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PREPARATION OF RADIOACTIVE "MIXED" WASTE SAMPLES FOR MEASUREMENT OF RCRA ORGANIC COMPOUNDS

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INTRODUCTION

A radioactive "mixed" waste typically contains alpha-, beta-, or gamma-emitting radionuclides and varying quantities of semivolatile or volatile organic species, some or all of which may be named specifically by the Resource Conservation and Recovery Act (RCRA). Because there are no acceptable means available currently for disposing of these mixed wastes, they are presently stored above-ground in sealed drums. For this reason, analytical procedures which can determine RCRA organics in radioactive waste are necessary for deciding the proper approach for disposal. An important goal of this work is the development of methods for preparing mixed waste samples in a manner which allows the RCRA organics to be measured in conventional organic analysis laboratories without special precautions.

Analytical procedures developed for handling mixed waste samples must satisfy not only the usual constraints present in any trace-level organic chemical determination, but also those needed to insure the protection of the operator from radioactive contamination. Consequently, procedures should be designed to use the least amount of radioactive sample commensurate with achieving acceptable sensitivity with the RCRA analytical methods. Furthermore, the usual laboratory glassware which would normally be used should be replaced with disposable materials wherever possible, in order to reduce the "clean-up" time required, and thereby reduce the operator's exposure to radioactivity. Actual sample handling should be reduced to the absolute minimum. Finally, the final isolate must exhibit a sufficiently low level of alpha, beta, or gamma activity to permit detailed characterization in a conventional organic analysis laboratory.

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Clearly, not all traditional or EPA-approved sample preparation procedures will prove satisfactory for mixed waste samples, given these additional restrictions. This paper describes our experiences in analyzing mixed waste aqueous and solid samples using a variety of conventional and supplemental procedures. In general, it is entirely feasible to prepare isolates from radioactive samples which are enriched in either semivolatile or volatile species, yet which are essentially decontaminated, and are therefore suitable for analysis in conventional organic analysis laboratories.

EXPERIMENTAL

Samples

The radioactive sludge materials tested were provided by the Operations Division of the Oak Ridge National Laboratory, Oak Ridge, TN.

Reagents, Solvents, and Radioactive Tracers

All solvents used in this work were purchased in "Distilled in Glass" purity from American Burdick & Jackson (Muskegon, MI), and were used as received.

Standard mixtures of the EPA priority polycyclic aromatic hydrocarbons (PAH), phenols, and organochlorine pesticides were purchased from Supelco, Inc. (Bellefonte, PA).

Acidic solutions with a known activity of the radionuclides Co-60 and Cs-137 were supplied by the Radiochemical and Activation Analysis Group, Analytical Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, TN.

The pH 4.9 leaching solution used is that described in EPA Method 1310, Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test (1). 64.3 mL of 1 N sodium hydroxide and 4.7 mL of glacial acetic acid were made to exactly 1 L with distilled water.

Equipment

Liquid-liquid extraction of aqueous samples with solvents of lesser specific gravity than water (e.g., pentane) were performed using a glass apparatus similar, but not identical, to Kontes Part No. K-584000 Continuous Extraction Apparatus (Vineland, NJ). Liquid-liquid extraction of aqueous samples with solvents of greater specific gravity than water (e.g., methylene chloride, were performed using Kontes Part No. K-584100, Continuous Extraction Apparatus.

All high-pressure liquid chromatographic separations and measurements were performed with a Beckman Model 334 Gradient Liquid Chromatograph (Berkeley, CA). This instrument was equipped with a UV detector (254 nm), a Spectra/Glo Filter Fluorometer (excitation filter, 280 nm; emission filter, "blue from fluorescamine") purchased from Gilson Medical Electronics (Middleton, WI), an HP 3390 integrator, and a stripchart recorder. The analytical column was protected by a 0.5 u porosity high-pressure inline filter (Scientific Systems, Inc., State College, PA, part nos. 05-0149 and 05-1055) and a guard column packed with Perisorb RP-18 (Upchurch Scientific, Inc., Oak Harbor, WA) connected in series. A Vydac No. 201 TP 5415 octadecyl column (The Sep/A/Ra/Tions Group, Hesperia, CA), 15 cm x 4.6 mm o.d., 5 u silica, was used for both PAH and phenol separations. The column was maintained at 30°C using a Model 7931 column heater purchased from Jones Chromatography (Littleton, Co). The methanol and water reservoirs, as well as the detectors, operated at room temperature. A 10 uL sample volume was introduced onto the column during each determination.

The solid phase extraction columns, manifold, pump, and pressure controllers were purchased from J. T. Baker, Inc. (Phillipsburg, NJ). Two manifolds and pressure controllers were used, such that the same one was always used for collecting organic analytes from radioactive aqueous samples, while the other was always used for eluting these analytes from loaded columns which had been essentially washed free of radioactivity.

The organochlorine pesticides were separated and quantitated according to EPA Method 608 using a Perkin Elmer Sigma 300 Capillary Gas Chromatograph (Norwalk, CT) equipped with a Model AS-300 autosampler (Perkin Elmer) and an SE-54 fused silica capillary column (0.25 u film thickness, 0.25 mm I.D., 30 m) purchased from Supelco, Inc. (Bellefonte, PA). The injector and electron capture detector temperatures were both 300 C. The column temperature was programmed from 140°C to 180°C at 4°C/min, and from 180°C to 250°C at 2°C/min. The oven temperature was held at 250°C for 5 min before returning to the starting temperature. A 3 uL injection was employed. The carrier gas was 90/10 (v/v) argon/methane, flowing at 5 mL/min. Data were collected, displayed, and analyzed using a Model 3000 Chromatography Data System (Nelson Analytical, Inc., Cupertino, CA) and an IBM PC/XT personal computer.

The collection of volatiles was performed using equipment custom-made at the Oak Ridge National Laboratory. A special Teflon sampling head equipped with a Teflon-faced silicone rubber septum screwed snugly onto a 40 mL EPA VOA sampling vial. The head provided a 10/32 screw port for a reusable "Fingertight" fitting, manufactured by Upchurch Scientific (Oak Harbor, WA). A length of capillary Teflon (1/16" o.d. x 0.3 mm i.d.) passed through the fitting into the VOA vial. The other end of the Teflon tubing was attached to a nitrogen cylinder with a flow controller. The head provided an additional port for a 1/8"-to-1/4" Swagelok reducing union; the 1/8" side was screwed into the collection head. The other end of the Swagelok union was connected to a 25 cm x 4.6 mm o.d. stainless steel column

dry-packed with Tenax GC 35/60 mesh, which was purchased from Alltech Associates (Deerfield, IL). A simple calibrated rotameter was connected to the free end of the Tenax trap with a piece of rubber tubing.

The volatiles were analyzed using a procedure based on EPA Method 8240; however, modifications were introduced to allow the volatile analytes to be desorbed directly from a stainless steel trap packed with Tenax GC. A Model LSC-2 Tekmar Liquid Sample Concentrator (Tekmar Co., Cincinnati, OH) was used to desorb the organics of interest. The existing 1/8" o.d. trapping column and corresponding column heater in the oven were both removed and replaced with the 1/4" o.d. sample trap and corresponding heater. Exactly 250 ng each of bromochloromethane, 1-chloro-2-bromo- propane, and 1,4-dichlorobutane from a Purgeables Internal Standard Mix-624 (Supelco,Inc., Bellefonte, PA) was sparged onto the Tenax sample trap using an 11 min wet purge and a 4 min dry purge. The internal standards and analytes present on the trap were desorbed at 180 C for 3 min using helium carrier gas. The components were swept onto the head of a 6.6' x 1/4" o.d. glass column packed with Carbopack B coated with 1% SP-1000 (Supelco) located in a Hewlett-Packard 5995 GC/MS. The oven temperature was programmed from 45°C (hold for 3 min) to 180°C (hold for 45 min) at a rate of 8°C/min. The analytes were detected by the mass spectrometer, which operated at 70 eV ionization potential, mass range of 35-260 amu, and a scan time of 0.24 scan/sec.

All measurements of gamma activity were performed using a proportional scintillation counter. Two sodium iodide crystal well counters heavily shielded with lead were coupled to an EG & G Ortec (Oak Ridge, TN) system consisting of two Model 776 Counter/Timer boards, two Model 478 0-2 keV bias supply boards, and a Model 779 Interface/Controller board. The counter exhibits a typical efficiency of ca. 35% for Cs-137.

All measurements of beta activity were performed using a beta RIDL Proportional Counter consisting of a Model 40-9B voltage module (operated at 2300 V), a Model 30-19 sensitivity module, and a Model 49-25 timing and readout module. The lead sample chamber was continuously sparged with 10% methane/90% argon during each measurement. The counter typically exhibits 8% counting efficiency for Sr-90.

Procedures

Soxhlet Extraction of Sludges. Approximately 10 g sludge were mixed with 20 g anhydrous sodium sulfate, then transferred to a pre-extracted cellulose Soxhlet thimble. The sludge was then extracted overnight with methylene chloride or pentane. The extract was concentrated to exactly 1 mL; portions were taken for gross beta and gross gamma counting (see below).

Leaching of Sludges. Approximately 10 g of sludge and 200 mL of the pH 4.9 acetic acid/sodium acetate solution were stirred briskly overnight using an overhead electric stirrer.

Extraction of the pH 4.9 Leachate. Exactly 100 mL of the pH 4.9 leachate described above were extracted continuously with pentane or methylene chloride overnight. The pentane extract was then concentrated using a Kuderna-Danish concentrator to exactly 1 mL. Aliquots were taken for gross beta and gross gamma counting (see below).

Solid Phase Extraction of EPA Priority Pollutant PAH from pH 4.9 Buffer. Three Baker-10 SPE 3 mL OCTADECYL columns were conditioned with methanol followed by 15% (v/v) isopropanol in water. An 18 mL portion of isopropanol was added to 100 mL water which had been spiked at 40-400 ppb with each PAH and 47,000 cpm (ca. 2200 Bq) Co-60. The resulting solution was passed through the OCTADECYL column using the "radioactive" manifold. The column was rinsed with a few mL of 15% (v/v) isopropanol in water. The PAH were eluted with exactly 1 mL of methylene chloride using the "clean" manifold (2). The final organic fraction was screened for gross gamma activity before performing the final quantitation by HPLC.

Solid Phase Extraction of EPA Organochlorine Pesticides from Water. Three Baker-10 SPE 3 mL OCTADECYL columns were conditioned with methanol followed by water. A 100 mL portion of distilled water which had been spiked with 5 ppb of organochlorine pesticides and 18,000 cpm (ca. 900 Bq) Cs-137 was passed through the OCTADECYL column using the "radioactive" manifold. The column was rinsed with a few mL of water and air-dried for a few minutes. The pesticides were eluted using 10 mL of hexane using the "clean" manifold (3). The final organic fraction was screened for gross gamma activity before performing the final quantitation by gas chromatography with electron capture detection.

Solid Phase Extraction of EPA Priority Pollutant Phenols from Water. Three Baker-10 SPE 3 mL or 6 mL OCTADECYL columns were conditioned with methanol and water adjusted to pH 2 with HCl. A 100 mL volume of water was spiked with 120 ug of each priority pollutant phenol and 5.5 E+6 cpm (ca. 2.6 E+5 Bq) Cs-137. The solution was adjusted to pH 2 with HCl, and the phenols were "salted out" by adding 25 g of NaCl. The test solution was then passed through the column, which was later rinsed with 0.01 M HCl and permitted to air-dry briefly using the "radioactive" manifold. The phenols were eluted with 5 mL of methanol using the "clean" manifold (4). The final organic fraction was screened for gross gamma activity before performing the final quantitation by HPLC.

HPLC Separation and Quantitation of EPA Priority Pollutant PAH. PAH were eluted from the HPLC column using a gradient which changed linearly from 70% methanol/water (hold for 10 min) to 99% methanol/water (hold for 10 min) over 15 min. Quantitation was performed using the integrator, which monitored the flurometer signal.

HPLC Separation and Quantitation of EPA Priority Pollutant Phenols. The phenols were eluted using a gradient program which changed linearly from 30% methanol/1% (v/v) acetic acid in water to 95% methanol/1% (v/v) acetic acid in water over 20 min. Quantitation was performed using the integrator, which monitored the UV detector signal (254 nm).

Collection of EPA Priority Volatiles from Water. Three 5 mL aqueous solutions containing 50 ppb each of the EPA volatiles and the purgeables surrogate standard were prepared in 40 mL EPA VOA vials. The Teflon sampling head was screwed onto the top of each vial, and a Tenax GC stainless steel trap was connected to the reducing union. The solutions were sparged with nitrogen at 90 mL/min for 15 min. The trap was then sealed and analyzed for volatiles, as described above. In separate experiments, 5 mL of unspiked water served as the blank, and 5 mL of water spiked with 5 E+5 Bq Co-60 served as the test for transfer of radiation. In the latter, a Tenax column was unpacked, and the front end of the column was removed and tested for gross gamma emission.

Determination of Gross Gamma Radioactivity. A known volume (usually 1 mL) of organic isolate was placed into a 10 x 75 mm glass test tube, which was then stoppered and placed into the well counter. Gross gamma activity was typically counted for 10 min and reported in counts per minute. The activity may be converted to Bq, but only assuming that the gamma emitter had the same efficiency as Cs-137 (standard available).

Determination of Gross Beta Radioactivity. Exactly 200 uL of organic isolate was placed onto a 25 mm o.d. watch glass, and the solvent was allowed to evaporate. The watch glass was then placed in a cardboard mount featuring a mylar film window. The mount was placed into the lead sample chamber, and gross beta activity was measured for 10 min. The activity is reported in Bq.

RESULTS AND DISCUSSION

The barrel sludge and filter cake samples studied, which were obtained from a process water treatment clarifier, exhibited a nominal beta activity of 20,000 Bq/g (mostly as Sr-90). Historically, similar samples exhibit not more than twice that value. The remaining solid is an impoundment pond sediment in which Sr-90 is also the principal radionuclide, but at a nominal activity of 2000 Bq/g.

The usual conditions employed for leaching a solid as described in EPA Method 1310 (1) were deemed unsuitable for these radioactive solids. The practice of tumbling 100 g of solid and 2 L of leaching solution for at least 18 hours in a glass jar presented a significant risk of seepage or radioactive spill. Thus, a substitution was made for Method 1310 in which (a) both the mass of sample and volume of liquid were scaled down by a factor of ten, and (b) brisk stirring of the sample and leach solution using an overhead electric laboratory stirrer was substituted for the tumbler. In this manner, the spirit of Method 1310 --intimate contact of the solid and leach solution --was maintained, while minimizing the likelihood of a radioactive spill.

The subsequent liquid-liquid extraction of the radioactive leachate also was modified. EPA Method 3510 (1), Separatory Funnel Liquid-Liquid Extraction, similarly was deemed unsuitable for radioactive liquids. EPA

Method 3520 (1), Continuous Liquid-Liquid Extraction, was substituted. In this procedure, the radioactive aqueous sample remains essentially undisturbed in a glass vessel, while the organic extraction solvent moves slowly through it. Continuous liquid-liquid extractors are available for solvents which have densities greater than water (e.g., methylene chloride) and less than water (e.g., pentane). In practice, the use of pentane yielded recoveries of test organic compounds comparable to those obtained with methylene chloride. The major advantage of pentane is that a smaller quantity of particulate matter is carried into the final organic extract compared to methylene chloride. Hence, a subsequent filtration step could be avoided. While many potential RCRA organic compounds are indeed less soluble in pentane than in methylene chloride, this deficiency may be readily overcome by simply extracting for a longer period of time than that used for methylene chloride. Furthermore, the RCRA compounds are frequently present at trace or ultratrace levels -- concentrations where the analyte would be reasonably soluble in pentane.

Table 1 follows the reduction in beta activity (due primarily to Sr-90) during the leaching of three test radioactive solids and the subsequent acid/base continuous extractions with pentane. Leaching with pH 4.9 sodium acetate/ acetic acid solution reduced the activity by an order of magnitude. The acid/base extractions of the leachate using either methylene chloride or pentane yielded a further reduction to near-background levels, thereby suitable for analysis in a conventional organic laboratory. Thus, traditional extraction procedures are capable of providing a concentrated organic extract containing the extractable organics without carrying over a significant quantity of beta activity from Sr-90, an alkaline-earth radionuclide. A cursory HPLC examination of these concentrates revealed no UV-absorbing organic compounds present in these samples at part-per-billion levels.

Sludge samples similar to those described above are also readily decontaminated when Soxhlet extracted with organic solvents such as methylene chloride. In a typical experiment, in which the final organic extract is concentrated to 1 mL, the total radioactivity present is <0.5 Bq/mL gross beta and <1 Bq/mL gross gamma (referenced to Cs-137) radiation over background -- an activity level entirely compatible with conventional organic analysis laboratories.

The potential advantages of solid phase extraction (SPE) warranted careful evaluation of the technique applied to simulated aqueous samples contaminated with radioactivity. As shown in Tables 2 and 3, SPE permitted near-quantitative recovery (>95%) of the EPA priority polycyclic aromatic hydrocarbons and the EPA organochlorine pesticides (both representing neutral compounds) at simulated environmental levels (40-400 ppb and 5 ppb, respectively) while simultaneously reducing the gamma activity present from a starting level of 20,000 or 40,000 cpm (ca. 1,000 or 2,000 Bq) to background or near-background levels (within instrumental uncertainty). The decontamination factors were ca. 2,000 for these samples. Note that in SPE, the sample preparation is complete in less than a half hour, more than one sample (up to ten) may be processed simultaneously, there is minimal cleanup

required (columns are disposable), and the operator's exposure to the radioactive sample is minimized. It is assumed (but not confirmed) that most water-soluble radionuclides would behave similarly to Cs-137 and Co-60, and can be removed almost completely from the final organic extract.

Solid phase extraction did not prove as successful in recovering the EPA priority pollutant phenols from a test mixture, even though the gamma activity was reduced from a starting value of 5.5 E+6 cpm (ca. 2.6 E+5 Bq) to background levels (within instrumental uncertainty). A variety of columns, including OCTADECYL (two sizes), OCTYL, and PHENYL, were tested; however, the OCTADECYL columns yielded the best recoveries, as shown in Table 4. Clearly, the columns tested did not recover either phenol or 4-nitrophenol quantitatively at the test level of ca. 1 ppm, although they did recover most of the other phenols at or somewhat below the 95% level. These data indicate that while SPE certainly does have advantages in the analysis of phenols in mixed waste aqueous samples, the OCTADECYL columns should not be considered optimal. Columns packed with resins or porous polymers might give better recoveries of phenols; however, it must be demonstrated that these columns will also yield a final extract with a near-background level of radiation.

The determination of volatile constituents in radioactive aqueous samples was simplified greatly using the custom-made sampling head and VOA vial containers. The recovery of many EPA priority volatile materials using the sampling head and a dry-packed Tenax GC trap was quite reasonable at the 50 ppb level, as shown in Table 5, while maintaining a very low level of activity in the Tenax trap itself. During one of the trials using only Co-60 tracer as the "volatile" analyte, the Tenax from the front 3-5 cm of the trap was removed after sparging and subjected to gross gamma counting. No gamma activity was detected. Reducing the sparge gas flow rate from 90 mL/min to 25-40 mL/min and the sparge time from 15 min to 11 min would have probably led to an improved recovery of volatiles without adding additional activity to the trap. Furthermore, if the liquid sample is sparged gently, the sampling head itself should remain noncontaminated despite repeated use. The only part of the sampler which would become contaminated, therefore, would be the capillary Teflon tubing which is in contact with the sample. Even here, contamination is minimized because the Teflon tubing is not wetted with water, and should absorb very little radioactive solution. Hence, it is quite feasible to sparge volatiles from a radioactive aqueous sample onto a Tenax trap, and take the trap into a conventional organic analysis laboratory for detailed characterization because the trap is itself noncontaminated.

In general, then, our preliminary experiences in preparing low-level radioactive samples for analysis suggest that concentrates not contaminated by the gamma- or beta-emitting alkaline or alkaline earth metals may be prepared readily without expensive and exotic equipment. In addition, several of the major pollutant compound classes, such as the PAH, organo-chlorine pesticides, and phenols, may be readily concentrated using simple solid phase sorbent technology.

Our experiences have not addressed the effect of these sample preparation procedures on several important classes of radionuclides. These include common low-energy beta emitters such as tritium, volatile beta/gamma emitters such as iodine-131, and transuranic radionuclides such as americium-241. Furthermore, samples contaminated with alpha emitters such as Pu-239 require special handling techniques, such as glove boxes and inert atmospheres, regardless of the activity level present. Nevertheless, the procedures described here should be considered a reasonable starting point for preparing suitable isolates of RCRA organic compounds. These procedures may also be entirely proper for preparing such isolates from alpha-contaminated samples provided that all manipulations are performed in a contained environment.

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Table 1

SUMMARY OF EXTRACTION AND LEACHING ACTIVITY DATA FOR THREE SOLIDS

Treatment		Pond Sludge 3513-A6, 30 g	Barrel 11 Sludge, 20 g	Filter Cake 20 g
Initial beta activity, Bq		62,400	316,000	426,000
Activity present after pH 5 leaching, Bq	(1)	1,200	34,000	39,200
	(2)	3,200	31,200	37,200
Activity present after acid/base extraction of the leachate with pentane, Bq	(1)	<22	< 4	0
	(2)	<26	<38	34
Activity present after acid/base extraction of the leachate with methylene chloride, Bq	(1)	< 1	120	53
	(2)	100	103	73

Table 2

Recovery of Priority PAH from 100 mL pH 4.9 Buffer

Compound	Spike Level 40-400 ppb			
	Trial 1	Trial 2	Trial 3	Mean % rec.
Naphthalene	83	83	73	80
Acenaphthene	90	83	80	84
Phenanthrene	100	95	90	95
Anthracene	93	70	68	77
Fluoranthene	89	85	81	85
Pyrene	100	100	100	100
BaA	98	98	95	97
Chrysene	98	95	98	97
B(b)fluoran	90	94	90	91
B(k)fluoran	93	95	93	94
BaP	93	95	93	94
Dibenz(a,h)anth	96	99	95	97
Benzo(ghi)peryl	96	91	90	92
Indeno[1,2,3-cd]pyrene	91	93	90	91

Samples spiked with 47,000 cpm (ca. 2200 Bq) Co-60. Final activity <10 cpm (ca. <0.5 Bq), with measurement limited by instrumental uncertainty. Decontamination factor ca. 2000.

Samples prepared using solid phase extraction, as described in text.

Table 3

Recovery of EPA Organochlorine Pesticides at Test Level of 5 ppb

	Recovery, % at 5 ppb	
	Lindane	Endrin
Hexane -1	89	88
Hexane -2	100	100
Hexane -3	98	100
Average Recovery, %	96	96

All samples spiked with 18,000 cpm (ca. 900 Bq) Cs-137. Final activity < 10 cpm (ca. <0.5 Bq). Estimated decontamination factor >2000.

Samples prepared by solid phase extraction, as described in text.

Table 5

Test of Sparger for Purgeables in Radioactive Aqueous Samples at 50 ppb

Compound	Recovery in ppb Trial 1	Trial 2	Trial 3	Avg % Rec.
Methylene chloride	28	240	23	194
Acetone	87	140	73	200
Carbon disulfide	14	10	10	23
1,1-Dichloroethene	20	16	15	34
1,1-Dichloroethane	27	24	22	49
1,2-Dichloroethene	25	22	20	45
Chloroform	14	34	30	52
1,2-Dichloroethane	32	28	26	57
2-Butanone	97	105	92	196
1,1,1-Trichloroethane	23	24	22	46
Carbon tetrachloride	25	25	25	50
Vinyl acetate	32	30	27	59
Bromodichloromethane	26	27	27	53
1,2-Dichloropropane	28	28	27	55
cis-1,3-Dichloropropene	21	30	27	52
Trichloroethene	29	29	27	57
Dibromochloromethane	18	32	30	53
1,1,2-Trichloroethane	24	33	31	59
Benzene	30	44	26	67
trans-1,3-Dichloropropene	20	23	21	43
Bromoform	6	31	28	43
4-Methyl-2-pentanone	37	51	46	89
2-Hexanone	22	55	51	85
Tetrachloroethene	9	33	32	49
1,1,2,2-Tetrachloroethane	21	21	22	43
Toluene	21	35	22	52
Chlorobenzene	10	22	21	35
Ethylbenzene	9	21	21	34
Styrene	5	17	16	25
Xylenes	7	26	18	34

Radioactive spike added: 5.0 E+6 Bq Cs-137.
Final activity: background (within instrumental uncertainty)
Estimated decontamination factor >1 E+6