

# CHARACTERIZATION OF RADIOACTIVE MIXED WASTES:

## THE SCIENTIFIC PERSPECTIVE\*

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W. H. Griest and J. R. Stokely, Jr.  
Analytical Chemistry Division  
Oak Ridge National Laboratory  
Oak Ridge, Tennessee 37831-6120

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### INTRODUCTION

This paper is concerned with the physical and chemical characterization of radioactive mixed wastes (RMW): what should be determined and how; the applications and limitations of current analytical methodologies, promising new technologies, and areas where further methodology research is needed. Constituents to be determined, sample collection, preparation, and analysis are considered. The scope concerns mainly low level and very low level RMW whose activities allow contact handling and analysis by Nuclear Regulatory Commission- or Agreement State-licensed commercial laboratories. It also includes high level RMW which will be characterized in laboratories with special shielded or contained facilities.

### WHAT SHOULD BE DETERMINED?

The characterization of RMW is driven by regulatory and treatability requirements. Currently, the regulatory requirements are defined jointly by the Environmental Protection Agency (EPA) and the Nuclear Regulatory Commission (NRC) under the Resource Conservation and Recovery Act (RCRA) and the Atomic Energy Act, respectively, with enforcement of both delegated to states having EPA Authorizations and NRC Agreements. Specific constituents and concentrations or activities are specified. From a scientific viewpoint, the Toxicity Characteristic Constituents and the TCLP scenario of waste co-disposal with municipal refuse in a landfill are not necessarily relevant to RMW disposal. The source terms (the wastes or their treated forms) and the localities of release to the environment (leaching and migration to drinking water supplies) are very different for RMW than for the solid wastes RCRA was intended to regulate. Making radioactivity a Toxicity Characteristic and setting de minimis levels of activity would remove the uncertainties leading to current "no added radioactivity" policies which classify all hazardous wastes as RMWs until they are proven otherwise.

Regulatory agencies should permit more realistic leaching procedures for RMW which reflect their ultimate forms and modes of disposal or storage. Simultaneously, it should be a high priority in the DOE to develop the scientific understanding of the leaching behavior and ultimate fate of the

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leached compounds in the different waste disposal and treatment options. For high level wastes, this should include evaluations of the changes in waste composition and properties over time which could affect the long-term waste performance (e.g., decay of radionuclides and radiolysis of organics). This knowledge could considerably simplify and "target" the waste characterization needed for evaluation of waste form ageing and environmental behavior. A good example is the concentrated brine leaching which will be evaluated at the Los Alamos National Laboratory (LANL) for assessing the suitability of radioactive wastes for disposal in the Waste Isolation Pilot Plant. Leachability and fate are included in the studies.

The constituents and properties which should be measured for waste treatability requirements are complex and not fully understood. The constituents and waste properties which affect the waste form for the major treatment technologies under consideration obviously must be identified and concentration and property range "windows" acceptable to the treatment processes must be determined. These will define the RMW characterization requirements from the treatability standpoint. An integrated approach was used by Rockwell International in selecting constituents and establishing detection limits for treatability and regulatory analyses of waste candidates for grouting (1). "Predictor" (or "group indicator") analyses and process knowledge also were included in their approach.

## THE ROLE OF PROCESS KNOWLEDGE

The RCRA allows process knowledge to be used for characterization where a well-defined process generated the waste. For RMW, process knowledge is of limited utility because low level wastes generally are not well-defined in composition and data for contaminated environmental samples or for wastes generated from the cleanup or other handling of high level wastes is usually nil. Historical records on old wastes (both low and high level) are usually sketchy at best. The main use of process knowledge in such cases appears to be in assisting characterization efforts by eliminating some constituents from analysis. For example, although many of the inactive nuclear waste storage tanks at the Oak Ridge National Laboratory (ORNL) lack useful source records, herbicides have been excluded from their RMW characterization because the total amount of herbicides used at the lab (estimated from laboratory purchase records) would not exceed the TCLP regulatory levels if disposed entirely in the tanks. This exclusion does not hold for all DOE sites.

## HOW CAN THE DETERMINATIONS BE MADE?

### SAMPLE COLLECTION

Collection of a representative sample of a waste is critical to generating valid characterization data. For low level wastes, sampling is conducted similarly to that for nonradioactive wastes but with the additional precautions and constraints of the ALARA principle (keeping radiation exposure and contamination of personnel and facilities as low as reasonably achievable). The main area requiring advances in strategy and technology for low level wastes is in sampling heterogeneous wastes. Drums or bags containing discrete items such as towels, gloves, bottles, and metal parts, or tanks with multiple phases (e.g., oily, aqueous, and sludge layers) are obviously heterogeneous. Other wastes

may lack obvious heterogeneity, such as single phases with composition gradients (as observed in an ORNL waste tank)(2). The current options for sampling heterogeneous wastes include segregation and subsampling, multiple sampling, or whole waste homogenizing or compositing and sampling. Heterogeneity also is a problem in sampling high level wastes (e.g., stratified wastes in the Hanford tanks). For situations where there are only a few heterogeneous samples to be analyzed, these approaches are feasible, but laborious. Where there are many samples to be collected and analyzed, current technology is not practical.

Physically collecting the sample, let alone even sufficient amounts or representative portions of samples from locations with restricted access, is a major technology area requiring development. The presence of high radiation fields (a major problem with high level waste tanks at some DOE sites such as the Hanford reservation) further complicates the problem. Volatile organics are a class of compounds particularly difficult to sample properly under such conditions. Radiation-hardened samplers with articulated arms are under development, but are critically needed now. Development of improved sampling equipment and their acceptance by regulatory agencies should be expedited.

## GENERAL ANALYTICAL CONSIDERATIONS

Although low level wastes are not simple to prepare and analyze, there are no unique "show stoppers" in their characterization. Regulatory sample preparation and determination methods such as those listed in the SW-846 manual (3) can be performed essentially without modification in radiochemical hoods, and the measurements can be conducted per the specified protocols with the instruments located in contamination zoned laboratories. The main operational difference from work with nonradioactive samples is the use of appropriate precautions in sample and extract handling and laboratory waste management to observe the ALARA principle and to minimize the spread of radioactive contamination. The major problem in the characterization of low level wastes will be the disposal of contaminated laboratory wastes. Often, secondary laboratory wastes generated in the preparation of RMW for analysis and in the clean-up of hoods after sample preparation can exceed the mass or volume of sample analyzed. This shows the need for the development and regulatory acceptance of "less waste intensive" sample preparation methods to reduce analytical costs.

**Sample Handling:** Low level samples can be contact-handled in radiochemical hoods. Higher activity wastes require handling and analysis with containment or shielding, which does limit sample operations and productivity. The permissible activities for radiochemical hood operations currently vary in magnitude as well as units of specification among the DOE laboratories. Generally, they range from ca. 0.1 to 45  $\mu\text{Ci}$  (microCuries) of activity and up to 1 g of total Pu in solution for alpha emitters, and dose rates of 10 to 500 mrem/hr at contact for beta/gamma emitters. Radiotoxicity and the complexity of the operation (and therefore the potential for a spill or other release of contamination) also are considered in health physics guidelines. For samples containing higher activities than permitted in radiochemical hoods, samples with alpha emitters are handled under containment in glove boxes to prevent the spread of highly radiotoxic nuclides whose radiation has little penetrating power. In contrast, a hot cell (or, at the least, sample shielding with lead bricks) is required for the much more penetrating gamma (and beta) emitters.

**Productivity:** Most any operation which can be performed on a bench or in a hood can be conducted in a glove box or hot cell. However, the methods and equipment must be adapted for the latter, highly skilled operators are required to safely perform the work, and productivity is reduced in the much more restricted working environment. With glove boxes, samples and reagents are bagged in

and prepared extracts and wastes are bagged out through sealed bag ports to prevent direct contact of the glove box atmosphere with the laboratory air. Sample manipulations are performed by reaching through heavy walled rubber gloves which severely restrict manual dexterity and freedom of motion. Sample introduction into hot cells is equally difficult and, in addition, the work is performed using master-slave remote manipulators which require a high degree of eye-hand coordination. Depending upon the complexity of the operation, productivity can be decreased (or, conversely, costs are increased) by factors of 2 to 5-fold for glove box operations and up to 10-fold for hot cell operations.

Holding Times: Another consideration is that regulatory holding times designed for environmental samples are very hard, if not impossible, to meet in some instances because of the delays caused by the additional sample characterization needed to define safe handling practices, and the reduced productivity from working under containment or shielding. The relevance of these holding times is highly questionable for RMW materials which in some cases have been in storage for decades. Reconsideration of this issue is needed.

## THE TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

Operationally, the TCLP (4,5) consists of two experimental procedures: leaching the wastes and analyzing the leachates for regulated constituents. For low level wastes which can be leached in a radiochemical hood, no major difficulties are anticipated in conducting the leaching. Cleaning the leaching equipment (especially the zero headspace extractor [ZHE]) will cause a waste disposal problem because of the generally messy nature of equipment cleanup operations and the radioactive contaminated lab wastes generated from the cleaning operations.

Because of the relatively large sample sizes (25 - 100g) and bulky, complex apparatus and equipment (e.g., the ZHE and tumbler) specified in the regulations, few laboratories have adapted the TCLP leaching procedures to glove box or hot cell operations for high level RMW. The nonvolatile leaching has been adapted to glove box and hot cell operations in only five DOE laboratories, with the main deviation from the EPA protocol being a reduction in scale. The Idaho National Engineering Laboratory (INEL) is the only laboratory conducting the full-scale nonvolatile leaching in a hot cell. The ZHE is much more complicated and difficult to adapt to glove box or hot cell use, but work in this area is underway. At least two companies are designing full-scale, lower cost ("disposable") ZHEs for hot cell use, and ORNL and INEL are designing ZHEs from alternate apparatus such as gas syringes. Reductions in TCLP scale to accommodate ALARA, limited sample availability, or more practical hot cell equipment for high level waste leaching must be developed and accepted by regulatory agencies. The recent draft of the joint EPA/NRC guidance on mixed waste testing (6) suggests a regulatory agency realization of this need. Until such equipment and procedures are developed and approved, the direct analysis option makes the most sense for RMW from both a scientific and a practical standpoint.

A final problem with the TCLP that is not specific to RMW is the interference of acetate with the analytical procedures. In particular, the acetate from the leaching fluids is carried into the semivolatile organics analysis (SVOA) extracts, where it seriously degrades the performance of the capillary GC-MS determinative method. It also consumes derivatizing agent in the preparation for herbicides determination by GC-ECD. The use of SW-846 GC methods 8040 (for phenols), a combination of 8121/8091/8081 (for hexachlorobenzene, dinitrotoluene, and the pesticides), a

modification of HPLC method 8123 (for herbicides), and 8260 (volatiles plus the remaining extractable organics) allow determination of all TCLP organics without interference from the acetate (7). The HPLC method is being further adapted to include all of the acidic extractable TCLP organics without any required sample preparation. Regulatory agency acceptance of HPLC methods for these compounds would permit faster and cheaper analyses with less laboratory waste and personnel exposure.

No difficulties are anticipated with the determination of the characteristics of corrosivity, flammability, or reactivity in low level RMW. Flammability determinations can be a problem when high level RMW samples require shielding or containment. INEL is developing a flash point apparatus with a pressure-sensing membrane to allow this determination to be conducted in a hot cell or glove box.

## ORGANIC CHEMICAL ANALYSIS

Sample Preparation: For low level RMW, current ultrasonic or soxhlet (solids) or separatory funnel or continuous extractor (aqueous liquids) SW-846 or Contract Laboratory Program methods can be used with little modification and careful handling and containment to minimize personnel exposure and prevent spread of contamination. However, considerable laboratory waste contaminated with radioactivity (therefore a low level waste and possibly RMW from toxic solvents or spikes) can be generated and will have to be disposed. With higher activity RMW, these problems are compounded with the difficulties of working in a glove box or hot cell, often with smaller sample amounts than specified in SW-846 protocols (which raises detection limits).

Several extraction technologies may be useful in low and high level RMW analysis, and expedited acceptance of them by regulatory agencies will help to alleviate these difficulties. Work at several laboratories (including EPA-sponsored studies) has demonstrated that a variety of analytes can be efficiently recovered from solids by supercritical fluid extraction (SFE). LANL recently has shown that carbon dioxide with a water modifier can efficiently extract many semivolatile organics (including phenols) from simulated RMW. The ability to extract relatively small masses of sample (in a glove box or hot cell, if need be) and analyze the extracts on-line via transfer lines to a GC-MS outside the box or cell greatly reduces lab waste generation, radiation exposure of staff, labor, and sample preparation/analysis time. For liquid samples, solid phase extraction using packed columns (as in some EPA 500 and 600 series methods), extraction membranes, and open plastic tubing (as at LANL) promise rapid and reproducible extractions with far less solvent usage and operator exposure than current solvent extraction methods. Both of these technologies can be conducted relatively easily under shielding or containment, and they could be readily automated to further improve productivity. Further development and optimization of these powerful tools, and their acceptance by regulatory agencies clearly should be a priority.

For higher level wastes, the presence of high concentrations of nitrite has caused problems in aqueous liquid sample preparation for the SVOA. Foaming during acidification and release of oxides of nitrogen, which creates artifacts by nitrating organic compounds (Westinghouse Hanford has observed nitration of at least two SVOA surrogate standard phenols), are some of the problems caused by nitrite (2). A means of selectively removing or lowering the nitrite, or of avoiding the need to acidify before extraction would be helpful in improving sample handling and reducing potential artifacts. Other problems encountered with high level wastes include high alkalinity of sludges and

also unidentified interferences in Hanford tank wastes which quickly degrade VOA and SVOA GC-MS performance. The latter is not removed by CLP purification methods. Clearly, there are major technology needs for clean-up of high level samples.

The automation of sample preparation and analysis will increase laboratory productivity and reduce operator exposure to radioactive samples. In the near term, only segments of procedures are automated (e.g., VOA purge and trap or gel permeation chromatography and fraction concentration), but in the long term, efforts such as the DOE Contaminant Analysis Automation Program should provide the methods and equipment. This effort should be continued.

Determination of Regulated Organic Compounds: Low level RMW do not present major problems in the organics determination steps, and regulatory agency protocols can be followed as prescribed. The instruments preferably should be located in contamination zoned laboratories to contain any radionuclides which may be carried over into sample extracts. The concentrated extracts are usually decontaminated by several orders of magnitude and thus could be analyzed in nonzoned laboratories (if the extracts are analyzed for gross alpha and gross beta activities to confirm decontamination), but it cannot always be assumed that complexing agents will not be present in the samples. Complexing agents would facilitate the transfer of radionuclides into the SVOA or PCB/pesticide extracts. The volatile organics analysis (VOA) must be conducted in a zoned laboratory because the sample preparation (purge and trap) is best carried out on-line with the GC-MS.

For high level RMW, sample amounts are often limited by compliance with the ALARA principle or by sampling constraints. This increases analysis reporting limits. Means of increasing the fraction of the SVOA extract (for example) injected into the GC-MS would offset the smaller sample sizes. Application of the SVOA extract to an adsorbent, evaporation of the solvent and thermal desorption into the GC-MS is being evaluated at ORNL. On-line SFC-GC-MS as being developed at LANL also may overcome this problem. Use of more sensitive MS instrumentation (i.e., the ion trap mass spectrometer) also should be examined. Validation of these methodologies and regulatory agency acceptance of their use is needed. Alternatively, it would be more reasonable for regulatory agencies to relax organic and inorganic compound regulatory limits for samples whose primary hazard is a high level of radioactivity.

DOE-Unique Organics: A major area requiring methods development is the determination of compounds which are unique to DOE RMW in their complexity and concentrations, i.e., chelators, extractants, decontamination agents, their radiolytic/chemical/thermal decomposition products, and chelated radionuclides. The organic matter suggested to be present in some DOE RMW by total organic carbon measurements is rarely accounted for by regulatory organics determinations (2,8), and in some cases is accounted for by these compounds. Although these compounds are not currently regulated, they are important for their influences upon waste treatment (e.g., grouting behavior) and mobility of radionuclides (e.g., environmental transport of radionuclides from leaks of radioactive wastes). They are not determined using current regulatory GC-MS methods because of their high polarity, water solubility, and thermal instability. At the present, their analysis is performed by an incompletely evaluated method including waste drying and derivatization followed by GC or GC-MS. High performance liquid chromatography and capillary zone electrophoresis interfaced with mass spectrometry appear to be good candidates for identifying and measuring DOE-unique compounds. The very small sample volume requirements (microliter volumes for nanoliter injections), high efficiency (up to one million plates per meter of column), and natural applicability to polar, water-soluble, ionic species make the latter particularly attractive. Development and validation of methods

will be needed for these specialized constituents.

## INORGANIC AND RADIOCHEMICAL ANALYSES

Sample Preparation Methods: Low level RMW sample preparation (where radiochemical hood containment is sufficient) for inorganic and radiochemical analyses presents few additional problems over nonradioactive samples. Analyses of low level wastes are readily feasible with current technologies, although there are areas where methodology improvement is desirable to facilitate their turn-around.

One problem unique to RMW is the spectral interferences caused by the presence of uranium, thorium, plutonium, and other actinides in sample digests analyzed for metals by inductively coupled plasma - atomic emission spectrometry (ICP-AES). The concentrations of actinides in the sample digests can be reduced using tri-n-octylphosphine oxide extraction or extraction chromatography on the unique EiChroM TRU•Spec chelator columns (9) developed at Argonne National Laboratory (ANL). Evaluation of these preparation methods is nearly complete at ORNL. Regulatory acceptance of these clean-up methods would improve ICP-AES performance with samples heavily contaminated with actinides.

A second problem unique to RMW, but limited to high level wastes, is the requirement to conduct the metals analyses of sample digests under containment or shielding. Many DOE laboratories have placed the ICP-AES, ICP-MS, or flame-AA burner, and the GF-AA or electrothermal vaporization ICP-MS furnace in glove boxes, and INEL has the only x-ray fluorescence source in a hot cell. It would greatly facilitate metals analyses of high level samples if methods were available to reduce the radionuclide content of the digests. Transuranics can be removed from the sample digests with essentially no effect upon RCRA and EPA Target Analyte List metals using TOPO extraction or the EiChroM columns, but a major technology development need is for methods to isolate or remove beta- and gamma-emitting radionuclides from regulated metals.

Sample dissolution/digestion preparation methods improvements would benefit both metals and radiochemical analyses. These include high pressure sealed tube dissolution methods, improved microwave dissolution methods, and methods for obtaining clean digests of organic matrices. Removal of salts from the digests also is highly desirable. The high solids content they contribute to digests interferes with metals analyses and especially gross alpha activity determinations. They also interfere with alpha spectrometry unless lengthy separations are performed. One promising method, the isolation of alpha emitters by extraction chromatography with the EiChroM TRU•Spec columns, should be validated for RMW and accepted by regulatory agencies.

With high level RMW samples, high concentrations of alkali metal nitrates in the sample digests cause corrosion of the graphite furnace in the GF-AA analysis of metals, and probably also of the electrothermal vaporization furnace of ICP-MS. A means of separating the heavy metals from nitrates would reduce graphite furnace corrosion and maintenance. Interferences in As and Se determinations by the hydride method have been observed at Westinghouse Hanford in samples with high Bi concentrations - possibly indicating the need to remove Bi (which also can consume hydride) from the digest.

The colorimetric determinations of cyanide and sulfide are subject to interferences from co-distilled compounds such as oxides of nitrogen and elemental chlorine and iodine. Methods to reduce the concentrations of these species would increase the reliability of colorimetric anion analyses. Preferably, however, regulatory agencies should accept ion chromatography for cyanide and sulfide analysis determinations in RMW because it is not subject to these interferences. Several laboratories are using this method successfully.

Inorganic and Radionuclide Determinations: No major problems exist for the inorganic and radiochemical determinations of low level wastes. However, laboratory operations would be considerably facilitated by more rapid, high sample load methods to measure gross alpha and gross beta activities. This would speed up sample turn-around because these are generally the first analyses conducted upon sample receipt in the laboratory and they are also usually the highest sample load. Automation of these determinations is called for. Sample preparation and transfer to counters (which already have automated sample changers) is a good candidate for robotics, and the prognosis for a near-term payoff is very good.

Both radiochemical and inorganic analyses of wastes would considerably benefit from the evaluation and regulatory agency acceptance of ICP-mass spectrometry (ICP-MS), particularly for multielement scanning and for the determination of long-lived, low specific activity radionuclides. Traditional sample preparation and long counting times lengthen turn-around times for determinations of radionuclides such as  $^{99}\text{Tc}$ ,  $^{237}\text{Np}$ , and  $^{129}\text{I}$ . Among the attractive features of ICP-MS are multielement capability, high sensitivity, small sample requirements (at least with electrothermal vaporization), direct analysis of solids and slurries with laser ablation or electrothermal vaporization, and its ease of automation. Also, RCRA metals probably could be determined with high accuracy using isotope dilution techniques. ICP-MS analytical technology should be a high priority for validation and acceptance by regulatory agencies.

For higher level RMW, determination methods for total fissile material by ICP-MS or by delayed neutron counting after neutron activation need to be evaluated and accepted.

## FIELD ANALYTICAL METHODS

Certainly, RMW characterization would benefit from the development, validation, and acceptance of methods for rapid sample screening and analysis in the field. These methods would eliminate sending samples with little or no organics, metals, or radionuclide content back to the laboratory for expensive analyses. Work is underway at ORNL and LANL with ion trap mass spectrometry, immunoassay, and other fieldable techniques for organics determinations. Field methods for rapidly screening samples in the field for selected metals and gross alpha and beta activity would be a useful adjunct to the organics screening methods. The latter also could reduce the time for laboratory radiochemical analyses needed for health physics protection. Examples of important work in this area include the method adaptation work which will be underway shortly at LANL for field gross alpha and gross beta measurements using the Protean system, and gross gamma and  $^3\text{H}$  measurements by other methods. A field test of a portable x-ray fluorescence screen for selected metals (10) was supported by the Army. The Nevada Test Site also is supporting work adapting the Frisch grid detector for alpha spectroscopy in the field.



## CONCLUSIONS

The main conclusions from the scientific perspective are that, although there are difficulties working with low level and very low level RMW, there are no major problems or "show stoppers" preventing their characterization in appropriately equipped and licensed commercial laboratories. The main problem probably will be the disposal of secondary laboratory wastes generated in the analysis of low level wastes. However, validation and regulatory agency acceptance of new technologies or adaptations of current technologies would greatly facilitate the characterization of the large sample load expected for DOE. Development and validation of field analytical methods also will be important in minimizing the costs of sample shipment and laboratory analyses and may help in meeting currently very restrictive holding time requirements. Higher level RMW requiring more stringent containment or shielding pose much greater characterization problems in their own right.

Two roles will be important in successfully meeting this challenge. DOE and other agencies must identify and adapt new laboratory and field analytical technologies, evaluate their performance, and transfer these technologies among the DOE sites and to the commercial sector. The EPA must facilitate the means of gaining regulatory agency acceptance of new and adapted methods. It also must officially recognize the limited applicability of the TCLP to RMW, and the high desirability of minimizing the characterization required of highly radioactive wastes.

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