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ADAPTATION OF SW-846 METHODOLOGY FOR THE ORGANIC ANALYSIS OF RADIOACTIVE MIXED WASTES

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ABSTRACT

Modifications to SW-846 sample preparation methodology permit the organic analysis of radioactive mixed waste with minimum personnel radiation exposure and equipment contamination. This paper describes modifications to SW-846 methods 5030 and 3510-3550 for sample preparation in radiation-zoned facilities (hood, glove box, and hot cell) and GC-MS analysis of the decontaminated organic extracts in a conventional laboratory for volatile and semivolatile organics by methods 8240 and 8270 (respectively). Results will be presented from the analysis of nearly 70 nuclear waste storage tank liquids and 17 sludges. Regulatory organics do not account for the organic matter suggested to be present by total organic carbon measurements.

INTRODUCTION

The closure and decommissioning of nuclear waste storage tanks at the Oak Ridge National Laboratory (ORNL) required the chemical, physical, and radiochemical analysis of highly radioactive liquids and sludges to determine their regulatory classifications and to aid in selection of appropriate treatment and disposal methods. A part of this characterization was the analysis of volatile and semivolatile organic compounds in the liquids and sludges.

Approved methodologies for the determination of regulated volatile and semivolatile organic (and other) compounds in wastes are described in the U. S. Environmental Protection Agency (EPA) Solid Waste Manual 846 (SW-846) (1). However, these methods were designed for nonradioactive wastes, and their direct application to radioactive wastes would result in the exposure of laboratory personnel to high radiation fields, and could contaminate personnel, equipment, and instruments with radionuclides.

We find that modifications can be made to SW-846 methods to limit radiation exposure and contamination in keeping with "ALARA" (As Low As Reasonably Achievable) policy, and yet achieve reasonable method performance. Exposures are minimized by traditional radiochemical means of shielding, minimizing the time of exposure to the sample, and maximizing the distance from the sample that the operator conducts the preparation. In addition, sample amounts must be reduced for some preparations.

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Our approach has been to prepare the samples using modified SW-846 methods in radioactivity zoned facilities such as radiochemical hoods, glove boxes, or hot cells. The organic extracts are effectively decontaminated of the bulk of the radionuclides, and they can be analyzed by SW-846 GC-MS methods in a conventional laboratory.

EXPERIMENTAL

Radioactive Waste Samples and Characteristics

The nuclear waste liquids analyzed in this project were collected by suction from underground storage tanks (2), some of which date to the 1940s. Sludges were collected using a coring device (2). Neither sample type was uniform in characteristics from layer to layer or tank to tank, and exhibited considerably diverse properties. The gross alpha activities ranged from <1 to 8.3×10^3 becquerels/mL (Bq/mL) for liquids and to 6.5×10^5 Bq/g for sludges. Gross beta/gamma activities were <20 to 3.6×10^6 Bq/mL for liquids and up to 5.9×10^7 Bq/g for sludges. The major radionuclides were ^{137}Cs, ^{90}Sr, and ^{60}Co. Lesser activities of other radionuclides such as ^{3}H and ^{233}U also were present. The sludges were also enriched in ^{244}Cm, ^{238}Pu, ^{239}Pu, and ^{241}Am. The pH of the liquids ranged from highly acidic (0.2) to highly alkaline (12.7), and total solids ran from 0.3 to 170 mg/mL. The dominant anions were sulfate (to 83,000 mg/L) and nitrate (to 31,000 mg/L). The total organic carbon contents were high: liquids were 10 to 12,000 mg/L and sludges were 4,000 to 28,000 mg/kg.

Radiochemical Facilities

The radioactivity of the samples required that they be prepared for organic analysis in radioactivity zoned facilities. Health Physics guidelines at ORNL (3) limit the total activity of "very high" radiotoxicity isotopes (such as ^{90}Sr) to 0.1 microcuries (uCi) for monitored benchtop sample preparation, 10 uCi for radiochemical hood work, and 10 millicuries (mCi) for glove box operations. Benchtop operations are performed in radioactive contamination-zoned laboratories with limited personnel access and periodic health physics monitoring for contamination. Samples, equipment, and staff cannot leave the lab without health physics screening for contamination. The radiochemical hood is located in the contamination-zoned laboratory, and consists of a normal stainless steel fume hood which exhausts through high efficiency particle filters and charcoal filters. Radioactive samples are contained in lead "pigs" when not being processed. The laboratory room air pressure is kept at a slightly lower level than that of surrounding halls or rooms to prevent spread of any airborne contamination. Laboratory room air exhausts through the hood. Glove boxes are located in another contamination-zoned laboratory and they consist of a sealed stainless steel box which is vented to an air exhaust header (also carefully filtered) maintained at lower pressure than the laboratory air. Samples are bagged in and out in such a manner that the glove box atmosphere never is in direct contact with or vents to the laboratory air. Sample manipulations are conducted with the protection of heavy rubber gloves protruding through one wall of the box and below viewing windows. Work with higher activities (>10 mCi) must be performed in hot cells, which have three foot thick concrete walls and remote manipulators. Personnel using all types of facilities wear special coveralls and shoes, and carry radiation dosimeters of several types. Cleanliness and a deliberate, unhurried, and carefully considered work plan are essential to successful operations. As noted in the Results and Discussion section, sample preparation was performed in all three types of facilities at the ORNL High Radiation Level Analytical Laboratory, depending upon the activity of the samples, and the operations performed.

Methods

The methodology described in this paper is based upon several SW-846 methods: 5030, 5040, and 8240 for volatile organic compounds in aqueous liquids; 3510 or 3550 and 8270 for semivolatile organic compounds in aqueous liquids and sludges (respectively). These methods were approached as prescribed in the SW-846 manual (1), and the original methods are not described here. Modifications necessary for limiting radiation exposure and contamination are discussed in the text. In some cases, EPA Contract Laboratory Program surrogate standards or matrix spike mixtures were used in method evaluation. Also, a modification was made to method 8015 to permit the direct aqueous injection GC analysis of mg/L concentrations of several alcohols and ketones.

RESULTS AND DISCUSSION

Volatile Organics Analysis (VOA) of Aqueous Liquid Wastes

The approach for the VOA entailed an off-line purge and trap (P/T) in a glove box located in a radiochemical laboratory. A glove box operation was required because the whole sample container (250 mL volume) had to be handled for the first opening for VOA. This step was followed by thermal desorption and a second purge and trap and gas chromatography-mass spectrometry (GC-MS) in a separate, conventional GC-MS laboratory. The procedure and apparatus are described in detail elsewhere (4). Briefly, 5 mL of aqueous waste sample and CLP surrogate standards were purged from a 40 mL VOA vial and a specially designed P/T head into a method 624 triple sorbent trap located outside of the glove box using method 5030 purging conditions. We have not detected the transfer into the trap of any radioactivity from waste samples. The internal standard was not added at this step to allow the recoveries of the two P/T stages to be differentiated. Matrix spiked samples and blanks also were prepared with the samples. The traps were screened by standard smear and probe procedures for external contamination, and were then transferred to a conventional GC-MS laboratory. Analysis was conducted by thermally desorbing the traps in a tube furnace at 182°C with a helium flow of 35 mL/min for 11 min. The effluent was bubbled through the Tekmar purging vessel which contained 5 mL of laboratory distilled water and CLP internal standards. This is similar to the desorption of volatile organics sampling train traps in method 5040, except that the internal standard was added to the water, and not swept into the triple sorbent trap prior to thermal desorption. The remainder of the analysis was conducted per method 8240 P/T GC-MS.

We find that this procedure performs quite well for the determination of volatile organic compounds in radioactive aqueous liquids. Keeping the same sample volume as stipulated in SW-846 permits the same reporting limits (i.e., 5-10 μg/L) as the conventional procedure. Using a VOA vial for the purging chamber allows foam (a problem with high salt content nuclear wastes) to disperse without contaminating the sampling head and transfer lines. A new vial is conveniently used for each analysis, preventing carryover of contaminants and minimizing equipment cleaning operations which are difficult to conduct in a glove box. The performance of the method as gauged by recoveries of surrogate standards and matrix spikes (Tables 1 and 2) is quite reasonable, considering the off-line procedure, two P/T steps, and the hostile sample matrix. The average recoveries generally fall within the QC Acceptance Limits for groundwater (1). The applicability of groundwater limits in methods 5030/8240 to aqueous nuclear wastes by modified 5030/5040/8240 has not been established, but the former are useful for comparison. The reason for the low bromofluorobenzene and chlorobenzene recoveries is not clear, but the similar results for the former in samples and blanks indicate that it is probably a characteristic of the modified apparatus, and not a sample matrix effect. The fact that bromofluorobenzene is the least volatile of the surrogate standards and that chlorobenzene has the highest boiling point of the matrix spiked compounds suggests that the sparging may not have been sufficiently vigorous for optimal recovery. Some of the matrix spike recovery data were discarded from the tabulation in Table 2 because they were inexplicably high (one for benzene, two for toluene, and one for chlorobenzene) or zero when the recoveries of the other matrix spikes were reasonable (two for toluene).

The method blanks were good, and showed only traces of methylene chloride and toluene (<20 μg/L each) and acetone (<40 μg/L). Other volatile organics were occasionally detected at levels of <20 μg/L. As reported elsewhere (4), the main volatile organics found in the samples were acetone (<5-600 μg/L), methylene chloride (8-1,000 μg/L), chloroform (3-400 μg/L), and methyl isobutyl ketone (<5-3,000 μg/L). Lesser concentrations of benzene, toluene, xylenes, trichloroethene, and tetrachloroethane also were determined in some samples.

Major Volatile Organic Compounds in Aqueous Liquids

The VOA was supplemented by a direct aqueous injection GC procedure similar to SW-846 method 8015. This analysis was conducted for two purposes: (a) to determine certain compounds which do not purge and cannot be determined by the VOA, e.g., methanol, and (b) to identify aqueous samples which were too concentrated in organic matter to P/T from a 5 mL volume. The latter was to protect the GC-MS from contamination from overloaded traps. A 1.5 mL volume of sample was taken at the time the P/T for VOA was conducted in the glove box, and a 3 μL aliquot of the sample was analyzed before the traps were taken to the GC-MS laboratory. The direct aqueous injection GC was performed using a GC located in a radioactivity contamination-zoned laboratory. The instrument was equipped with the same column packing as the method 8240 GC-MS (i.e., 0.125 in. OD x 8 ft. stainless steel packed with 1% SP-100 on 60/80 mesh Carbopack B), and a flame ionization detector, but was temperature programmed differently (70°C for 2 min, then program to 220°C at 16°C/min and hold at 220°C for 16 min). Three μL were injected using the solvent-flush technique (water), and peaks were identified using retention time. Quantitation was by the method of external standards versus 4 concentration levels of standards ranging from 3 to 40 mg/L.

This supplementary method proved to be quite valuable for the analysis of radioactive aqueous liquids. The method determined several alcohols which the VOA could not analyze, and also provided a better quantitation of ketones at mg/L concentrations which were above the calibration range of the VOA. Methanol and ethanol were found in several samples at concentrations up to ca. 40 mg/L. In two aqueous samples which were overlayered with an organic solvent mixture direct aqueous injection GC, was valuable in measuring alcohol and ketone concentrations of 1,000 to 2,000 mg/L. The detection limit of the method was ca. 1 mg/L, and the recoveries of matrix spikes averaged >90% for all analytes except for allyl alcohol (86%), because of peak tailing for the latter.

Semivolatile Organics Analysis (SVOA) of Aqueous Liquid Wastes

This sample preparation was conducted in a radiochemical hood after the sample had been opened, the P/T for VOA completed, and an analysis of gross alpha and beta/gamma activity had been conducted. A 20 mL volume of sample and a 40 mL VOA vial were utilized instead of the 1 L volume and separatory funnel stipulated by SW-846 method 3510 because the radioactivity of the latter was generally too great for a hood, and separatory funnel extractions in a hot cell were considered impractical for extensive numbers of samples. This reduced the reporting limits 50-fold, to 500-2,500 μg/L. The sample was spiked with CLP surrogate standards (and matrix spikes, when required) and three extractions were made with 5 mL of methylene chloride. The initial pH of the sample determined the pH of the first set of extractions. Three distinct cases were observed: pH <2, pH ca. 6-9, and pH >10. When the pH was <2, the sample was extracted as is to recover an acid/neutral fraction, and then the pH was adjusted to >10 with 1 M sodium hydroxide for extraction of the base fraction. When the initial pH was >10, the base/neutral fraction was extracted first, before pH adjustment and recovery of the acid fraction. For samples with pH 6-9, the pH was first adjusted to >10 and treated as noted above. The extractions were performed by gently tumbling the vial ca. 30 times. More vigorous agitation caused emulsion problems. In some cases, emulsions formed in spite of the gentle agitation, and centrifugation was required to break the emulsion. Other types of complicating sample behavior observed with nuclear wastes included evolution of oxides of nitrogen, precipitation, and significant buffering capacity.

Generation of oxides of nitrogen during acidification suggests the presence of nitrite in the samples, and raises the possibility of artifact formation. In a few cases, precipitates formed upon acidification. Also, a few of the initially alkaline samples required up to ca. 7 mL of 12 M hydrochloric acid, versus the 1-2 mL typically needed for adjustment to a pH of <2. The methylene chloride layers were recovered with a Pasteur pipette, and were separated from traces of water by passing through a disposable 10 mL polypropylene syringe fitted with a 0.45 um porosity Acrodisc CR Teflon membrane filter. The fractions were combined, reduced to a 1 mL volume under dry, flowing nitrogen gas, and were then transferred to autosampler vials and the CLP semivolatile organic internal standard solution was added.

Two characterizations were conducted before the SVOA extracts were transferred to the GC-MS laboratory. A GC equipped with an autosampler and a flame ionization detector, located in the contamination-zoned laboratory, was used to prescreen the samples to identify those which did not require GC-MS analysis and also those which required dilution to prevent overloading and organic contamination of the GC-MS. For the aqueous nuclear wastes, the latter was not a problem, and the main use was to screen out samples. The criteria used here was that if the sample did not contain any analyte peaks (different from those in a blank sample) greater than the responses of 4 mg/L (injected concentrations) of CLP Target Compound List (TCL) base/neutral and acid standards, TCL pesticide standards, or other selected Appendix VIII compounds (corresponding to a concentration in the original aqueous sample of 200 μg/L), then it did not require GC-MS analysis. In practice, less than 20% of the samples were rejected at this point. The SVOA extract also was sampled for gross alpha and beta/gamma activity determination. This characterization was conducted to prevent contamination of the GC-MS laboratory. For aqueous samples, the decontamination factors ranged from 2 to 4 orders of magnitude, and very little radioactivity typically carried over into the SVOA extract. Typically, the samples contained less than 0.01 uCi of radioactivity. In the very few cases where greater activity was observed, a 100 uL aliquot of the SVOA extract or a 1 mL aliquot of a 1:10 dilution (the latter re-fortified with internal standard) were sent to the GC-MS laboratory. It should be pointed out that this reported level of decontamination must never be assumed. The presence of large amounts of chelators or extractants in wastes conceivably could enhance carry-over of radionuclides. The GC-MS analysis was conducted as required by method 8270, except that it was not possible for the base/neutral and acid fractions to be combined and internal standard added at that point.

The modified procedure performed well in comparison with SW-846 QC Acceptance Limits for the conventional procedures. The recoveries of surrogate standards and matrix spikes are listed in Tables 3 and 4. The average recoveries fall well within the QC limits. Although all of the base/neutral compound recoveries were used for the tabulation, some of the acid compound data were deleted because of possible preparation or analysis problems. It is possible that the high initial pH of some samples caused the destruction of the phenolic surrogates and matrix spikes (a matrix effect). Quantitation of at least one phenol, pentachlorophenol historically is difficult. The method blanks were free of TCL compounds, except for traces of the ubiquitous phthalates, di-n-butyl (110 μg/L) and di-n-octyl (170 μg/L). Low concentrations (ca. 100-200 μg/L) of hydrocarbons were observed in all samples and blanks, and were contributed by the Teflon filter assembly. They did not interfere with the identification and quantitation of the TCL compounds. The only SVOA hit exceeding the reporting limit was benzoic acid (2,900 μg/L). Low levels (ca 20 - 300 μg/L) of 2-nitrophenol, 2,4-dinitrophenol, 2,4,5-trinitrophenol, and 2-4 ring polycyclic aromatic hydrocarbons also were determined. The main semivolatile organic compound found was the tentatively identified compound tributyl phosphate (2,000-30,000 μg/L). This compound was not present in the computerized spectral library, and was initially identified manually. It is an important component of the Purex process, and its presence was expected. Dibromonitrophenol also was detected in two samples (700 μg/L). The obvious generation of oxides of nitrogen during the acidification of some samples raises the question of at least some of these nitro-derivatives being artifactual. Our experiences with the SVOA of aqueous liquids are described in more detail elsewhere (5).

SVOA of Sludges

The SVOA of waste tank sludges followed methods 3550 and 8270 with the main exception being the masses extracted. The aliquots varied from ca. 2 to 20 g because of the limited amounts of sample available. The reporting limits accordingly ranged from 500 - 2,500 μg/kg to 5,000 - 25,000 μg/kg. The sludge sample extractions with larger masses of sample (10-20 g) or with the highly radioactive samples were conducted remotely in a hot cell, while the smaller masses (e.g., 2-5 g) were extracted in a radiochemical hood. The extracts were decanted from ultrasonic extractions conducted in a beaker, and were filtered through a medium porosity sintered glass funnel. Extractions conducted directly in the sintered glass funnel resulted in plugging of the filter. The composited extracts in the hot cell were sampled and analyzed for gross alpha and beta/gamma activity before transfer to a radiochemical hood for volume reduction (with flowing nitrogen gas) to 1 mL, transfer to an autosampler vial, and addition of the CLP SVOA internal standards. The concentrated extracts were sampled for gross alpha and beta/gamma activity and were screened with the GC before transfer to the GC-MS laboratory. In contrast to the behavior with the aqueous liquids, several of the extracts from the more highly radioactive sludges contained appreciable amounts of radioactivity (ca. 0.1 uCi), and 100 uL aliquots were transferred to small-volume autosampler vials to limit the activity taken to the GC-MS laboratory. Also, some of the extracts of the 20 g samples contained too high background levels of chromatographable organic matter, and 1:10 dilutions had to be made before GC-MS. The background was a very complex hydrocarbon mixture.

The performance of surrogate standards in this procedure is shown in Table 5. The recoveries for sludge samples are lower than for the aqueous liquids, and the reproducibilities are poorer. Problems were experienced with the recoveries of acidic compounds, and this undoubtedly was a result of the alkaline nature of the sludges, and the lack of pH adjustment procedures for solid samples in SW-846. Improved methods for semivolatile organics extraction of sludges are needed. The blanks were clean relative to the sludge extracts. The main compounds determined in the sludges were 4-6 ring polycyclic aromatic hydrocarbons (at concentrations of a few hundred to 240,000 μg/kg), phthalates (e.g., bis-[2-ethylhexyl]phthalate at 57,000 μg/kg in one sludge), and tributylphosphate (2-300 mg/kg). The relative accumulation of polycyclic aromatic hydrocarbons in the sludges probably reflects their low aqueous solubilities and salting-out from the high ionic strength liquids.

Total Organic Carbon (TOC) Accounting

TOC was estimated by another group using method 9060. An important conclusion from the comparison of the TOC data with the regulatory organics analyses is that the latter does not account for the organic matter suggested to be present by the former. For approximately 50% of the aqueous liquid samples, the TOC accounting was $< 5\%$, and for 78% of the samples, the accounting was $< 20\%$. For sludges, the TOC accounting was even poorer for 95% of the sludge samples, the accounting was $< 5\%$. The unaccounted organic matter seems to consist of highly polar, water soluble organics arising at least in part from the degradation of chelators, extractants, and other compounds used in the nuclear industry (2, 6, 7). For example, we have found (2,6) that a simple trimethylsilylation of the regulatory SVOA extract can double the organic matter visualized, and even more can be detected by evaporation and derivatization of the aqueous sample (6,7). Certainly, this is an area for much further work.

SUMMARY

SW-846 sample methodology can be adapted to radiochemical facility use for the preparation of highly radioactive samples for VOA and SVOA. Analyses of regulated volatile and semivolatile organics can be conducted with minimal personnel radiation exposure and instrument or equipment contamination and with acceptable method performance.

Improvements in instrumental sensitivity are needed to improve the detection limits for SVOA where limited by sample amount. Development of new extraction technology applicable to hot cell or glove box use with larger sample aliquots also would improve SVOA sensitivity. Sludges in particular would benefit from extraction methodology including pH adjustment.

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TABLE 1. RECOVERIES OF VOA SURROGATE STANDARDS FROM RADIOACTIVE AQUEOUS LIQUID WASTE

	Recovery[b], %		
Standard[a]	Wastes (n = 65)	Blanks (n = 7)	QC Limits
Toluene-d_8	89 ± 17	91 ± 11	88 - 110
Bromofluorobenzene	59 ± 15	61 ± 12	86 - 115
1,2-Dichloroethane-d_4	81 ± 11	86 ± 13	76 - 114

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[a] Spiked at concentration of 50 μg/L

[b] Average ± standard deviations for samples and blanks, and ranges of QC Acceptance Limits.

TABLE 2. RECOVERIES OF VOA MATRIX SPIKES FROM RADIOACTIVE AQUEOUS LIQUID WASTE

	Recovery[b], %	
Spike[a]	Waste (n = 20)[c]	QC Limits[d]
1,1-Dichloroethene	105 ± 18	D - 234
Trichloroethene	87 ± 14	71 - 157
Benzene	89 ± 15[e]	37 - 151
Toluene	81 ± 20[f]	47 - 150
Chlorobenzene	67 ± 13[e]	37 - 160

[a] Spiked at 50 μg/L Concentration.

[b] Averages ± Standard deviations for waste and ranges for QC Acceptance Limits.

[c] n = 20 except as noted.

[d] for 20 μg/L Spikes.

[e] n = 19.

[f] n = 15.

TABLE 3. SVOA SURROGATE STANDARD RECOVERIES FROM RADIOACTIVE AQUEOUS LIQUID WASTES

	Recovery[b], %		
Standard[a]	Wastes (n = 67)	Blanks (n = 8)	QC Limits
Nitrobenzene-d$_5$	70 ± 15	65 ± 18	35 - 114
2-Fluorobiphenyl	64 ± 14	58 ± 13	43 - 116
Terphenyl-d$_{14}$	88 ± 17	85 ± 18	33 - 141
Phenol-d$_5$	53 ± 12[c]	49 ± 12	10 - 94
2-Fluorophenol	48 ± 11[c]	42 ± 8	21 - 100
2,4,6-Tribromophenol	71 ± 17[c]	69 ± 14	10 - 123

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[a] Base/neutral and acid compounds spiked at 5 and 10 mg/L concentrations, respectively.
[b] Averages ± standard deviations for wastes and blanks and ranges for QC Acceptance Limits.
[c] n = 63.

TABLE 4. SVOA MATRIX SPIKE RECOVERIES FROM RADIOACTIVE AQUEOUS LIQUID WASTE

	Recovery[b], %	
Spike[a]	Wastes (n = 14)	QC Limits[c]
1,4-Dichlorobenzene	50 ± 10	20 - 124
N-nitroso-di-n-propylamine	66 ± 10	D - 230
1,2,4-Trichlorobenzene	53 ± 12	44 - 142
Acenaphthene	64 ± 13	47 - 145
2,4-Dinitrotoluene	71 ± 18	39 - 139
Pyrene	78 ± 12	52 - 115
Phenol	40 ± 9[d]	5 - 112
2-Chlorophenol	43 ± 7[d]	23 - 134
4-Chloro-3-methylphenol	55 ± 19[d]	22 - 147
4-Nitrophenol	69 ± 31[d]	D - 132
Pentachlorophenol	74 ± 32[e]	14 - 176

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[a] Base/neutral and acid compounds spiked at 5 and 10 mg/L concentrations, respectively.
[b] Averages ± standard deviations for wastes and ranges for QC Acceptance Limits.
[c] For 100 µg/L spikes.
[d] n = 10.
[e] n = 8.

TABLE 5. RECOVERIES OF SVOA SURROGATE STANDARDS FROM RADIOACTIVE WASTE SLUDGES

	Recovery[b], %		
Standard[a]	Sludges (n = 19)	Blank (n = 1)	QC Limit
Nitrobenzene-d_5	18 ± 17	33	23 - 120
2-Fluorobiphonyl	37 ± 19	35	30 - 115
Terphenyl-d_{14}	66 ± 20	99	18 - 137
Phenol-d_5	47 ± 20	46	24 ± 113
2-Fluorophenol	35 ± 19	39	25 - 121
2,4,6-Tribromophenol	37 ± 18	92	19 - 122

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[a] Base/neutral and acid compounds spiked at concentrations of 1 and 2 mg/kg (20 g sample) to 10 and 20 mg/kg (2g sample), respectively.

[b] Averages ± standard deviations for sludges, one result for blank, and range for QC Acceptance Limits.