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# LESSONS LEARNED FROM RADIOACTIVE/MIXED WASTE ANALYSES AT EG&G IDAHO, INC.

Richard J. Murphy
Shelly J. Sailer
Joseph T. Bennett
Janine S. Arvizu

Idaho National Engineering Laboratory
EG&G Idaho, Inc.
P.O. Box 1625
Idaho Falls, Idaho 83415-4123

## INTRODUCTION

For the past 30 years extensive chemical characterizations of environmental and waste samples have been performed by numerous academic, commercial, and government analytical chemistry laboratories for the purposes of research, monitoring, and compliance with regulations. The vast majority of these analyses, however, has been conducted on samples containing natural concentrations of radioactive constituents. It is only within the last decade that a small number of laboratories have been conducting extensive chemical characterizations of highly radioactive samples and consequently have begun to identify many special requirements for the safe and accurate conduct of such analyses.

Experience gained from chemical analyses of radioactively contaminated samples has indicated special requirements and actions needed in the following three general areas:

- Sample collection and preservation
- Chemical analysis protocols
- Disposal of waste from chemical analyses

In this paper we will summarize the experience and findings acquired from four years of radioactive sample analyses by the Environmental Chemistry Unit, an analytical chemistry laboratory of EG&G Idaho, Inc.[a] at the Idaho National Engineering Laboratory.

## LABORATORY CAPABILITIES AND EXPERIENCE

The Environmental Chemistry Unit (ECU) of EG&G Idaho, Inc. provides analytical chemical services to support environmental, health and safety, and research and development activities at the Idaho National Engineering Laboratory (INEL) and within the U.S. Department of Energy (DOE). The laboratory is currently equipped to analyze elemental, inorganic, organic, and physical parameters in a wide variety of matrices including radioactively contaminated samples. Chemical analyses performed range from traditional wet chemical procedures to trace level analyses utilizing state-of-the-art instrumentation. The principal analytical techniques available are atomic absorption spectrometry (AAS), gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), ion chromatography (IC), inductively coupled plasma/atomic emission spectrometry (ICP/AES), and inductively coupled plasma/mass spectrometry (ICP/MS). Analytical chemical services and research activities include environmental monitoring, waste characterization, analytical method development, analytical program design and operation, sampling plan review, and data quality assessment.

The ECU laboratory was established in 1985 with a small staff and a few pieces of equipment to chemically characterize liquid effluent from INEL facilities to comply with the Clean Water Act. In 1986 the laboratory began to expand in both staff and instrumentation as a participant in the DOE initiated Environmental Survey Program to identify environmental issues at all DOE sites through a sampling and analysis program. Between 1986 and 1989 the primary emphasis of laboratory work was on samples of the DOE Environmental Survey Program, but some samples from INEL were also analyzed. During this period the percentage of samples analyzed that were radioactive was about 20%. Since 1989 the bulk of the laboratory's analyses has been on samples from the INEL with the percentage of the samples which are radioactive being roughly 35% and steadily increasing.

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## SPECIAL REQUIREMENTS FOR RADIOACTIVE SAMPLE ANALYSES

The ECU has identified numerous special requirements for analyses of radioactive/mixed waste samples. These opinions were derived from analyses of radioactive samples ranging from naturally occurring concentrations up to approximately 1 R/hr (i.e., Roentgen per hour). The predominant radionuclides present in the samples were transuranics and fission products with all the normally associated types of emissions (i.e., alpha particles, beta particles, neutrons, gamma rays, X-rays).

For the safe and efficient analysis of radioactive/mixed waste samples, the ECU has identified six categories of special requirements, which are as follows:

- General considerations
- Sample collection, preservation, and transport
- Receiving and storage of radioactive samples
- Preparation of radioactive samples for chemical analysis
- Instrumental chemical analysis of radioactive samples
- Disposal of radioactive samples and radioactive waste

Specific requirements, problems, and needs for each of these categories are provided in Tables 1-6.

## FUTURE NEEDS AND RECOMMENDATIONS

In 1989 environmental monitoring, environmental restoration and waste management activities at DOE sites generated approximately two million potentially radioactively contaminated samples, and it is projected that during the next decade the number of such samples may increase by an order of magnitude or more. It is also likely that in future samples the level of radioactive contamination will substantially increase as the most difficult environmental restoration and waste management problems at DOE sites are undertaken. Presently about 45 analytical chemistry laboratories within the DOE and seven commercial laboratories provide support for these DOE activities; clearly the capacity of the currently participating laboratories will not be adequate for the projected analytical chemistry needs of DOE during the next decade.

The projected increase in both the number of samples and level of radioactive contamination of samples requiring analysis to support DOE environmental and waste management projects requires immediate attention. To adequately and cost effectively fulfill these analytical chemistry needs of DOE during the next decade, a systematic plan is needed to investigate and implement programs to accomplish the following:

- Identify and prioritize environmental and waste management problems
- Quantify specific sampling and analytical chemistry needs
- Establish standardized systems or programs for application at analytical chemistry laboratories throughout DOE for laboratory management, quality assurance, data quality objectives, and data reporting requirements
- Identify and develop required sample handling techniques
- Identify and develop required analytical chemistry methods
- Design and construct needed sample handling facilities
- Design and construct needed analytical chemistry facilities with emphasis on remote handling and automation
- Minimize waste generation and develop waste treatment technologies

The key to the long-term resolution of DOE's current environmental restoration and waste management problems is team work. More frequent collaboration among individual laboratories is needed, and increased management involvement by DOE Headquarters is essential.

Table 1. General considerations and special requirements for analysis of radioactive samples

| Requirement | Problem | Need |
|---|---|---|
| Cost | Increased labor hours | Training, testing, protection |
| Cost | Mixed and nonrad | Redundant hardware |
| Deadlines | Long analysis time | New methods, planning |
| Facility | Irradiation | Proper construction, shielding |
| Facility | Contamination | HVAC, remote facilities |
| Facility | Inefficient design | Proper design, construction |
| Facility control | Release | Proper construction, HVAC, |
| Personnel Protection | Exposure | Training, equipment |
| Personnel | Staffing | Training, recruitment |
| Planning | Unexpected | Contingencies |
| Quality Assurance | Variable requirements | Standardization |
| Regulatory | RCRA vs. CERCLA | Collaboration |

Table 2. Special requirements for sample collection, preservation, and transport of radioactive samples

| Requirement | Problem | Need |
|---|---|---|
| Chemical preservation | Exposure | Personal protective equipment, remote handling |
| Personnel protection | Exposure | Training, monitoring equipment |
| Personnel protection | Exposure | Personal protective equipment |
| Sampling methodologies | Exposure, release | Reduce sample sizes, remote sampling, containment |
| Sample transportation | Exposure, regulations | Shielding, container safety |

Table 3. Special requirements for receiving and storage of radioactive samples

| Requirement | Problem | Need |
|---|---|---|
| Hold times | Radiological analyses | Hold time studies |
| Hold times | Radiological analyses | Faster radiological methods |
| Refrigeration | Irradiation, contamination | Equipment |
| Sample control, transfer | Exposure | Remote facilities, HVAC |

Table 4. Special requirements for preparation of radioactive samples for chemical analysis

| Requirement | Problem | Need |
|---|---|---|
| Concentration | Increased exposure | New, adjusted methods |
| Detection limit | Decreased sample size | Meet regulatory requirements |
| Digestion/extraction | Producing mixed waste | New prep methods, development |
| Digestion/extraction | Radionuclide carryover | New prep methods, development |
| Digestion/extraction | Contaminated labware | Multiple, disposable sets |
| Digestion/extraction | Exposure | New prep method (microwave) |
| Filtration | Concentration of radionuclides | New methods |
| Percent Moisture | Drying alpha particle emitting samples | Remote, ventilated ovens |

Table 5. Special requirements for instrumental chemical analysis of radioactive samples

| Requirement | Problem | Need |
|---|---|---|
| Detection limit | Sample size | Multi-analyte methods |
| Documentation/reporting | Volume, compatibility | Standardization |
| Data quality objectives | Variable | Standardization, collaboration |
| GC/MS | GC, source, analyzer contamination | Isolated, dedicated instruments |
| ICP | High temperature, volatilization | Large spares inventory |
| ICP | Chemical interference | Alternative methods, Development |
| ICP/MS | Unnatural isotopic abundances | Method development |
| Instrumental analysis | Contamination and irradiation | Multiple and remote instruments |
| Ion chromatography | Nuclides trapped in column | Method development, multiple instruments |
| Purge and trap | Chemical contamination in hot cell | Several remote facilities |
| Screening methods | Availability, acceptability | Method development |

Table 6. Special requirements for disposal of radioactive samples and radioactive waste

| Requirement | Problem | Need |
|---|---|---|
| Process, Treatment | Treatment permit | Process precipitation, exchange |
| Storage | RCRA vs. ALARA (i.e. safety) | Collaboration, standardization |
| Waste disposal | Method required volumes | New methods |
| Waste disposal | Mixed streams | Waste tracking |

# END

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