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Wastewater Standards and Extraction Chemistry in Validation of Microwave-Assisted EPA Method 3015A

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The difficulties associated with the control and transfer of environmental leach methods are discussed. Optimized EPA Method 3015A, a microwave-assisted leach of wastewater and drinking water matrices and aqueous extracts, is evaluated. The option to add HCl in addition to HNO₃ provides better complexation and recovery of certain metals that are regulated by the Resource Conservation and Recovery Act (RCRA) than the original HNO₃-only Method 3015. Also discussed is the preparation and appropriate use of "simulated" wastewater standards. Standard reference materials for a wastewater matrix are unavailable, and this novel approach provides NIST-traceability of results for the first time on this matrix type. Leach concentrations from these "simulated" standards were determined using both the 5 mL HNO₃ and the 4 mL HNO₃ and 1 mL HCl leach options of new Method 3015A. Validation of the new mixed-acid option of Method 3015A has been provided by evaluating its performance on the 23 elements for which original Method 3015 was validated. In addition, validation is provided for boron, mercury, and strontium, elements that were not validated in original Method 3015. Method 3015A has been developed into a method capable of evaluating 26 elements in a single, efficient, 20-min procedure.

Introduction

Leach methods pose unique quantification problems when comparing analyte concentrations. In contrast to total decomposition methods, which give absolute quantities of analyte levels, leach methods provide only relative concentrations based on the procedure used and conditions achieved. Elemental leach methods are acid extractions which are meant to provide a measure of analyte solubilization under "worst-case scenario" environmental conditions. Leach results are highly dependent on acid chemistry and extraction conditions. Because no absolute answer is provided by leach methods, the precision of the results is used as an expression of the accuracy. The dependence of leachable analyte concentrations on extraction conditions (1) makes the reproducible control and transfer of leach methods much more difficult than for total decomposition methods. Microwave digestion with temperature feedback control has demonstrated more precise temperature control than is possible on a hot plate and is more appropriate for

TABLE 1. Comparison of EPA SW-846 Leach Methods for Aqueous Samples

	EPA Method 3015	EPA Method 3015A
matrices	aqueous samples and extracts	aqueous samples and extracts drinking water
reagents	45 mL aqueous sample 5 mL of HNO ₃	45 mL aqueous sample 5 mL of HNO ₃ or 4 mL of HNO ₃ + 1 mL of HCl
heating profile	heat to 160 °C in 10 min slow rise to 170 °C in next 10 min	heat to 170 ± 5 °C in 10 min hold 170 ± 5 °C for 10 min
analytes	23 elements (not validated for B, Hg, Sr)	26 elements (B, Hg, Sr added)

reproducible control and transfer of environmental leach methods.

The Environmental Protection Agency's SW-846 manual of methods includes one microwave-assisted acid leach method for the preparation of aqueous samples and extracts for subsequent elemental analysis. This method, EPA Method 3015, is a closed vessel microwave leach valid for the analysis of 23 elements. The advantages of using closed vessels and microwave heating have been discussed (2, 3). The added advantage of reproducible temperature control, typically ±2.5 °C, makes microwave heating the method of choice for optimum control and transfer of standard methods among analysts, laboratories, and even foreign countries, compared to the wide variability and subjectivity associated with hot-plate methods. Method 3015 has been a promulgated sample preparation method for several years (4, 5). Originally, this method was limited by the EPA to the exclusive use of HNO₃. A HNO₃–HCl mixed-acid leach was avoided to reduce the potential for interferences from HCl and its resulting species on some analytical detection methods such as graphite furnace atomic absorption spectroscopy (GFAAS) and inductively coupled plasma mass spectrometry (ICP-MS). However, by restricting the reagents used in leach methods to HNO₃, limits are placed on the chemistry of the method (2). It has been shown that some analytes are unstable either in the oxidizing environment of an HNO₃-only leach (6–11) or in environments containing trace amounts of chlorides (12). Solution stabilities and recoveries of these analytes can be increased by raising the concentration of chlorides in the leach solution. To enhance recoveries of certain analytes through complexation by HCl, the new Method 3015A, which provides the option to perform either a HNO₃-only or a mixed-acid HNO₃–HCl leach, was proposed for the leaching of aqueous samples and extracts. Table 1 compares the protocols of original Method 3015 and the recently updated Method 3015A.

This work describes the importance of reproducible control and transfer of leach methods and evaluates the performance of both the HNO₃–HCl mixed-acid option and the HNO₃-only option of the new EPA Method 3015A. One goal was to provide validation of the new method using several standards consisting of various matrix types. Concurrent with this validation is discussion of the preparation and use of "simulated" wastewater standards consisting of standard reference materials (SRMs) mixed with 18 MΩ·cm water. The absence of a suitable wastewater standard

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reference material for these validation studies prompted the development and evaluation of these simulated SRM-wastewater standards. A second goal was to provide method validation for the elements boron, mercury, and strontium, three analytes which were never validated for the original Method 3015. This study shows that the mixed-acid option provides an appropriate chemistry for the recovery of 26 elements using a single, 20-min microwave leach.

Experimental Section

Microwave leaches were performed using the MLS 1200-Mega with the Microwave Digestion Rotor MDR-300/10 from Milestone USA (Monroe, CT) and the MDS-2100 with HDV (heavy duty vessels) from CEM Corp. (Matthews, NC). Concentrated acids (ACS reagent grade) were purchased (Fisher Scientific, Pittsburgh, PA) and were purified by sub-boiling distillation using a DuoPUR quartz still (Milestone USA, Monroe, CT) prior to use. The SRMs used in this study, SRM 2704 (Buffalo River Sediment), SRM 4355 (Peruvian Soil), SRM 1084a (Wear-Metals in Lubricating Oil), and SRM 1634c (Trace Metals in Fuel Oil), were purchased from the National Institute of Standards and Technology (NIST) in Gaithersburg, MD. These SRMs were also used in the study which validated EPA Method 3051A (2, 13) and the round-robin study validating EPA Method 3051 against Method 3050B (14, 15). Therefore, an abundance of data exists for comparison and validation of updated Method 3015A. To minimize the potential for cross-contamination, all microwave vessels and labware were cleaned using either hot or room-temperature acid cleaning procedures between uses. All procedures prior to and following microwave leaching were performed in a class 100 clean laboratory (3).

Prior to validation of Method 3015A, an appropriate standard material had to be developed. At the time this method was under development, no SRMs representative of a wastewater matrix were available from NIST. Typically, a wastewater matrix is composed of an aqueous portion along with some percentage of an associated solid portion, which is usually suspended matter and some dissolved fraction. This composition increases the complexity associated with quantifying leach results, which are meant to show the quantities of analyte that may become available from the entire matrix under the worst-case scenario conditions simulated by concentrated nitric (and hydrochloric) acids. However, when the solid component of the wastewater is a soil, sediment, or similar matrix that is susceptible to "environmental" reagents, elements may become available from both the solid portion and the aqueous portion. Therefore, some way of quantifying the leach recoveries from each component of the wastewater against a certified standard was needed. For this reason, simulated wastewater standards were created using solid SRMs and ultrapure water.

These simulated standards were prepared by mixing an appropriate amount (typically around 400 mg) of the solid SRM with 45 mL of 18 M Ω ·cm water in each microwave vessel. Each standard was prepared individually rather than preparing a large batch of bulk wastewater standard and using aliquots of that sample. Subsampling from a slurry may be appropriate in the preparation of a new standard but not in the use of these already-certified standards because of sample heterogeneity issues. Subsampling would lead to possible alteration of the size and content fractions of the certified material and lead to questionable transfer of the SRM. The degree of heterogeneity of solid SRMs is well-established and is reported on the certificate of analysis. By using a mass greater than the minimum certified homogeneous amount of SRM (approximately 250 mg) in each vessel, a representative sample that conforms to the certificate of analysis was produced in each case. Therefore, the traceability of the total concentration in the sample is maintained. When

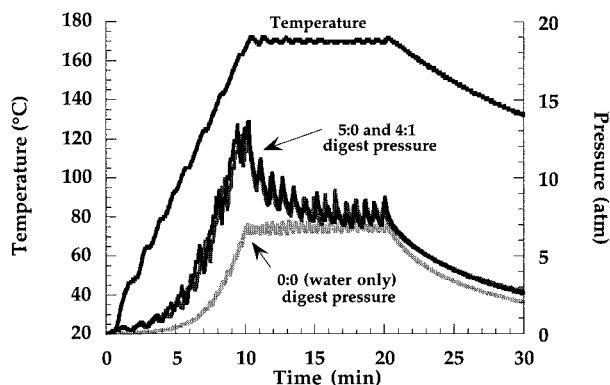


FIGURE 1. Typical temperature and pressure profile for the leaching of a simulated wastewater sample (~400 mg of SRM 2704 in 45 mL of 18 M Ω ·cm H₂O) using both digest options of EPA Method 3015A (13).

attempting to subsample from a larger quantity of a wastewater (or a similar "slurry-type" of matrix), it would be inherently more difficult to guarantee that each aliquot contained enough particles to ensure that heterogeneity problems are minimized. Minimizing heterogeneity problems is especially important when attempting to statistically validate the new method.

Wastewaters of three matrix types were created: a sediment-wastewater (using SRM 2704), a soil-wastewater (SRM 4355), and an oil-wastewater (SRM 1084a). These samples were leached using both reagent options of Method 3015A, according to method protocol (Table 1). After appropriate filtration and sample dilution, elemental analysis was performed using either flame atomic absorption spectroscopy (FAAS) (AAS 1100, Perkin-Elmer) or ICP-MS (PlasmaQuad II, VG Elemental). In addition, mercury analysis was performed using the dedicated Hg analyzer DMA-80a (Milestone USA, Monroe, CT). These Hg determinations followed procedures documented in draft Method 7473 in Update IVA of EPA's SW-846 manual of methods (4, 16, 17).

Results and Discussion

General Safety. The typical temperature and pressure profiles for both the HNO₃-only and the HNO₃-HCl leaches of the SRM 2704-wastewater sample are shown in Figure 1. For comparison's sake, the profile for the heating of SRM 2704 in 18 M Ω ·cm H₂O is also shown. Addition of acids causes the pressure to increase by approximately 8 atm owing to the production of several reactive, gaseous species upon heating. These species include chlorine gas (Cl₂) and nitrosyl chloride (NOCl), which further decomposes into the gaseous products Cl₂ and NO (2, 3, 18, 19). It should be noted that these conditions remain similar regardless of the matrix type used to create the wastewater (i.e. soil-type wastewater or oil-type wastewater) and regardless of the acid ratio used in the leach. Also, the pressures attained inside the vessel are highly dependent on the material and thickness of the vessel and casing components. Vessels from different manufacturers and different designs have different heat loss characteristics and resultant pressures at a given temperature, as discussed elsewhere (2, 3). From a safety standpoint, the pressures reached during the wastewater leach require vessels having pressure capabilities of at least 30 atm (435 psi). Prior vessel specification in Method 3015 was 10 atm (110 psi).

Another safety precaution specified in new Method 3015A is that aqueous samples cannot contain more than 1% suspended solids or oxidizable material. This precaution was added to prevent analysts from mistakenly attempting to leach 45 mL of a highly reactive, organic matrix (for example, adding concentrated HNO₃ and HCl to 45 mL of a light liquid

oil or alcohol sample and heating). Reactions of this type are dangerous because of the extreme amount of pressure that would be produced in a very short time. The shock waves resulting from this reaction could potentially lead to catastrophic vessel failure or an explosion. Safety issues regarding microwave heating have been recently compiled and reviewed and are addressed in detail elsewhere (20). According to method specifications, the maximum amount of organic solids present should be no more than 0.45 g. Also, should a vigorous reaction occur upon addition of the acids, the analyst is instructed to treat the sample as a potential safety hazard and to use another method of sample preparation such as EPA Methods 3051A or 3052.

Vessel Materials. In addition to vessel pressure capabilities, the type of equipment specified in the method has also been updated. This is to allow the use of vessels constructed from advanced materials. The original Method 3015 specified that the digestion vessels be made of "fluorocarbon PFA or TFM" material (4). Updated Method 3015A specifies only that vessels be composed of "microwave transparent and reagent resistant materials" and gives PFA, TFM, and quartz as examples of vessel liner materials (4). This permits the method to be virtually independent of microwave equipment and manufacturer, provided that the equipment meets the minimum safety and performance requirements of the method.

Heating Profile. As shown in Table 1, the temperature profile specified in Method 3015A has also been changed. The profile previously specified in Method 3015 was to heat the samples to $160 \pm 4^\circ\text{C}$ in 10 min and then to allow the samples to slowly rise to $165\text{--}170^\circ\text{C}$ during the next 10 min. The new specification in Method 3015A is to heat the samples to $170 \pm 5^\circ\text{C}$ in 10 min and to hold the samples at $170 \pm 5^\circ\text{C}$ for the remaining 10 min. Not only does this new profile account for different manufacturers methods of temperature control but also it allows more precise reproduction of the leach heating profile. Some manufacturers treat the temperature specification as an upper limit. Microwave power is turned off once the controlling temperature has been reached and turned on after the temperature has dropped several degrees below the control temperature. Other manufacturers treat the controlling temperature as a mean value, turning the power on and off to maintain the temperature in a certain range about the mean controlling temperature. The new specified temperature of $170 \pm 5^\circ\text{C}$ allows better compliance by microwave manufacturers. In another change, the "slow rise" heating step in original Method 3015 was removed. Under the former conditions, it was up to the analyst to decide what was considered to be a slow rise in temperature from 160°C up to 170°C , adding unwanted subjectivity to the method. With updated Method 3015A, reaching and holding the same temperature allows more precise control of heating. This is important because control of temperature has been determined to be one of the most important parameters in obtaining precise leach results (3). And since precision is used as accuracy when comparing leach results, this precise temperature control becomes even more significant.

Development of Standards. As this was the first time that simulated wastewaters were created and used as standards, the validity of this approach was evaluated. Table 2 shows the results using the mixed-acid option of aqueous microwave leach Method 3015A, compared to mixed-reagent leach Methods 3051A and 3050B for samples of SRM 4355. Methods 3051A and 3050B are promulgated leach methods for solid materials with an abundance of data which can be used to compare and validate the procedure for creating simulated standards. Despite the presence of 45 mL of water in Method 3015A, all three leach methods consistently extract the same amount of metals from the matrix at the 95% confidence

TABLE 2. Comparison of Leachable Analyte Concentrations from SRM 4355 (Peruvian Soil) and SRM 4355-Wastewater^a Using EPA Leach Methods 3015A, 3051A, and 3050B (2, 13–15)

element	3015A Leach ^b	3051A Leach ^c	3050B Leach ^d
Co	10.4 ± 0.41	10.3 ± 0.24	13.7 ± 2.8
Cd	1.28 ± 0.33	0.85 ± 0.17	1.03 ± 0.08
Cr	17.7 ± 1.2	18.9 ± 0.7	17.1 ± 0.9
Pb	136 ± 3	130 ± 4	131 ± 5
Ni	10.8 ± 3.5	11.2 ± 0.4	9.93 ± 1.45

^a Method 3015A sample was ~400 mg of SRM 4355 mixed with 45 mL of 18 MΩ·cm H₂O; Methods 3051A and 3050B samples were ~500 mg of SRM 4355. ^b Results reported in mean μg/g analyte ±95% confidence interval. ^c Results from Method 3051A validation study (13). ^d Results from Method 3051 vs Method 3050B validation study (14, 15).

TABLE 3. Comparison of Leachable Analyte Concentrations from SRM 2704 (Buffalo River Sediment)-Wastewater Using Both Digest Options of EPA Method 3015A (13)^a

element	5:0 Leach	4:1 Leach	total ^b
Ag	0.31 ± 0.05	0.41 ± 0.09	<4
Co	12.0 ± 0.3	11.5 ± 1.0	14.0 ± 0.6
Cu	92.7 ± 3.1	89.2 ± 6.8	98.6 ± 5.0
Ni	39.6 ± 2.5	41.3 ± 1.7	44.1 ± 3.0
Pb	155 ± 2	154 ± 6	161 ± 17
V	6.18 ± 2.5	14.6 ± 2.4	95 ± 4
Zn	418 ± 12	412 ± 31	438 ± 12

^a Results reported in mean μg/g analyte ±95% confidence interval. ^b Total certified analyte concentration taken from NIST SRM Certificate of Analysis.

interval. These results demonstrated for the first time that the approach of creating simulated wastewaters containing some matrix-specific solid component was appropriate. This approach allows analysts to create simulated wastewater standards using solid reference materials that closely match the solid component of the sample wastewater matrix. This approach also provides NIST-traceable leach results for a standard of a matrix type which previously did not exist. For example, a reference standard for a wastewater consisting of suspended oil particles could be simulated by mixing a comparable amount of an oil SRM with ultrapure water. The same could be done with soils, sediments, and other specific solids. Most environmental SRMs contain fractions of carbonaceous material, chlorides, etc., a portion of which may transfer to the aqueous portion when mixed with water. Having species present in both the solid and aqueous portions is typical of what may be expected with an actual wastewater sample, thus providing a standard that is truly representative of this sample type.

Leach Chemistries. As mentioned, certain analytes are not stable either in an oxidizing environment (Sb) or in environments containing trace quantities of chlorides (Ag). The chemistry of a HNO₃-only leach is deficient when it comes to this type of analyte. However, by including the option to add HCl in the leach method, better recoveries are achieved for those analytes which are complexed by Cl⁻ and therefore more stable in solution. Specific discussion of the chemistry of these elements as it pertains to leach recoveries, including pertinent chemical reactions, and overall improvement of the chemistries of leach methods has been described in detail elsewhere (2).

Leach Recoveries. Leach recoveries using both reagent options of Method 3015A for the sediment-wastewater matrix are shown in Table 3, while recoveries for the soil-wastewater are shown in Table 4. For both of these similar matrix types, the results demonstrate that recoveries of some "problem"

TABLE 4. Comparison of Leachable Analyte Concentrations from SRM 4355 (Peruvian Soil)-Wastewater Using Both Digest Options of EPA Method 3015A (13)^a

element	5:0 Leach	4:1 Leach	total ^b
Ag	1.31 ± 0.12	1.62 ± 0.11	(1.9)
Cd	1.30 ± 0.25	1.28 ± 0.33	(1.5)
Co	10.5 ± 0.34	10.4 ± 0.41	14.8 ± 0.76
Cu	70.0 ± 1.9	70.6 ± 3.0	77.1 ± 4.7
Mo	0.99 ± 0.06	1.1 ± 0.11	(1.7)
Ni	12.2 ± 1.2	13.1 ± 1.9	(13)
Sb	3.7 ± 0.30	5.2 ± 0.53	14.3 ± 2.2

^a Results reported in mean $\mu\text{g/g}$ analyte $\pm 95\%$ confidence interval.

^b Total certified analyte concentration taken from NIST SRM Certificate of Analysis. Values in parentheses indicate reference concentrations.

TABLE 5. Comparison of Leachable Analyte Concentrations from SRM 1084A (Wear-Metals in Lubricating Oil)-Wastewater Using Both Digest Options of EPA Method 3015A (13)^a

element	5:0 Leach	4:1 Leach	total ^b
Al	102 ± 12	96.8 ± 7.0	(104)
Cr	100 ± 16	95.0 ± 5.6	98.3 ± 0.8
Cu	93.6 ± 6.0	92.0 ± 7.8	100 ± 1.9
Mg	87.3 ± 16	91.5 ± 6.9	99.5 ± 1.7
Mo	83.8 ± 5.1	88.3 ± 4.4	100.3 ± 1.4
Ni	92.9 ± 9.9	96.1 ± 10.8	99.7 ± 1.6
Pb	88.0 ± 2.6	89.1 ± 3.4	101.1 ± 1.3

^a Results reported in mean $\mu\text{g/g}$ analyte $\pm 95\%$ confidence interval.

^b Total certified analyte concentration taken from NIST SRM Certificate of Analysis. Values in parentheses indicate reference concentrations.

analytes (e.g. Ag, Sb, V) have increased with the mixed-acid leach, while recoveries of other analytes (e.g. Co, Ni, Pb) have been preserved (i.e. losses have not occurred).

Leach recoveries for the oil-wastewater matrix are shown in Table 5. Recoveries using either leach option demonstrate statistical equivalence at the 95% confidence interval, signifying that the mixed-acid leach does not introduce a negative bias in recoveries.

Expanded Analyte Validation. Originally, EPA Method 3015 was not validated for the preparation and subsequent evaluation of boron, mercury, and strontium. This means that some other sample preparation method in addition to Method 3015 would have to be used for analysis of these three elements. At one time, it was acceptable to require many different test methods to be used to evaluate a single waste material. Today, the preferred strategy is the development of more robust procedures capable of evaluating all the inorganic elements of interest. Table 6 demonstrates the leach recoveries of these three analytes using both reagent options for the sediment-wastewater and the soil-wastewater. The recoveries using Method 3015A are compared to those using promulgated EPA Method 3051A to leach the solid sediment or soil sample. Because Method 3051A is validated for B, Hg, and Sr, it was used to validate the wastewater results. The table shows that recoveries for all three analytes are statistically equivalent at the 95% confidence interval. Similar recoveries are provided by both the HNO_3 -only and the mixed-acid wastewater leach, demonstrating that either reagent combination is appropriate for the analysis of these three analytes. Thus, the list of validated analytes for wastewater preparation has been updated and now includes 26 elements.

Method Control and Transfer. Because of the special conditions under which leach results are compared, the reproducibility of the reaction conditions is critical. Also, it is the practice of some testing labs and agencies in foreign

TABLE 6. Performance and Validation of Method 3015A versus EPA Method 3051A for the Recovery of Previously Nonvalidated RCRA-Regulated Elements (13, 16)^a

element	5:0 Leach	4:1 Leach	EPA 3051A ^b
SRM 2704-Wastewater			
B	23.8 ± 3.0	30.6 ± 8.3	18.4 ± 4.4
Hg	1.49 ± 0.14	1.77 ± 0.28	1.75 ± 0.07
Sr	41.9 ± 1.3	49.0 ± 1.6	43.2 ± 4.5
SRM 4355-Wastewater			
B	32.9 ± 2.1	31.8 ± 2.7	28.4 ± 5.6
Hg	0.82 ± 0.08	0.83 ± 0.07	0.88 ± 0.15
Sr	136 ± 14.5	138 ± 13.8	108 ± 19.1

^a Results reported in mean $\mu\text{g/g}$ analyte $\pm 95\%$ confidence interval.

^b EPA Method 3051A leaches were performed using the 9 mL of HNO_3 to 3 mL of HCl acid ratio option on ~0.35 g samples of solid SRM 2704 and SRM 4355.

countries to adopt EPA methods as their standard methods. Thus, the development of reproducible standard methods has both national and worldwide importance. This requires standard methods which provide reproducible control of extraction conditions and accurate transfer. Use of microwave energy and temperature feedback control exceeds other methods in terms of the reproducibility of conditions and ease of method transfer from one user to another. Temperature, the parameter which controls reaction rates, can be maintained more accurately, resulting in more precise results.

Summary

The usefulness and applicability of creating specific types of simulated wastewater standards by mixing solid SRMs and 18 M Ω -cm water has been demonstrated. This technique establishes NIST-traceable standards for a matrix which previously was unavailable. This approach may also prove to be useful with reference materials from other international standards organizations, thus permitting wastewater methods to provide internationally referenceable results. Also shown is the effectiveness of adding HCl in Method 3015A to enhance the recoveries of specific analytes for the leaching of aqueous samples (wastewaters, drinking waters, and aqueous extracts). Leach data for three simulated wastewater matrices using the mixed-acid leach Method 3015A shows that this reagent combination provides enhancement in recoveries for "problem" analytes while preserving the recoveries of other analytes. Thus, the mixed-acid option allows the analyst to improve the chemistry of the wastewater leach method for recovery of certain analytes, when necessary. In addition, B, Hg, and Sr have been validated and added to the analyte list for Method 3015A. This improves the efficiency of the method and permits the sample preparation for analysis of 26 elements with a single, reproducible, 20-min microwave-assisted leach procedure.

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