intercomparison Studies In-vivo Program..... | A-10 |
| Table E-3 | Quality Assurance/Quality Control for Internal Dosimetry 2005-2006 Audits..... | A-12 |
| Table E-4 | Blind Check 2005 Environmental Chemistry Inorganic Analyses | A-13 |
| Table E-5 | Radiochemistry NIST-RIP and MAPEP Intercomparison Results..... | A-16 |
| Table E-6 | An Example of the Daily Performance Tests for ICP-MS | A-26 |

List of Figures

| | | |
|------------|--|-----|
| Figure 1.1 | High Volume Ambient Aerosol ^{239,240} Pu Activity Concentration | 1-6 |
| Figure 1.2 | High Volume Ambient Aerosol ^{239,240} Pu Activity Density..... | 1-7 |
| Figure 1.3 | High Volume Ambient Aerosol ²⁴¹ Am Activity Concentration | 1-8 |

| | | |
|------------|--|------|
| Figure 1.4 | High Volume Ambient Aerosol ²⁴¹ Am Activity Density | 1-9 |
| Figure 1.5 | High Volume Ambient Aerosol Mass Concentrations | 1-10 |
| Figure 1.6 | Whisker Plots Showing High Volume Ambient Aerosol ^{239,240} Pu Activity Concentrations and Densities..... | 1-11 |
| Figure 1.7 | Whisker Plots Showing High Volume Ambient Aerosol ²⁴¹ Am Activity Concentrations and Densities..... | 1-12 |
| Figure 1.8 | Whisker Plots Showing High Volume Ambient Aerosol Mass Concentrations | 1-13 |
| Figure 2.1 | Fixed Air Samplers at Station A | 2-9 |
| Figure 2.2 | Flow Diagram Showing the Handling and Analysis of the Aerosol Sample Filters from Station A | 2-10 |
| Figure 2.3 | Timeseries Plots of Gross Alpha Activity Densities and Activity Concentrations | 2-11 |
| Figure 2.4 | Timeseries Plots of Gross Beta Activity Densities and Activity Concentrations | 2-12 |
| Figure 2.5 | Timeseries Plot of Aerosol Mass Loadings | 2-13 |
| Figure 2.6 | Beta Activity vs. Alpha Activity in Fixed Air Samples from Station A | 2-14 |
| Figure 2.7 | Concentration of Selected Elements (Al, Mg, Na) in WIPP Exhaust Air | 2-15 |
| Figure 2.8 | Concentration of Selected Elements (U, Th, Pb, Cd) in WIPP Exhaust Air | 2-15 |
| Figure 3.1 | Soil Sampling Locations in the Vicinity of the WIPP | 3-4 |
| Figure 3.2 | Mean Concentrations of Radionuclides in Soil Samples from Near Field Grid Collected during 1998-2001 Compared to 2005 | 3-5 |
| Figure 4.1 | Concentrations of Metals in Gnome Soils | 4-11 |
| Figure 4.2 | Isotopic Ratios Versus Activity Concentrations for Gnome and WIPP Samples..... | 4-15 |
| Figure 5.1 | Soil Profile 3 Before Sampling..... | 5-10 |

| | | |
|-------------|--|------|
| Figure 5.2 | Soil Profile 4 After Sampling | 5-10 |
| Figure 5.3 | Distribution of ¹³⁷ Cs Expressed as a Function of Depth in Four Profiles | 5-11 |
| Figure 5.4 | Distributions of ¹³⁷ Cs and ^{239,240} Pu expressed as a Function of Depth in the Top 10 cm of Soil | 5-12 |
| Figure 5.5 | Correlation Between ^{239,240} Pu and ¹³⁷ Cs | 5-13 |
| Figure 5.6 | Distribution of Clay Content Expressed as a Function of Depth in Four Profiles..... | 5-13 |
| Figure 5.7 | Distribution of ²⁴¹ Am Expressed as a Function of Depth in the Top 4 cm of Soil..... | 5-14 |
| Figure 6.1 | Surface Water and Sediment Sampling Locations at Brantley Lake..... | 6-4 |
| Figure 6.2 | Surface Water and Sediment Sampling Locations at Red Bluff Reservoir..... | 6-5 |
| Figure 6.3 | Surface Water and Sediment Sampling Locations at Lake Carlsbad | 6-6 |
| Figure 6.4 | ^{239,240} Pu in Carlsbad Drinking Water from 1998 to 2005 | 6-8 |
| Figure 6.5 | ^{239,240} Pu in Regional Drinking Water from 1998 to 2005 | 6-9 |
| Figure 6.6 | ²³⁸ Pu in Regional Drinking Water from 1998 to 2005..... | 6-10 |
| Figure 9.1 | The RACER Project is Designed to Both Assess Risks and to Help Manage and Reduce those Risks..... | 9-2 |
| Figure 9.2 | The Web-based Interface to the RACER Database Developed by CEMRC..... | 9-3 |
| Figure 9.3 | Typical Tables of Data Selected from the RACER Online Database..... | 9-3 |
| Figure 9.4 | Typical Timeseries Graph Produced Using the RACER Online Database Tool | 9-4 |
| Figure 9.5 | MapSelect is Integrated with the RACER Database to Provide Mapping and Spatial Analyses Functions | 9-5 |
| Figure 10.1 | Collage of Activities During a Dirty Bomb Training Course at CEMRC..... | 10-5 |

| | | |
|---------------|---|-------|
| Figure 11.1 | Engineering Drawing of a Shrouded Aerosol Sampling Probe of the Type Used for Effluent Monitoring at the WIPP | 11-21 |
| Figure 11.2 | Probe One-third Blocked, Prior to Deployment | 11-22 |
| Figure 11.3 | Probe One-third Blocked, After Deployment | 11-22 |
| Figure 11.4 | Probe Two-thirds Blocked, Prior to Deployment | 11-23 |
| Figure 11.5 | Unblocked (Control) Probe as Recovered September 12, 2005 | 11-23 |
| Figure 11.6 | Probe Two-thirds Blocked, As Recovered..... | 11-24 |
| Figure 11.7 | Probe Two-thirds Blocked, As Recovered (Outlet Side View) | 11-24 |
| Figure 11.8 | Probe Fully Occluded, Prior to Deployment..... | 11-25 |
| Figure 11.9 | Probe Fully Occluded, Prior to Deployment (Reverse View) | 11-25 |
| Figure 11.10 | Probe Fully Occluded, As Recovered..... | 11-26 |
| Figure 11.11 | Probe Fully Occluded, As Recovered..... | 11-26 |
| Figure 11.12 | Sample Volumes for all Samples | 11-27 |
| Figure 11.13 | Mass Concentrations for all Samples..... | 11-27 |
| Figure 11.14 | Sample Volumes for Matched Samples | 11-28 |
| Figure 11.15 | Mass Concentrations for Matched Samples..... | 11-28 |
| Figure 11.16 | Mass Concentrations for Matched Samples from the Experimental/ Occluded Sampler vs. the Sampler of Record | 11-29 |
| Figure 11.17 | Gross Alpha and Beta Activities for all Sample Filters..... | 11-29 |
| Figure 11.18 | Gross Alpha and Beta Activity Concentrations for all Sample Filters | 11-30 |
| Figure 11.19 | Gross Alpha and Beta Activity Densities for all Sample Filters | 11-30 |
| Figure 11.20 | Gross Alpha and Beta Activity Densities Plotted on a Logarithmic Scale for all Sample Filters | 11-31 |
| Fig. 11.21a-e | Timeseries Plots of Group A Trace Elements..... | 11-32 |
| Fig. 11.21f-i | Timeseries Plots of Group A Trace Elements..... | 11-33 |

| | | |
|---------------|--|-------|
| Fig. 11.22a-e | Timeseries Plots of Group B Trace Elements..... | 11-34 |
| Fig. 11.22f-j | Timeseries Plots of Group B Trace Elements..... | 11-35 |
| Figure 11.23 | Timeseries Plot of Mass Concentrations vs. Time for the Sampler of Record and Back-Up Sampler at Station A | 11-36 |
| Fig. 11.24a-c | Selected Monthly Plots of Mass Concentrations vs. Time for the Sampler of Record and Back-Up Sampler at Station A..... | 11-36 |
| Fig. 11.25a-b | Timeseries Plots for the Sampler of Record and Back-Up Sampler at Station A | 11-37 |
| Figure 11.26 | Bivariate Fit of Back-up Filter versus Primary Filter Mass Concentrations | 11-28 |
| Figure 11.27 | Bivariate Fit of Back-Up Filter versus Primary Filter Gross Alpha Activities..... | 11-39 |
| Figure 11.28 | Bivariate Fit of Back-Up Filter versus Primary Filter Gross Beta Activities..... | 11-40 |
| Figure E.1 | Comparison of Results for Ten Internal Dosimetry Laboratories in the U.S. During 2006 by the ORNL Intercomparison Studies In-vivo Program..... | A-11 |

Acronyms and Abbreviations

| | |
|-------------------|--|
| μBq | microBecquerel |
| μm | micrometer |
| AA, AAS | atomic absorption, atomic absorption spectrometry |
| Ag | silver |
| Al | aluminum |
| Am | americium |
| As | arsenic |
| ASTM | American Society for Testing and Materials |
| Ba | barium |
| Be | beryllium |
| Bq | Becquerel |
| C | centigrade |
| Ca | calcium |
| Cd | cadmium |
| Ce | cerium |
| CEMRC | Carlsbad Environmental Monitoring & Research Center |
| CEMRP | Carlsbad Environmental Monitoring & Research Program |
| CFR | Code of Federal Regulations |
| Ci | Curie |
| CLP | Contract Laboratory Program |
| cm | centimeter |
| Cm | curium |
| Co | cobalt |
| Cr | chromium |
| CRDL | Contract Required Detection Limit |
| CRM | certified reference materials |
| Cs | cesium |
| Cu | copper |
| DL | detection limit |
| DOE | U.S. Department of Energy |
| Dy | dysprosium |
| EM | Environmental Monitoring |
| EML | Environmental Monitoring Laboratory |
| EPA | U.S. Environmental Protection Agency |
| Er | erbium |
| ERA | Environmental Research Associates |
| Eu | europium |
| F | fluoride |
| Fe | iron |
| FY | fiscal year |
| g | gram |
| Gd | gadolinium |
| Ge | germanium |
| GPS | global positioning satellite |
| HCl | hydrochloric acid |
| HClO ₄ | perchloric acid |
| HF | hydrofluoric acid |
| Hg | mercury |

| | |
|-------------------------------|---|
| HNO ₃ | nitric acid |
| H ₂ O ₂ | hydrogen peroxide |
| hr | hour |
| IC | ion chromatography |
| ICP-MS | inductively coupled plasma-mass spectrometry |
| K | potassium |
| km | kilometer |
| L | liter |
| La | lanthanum |
| LaF ₃ | lanthanum fluoride |
| lb | pound |
| LDBC | "Lie Down and Be Counted" |
| LFB | Laboratory Fortified Blank |
| LFM | Laboratory Fortified Matrix |
| Li | lithium |
| LRB | Laboratory Reagent Blanks |
| m | meter |
| mb | millibar |
| MBL | mobile bioassay laboratory |
| mBq | milliBecquerel |
| MDC | minimum detectable concentration |
| Mg | magnesium |
| min | minute |
| MJ | megajoule |
| mL | milliliter |
| mm | millimeter |
| Mn | manganese |
| Mo | molybdenum |
| Na | sodium |
| NaOH | sodium hydroxide |
| Nd | neodymium |
| Ni | nickel |
| NIST | National Institute of Standards and Technology |
| nm | nanometer |
| NMSU | New Mexico State University |
| Np | neptunium |
| ORNL | Oak Ridge National Laboratory |
| p | probability |
| Pa | protactinium |
| Pb | lead |
| pH | scale indicating acidity or alkalinity of a substance |
| PM ₁₀ | particulate matter smaller than 10 micrometers in aerodynamic diameter |
| PM _{2.5} | particulate matter smaller than 2.5 micrometers in aerodynamic diameter |
| Pr | praseodymium |
| PRB | Program Review Board |
| Pu | plutonium |
| QA | quality assurance |
| QAP | quality assurance program |
| QC | quality control |
| RIP | Radiochemistry Intercomparison Program |
| Ru | ruthenium |
| SAB | Science Advisory Board |

| | |
|------------------|--|
| Sb | antimony |
| Sc | scandium |
| SD | standard deviation |
| Se | selenium |
| SE | standard error |
| sec | second |
| Sm | samarium |
| Sn | tin |
| Sr | strontium |
| T _{1/2} | half-life |
| Th | thorium |
| Ti | titanium |
| TIMS | thermal ionization mass spectrometry |
| Tl | thallium |
| TSP | total suspended particulates |
| U | uranium |
| UVB | Ultra-Violet B |
| V | vanadium |
| W | watt |
| WERC | Waste-management Education & Research Consortium |
| WID | Waste Isolation Division |
| WIPP | Waste Isolation Pilot Plant |
| WTS | Westinghouse TRU Solutions |



New VOC GC-MS Laboratory



Light Hall – Home of CEMRC



Plutonium-Glove Box in New Radiochemistry Laboratory



Inductively Coupled Plasma-Mass Spectrometer

FOREWORD

This report was written, edited and produced collaboratively by the staff of the Carlsbad Environmental Monitoring & Research Center (CEMRC), who are hereby acknowledged for their contributions to the report and the project activities described herein. The first Section is an overview of the current program activities, structure, resources and quality assurance. The second section consists of data summaries as specific chapters containing methods and descriptions of results of studies in the WIPP Environmental Monitoring project and other activities at CEMRC during 2005/2006.



Oxford/Canberra Oasis Alpha Spectrometer



GC-MS for Head Space Gas

Production of this report is supported as part of the Carlsbad Environmental Monitoring and Research Center, a grant from the U.S. Department of Energy to New Mexico State University (DE-FG04-91-AL74167). The issuance of this report and other publications fulfills a CEMRC mission in making the results of CEMRC research available for public access.



Hivol air samplers near WIPP



Lung Counter

This year's cover photograph is of the entrance to the CEMRC facility. The Foreword shows various equipment and laboratories used by scientists at CEMRC.



A citizen getting a whole body count



Student Training in Radiological Issues

OVERVIEW

Current Program Status

HISTORY

The Carlsbad Environmental Monitoring and Research Center (CEMRC) was established in 1991 with a grant from the U.S. Department of Energy (DOE). The primary goals of the CEMRC are to:

- Establish a permanent center of excellence to anticipate and respond to emerging health and environmental needs, and
- Develop and implement an independent health and environmental monitoring program in the vicinity of the DOE Waste Isolation Pilot Plant (WIPP), and make the results easily accessible to all interested parties.

CEMRC is a division of the College of Engineering at New Mexico State University (NMSU). Under the terms of the grant from DOE, the design and conduct of research for environmental monitoring at the WIPP are carried out independently of the DOE, and the production and release of resulting reports do not require DOE review or approval. A brief history of the CEMRC is presented in Appendix A.

The CEMRC is operated as a research institute within NMSU, supported through grants and service contracts. The CEMRC's primary objectives are to:

- Provide for objective, independent health and environmental monitoring;
- Conduct research on environmental phenomena, with particular emphasis on natural and anthropogenic radionuclide chemistry;

- Provide advanced training and educational opportunities;
- Develop improved measurement methods, procedures and sensors; and
- Establish a health and environmental database accessible to all sectors.

Over the last three years, about 60% of CEMRC funding has come from more direct contract support of WIPP by providing facility and scientific support to entities such as Los Alamos National Laboratory (LANL) and Washington TRU Solutions (WTS), such that the monitoring mission dropped from 100% of the Center's activities to about 40%.

KEY ACTIVITIES

The following is a summary of several key activities that are necessary to achieve the goal of establishing and developing CEMRC. Activities to achieve the goal of monitoring in the vicinity of the WIPP are presented in the following section (WIPP Environmental Monitoring Project).

- 1. Assemble a team of highly qualified research scientists and support staff capable of carrying out current and future projects.**

At the end of FY2006, the CEMRC employed 27 personnel (Table 2). Two positions were in recruitment.

- 2. Create state-of-the-art laboratory facilities capable of supporting advanced studies in areas of scientific specialization.**

In January 1997, the CEMRC was relocated to Light Hall, a new 26,000 ft²

laboratory and office facility constructed adjacent to the NMSU-Carlsbad branch campus. In 2005 and 2006, three significant facility upgrades occurred. The first was a radiochemistry laboratory designed for investigating the chemistry of plutonium, uranium and other actinides, particularly for performance assessment studies for the WIPP site by LANL Carlsbad Operations. 600 ft² of laboratory space was separated from the surrounding labs, and new fume hoods and glove boxes feeding into a new HEPA-filtered isolated ventilation system were installed along with new benchwork. Funding for this upgrade was obtained from the City of Carlsbad with additional funds from NMSU CEMRC and LANL.

The second was the construction of an 800 ft² organic chemistry laboratory dedicated to the measurement of volatile organic compounds (VOCs). Over 100 linear feet of benchwork was installed, and a bank of gas chromatograph – mass spectrometers (GC-MS), were set up to measure constituents such as methylene chloride, benzene, carbon tetrachloride, hydrogen, and methane, in WIPP underground air and waste drum head space gas. Funding for this upgrade was obtained from DOE through WTS with additional funds from NMSU CEMRC.

Third, a new ventilation system specifically designed to handle high acid volumes was installed in the primary laboratory wing to accommodate WTS radiochemical operations. Funding for this upgrade was provided by DOE through WTS.

During these 2005/2006 construction activities, the WIPP Environmental Monitoring project at CEMRC was shut down for about 8 months.

The CEMRC's scientific activities are organized into major areas of specialization, with corresponding assignment of staff roles and responsibilities. Although some of the CEMRC's projects involve only one or two of the program areas, all of the program areas collaborate in carrying out the WIPP Environmental Monitoring project. The five scientific program areas include (1) radiochemistry, (2) environmental chemistry, (3) informatics and modeling, (4) internal dosimetry, and (5) field programs. Detailed descriptions of each program area and associated facilities and instrumentation are on the CEMRC web site at <http://www.cemrc.org>.

3. Establish grants and contracts to replace the original grant.

Table 1 gives a summary of CEMRC's budget over the last several years, including 2007. The following is a list of grants and contracts generated during FY2005 and 2006.

DOE CBFO

- 2005 - \$1.2 million for WIPP Environmental Monitoring
- 2006 - \$1.2 million for WIPP Environmental Monitoring

Washington TRU Solutions

- 2005 - \$1.0 million for Technical Services to WTS
- 2006 - \$350,000 for VOC analyses
- 2006 - \$432,000 for construction and equipping a new VOC laboratory
- 2006 - \$85,000 for Whole Body Counting of selected WTS employees
- 2006 - \$225,000 certification for Head Space Gas Analyses under DOE's PDP

Los Alamos National Laboratory

- 2005 - \$910,000 for Actinide Chemistry scientific support
- 2006 - \$801,000 for Actinide Chemistry scientific support

Sandia National Laboratory

- 2005 - \$90,000 for Performance Assessment Scientific Support
- 2006 - \$89,000 for Performance Assessment Scientific Support

City of Carlsbad

- 2005 - \$350,000

OCUP

- 2006 - \$137,899

Rodent

- 2005 - \$50,000

PuDust

- 2005 - \$101,000
- 2006 - \$101,000

Waste Control Specialists

- 2005 - \$47,250
- 2006 - \$52,500

Other

- 2005/2006 - \$35,000

- 4. Establish effective liaisons with leading research groups and laboratories to facilitate shared services and collaborative research.**

In response to the need for expanding the CEMRC research role, the Center has developed a partnership with LANL to conduct actinide chemistry research for WIPP recertification, and with WTS radiochemistry group to support compliance activities such as radiobioassay and WIPP permit-required environmental monitoring. Agreements

and partnerships were also developed with New Mexico Tech in Socorro, the New Mexico Military Institute in Roswell, and the National Guard 64th Civil Support Team and Rio Rancho, to develop emergency radiological response training.

- 5. Publish research results and create a database management system to provide access to information generated by the CEMRC.**

CEMRC staff authored or co-authored many presentations at international, national and regional scientific meetings and 18 papers were published in peer-reviewed scientific journals and books during 2005/2006 (Appendix C). A cumulative list of publications by CEMRC staff since 1996 can be obtained by request, as can previous CEMRC annual reports and other CEMRC information.

- 6. Establish regional, national and international outreach and collaboration.**

During 2005/2006, the CEMRC hosted various colloquia presented by visiting scientists, was involved in other outreach activities including presentations for local civic and professional groups and exhibits for various school, and community events some of which are listed in Appendix D. As described in a later section, over 1000 volunteers from the local community have participated in the “Lie Down and Be Counted” project.

7. Implement programs to offer visiting scientists training in specialized research techniques and methodologies and to involve CEMRC resources and personnel in providing educational opportunities for students nationwide.

During 2005/2006, five undergraduate students worked in laboratory aide positions at the CEMRC; these positions provided training and basic skills development relevant to the position assignments. Visiting scientists on sabbatical leave also worked at CEMRC in support of various research projects (Appendix B).

Table 1: CEMRC Budget Changes Since FY2002 (in \$thousands)

| Funding | 2002 | 2003 | 2004 | 2005 | 2006 | 2007 |
|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| DOE | \$2,461 | \$2,450 | \$698 | \$1,200 | \$1,200 | \$1,200 |
| LANL | 0 | 0 | 175 | 910 | 801 | 848 |
| WTS | 0 | 0 | 1,100 | 1,000 | 1,092 | 1,115 |
| Carlsbad | 0 | 0 | 0 | 350 | 0 | 0 |
| SNL | 0 | 0 | 184 | 90 | 89 | 268 |
| Other | 200 | 148 | 229 | 346 | 179 | 192 |
| TOTAL | \$2,661 | \$2,598 | \$2,386 | \$3,896 | \$3,361 | \$3,623 |

Table 2: Listing of CEMRC Staff as of September 30, 2006

| Name | Position |
|--------------------|--|
| Arimoto, Richard | Senior Scientist |
| Ballard, Sally | Environmental Scientist |
| Brown, Becky | Administrative Services Manager |
| Bill Brown | Facilities Manager |
| Conca, James | Director |
| Ganaway, David | Environmental Scientist |
| Garrett, Fran | Secretary |
| Greene, Chris | Physicist Scientist |
| Hudston, Lisa | Environmental Scientist |
| Khaing, Hnin | Environmental Scientist |
| Kirchner, Thomas | Computer & Information Systems Manager |
| Kirchner, Vicki | Technology Specialist |
| Marple, Julia | Chemical Technician |
| McCauley, Sharyl | Quality Assurance Manager |
| Monk, James | Environmental Scientist |
| Najera, Angela | Secretary |
| Nesbit, Curtis | Environmental Scientist |
| Pennock, Karl | Environmental Scientist |
| Porter, Ruthie | Records Technician |
| Sage, Sondra | Physical Scientist |
| Schoep, David | Radiation Safety Training Specialist |
| Sneller, Michele | Chemical Technician |
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WIPP Environmental Monitoring Project

PROJECT CONCEPT

As defined in the original grant, the purpose of the WIPP EM project is to establish and maintain independent environmental research and monitoring in the vicinity of the WIPP and to make the results easily accessible to all interested parties. This project was implemented during the WIPP pre-disposal phase, and is now continuing during the operational (disposal) phase. The WIPP EM project is organized and carried out independent of direct oversight by DOE, and the project does not provide data to any regulatory body to meet the compliance demonstration requirements applicable to the WIPP. Analytical results and interpretations from the WIPP EM are published by CEMRC to inform the public and particularly the environmental science community.

A detailed description of the WIPP EM concepts, sampling design and baseline studies is presented on the CEMRC web page. The following is a summary of 2005/2006 activities for each major environmental medium in the WIPP EM. It is important to note that nuclear waste first began being received at WIPP on March 26, 1999. Mixed waste was first received by the WIPP on September 9, 2000, and higher-activity waste (called remote handled or RH waste) was first received at the beginning of 2007. Results summarized in this report cover samples collected through December 2006.

Based on the radiological analyses of monitoring phase samples (collected since March 26, 1999) completed to date for area residents and for selected aerosols, soils, drinking water and surface water,

there is no evidence of increases in radiological contaminants in the region of the WIPP that could be attributed to releases from the WIPP. Levels of radiological and non-radiological analytes measured in 2005 and 2006 were within the range of baseline levels measured previously by CEMRC for the targeted analytes, and are within the ranges measured by other entities at the State and local levels since well before disposal phase operations began in 1999.

In the summer of 2001, the Carlsbad Field Office (CBFO) of DOE requested CEMRC to investigate whether the Center's direction could become more closely aligned with scientific and analytical activities foreseen by the CBFO to support the safe and efficient operation of the WIPP. To further develop the CEMRC program, during 2005/2006 the Center has been working with the CBFO management to define research and analytical tasks that will address such needs. This redirection permits CEMRC to pursue new research avenues aggressively in partnership with (versus independent of) the DOE community.

The tasks requested to be performed by the CEMRC during 2005/2006 included:

- Analytical and scientific support for the LANL Actinide Chemistry and Repository Science Program including construction of a new actinide chemistry laboratory during 2005 and 2006 that is focused on research with Pu and other actinide elements under WIPP conditions
- Identification and quantification of Gnome-derived radionuclides

- Environmental safety and health support for WIPP operations including providing WTS scientists with radio-chemical laboratory space, office space and safety support for environmental compliance and monitoring activities; measurement of VOCs in WIPP underground air for permit compliance, and development and certification of the capability to measure head space gas constituents in WIPP waste drums, including construction of an 800 ft² organics laboratory during 2006. A summary of the progress made on these tasks is also provided in this report.

ORGANIZATION OF THE MONITORING PROGRAM

The scheduling and management of sample analyses collected in the WIPP EM project are based on (1) priorities for providing information to the public, (2) relative risks of human exposure to contaminants among the various media sampled, (3) needs for data validation and verification prior to release, (4) time constraints resulting from sample preparation and analysis procedures, (5) funding changes, and (6) time and resource coordination among the other programs in the facility.

During 2003/2004, the elements of the monitoring project were reviewed and evaluated as part of the strategic planning for CEMRC activities over the next few years. A re-definition of the scope of the monitoring program has been driven by two factors - (1) the diminishing resources available for the monitoring work, and (2) the increased emphasis at CEMRC on direct research and technical support of WIPP operations. The challenge that has faced CEMRC during 2005/2006 has been to restructure and optimize the WIPP EM

in order to maintain a long-term environmental monitoring program that will contribute to the public's confidence in the safe operation of the WIPP, and identify missing elements in our understanding of the WIPP environment that are not addressed by the ongoing and proposed long-term monitoring studies.

A major reduction in the resources devoted to the WIPP EM was proposed by CEMRC through a cut back in the frequency of sampling of the various media and by reducing the number of target analytes. The justification for this reduction is based on the fact that, to date, there has been no evidence for any perturbation to drinking water, soils, surface water or sediments caused by the WIPP operations. Studies of airborne particulate matter (aerosols) will continue to be the major focus of the CEMRC's monitoring efforts because, in the event that radioactive or chemical contaminants are released from WIPP, these materials could be rapidly dispersed through the atmosphere and spread throughout the environment. In addition, monitoring of the public through the Lie Down and Be Counted program is of the utmost importance as humans are the most important target whatever will be the transmission vector for contaminants.

Past public surveys indicated that air monitoring and direct monitoring of people (whole body counting), followed by monitoring of drinking water, were the areas of greatest public interest. While it is highly unlikely that any chemical impacts of the WIPP will be detected through analyses of media other than air and people, CEMRC considers there is value in continued monitoring of soils, water and sediments, and vegetation and biota in some form and frequency. Thus, a program has been recommended, and will

be revised yearly with input from various stakeholders, in which one of the media other than air and people are sampled each year on a rotating basis.

The continuation of the WIPP EM and new WIPP-related projects reflect the Center's commitment to ensuring that the public, workers, and the environment are protected from exposure to contaminants. It is likely that additional adjustments to the WIPP EM will be needed as the Center's capabilities continue to evolve and the other programs supporting the WIPP also move in new directions.

AEROSOLS

Aerosol particle sampling is conducted at four locations, with samplers operating continuously at each location. The locations include a port inside the WIPP exhaust shaft (Station A, FAS samples), a site approximately 0.1 km northwest (downwind) of the WIPP exhaust shaft (On Site station), a site approximately 1 km northwest (downwind) of the WIPP (Near Field station), and a site approximately 19 km southeast (upwind) of the WIPP (Cactus Flats station). In November 2006, CEMRC began collecting samples at a point inside the WIPP exhaust but after the filtration system (Station B). The schedule for analysis of these samples will be decided based upon discussions with stakeholders.

Continuous sampling of aerosol particles was conducted through December 2006. Analyses of all particle samples collected through December 2005 for both radiological and non-radiological constituents have been completed and are reported herein. All FAS samples from 2006 have been analyzed with respect to gross alpha and beta and are reported herein, while 2006 samples from the other

aerosol sites along with spectroscopy on the 2006 FAS samples are still being analyzed.

SOILS

Soil samples were collected during 2005, and measurements were made for ^{137}Cs , ^{208}Tl , ^{212}Pb , ^{212}Bi , ^{214}Bi , ^{214}Pb , ^{228}Ac , and ^{40}K . The analyses for ^{234}U , ^{235}U , ^{238}U , ^{230}Th , ^{232}Th , ^{228}Th , $^{239,240}\text{Pu}$, ^{241}Am are not reported because of low yields. Soil samples were collected during 2006 and have been archived for future analysis to be based upon programmatic decisions.

SURFACE WATER AND SEDIMENTS

Surface waters and sediments were collected during 2005 from three regional reservoirs situated on the Pecos River - Brantley Lake, Red Bluff and Lake Carlsbad. Surface waters were analyzed for inorganic constituents.

DRINKING WATER

The WIPP EM studies of ground water focus on the major drinking water supplies used by communities in the WIPP region because these are often perceived by the public as a potential route for contaminants to reach humans. Five community supplies of drinking water (representing three major regional aquifers) are included in routine sampling, including Carlsbad, Loving/Malaga, Otis, Hobbs and a secondary source for Carlsbad. One private water well (representing a fourth aquifer) that is located within 16 km of the WIPP is also sampled. During 2005, drinking water samples were collected in the spring at five of the six drinking water supplies (the sixth was dry), and results of radiological

and non-radiological analyses are reported herein for 2005.

BIOTA

During 2005/2006, no vegetation samples were collected or analyzed.

HUMAN POPULATION

The *Lie Down and Be Counted* (LDBC) project serves as a component of the WIPP EM that directly addresses the general concern about personal exposure to contaminants shared by residents who live near DOE sites. As in other aspects of the WIPP EM, *in vivo* bioassay testing was used to establish a baseline profile of internally-deposited radionuclides in a sample of local residents before disposal phase operations began, and has continued into the disposal phase to the present. The sampling design includes solicitation of volunteers from all segments of the

community, with sample sizes sufficient to meet or exceed a 15% range in margin of error for comparisons between major population ethnicity and gender categories as identified in the 1990 census. Radiobioassays of the original volunteer cohort have been ongoing since July 1999. New volunteers will continue to be recruited each year to establish new study cohorts and replace volunteer attrition. Results of the LDBC project through December 2006 are reported herein.

RADIOCHEMICAL AND ACTIVITY UNITS

The primary unit of activity, or radioactivity, used in this report is the becquerel (Bq) which is equal to one disintegration of a nucleus per second. This disintegration gives rise to ejection of a particle or ray of ionizing radiation, either an alpha, beta, neutron, or gamma.

Quality Assurance

The CEMRC is subject to the policies, procedures and guidelines adopted by NMSU, as well as state and federal laws and regulations that govern the operation of the University. The management of CEMRC is committed to conducting a well-defined quality assurance program, incorporating good professional practice and focusing on the quality of its testing and calibration in research and service to sponsors. CEMRC technical programmatic areas in 2005-2006 included: Environmental Chemistry, Organic Chemistry, Radiochemistry, Field Programs, Informatics and Modeling and Internal Dosimetry. The development and implementation of an independent health and environmental monitoring program has been CEMRC's primary activity since establishment.

PROJECT REPORTING REQUIREMENTS

Since its inception, CEMRC's WIPP Environmental Monitoring Program (WIPP-EM) has been conducted as a scientific investigation, that is, without any compliance, regulatory, or oversight responsibilities. As such, there are no specific requirements for reporting data other than good scientific practices. An example of reporting decisions made by CEMRC for this program is whether to correct or not correct data for blanks. The decision to subtract blanks from the monitoring data was made by the senior staff in the mid-1990s because the consensus opinion was that this procedure provided the best means for determining the analytes' true concentrations, i.e. bias-

free estimates of the values. The practice of correcting environmental data for blanks is well established, as described by the International Union of Pure and Applied Chemistry (IUPAC) and The International Organization for Standardization (ISO). See also <http://epa.gov/waterscience/methods/det/fa/ca/mtg20051208/blank.html>

QUALITY ASSURANCE PROGRAM

Beginning in early 2002, a significant effort was devoted to refining CEMRC's quality system to meet applicable requirements of the U.S. DOE Carlsbad Field Office (CBFO) Quality Assurance Program Document (QAPD, CAO-94-1012). This effort was in response to the CBFO's request for a change in CEMRC's direction to allow it to become more closely aligned with scientific and analytical activities seen by CBFO to support the safe and efficient operation of WIPP. As a result, CEMRC produced a center-wide Quality Assurance Plan (QAP) CP-QAP-004, which was subsequently submitted to and approved by DOE.

Internal surveillances were performed during 2005/2006 on the following programmatic areas: Environmental Chemistry, Field Programs, Informatics and Modeling, Internal Dosimetry Organic Chemistry and Radiochemistry. In addition, internal surveillances were performed in the Administrative, Quality Assurance areas as well as on Document Control and maintenance of Scientific Notebooks. A summary of 2005/2006 audits is reported in Appendix E.

QUALITY ASSURANCE/ QUALITY CONTROL FOR ORGANIC CHEMISTRY

The following audits were conducted on the Organic Chemistry group:

- A VOCs Confirmatory Monitoring Audit, by WTS QA, February 2005 and February 2006. Both audits were passed. These audits were routine yearly program audits conducted in compliance with contract requirements.
- CEMRC QA audits were conducted on the OC group July 9-11, 2005, and June 13-14, 2006. Both audits were passed and were conducted in compliance with the Center's QAP.

QUALITY ASSURANCE/QUALITY CONTROL FOR RADIOANALYSES

Routine quality assurance/quality control activities conducted for radioanalyses include tracking and verification of analytical instrument performance, use of American Chemical Society certified reagents, use of National Institute of Standards and Technology (NIST) traceable radionuclide solutions and verification testing of radionuclide concentrations for tracers not purchased directly from NIST or Eckert and Ziegler Analytics. When making laboratory solutions, volumes and lot numbers of stock chemicals are recorded. Prior to weighing radionuclide tracers and samples, the balance being used is checked using NIST traceable weights.

Control checks were performed on all nuclear counting instrumentation each day or prior to counting a new sample. The type of instrument and methods used for performance checks were as follows: for the Protean 9604 gas-flow α/β proportional counter used for the FAS program, efficiency control charting was performed using ^{239}Pu and ^{90}Sr check sources along with ensuring that α/β cross-

talk was within limits. Sixty-minute background counts were recorded daily. Two blanks per week for the FAS program were counted for 20 hours and were used as a background history for calculating results.

Routine background determinations were made on the HPGe detector systems by counting blank samples, and the data was used to blank correct the sample concentrations.

For the Oxford Oasis alpha spectrometer, efficiency, resolution and centroid control charting was performed using ^{148}Gd check sources on a regular basis. Before each sample count, pulser checks were performed to ensure acceptable detector resolution and centroid. Blanks counted for 5 days were used as a background history for calculating results.

During 2004 - 2006, CEMRC participated in the NIST Radiochemistry Intercomparison Program (NIST-RIP) and the Mixed-Analyte Performance Evaluation Program (MAPEP-05) for soil, air filter and water analysis. In NRIP, there were a variety of matrices to analyze. Isotopes of interest in the NRIP studies were ^{234}U , ^{238}U , ^{238}Pu , ^{239}Pu , ^{230}Th , ^{241}Am , and ^{90}Sr . In the 2005 study, CEMRC performed consistently well when analyzing ^{234}U , ^{238}U , ^{238}Pu , ^{239}Pu and ^{241}Am , with the experimental values nearly matching the NRIP reported values. The other two isotopes, ^{230}Th and ^{90}Sr performed less well, with some experimental values exceeding the lower limit of acceptability. However, replicate samples of these analytes were within the acceptable range.

For MAPEP, the matrices selected were air filters, soils, and water and the isotopes were $^{233/234}\text{U}$, ^{238}U , ^{238}Pu , $^{239/240}\text{Pu}$, ^{241}Am , and ^{90}Sr . The analyses were carried out

using CEMRC's actinide and separation procedures, and were treated as a regular sample set to test regular performance. CEMRC's results were consistently close to the known value, with only two "Not Acceptable" results on strontium analysis.

Results for NRIP and MAPEP are given in Appendix E.

QUALITY ASSURANCE/QUALITY CONTROL FOR ENVIRONMENTAL CHEMISTRY INORGANIC ANALYSES

The analytical methods employed for inorganic analyses in the environmental chemistry program at CEMRC are based, when applicable, on various standard procedures (EPA/600/4-79-020, 1983; EPA/SW-846, 1997; American Public Health Association, 1981). For some matrix/analyte combinations, appropriate external standard procedures do not exist, and for those cases, specialized procedures have been developed to meet the needs of the WIPP EM and other research projects.

INSTRUMENTATION

A DIONEX 500 ion chromatography (IC) system is used to determine the concentrations of a suite of anions, including nitrate, nitrite, sulfate, chloride, fluoride, and phosphate in water samples and aqueous extracts of aerosol samples, soils, and sediments. Configured differently, the same instrumentation can be used to determine the concentrations of several cations (calcium, magnesium, sodium, ammonium and potassium). The anion analyses are performed with the use of AS11 and AS14 anion exchange columns and AG11 and AG14 guard columns, with chemical suppression and conductivity detection. The cations are determined using a CG12A guard column

and a CS12A analytical column, with the same type of chemical suppression and conductivity detection.

Inorganic analyses were performed using Perkin-Elmer Elan 6000 and 6100 inductively-coupled plasma mass spectrometers (ICP-MS). Regular QC verifications and batch QC provide records of sample performance data. For all environmental chemistry analyses, QC samples are analyzed with each sample batch as an indicator of the reliability of the data produced. The types, frequencies of analysis, and limits for these QC samples have been established in a set of standard operating procedures. Extraction QC samples include Laboratory Reagent Blanks, or LRBs (for aerosol and FAS samples, unused cellulose ester filters were used as LRB samples), Laboratory Fortified Blanks, or LFBs (a cellulose ester CRM, "Trace Metals on Filter Media" from High Purity Standards in Charleston, South Carolina, was used for QC of aerosol sample metals analyses), duplicates and Laboratory Fortified Matrix (LFM) samples. In cases where duplicate aliquots from the original sample were not feasible (such as aerosol filters), separate aliquots of the sample extract were analyzed for the duplicate and LFM analyses. The digestion QC parameters used for the evaluation of constituents in water, soils, and sediments were based on concepts in EPA Contract Laboratory Program (EPA 540/R-94013, 1994); and SW-846 methods (EPA/SW-846, 1997). No comparable control parameters presently exist for aerosol samples. All constituents values were reported relative to the method detection limit as determined by the method outlined in 40 CFR 136, Appendix B.

For each ICP-MS analysis, the QC requirements are as follows: 1) A spiked

blank (LFB, or laboratory fortified blank) is prepared identically to a sample for every batch (ten samples) and its percent recovery must be within 15%. 2) A batch blank (LRB, or laboratory reagent blank) is prepared and analyzed for every ten samples, and its value must be lower than the method detection limit (MDL). If the value is higher than the MDL, the entire batch is reanalyzed up to 3 times. If the value consistently falls above the MDL, blank subtraction may be performed on the samples in that batch, or the data for the analyte(s) in question are flagged or not reported, at the discretion of the lead scientist. 3) One duplicate sample for every ten samples is either collected in the field or two aliquots from a single field sample are prepared and analyzed identically. The percent difference between duplicates must be within 20%. 4) One laboratory fortified sample matrix (LFSM) is prepared for each batch of 10 samples by spiking a sample with a known amount of standard. The percent recovery for the spike must fall within 15% of the expected value. 5) After calibration, an initial calibration verification (ICV) standard from a different lot number and/or manufacturer of the calibration standards is analyzed, and the value must fall within 10% of the expected value. If one or more analytes falls outside of the expected range, recalibration is performed or the analyte(s) in question are either flagged as having a greater uncertainty or are not reported. 6) A mid-range calibration standard is reanalyzed every ten samples and the percent recovery must be within 15% of the true value. 7) The calibration blank is reanalyzed immediately after calibration and then every ten samples thereafter (including batch blanks and batch spiked blanks) and must be less than 3 times the instrument detection limit. 8) The relative percent difference between the 3 replicate sweeps

of the instrument for each analyte must be less than 20%. 9) The correlation coefficient for the linear regression of the calibration curve must be greater than 0.995. 10) All samples and standards are spiked with an internal standard (usually indium), and the percent recovery of the internal standard must lie between 60% and 125% of the value measured in the calibration blank.

Independent quality assurance samples are obtained and analyzed to verify the performance of the instrumentation and the proficiency of the analyst. Reference samples (obtained from an outside source or prepared internally, with true values known at the time of analysis) are the primary method used to perform this function at CEMRC. Occasionally, blind samples (obtained from an outside source, with true values not known at the time of analysis) are used. However, since blind samples are usually diluted many times, the instrument is not optimized for any one or group of elements, and the instrument measures such a large number of analytes at one time at near their MDCs, several analytes often exceed the acceptable range by several percent, in particular aluminum, beryllium, cobalt, iron, chlorine and fluorine. This increases the overall uncertainty of the analyses. Examples of results from a reference sample and a blind sample (from the Environmental Resource Associates [ERA] WatR™ Supply Proficiency Testing Study) for 2005 are given in Appendix E. Table E-4 shows that, of the analytes run, Al, Be, Co and Fe were not within the acceptable range of approximately $\pm 10\%$ in 2005 and Br, and Cl were not within the acceptable range in 2006. Instead, these analytes were between $\pm 10\%$ and $\pm 20\%$. In this report, assume that these analytes have a $\pm 20\%$ uncertainty associated with their values. In 2006, F had an uncertainty of 25%,

therefore, for this report assume F has a $\pm 25\%$ uncertainty associated with its values. Table E-6 gives an example of the daily performance tests for ICP-MS.

QUALITY ASSURANCE/QUALITY CONTROL FOR FIELD SAMPLING

For the collection of most WIPP EM samples, no external standard procedures are considered completely appropriate for the objectives of the studies. In these cases, customized plans are developed and documented. After the activity is completed, the plan is revised to reflect any departures from the original plan, and documented to file. For most environmental media, the sampling plans combine selected standard procedures with specific adaptations to address scientific objectives of interest. For example, procedures for collection and preservation of samples for compliance with Safe Drinking Water Act requirements are applied to the collection of drinking water and surface water samples, but the locations of sample collection are selected on the basis of other criteria. Likewise, high-volume air samplers are operated to meet an EPA standard of $1.13 \text{ m}^3 \text{ min}^{-1}$, but the frequency of filter replacement is based on optimal loading for radioanalysis.

Logbooks are maintained by technical staff in field operations to record locations and other specifics of sample collection, and data on instrument identification, performance, calibration and maintenance. Data generated from field sampling equipment are error-checked by using routine cross checks, control charts and graphical summaries. Most data collected in written form are also entered in electronic files, and electronic copies are crosschecked against the original data

forms. All electronic files are backed up daily.

Calibration and maintenance of equipment and analytical instruments are carried out on predetermined schedules coinciding with manufacturer's specifications or modified to special project needs. Calibrations are either carried out by equipment vendors or by CEMRC personnel using certified calibration standards.

QUALITY ASSURANCE/QUALITY CONTROL FOR INTERNAL DOSIMETRY

The *in vivo* bioassay program at CEMRC participates in the Department of Energy's *In Vivo* Laboratory Accreditation Program (DOELAP) via WIPP, and is currently accredited as a service laboratory to perform the following direct bioassays:

- Transuranium elements via L x-ray in lungs
- ²⁴¹Am in lungs
- ²³⁴Th in lungs
- ²³⁵U in lungs
- Fission and activation products in lungs including ⁵⁴Mn, ⁵⁸Co, ⁶⁰Co and ¹⁴⁴Ce
- Fission and activation products in total body including ¹³⁴Cs and ¹³⁷Cs

Under DOELAP, the *in vivo* bioassay program is subject to the performance and quality assurance requirements specified in *Department of Energy Laboratory Accreditation Program for Radiobioassay* (DOE-STD-1112-98) and *Performance Criteria for Radiobioassay* (ANSI-N13.30). A DOELAP testing cycle was completed in 2005-2006 that included counting phantoms representative of each of the categories listed above.

To evaluate system performance, quality control data were routinely collected throughout the year in order to verify that the lung and whole body counting system was operating as it was at the time the system was calibrated. Quality control parameters that track both overall system performance and individual detector performance were measured. Quality control parameters tracked to evaluate individual detector performance, included:

- Net peak area, peak centroid and peak resolution (FWHM) across the energy range of the spectrum,
- Detector background

Quality control parameters tracked to assess overall system performance included:

- Mean weighted activity of a standard source
- Summed detector background

In addition, calibration verification counts were routinely performed using NIST-traceable standards and phantoms.

The Internal Dosimetry program also participated in an intercomparison study program for whole body counting administered by Oak Ridge National Laboratory (ORNL). Under this program bottle phantoms containing unknown amounts of ¹³⁷Cs, ⁶⁰Co, ⁵⁷Co, ⁸⁸Y and ¹³³Ba are sent to CEMRC, quarterly. The phantoms were counted on the lung and whole body counting system and the measured activities were reported back to ORNL and compared against the known activities. An example of results for one quarter is shown in Appendix E. For all years since CEMRC has participated in the ORNL program, CEMRC has consistently out-performed all other laboratories in this area.

INDEPENDENT REVIEWS

Periodically, CEMRC has had independent review panels and scientific advisory boards that have addressed scientific and programmatic issues and questions, and have provided guidance and recommendations. In 2007, a review panel made up of chemistry professors from the main campus of NMSU (Prof. Gary Eiceman, Department of Chemistry and Biochemistry, Prof. Gary Rayson, Department of Chemistry and Biochemistry, and Dr. F. William (Bill) Boyle, of the SWAT - Soil, Water, and Air Testing laboratory) performed a scientific review centering on two primary QA questions concerning data generated in 2005/2006: 1) should instrument blanks be subtracted or corrected for in the final values of data from analyses such as ICP-MS, and 2) what constitutes valid resolution of failures for certain analytes on external performance tests and what impact should this have on reported data for research projects.

The panel commended the staff at CEMRC for having developed and followed criteria for quality assurance and quality control during the acquisition of sample analyses.

The consensus of the panel was that every effort should be made in the reporting of analysis results to provide sufficient information for any reader to arrive at an interpretation of those results. Regarding the use of blank subtraction in data processing, it was recommended that measurements arising from reagent blanks not be systematically subtracted from all subsequent sample measurements, but that those values be included in any reports to enable the most accurate interpretation of the results. Although blank subtraction is a

common data analysis procedure, the often extremely low concentrations that can be encountered with samples analyzed at CEMRC necessitates a post-calibration processing of blank measurements with the requisite statistical evaluations. In response to this panel recommendation the ICP-MS results tabulated in this report include a column showing the blank values for each analysis.

The panel felt that the inability of any laboratory to agree with “acceptable” external values for a concentration of targeted analytes within performance evaluation samples should not be considered as an indictment of their abilities to generate accurate results. It should, however, be interpreted as an opportunity to re-evaluate the QA/QC protocols and criteria for acceptable performance. Results generated following a “failed” performance evaluation sample should be reported with an indication of that non-agreement with “known” samples and a discussion of the causes of such results including efforts made to address any concerns revealed during post-PE sample internal evaluations. Simply stated, it is the opinion of the panel that full disclosure of all results, both positive and “negative,” should be provided in any and all reports, a practice that CEMRC has always followed. As a result, the performance tests in 2005 and 2006 are presented in Appendix E with all results, including those several analytes that were outside the acceptable ranges as discussed above.

CEMRC thanks the panel members for their time and effort. CEMRC intends to continue these types of periodic reviews in the future.

