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**WASTE MINIMIZATION THROUGH HIGH-PRESSURE MICROWAVE
DIGESTION OF SOILS FOR GROSS α/β ANALYSES**

by

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April 1995

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ABSTRACT

As a result of the U.S. Department of Energy's (DOE) environmental restoration and waste management activities, laboratories receive numerous analytical requests for gross α/β analyses. Traditional sample preparation methods for gross α/β analysis of environmental and mixed waste samples require repetitive leaching, which is time consuming and generates large volumes of secondary wastes. An alternative to leaching is microwave digestion. In the past, microwave technology has had limited application in the radiochemical laboratory because of restrictions on sample size resulting from vessel pressure limitations. However, new microwave vessel designs allow for pressures on the order of 11 MPa (1500 psi). A procedure is described in which microwave digestion is used to prepare environmental soil samples for gross α/β analysis. Results indicate that the described procedure meets performance requirements for several soil types and is equivalent to traditional digestion techniques. No statistical differences at the 95% confidence interval exist between the measurement on samples prepared from the hot plate and microwave digestion procedures for those soils tested. Moreover, microwave digestion allows samples to be prepared in a fraction of the time with significantly less acid and with lower potential of cross-contamination. In comparison to the traditional hot plate method, the waste volumes required for the microwave procedure are a factor of 10 lower, while the analyst time for sample processing is at least a factor of three lower.

I. INTRODUCTION

Gross α/β analysis is a routine radioanalytical procedure. This analysis is frequently requested as a screening procedure prior to initiating organic and inorganic analyses and, therefore, becomes a rate-determining step in these analytical protocols. The traditional sample preparation procedure for gross α/β is based upon repetitive leaching in open vessels on hot plates. This digestion method adequately extracts contaminant activity but is not a total dissolution procedure. These hot plate procedures are time-consuming and contribute to the long turnaround times associated with gross α/β analyses. Moreover, these hot plate methods generate significant quantities of secondary wastes and fume hood emissions.

The use of microwave energy as a heat source for open-vessel sample digestion was first described over 20 years ago.^{1,2} However, hot plate digestion remained the method of choice until the late 1980s when closed-vessels systems revitalized interest in microwave sample digestion procedures.³⁻⁵ These closed vessels allowed for higher pressures to be used, resulted in temperatures greater than the acid's boiling point, and subsequently accelerated the digestion process.⁶ In 1990, the Environmental Protection Agency (EPA) in its Contract Laboratory Program (CLP)⁷ approved closed-vessel microwave digestion of soils for inorganics analysis.

These early closed-vessel systems were only applicable to small sample sizes because of vessel pressure limitations. As a result, microwave dissolution techniques were not practical for many radiochemical procedures where larger sample sizes are needed to achieve required detection limits. However, recent advances in vessel design allow for pressures on the order of 11 MPa (1500 psi).⁸ These high-pressure, closed-vessel systems allow for larger sample sizes, shorter processing times, and reliable sample digestion, while dramatically reducing secondary wastes and acid fume emissions. Additionally, vessel composition has been improved. Noltner

et al. have demonstrated that Tetrafluorometoxil™ (TFM) vessels exhibit significantly lower memory than vessels produced from the more traditional Perfluoroalcoholoxil™ (PFA).⁹ This lower memory results in lower blank values, and thereby lower limits of detections, a clear advantage for environmental laboratories. The TFM vessels are also able to withstand higher pressures and temperatures than the PFA vessels.

A procedure is described in which a high-pressure microwave digestion system is used to prepare environmental soil samples for gross α/β analysis. Both qualitative and quantitative aspects of the procedure are addressed. Results indicate that the described procedure meets performance requirements for several soil types, is equivalent to traditional digestion techniques, and allows samples to be prepared in a fraction of the time of hot plate methods, with significantly less acid and less opportunity for cross-contamination.

II. EXPERIMENTAL

A. Apparatus

A Milestone MLS-1200 MEGA Microwave Digestion System with the newly developed MDR (Microwave Digestion Rotor) technology was used in this study. A polypropylene rotor holds six high-pressure TFMTTM vessels, which are designed to tolerate exposure to aggressive acids at elevated pressures. The pressure inside the vessels may reach 11 MPa before any of nine safety control features are implemented. The power of the magnetron is 1200 W, with 1000 W delivered inside the working chamber. The microwave emission is "unpulsed" in the 250 W mode, but "pulsed" at other wattages. The "unpulsed" 250 W mode assures controlled oxidation of organics. An Exhaust Module 30 was used for fume ventilation into a chemical hood. The microwave and accessories were purchased through Buck Scientific (East Norwalk, CT).

A Tennelec Low Background 5100 Series II proportional counter with an automated programmable sample changer was used for the gross α/β determinations. The detector was a 5.72-cm diameter chamber with a MylarTM window. Typical background counting rates were <0.10 and <1.0 counts per minute (cpm) for α and β determinations, respectively.

B. Materials

A variety of soil types of potential interest to DOE were selected for testing, including a National Institute of Standards and Technology (NIST) reference soil from the Rocky Flats Plant (SRM 4353), several environmental soils (labeled Mound, Fernald, ANL-East, ANL-West, and RFP) and several contaminated soils from selected DOE sites (labeled Con 1 through Con 10). All reagents were American Chemical Society reagent grade and were utilized as received.

C. Hot Plate Digestion Procedure

Soils were dried at 110°C for at least 48 hours (or until constant weight was obtained) and were subsequently homogenized. Two grams or less of sample was employed in each analysis. The sample was leached with 50 mL of 6 M HCl at near boiling for 90 min. The solution was allowed to cool, and the supernatant was separated by centrifugation. The sample was returned to the original beaker and leached with 50 mL of 6 M HCl and two 50 mL volumes of 3 M HCl-4 M HNO₃. The leachates were combined and evaporated to near dryness. The residue was dissolved in 8 M HNO₃ and evaporated. The final residue was dissolved in a small quantity of 1 M HNO₃. This solution was quantitatively transferred to a 25 mL volumetric flask and diluted to the desired volume with deionized water. A known aliquot was evaporated onto a pre-weighed 5.1-cm diameter stainless steel planchet. The planchet was dried to constant weight and counted for gross α/β activity on a 2π proportional counter.

D. Microwave Digestion Procedure

The microwave digestion protocol is summarized in Table 1. Soils were dried at 110°C for at least 48 hours (or until constant weight was obtained) and were homogenized. Two grams or less of sample was used in each analysis. The sample was transferred to a clean TFM™ vessel and treated with 5 mL concentrated HNO₃, 2.5 mL concentrated HCl, and 0.5 mL 30% H₂O₂. The vessel was capped and placed into the microwave rotor apparatus. The sample was irradiated and subsequently cooled in a cold water bath. The sample was removed from the vessel, and the leachate separated by centrifugation. The residue was washed with 5 mL of 8 M HNO₃ and centrifuged. The supernatants were combined and diluted to 25 mL with deionized water. A counting planchet was prepared, as described above (Sec. II C).

Table 1. Microwave Digestion Protocol

| Step | Wattage | Time (minutes) |
|------|---------|-------------------|
| 1 | 250 | 2 |
| 2 | 0 | 2 |
| 3 | 250 | 6 |
| 4 | 400 | 5 |
| 5 | 0 | 1 |
| 6 | 600 | 5 |
| 7 | 0 | 5 |

III. RESULTS AND DISCUSSION

Certified soil standard reference materials are not available for gross α/β analyses.

However, NIST SRM 4353 is routinely used in our laboratory to monitor the reproducibility of such analyses. "Expected" values of 14 ± 3 pCi/g α and 12 ± 2 pCi/g β have been determined from vigorous, repetitive leachings of 15 aliquots of this soil. The average sample size was 12 g.

Table 2 lists gross α/β results for typical environmental samples prepared by hot plate and microwave methods. The reported α and β activity levels are consistent with the activity levels expected from naturally occurring radionuclides (U, Th, and Ra isotopes). The large uncertainties reported are primarily the result of poor counting statistics from these low activity samples. The values obtained for NIST SRM 4353 agree favorably with the "expected" results. Table 3 gives gross α/β results from a series of contaminated soils prepared by the hot plate and microwave methods. Paired, two-tailed *t*-tests indicate no significant differences at the 95% confidence interval in the results from the hot plate and microwave digestion methods for the environmental and contaminated soils.

Table 2. Gross α/β Analyses by Hot Plate and Microwave Digestion Methods: Environmental Soils

| Gross α , pCi/g $\pm 2\sigma^a$ | | | | |
|--|------------------|--------------------|------------------|--------------------|
| Sample Type | Hot Plate Method | Number of Analyses | Microwave Method | Number of Analyses |
| NIST SRM 4353 ^b | 15 \pm 5 | 6 | 18 \pm 5 | 14 |
| Fernald | 9 \pm 7 | 2 | 9 \pm 5 | 5 |
| Mound | 22 \pm 9 | 4 | 13 \pm 7 | 5 |
| ANL-East | 11 \pm 6 | 4 | 12 \pm 5 | 7 |
| ANL-West | 14 \pm 9 | 2 | 11 \pm 5 | 4 |
| RFP | 13 \pm 6 | 2 | 11 \pm 4 | 4 |

| Gross β , pCi/g $\pm 2\sigma^a$ | | | | |
|---------------------------------------|------------------|--------------------|------------------|--------------------|
| Sample Type | Hot Plate Method | Number of Analyses | Microwave Method | Number of Analyses |
| NIST SRM 4353 ^c | 14 \pm 4 | 6 | 11 \pm 3 | 14 |
| Fernald | <6 | 2 | 10 \pm 3 | 3 |
| Mound | 16 \pm 6 | 4 | 19 \pm 4 | 5 |
| ANL-East | 12 \pm 5 | 4 | 16 \pm 3 | 7 |
| ANL-West | 14 \pm 6 | 2 | 10 \pm 2 | 4 |
| RFP | 7 \pm 6 | 2 | 6 \pm 2 | 4 |

^aMean and standard deviation, where σ is based upon propagation of counting statistical error.

^b"Expected" value (α) = 14 \pm 3 pCi/g.

^c"Expected" value (β) = 12 \pm 2 pCi/g.

Table 3. Gross α/β Analyses by Hot Plate and Microwave Digestion Methods: Contaminated Soils

| Gross α , pCi/g $\pm 2\sigma^a$ | | |
|--|------------------|------------------|
| Sample ID | Hot Plate Method | Microwave Method |
| CON 1 | 320 \pm 34 | 354 \pm 35 |
| CON 2 | 174 \pm 26 | 191 \pm 26 |
| CON 2 (Duplicate) | -- | 220 \pm 30 |
| CON 3 | 183 \pm 26 | 202 \pm 27 |
| CON 4 | 436 \pm 38 | 384 \pm 41 |
| CON 5 | 150 \pm 25 | 440 \pm 40 |
| CON 6 | 50 \pm 15 | 30 \pm 10 |
| CON 7 | 485 \pm 45 | 375 \pm 45 |
| CON 8 | 180 \pm 25 | 153 \pm 22 |
| CON 9 | 530 \pm 40 | 380 \pm 40 |
| CON 10 | 320 \pm 30 | 560 \pm 45 |
| CON 10 (Duplicate) | 430 \pm 40 | 480 \pm 40 |
| Gross β , pCi/g $\pm 2\sigma^a$ | | |
| Sample ID | Hot Plate Method | Microwave Method |
| CON 1 | 31 \pm 7 | 32 \pm 7 |
| CON 2 | 22 \pm 7 | 23 \pm 7 |
| CON 2 (Duplicate) | -- | 24 \pm 6 |
| CON 3 | 27 \pm 8 | 38 \pm 8 |
| CON 4 | 15 \pm 4 | 18 \pm 6 |
| CON 5 | 13 \pm 5 | 29 \pm 7 |
| CON 6 | 9 \pm 5 | 13 \pm 5 |
| CON 7 | 30 \pm 8 | 32 \pm 13 |
| CON 8 | 25 \pm 8 | 18 \pm 7 |
| CON 9 | 34 \pm 6 | 23 \pm 7 |
| CON 10 | 30 \pm 6 | 34 \pm 7 |
| CON 10 (Duplicate) | 38 \pm 8 | 30 \pm 7 |

^aUncertainty based upon propagation of counting statistical error.

Data used to evaluate the precision of the methods are listed in Table 4. Five aliquots of NIST SRM 4353 were analyzed for gross α/β using each sample digestion method. The reproducibility for each method was good, and the values obtained agreed favorably with the "expected" values.

Table 4. Reproducibility of the Hot Plate and Microwave Digestion Methods for Gross α/β Determinations

| Sample Type | Gross α , pCi/g $\pm 2\sigma^a$ | | Gross β , pCi/g $\pm 2\sigma^a$ | |
|---------------|--|------------------|---------------------------------------|------------------|
| | Hot Plate Method | Microwave Method | Hot Plate Method | Microwave Method |
| NIST SRM 4353 | 16 \pm 5 | 16 \pm 5 | 11 \pm 4 | 12 \pm 4 |
| | 15 \pm 5 | 24 \pm 5 | 8 \pm 4 | 12 \pm 4 |
| | 9 \pm 3 | 14 \pm 4 | 8 \pm 3 | 10 \pm 4 |
| | 20 \pm 5 | 22 \pm 5 | 9 \pm 4 | 11 \pm 3 |
| | 14 \pm 4 | 18 \pm 5 | 13 \pm 4 | 17 \pm 4 |
| Avg. = | 15 \pm 5 | 19 \pm 5 | 10 \pm 4 | 12 \pm 4 |
| RSD = | 4.0 | 4.1 | 2.2 | 2.7 |

^aUncertainty is based upon propagation of counting statistical error.

An evaluation of the data in Tables 2-4 indicates that the gross α determination exhibits more variation between digestion methods and between duplicates than does gross β . These variabilities may be a result of sample inhomogeneity and/or self-absorption effects caused by solids on the counting planchets. Because sample sizes of only one to two grams were used, it would not be unreasonable to expect inhomogeneities. This is especially true for the

contaminated samples, since "hot spots" are not uncommon in these types of samples. Although self-absorption curves are used to correct for changes in counting efficiencies due to solids on the counting planchets, it is difficult to exactly reproduce the counting geometry with every sample. This phenomenon attributes to the imprecision in these analyses.

Other sample preparation variables were also evaluated. A small set of samples was ashed at 510°C to destroy organics prior to digestion. The gross α/β results from these ashed samples were equivalent to results obtained from non-ashed aliquots. Additionally, the effect of HF acid in the microwave digestion protocol was examined. One milliliter of concentrated HF was added to the mixed acid-peroxide solution described previously (Sec. II. D) and gross α/β was subsequently determined. Results for gross α were equivalent for the digestion solutions with and without HF. However, for several samples, β results were significantly higher for those aliquots which were treated with HF. This finding indicates that HF may help to solubilize certain species not affected by the traditional acid mixtures.

Typical blank values for the microwave digestion procedure are reported in Table 5. Blank samples consist of all reagents processed through the sample preparation and analysis procedures. Blank determinations were also performed following analyses of the contaminated samples listed in Table 3. The TFM™ vessels appeared to exhibit no memory, as predicted by Noltner et al.⁹

Table 5. Typical Blank Values

| Microwave Environmental Samples | | | |
|---|------------------------------------|--|------------------------|
| Gross α (pCi/g $\pm 2\sigma$) | MDA^a (pCi/g) | Gross β (pCi/g $\pm 2\sigma$) | MDA (pCi/g) |
| 0.9 \pm 0.9 | 1.3 | -0.7 \pm 1.4 | 2.8 |
| 0.0 \pm 0.7 | 1.4 | 2.9 \pm 1.7 | 2.8 |
| 0.6 \pm 0.9 | 1.4 | 0.0 \pm 1.5 | 2.8 |
| 0.1 \pm 0.7 | 1.3 | 0.2 \pm 1.5 | 2.8 |
| 0.1 \pm 0.8 | 1.4 | 1.2 \pm 1.6 | 2.8 |
| Microwave Contaminated Samples | | | |
| Gross α (pCi/g $\pm 2\sigma$) | MDA (pCi/g) | Gross β (pCi/g $\pm 2\sigma$) | MDA (pCi/g) |
| 0.9 \pm 1.7 | 2.8 | -3.6 \pm 3.5 | 8.1 |
| 2.2 \pm 2.2 | 2.8 | 2.0 \pm 4.1 | 8.1 |
| 0.3 \pm 1.4 | 2.8 | -5.3 \pm 3.2 | 8.1 |
| 1.2 \pm 1.9 | 2.8 | -4.6 \pm 3.3 | 8.1 |
| 1.2 \pm 1.8 | 2.8 | 0.1 \pm 3.9 | 8.1 |
| 0.6 \pm 1.6 | 2.8 | -2.3 \pm 3.6 | 8.1 |
| Hot Plate Environmental Samples | | | |
| Gross α (pCi/g $\pm 2\sigma$) | MDA (pCi/g) | Gross β (pCi/g $\pm 2\sigma$) | MDA (pCi/g) |
| -0.6 \pm 1.1 | 2.5 | -1.4 \pm 3.6 | 6.1 |
| 1.0 \pm 2.1 | 3.7 | 1.0 \pm 4.2 | 8.5 |
| 0.3 \pm 1.2 | 2.0 | 1.2 \pm 3.1 | 5.7 |

^aMinimum detectable activity (MDA) was calculated as described in Ref. 10. This value is a function of instrument background and counting times. Therefore, shorter counting times produce higher MDA values, as reflected in the above data.

In conclusion, microwave digestion using a high-pressure, closed-vessel system clearly produces comparable analytical data to traditional hot plate methods for gross α/β

determinations of soils. Moreover, microwave digestion offers significant waste minimization advantages. These waste minimization figures of merit are described in Table 6. As shown, microwave digestion dramatically reduces sample preparation time, secondary acid wastes, and consumable supplies. Analyses are completed faster, with fewer wastes. Therefore, microwave digestion is recommended for gross α/β analyses.

Table 6. Waste Minimization Figures of Merit

| | Traditional Hot Plate Method | High Pressure Microwave Method |
|--------------------------------------|---|---|
| Sample Preparation Time ^a | 60 min | 20 min |
| Minimum Turnaround Time ^b | 3 days | 1 day |
| Secondary Acid Wastes ^a | 225 mL | 25 mL |
| Consumable Supplies ^a | 3 Beakers 1 Volumetric Flask 2 Centrifuge Tubes | 1 Volumetric Flask 1 Centrifuge Tube |

^aPer sample
^bDependent upon batch size

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