



Project Summary

USEPA Method Study 36 SW-846 Methods 8270/3510 GC/MS Method for Semivolatile Organics: Capillary Column Technique; Separatory Funnel Liquid-Liquid Extraction

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An interlaboratory collaborative study was conducted on SW-846 Methods 8270/3510 entitled "GC/MS Method for Semivolatile Organics: Capillary Column Technique; Separatory Funnel Liquid-Liquid Extraction" to determine the precision and recovery of 59 semivolatile organic compounds in reagent water, ground water and leachate. SW-846 Methods 8270/3510 include instructions for quality control, sample preparation, and analysis by gas chromatography mass spectrometry (GC/MS).

The study design was based upon Youden's non-replicate plan for collaborative tests of analytical methods. The test waters: reagent water used as a control, ground water, and hazardous waste leachate were spiked with 59 semivolatile compounds at six concentration levels (three Youden pairs). In the study of SW-846 Methods 8270/3510, ten laboratories extracted the test waters with methylene chloride, concentrated the extract to 1 mL and analyzed the extract for 59 semivolatile compounds by GC/MS. The results were analyzed using USEPA computer programs entitled "Interlaboratory Method Validation Study" (IMVS) which produced measures of precision and recovery for the 59 semivolatile compounds in

each water type and compared the performance of the method among water types.

The study was conducted under the direction of the Quality Assurance Research Division, Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Cincinnati) Environmental Protection Agency, under Contract No. 6803-3254. Analytical work was completed as of September 1987. The report covers a period from September 10, 1986 to December 21, 1987.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Cincinnati, OH to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The Hazardous Waste Management Facility Permit Regulations promulgated in July, 1982 (40 CFR 265) establishes performance standards for the monitoring of ground waters, wastewaters and solid wastes at hazardous waste sites. To facilitate these standards, chemical and physical analyses are required to assess the degree of ground water contamination at and around the site. The Manual:

[Test Methods for Evaluating Solid Waste Physical and Chemical Methods, (SW-846), November 1986, Third Edition], provides a unified, up-to-date source of information on sampling and analyses related to compliance with Resource Conservation and Recovery Act (RCRA) regulations. The success of pollution control activities, particularly when legal action is involved, depends upon the reliability of the data generated by the laboratories, therefore it is important to evaluate the analytical methods fully. This is best done through interlaboratory method validation studies.

The Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL Cincinnati), develops/selects analytical methods and provides quality assurance (QA) support to agency programs involving water and waste regulations. In EMSL-Cincinnati, the responsibility for providing QA support is assigned to the Quality Assurance Research Division (QARD). Its QA program is designed to establish the reliability and legal defensibility of waste and waste data collected by the Agency, the state regulating authorities, the private sector, and commercial laboratories performing compliance analyses. One of QARD's QA activities is to conduct interlaboratory method validation studies to evaluate analytical methods selected for the Agency's operating programs such as the Office of Solid Waste.

This report describes an interlaboratory method validation study for SW-846 Method 8270/3510, "GC/MS Method for Semivolatile Organics: Capillary Column Technique; Separatory Funnel Liquid-Liquid Extraction" for the analyses of 59 semivolatile compounds.

The objective of the study was to characterize the behavior of Method 8270/3510, when performed by multiple laboratories, in terms of recovery, overall and single-analyst precision and the effect of water type on recovery and precision. The study was conducted with the cooperation of ten participating laboratories under the direction of the Quality Assurance Research Division (QARD), EMSL-Cincinnati. The Bionetics Corporation, as primary contractor to QARD, was responsible for the collection and characterization of test waters, preparation of samples, user instructions and report forms, distribution of samples, and screening the returned data for gross errors. The raw data were evaluated statistically by the QARD using computer programs entitled, "Interlaboratory Method Validation Studies" (IMVS). Upon review of the draft report by EMSL-

Cincinnati, The Bionetics Corporation prepared the final report.

Description of Study

Two SW-846 methods were investigated in this study. Method 3510 entitled "Separatory Funnel Liquid-Liquid Extraction" was used for the extraction procedure and Method 8270 entitled "GC/MS Method for Semivolatile Organics: Capillary Column Technique" was used for the determinative step.

Method 8270 is applicable to Appendix IX semivolatile compounds. However, this study was limited to those compounds not previously covered under USEPA Method Study 30 on Method 625 a similar GC/MS method. Method 625 uses packed column chromatography while Method 8270 uses capillary column chromatography. It was assumed there would be no significant difference in the computed statistics of the compounds covered by both methods therefore these organics were not included in this study. Of the remaining 72 compounds, 13 were unsuitable for the study. Table 2 lists these compounds and the reasons they were excluded. The remaining 59 compounds were divided into three mixes for this method study.

Method Summary

One liter of sample water was extracted three times with 60 mL portions of methylene chloride at pH > 11 then re-extracted three times with 60 ml portions of methylene chloride of pH < 2. The collected extracts were passed through sodium sulfate then concentrated to 1.0 mL using a Kuderna-Danish apparatus. The concentrated extracts were injected into a GC/MS instrument equipped with a 30 meter narrow bore DB-5 fused silica capillary column or equivalent.

Study Design

The design of the study was based upon Youden's non-replicate design for collaborative evaluation of analytical methods. In this design, sample solutions are prepared in pairs, such that the analyte concentrations of the pairs vary between 5-20% of the mean of the pairs. For this study, spiking solution concentrates of the 59 compounds were prepared at six concentration levels, as three Youden pairs. The spiking solutions were divided into three mixes to facilitate chromatographic resolution during analyses and to maximize compound solubility at high concentrate levels. The ampul concentrates were used to fortify three water matrices before extraction and analyses. Analysts from ten

participating laboratories were directed to extract and analyze each sample an report one value for each analyte at each concentration level. Analyses in reagent water were used to evaluate the proficiency of the method on a sample free of interferences; analyses in the other waters were intended to reveal the effects of interferences on the method.

Each participating laboratory was required to analyze a matrix blank and quality control sample. Acceptance limits were provided with the quality control sample to give the participating laboratories their "in-control" limits. Each laboratory also received a set of standard solutions for use in preparation of calibration curves. The cost and limited availability of several of the compounds included in this study necessitated this course of action.

Verification Analyses

Ampulled samples and standard solutions were analyzed by Bionetics personnel prior to distribution and compared to freshly prepared standards to verify that the solutions were properly prepared and stable. At the conclusion of the method study, the ampulled samples and standard solutions were again analyzed versus freshly prepared standards to verify the stability of each compound throughout the period of the study.

Selection of the Matrix Waters

Two waters were collected from hazardous waste sites for use in this study. A ground water sample was collected from a monitoring well at sanitary landfill and a leachate sample was collected from a closed cell at hazardous waste landfill.

GC/MS analyses of the ground water sample revealed no background concentrations of the 59 analytes in the study. The leachate sample, on the other hand, contained phenol and phenolic compounds. This matrix was ultimately diluted to 10% leachate to reduce the phenolic background concentration to levels that would not saturate the instrument detector. Interferences with the study compounds were not found.

Selection of Participating Laboratories

The Quality Assurance Research Division (QARD) of EMSL-Cincinnati was responsible for the selection of the participating laboratories. As per the standard competitive bid process, an abstract of the scope of work was announced in the Commerce Business Daily. Over 100 laboratories requested

the complete request for proposal (RFP) which included the evaluation criteria upon which the offeror would be scored.

Submitted technical proposals were evaluated based upon laboratory experience and quality control practices. Laboratories whose proposals were acceptable were evaluated further in a preaward performance evaluation study. The participants selected for the formal study were the ten laboratories with acceptable proposals who performed best in the preaward study.

Results and Discussion

The objective of this study was to characterize the performance of SW-B46 Method 8270/3510 in terms of recovery, overall precision, single analyst precision and the effect of water type on recovery and precision.

The calculated mean recoveries for reagent water indicate that the data presented in this report meet the objectives outlined for this study. The pooled mean recovery for all 58 compounds, excluding 3-methylcholanthrene, was 77.9% with a standard deviation of 13.3%. Forty-four compounds had recoveries within one standard deviation of the pooled mean. The average recovery of these 44 compounds was 80.2% with a standard deviation improving to 6.3%. The small improvement in the pooled mean recovery with a large improvement in the standard deviation, 13.3% to 6.3%, illustrates how tightly the data are distributed.

Generally, the ground water matrix had a lower mean recovery, 73.5%, than reagent water with 77.9% and slightly more variable data with a standard deviation of 16.6% versus 13.3% for reagent water. The leachate matrix had mean recoveries comparable to reagent water with 78.9% and a standard deviation of 14.9%.

Several compounds presented problems during the study thus making their regression equations suspect. Chlorobenzilate suffered from extremely poor extraction efficiency. The standard solution for 3-methylcholanthrene dehydrogenated in the methylene chloride causing high sample recoveries. Three compounds: 7,12-dimethylbenz(a)anthracene, a,a-dimethylphenethylamine, dibenzo(a,e)pyrene and tris(2,3-dibromopropyl) phosphate suffered from poor response and erratic chromatography.

The preliminary stability studies revealed that the aromatic amines were with the acetone solvent and eluting as two peaks. The main peak was the

aromatic amine and the second was the reaction product. The amines were combining with the acetone and, because they were stabilized by an aryl group, formed stable Schiff bases.

To prevent this reaction from interfering with the 8270 analyses, the aromatic amines were prepared as a separate concentrate (mix 1) with methanol as the solvent. The Schiff base reaction was not found to occur in this solvent. The remaining compounds were dissolved using acetone as the solvent.

Several compounds were only available as a mixture of isomers or as impure compounds. They were: di-allate, two isomers approximately 55% and 45%; isosafrole, two isomers 80% and 20%; pronamide with two impurities at 1% and 24% with the main peak at 75%; 2,3,4,6-tetrachlorophenol 80% pure with a 20% impurity peak; tris(2,3-dibromopropyl)phosphate 62% pure with two impurity peaks at 25% and 13%.

The analytical data, processed through the IMVS programs, were subjected to three outlier tests. First, the Youden's Laboratory Ranking Procedure was used to detect and reject data having a large systematic error associated with a particular laboratory. If a laboratory reported the majority of their data for a particular compound-water combination biased either high or low, compared to the other laboratories, this laboratory would fail the lab ranking procedure and all of its data would be rejected for that compound-water combination. Next, zero, negative and non-detected data were rejected. Finally, the Thompson outlier test was used to reject individual outliers.

For the study, the IMVS computer programs rejected 17.0% of the 10,620 data points submitted. The percentage of rejected data did not vary significantly among the three water types. Reagent water had the lowest number of rejected data, 519 (4.9%), ground water had 695 (6.5%) rejected data and leachate had 595 (5.6%) rejected data.

The average number of rejected data for all 59 compounds was 30.7. The compound with the fewest number of rejected data was safrole at 6 and the compound with the highest number of rejected data was chlorobenzilate at 56. For purposes of this report, a compound was considered to have excessive outliers if the number of rejected data was greater than 45 (25% of the 180 submitted data points). Using this guideline, only five compounds fell in this category: 2-sec-butyl-4,6-dinitrophenol (49), methapyrilene (51), tris(2,3-dibromopropyl)phosphate (51), 1,3,5-

trinitrobenzene (53) and chlorobenzilate (56).

The laboratory ranking procedure accounted for 90% of the rejected data points or 1620 out of the 1809 total data points rejected. Thompson's individual outlier test accounted for the remaining 10% of the rejected data.

Of the ten laboratories participating in the study, Lab 2, Lab 4, and Lab 5, accounted for 46.5% of the rejected data. Laboratory 2 had the highest number of rejected data, 297, which resulted from their poor performance in the lab ranking test. Four laboratories (6,8,9, and 10) had 5% or less of their submitted data rejected.

The overall precision, expressed as percent relative standard deviation (%RSD) was 22% for reagent water, 23% for the leachate matrix and 23% for ground water. These averages did not include 3-methylcholanthrene (104%), or chlorobenzilate (68%). Aniline had the lowest mean %RSD across water types at 9% while chlorobenzilate had the highest at 68%. Nine compounds: a,a-dimethylphenethylamine (29%), p-nitroaniline (34%), methapyrilene (27%), benzidine (54%), tris(2,3-dibromopropyl)phosphate (52%), chlorobenzilate (68%), 7,12-dimethylbenz(a)anthracene (50%), dibenzo(a,e)pyrene (49%), and 4-nitroquinoline-N-oxide (27%) had mean overall %RSDs greater than 25% across all water types.

The average single-analyst precision, expressed as %RSD-SR, was 10% for reagent water, 13% for ground water and 10% for leachate. These precisions, lower than overall RSDs, were expected since within-laboratory precision was expected to be better than between-laboratory precision. The mean %RSD-SR ranged from 6% to 33% for all water types.

Twenty-one compounds showed statistically significant matrix effects in the ground water sample yet no matrix effects were found in the leachate.

Ten of these compounds: N-nitrosomethylethylamine, N-nitrosopyrrolidine, 2-nitroaniline, 4-aminobiphenyl, benzyl alcohol, 2-methylphenol, acetophenone, N-nitrosopiperidine, 2,4,5-trichlorophenol, and 1,2-dinitrobenzene had statistically significant matrix effects that were not considered to be of practical importance. These compounds exhibited similar analyte recoveries for the four highest sample concentrates (Ampuls 1,2,3,4) whether extracted from reagent water or ground water. Their overall precisions were also similar. The two lowest sample concentrates for ground water (Ampuls

5,6), however, showed marked differences in recovery versus reagent water. The precisions of these two ampuls were also poorer than those of reagent water. The regression equations generated by the IMVS series of programs used a weighted least squares approach thus giving low concentration samples equal weight with high concentration samples. The poorer recoveries with the low concentration samples from the ground water have adversely affected the statistics even though the other concentration levels showed comparable recoveries to the reagent water. Since these differences were only observed at the low end of the concentration range, the matrix effects were not deemed of practical significance. In all cases the mean recoveries for these compounds were no greater than 6% different between reagent water and ground water.

One compound, *a,a*-dimethylphenethylamine, showed a statistically significant matrix effect, but was judged to be of no practical significance. The retained data for *a,a*-dimethylphenethylamine in all three waters were extremely variable with the majority of the %RSDs between 50%-89%. Even the reagent water recoveries were variable. This extreme variability between the mean recoveries and the overall precision makes statistical inference difficult for this compound.

The remaining ten compounds: naphthylamine, benzidine, 3,3'-dimethylbenzidine, 4,4'-methylene bis(2-chloroaniline), 3,3'-dimethoxybenzidine, pronamide, tris(2,3-dibromopropyl)phosphate, kepone, N-nitrosomorpholine, and 4-methylphenol had statistically significant matrix effects that were judged to be of practical importance. In all cases ground water was the affected water type. These compounds had calculated mean recoveries which differed more than 10% from reagent water even though the precision values were similar. Also, the leachate mean recoveries paralleled the reagent water mean recoveries which supported the view that the ground water matrix was indeed different from reagent water.

Conclusions

The objective of this study was to characterize the performance of SW-846 Methods 8270/3510 in terms of recovery, overall precision, single analyst precision and the effect of water types on recovery and precision. Through the IMVS series of computer programs, statistical analyses of 10,620 analytical values

provided estimates of recovery and precision expressed as regression equations. These equations regressed recovery against the true (known) value and overall precision and single-analyst precision against mean recovery for each compound in a given water type. The linear regression equations obtained from this study, and presented in Table 1, can be used to predict the recovery and precision of the compounds studied at concentrations in the ranges investigated in this study.

For the entire project, the IMVS computer programs rejected 17% of the 10,620 data points submitted. The percentage of rejected data did not vary significantly across water types.

The pooled mean recovery for 58 compounds, in reagent water, excluding 3-methylcholanthrene, was 77.9% with a standard deviation of 13.3%. Three compounds had recoveries less than 60%. They were chlorobenzilate (26.9%), 7,12-dimethylbenz(a)anthracene (51.3%) and 1,3,5-trinitrobenzene (54.8%).

The overall standard deviation expressed as the percent relative standard deviation (%RSD) ranged from 9% to 58% across the three waters tested, except for 3-methylcholanthrene at 104%. The single-analyst precision expressed as the percent relative standard deviation (%RSD-SR) ranged from 6% to 33% across the three waters tested. In all cases, the highest %RSD-SR (poorest precision) was associated with the lowest Youden pair concentration.

Statistical comparisons of the effect of water type were performed on all samples. The ten compounds listed below were found to have statistically significant matrix effects that are of practical significance.

2-Naphthylamine
Benzidine
3,3'-Dimethylbenzidine
4,4'-Methylene bis(2-chloroaniline)
3,3'-Dimethoxybenzidine
Pronamide
Tris(2,3-dibromopropyl)phosphate
Kepone
N-Nitrosomorpholine
4-Methylphenol

Several compounds did not perform well during this method validation study. 3-Methylcholanthrene was unstable in the methylene chloride standard mixture provided with the study. All data generated for this compound were unusable. Chlorobenzilate had the lowest recovery, 27%, from reagent water of the 59 compounds tested. The uniform low recoveries among all six concentration levels point to poor extraction efficiency.

Compounds exhibiting a high degree of variability as a result of poor chromatography were 7,12-dimethylbenz(a)anthracene, 1,3,5-trinitrobenzene, tris(2,3-dibromopropyl)phosphate, dibenzo(a,e)pyrene, benzidine, 3,3'-dimethylbenzidine, 3,3'-dimethoxybenzidine, and 4-nitroquinoline-N-oxide.

Seven compounds: N-nitrosodibutylamine, 1,2-diphenylhydrazine, 2-methylnaphthalene, 1,2,4,5-tetrachlorobenzene, dibenzofuran, dihydrosafrol and safrole appeared to have saturated the detector at the highest Youden concentration samples as indicated by low mean recoveries for the high concentration samples and uniform recoveries for the middle and low concentration samples.

Recommendations

SW-846 Methods 8270/3510 are recommended for the analyses of Appendix IX compounds with the exception of the analytes listed in Table and with the following observations.

- * The strongly acidic and basic conditions encountered during the extraction procedure were postulated to have caused chlorobenzilate, methylmethane sulfonate, N-nitrosomorpholine, hexachloropropene and 7,12-dimethylbenz(a)anthracene to have poor extraction efficiencies. Milder extraction conditions may improve extraction recoveries for these compounds.
- * Stability studies revealed that aromatic amines were reacting with the solvent, acetone, to form Schiff bases. Changing the solvent to methanol remedied the immediate problem but this situation should be addressed in the final method write-up.
- * 3-Methylcholanthrene (molecular weight 268) dehydrogenated in methylene chloride to form a compound whose molecular weight was 266. The causes of this reaction have not been investigated. Quantitation by GC/MS is not possible when a compound's structure changes in standard solution. Further analytical work is recommended to examine this reaction. Analysts should be alerted to this situation in the final method write-up.
- * SW-846 Method 8270 recommends use of a DB-5 fused silica column with a film thickness of 1.0 μm . This preliminary analytical work performed under another contract used DB-5 column with a film thickness of 2.5 μm . Further

analytical work is recommended to determine which film thickness would offer the greater advantages.

Table 1. Study 36, SW 846 Method 8270, Semi-Volatiles, GC-MS Weighted Linear Regression Equations for Mean Recovery and Precision (in UG/L)

WATER TYPE	M-NITROSOMETHYLETHYLAMINE (9.60 - 480.00)	M-NITROSODIETHYLAMINE (9.60 - 480.00)	ANILINE (9.60 - 480.00)	M-NITROSOPYRROLIDINE (9.60 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.154X + 0.16	SR = 0.064X + 0.54	SR = 0.080X + 0.42	SR = 0.088X + 0.29
OVERALL PRECISION	S = 0.228X + 0.90	S = 0.160X + 1.34	S = 0.226X + 0.32	S = 0.235X + 0.69
MEAN RECOVERY	X = 0.749C - 1.91	X = 0.843C - 2.14	X = 0.674C - 0.89	X = 0.742C - 2.17
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.102X + 0.04	SR = 0.094X + 0.01	SR = 0.117X + 0.30	SR = 0.172X - 0.49
OVERALL PRECISION	S = 0.128X + 0.98	S = 0.147X + 1.05	S = 0.115X + 1.80	S = 0.251X - 0.51
MEAN RECOVERY	X = 0.779C - 0.09	X = 0.828C - 0.54	X = 0.756C - 0.56	X = 0.782C - 0.07
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.087X + 0.37	SR = 0.074X + 0.41	SR = 0.083X + 0.45	SR = 0.097X + 0.21
OVERALL PRECISION	S = 0.137X + 1.54	S = 0.147X + 1.51	S = 0.159X + 0.28	S = 0.227X - 0.35
MEAN RECOVERY	X = 0.744C - 1.24	X = 0.839C - 2.38	X = 0.818C - 1.15	X = 0.863C - 2.29
WATER TYPE	A.A-DIMETHYLPHENETHYLAMINE (20.00 - 480.00)	P-CHLOROANILINE (20.00 - 480.00)	2-NITROANILINE (20.00 - 480.00)	M-NITROSODI-N-BUTYLAMINE (9.60 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.167X + 2.04	SR = 0.053X + 1.36	SR = 0.085X - 0.31	SR = 0.075X + 1.52
OVERALL PRECISION	S = 0.353X + 0.96	S = 0.139X + 0.07	S = 0.178X + 0.15	S = 0.203X + 0.48
MEAN RECOVERY	X = 1.008C - 10.16	X = 0.821C - 1.35	X = 0.737C - 7.75	X = 0.985C + 4.34
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.372X - 1.20	SR = 0.077X + 0.49	SR = 0.085X + 0.14	SR = 0.116X - 0.89
OVERALL PRECISION	S = 0.472X + 2.50	S = 0.124X + 3.59	S = 0.178X - 0.72	S = 0.201X - 0.64
MEAN RECOVERY	X = 0.780C - 9.32	X = 0.785C - 1.28	X = 0.712C - 5.54	X = 0.941C + 6.38
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.148X + 1.91	SR = 0.066X + 0.15	SR = 0.068X - 0.09	SR = 0.084X + 0.10
OVERALL PRECISION	S = 0.390X + 2.66	S = 0.125X + 0.10	S = 0.134X - 0.25	S = 0.179X - 0.11
MEAN RECOVERY	X = 0.890C - 12.40	X = 0.865C - 1.46	X = 0.803C - 8.74	X = 0.990C + 4.37
WATER TYPE	3-NITROANILINE (20.00 - 480.00)	2-NAPHTHYLAMINE (9.60 - 480.00)	S-NITRO-O-TOLUIDINE (9.60 - 480.00)	P-NITROANILINE (48.00 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.078X + 2.28	SR = 0.123X + 1.45	SR = 0.090X + 1.37	SR = 0.073X + 7.96
OVERALL PRECISION	S = 0.257X + 1.59	S = 0.290X + 0.47	S = 0.250X + 0.60	S = 0.457X - 7.08
MEAN RECOVERY	X = 0.920C - 5.33	X = 0.890C - 2.71	X = 0.852C - 1.80	X = 0.934C - 6.71
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.123X + 0.22	SR = 0.144X + 0.75	SR = 0.139X + 0.22	SR = 0.134X + 0.33
OVERALL PRECISION	S = 0.194X + 1.70	S = 0.241X + 2.12	S = 0.160X + 1.20	S = 0.192X + 3.99
MEAN RECOVERY	X = 0.865C - 5.51	X = 0.775C - 4.84	X = 0.812C - 1.29	X = 0.803C - 6.64
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.085X + 0.87	SR = 0.089X + 0.63	SR = 0.088X + 0.62	SR = 0.114X + 2.99
OVERALL PRECISION	S = 0.248X + 0.18	S = 0.207X + 1.70	S = 0.315X + 0.04	S = 0.371X - 5.21
MEAN RECOVERY	X = 0.945C - 5.32	X = 0.911C - 3.34	X = 0.958C - 2.34	X = 0.871C - 8.11

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

Table 1. (Continued)

WATER TYPE	1,2-DIPHENYLHYDRAZINE	4-AMINODIPHENYL	METHAPYRILENE	BENZIDINE
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(9.60 - 480.00)	(9.60 - 480.00)	(48.00 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.055X + 0.58	SR = 0.085X + 1.08	SR = 0.103X + 1.37	SR = 0.187X - 1.83
OVERALL PRECISION	S = 0.266X - 0.90	S = 0.254X + 0.99	S = 0.314X + 1.16	S = 0.417X - 1.36
MEAN RECOVERY	X = 0.766C + 1.61	X = 0.845C - 1.46	X = 1.003C - 5.69	X = 1.074C - 27.10
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.167X - 1.00	SR = 0.112X + 0.59	SR = 0.128X - 0.21	SR = 0.335X + 1.54
OVERALL PRECISION	S = 0.254X - 1.23	S = 0.249X + 1.32	S = 0.302X - 0.04	S = 0.733X - 0.28
MEAN RECOVERY	X = 0.744C + 1.16	X = 0.820C - 4.30	X = 0.854C - 1.51	X = 0.609C - 27.60
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.065X + 0.09	SR = 0.081X + 0.46	SR = 0.232X + 0.36	SR = 0.320X - 0.80
OVERALL PRECISION	S = 0.218X - 0.64	S = 0.167X + 1.34	S = 0.298X + 1.93	S = 0.473X + 7.50
MEAN RECOVERY	X = 0.765C + 1.82	X = 0.895C - 1.32	X = 0.799C - 4.52	X = 1.028C - 28.00
WATER TYPE	P-DIMETHYLAMINOAZOBENZENE	3,3-DIMETHYLBENZIDINE	4,4-METHYLENE BIS(2-CHL 3,3-DITHIOXYBENZIDINE)	
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(48.00 - 480.00)	(48.00 - 480.00)	(48.00 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.062X + 0.57	SR = 0.118X + 3.26	SR = 0.065X + 2.64	SR = 0.087X + 3.64
OVERALL PRECISION	S = 0.221X - 0.20	S = 0.208X + 2.46	S = 0.203X - 3.68	S = 0.202X + 6.02
MEAN RECOVERY	X = 0.856C - 1.75	X = 0.979C - 18.00	X = 1.016C - 12.67	X = 0.908C - 28.71
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.120X - 0.49	SR = 0.154X + 2.44	SR = 0.091X + 3.26	SR = 0.163X + 3.32
OVERALL PRECISION	S = 0.127X - 0.94	S = 0.313X + 5.56	S = 0.172X + 2.28	S = 0.344X + 3.51
MEAN RECOVERY	X = 0.828C - 1.05	X = 0.788C - 28.75	X = 0.878C - 17.20	X = 0.796C - 30.94
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.059X + 0.15	SR = 0.084X + 7.90	SR = 0.094X + 0.51	SR = 0.078X + 4.78
OVERALL PRECISION	S = 0.155X + 1.01	S = 0.178X + 5.08	S = 0.144X - 0.39	S = 0.217X + 7.79
MEAN RECOVERY	X = 0.856C - 1.44	X = 0.957C - 17.91	X = 0.932C - 7.59	X = 0.961C - 24.97
WATER TYPE	2-METHYLNAPHTHALENE	METHYL METHANE SULFONATE	M-NITROSOPHOLINE	4-METHYLPHENOL
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(9.60 - 480.00)	(9.60 - 480.00)	(20.00 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.063X + 0.49	SR = 0.077X - 0.11	SR = 0.060X + 0.87	SR = 0.061X + 2.26
OVERALL PRECISION	S = 0.153X - 0.00	S = 0.087X + 0.66	S = 0.137X + 0.84	S = 0.135X + 2.80
MEAN RECOVERY	X = 0.797C + 1.59	X = 0.643C - 0.71	X = 0.652C - 1.22	X = 0.782C - 1.87
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.103X - 0.55	SR = 0.096X + 1.28	SR = 0.090X + 0.50	SR = 0.039X + 3.85
OVERALL PRECISION	S = 0.153X - 0.14	S = 0.175X + 0.69	S = 0.203X - 0.56	S = 0.141X + 4.02
MEAN RECOVERY	X = 0.806C + 1.86	X = 0.609C - 0.67	X = 0.780C + 0.18	X = 0.778C + 13.23
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.097X - 0.39	SR = 0.078X + 0.27	SR = 0.081X + 0.39	SR = 0.073X + 0.75
OVERALL PRECISION	S = 0.182X - 0.43	S = 0.146X + 0.03	S = 0.261X + 0.07	S = 0.244X - 0.82
MEAN RECOVERY	X = 0.802C + 1.34	X = 0.682C - 1.11	X = 0.710C - 0.81	X = 0.834C - 1.48

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

Table 1. (Continued)

WATER TYPE	2,6-DICHLOROPHENOL	1,2,4,5-TETRACHLOROBENZEN ISOSAPROLE	DIBENZOPURAN
APPLICABLE CONC. RANGE	(20.00 - 480.00)	(9.60 - 480.00)	(9.60 - 480.00)
REAGENT WATER			
SINGLE-ANALYST PRECISION	SR = 0.049X + 0.57	SR = 0.097X - 0.27	SR = 0.092X - 0.49
OVERALL PRECISION	S = 0.096X + 1.90	S = 0.157X + 0.37	S = 0.143X - 0.16
MEAN RECOVERY	X = 0.858C + 0.89	X = 0.694C + 1.47	X = 0.824C + 2.05
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.056X + 0.85	SR = 0.071X + 0.05	SR = 0.033X + 0.31
OVERALL PRECISION	S = 0.136X - 0.13	S = 0.093X + 1.16	S = 0.113X + 0.08
MEAN RECOVERY	X = 0.833C + 2.90	X = 0.647C + 1.19	X = 0.736C + 2.18
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.064X + 0.08	SR = 0.073X + 0.49	SR = 0.066X + 0.11
OVERALL PRECISION	S = 0.196X - 0.82	S = 0.184X + 0.66	S = 0.207X - 0.62
MEAN RECOVERY	X = 0.892C - 0.84	X = 0.687C + 1.33	X = 0.813C + 1.46
WATER TYPE	N-NITRODIPHENYLAMINE	PROMAMIDE	2-ACETYLAMINOFLOURENE
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(20.00 - 480.00)	(48.00 - 480.00)
REAGENT WATER			
SINGLE-ANALYST PRECISION	SR = 0.141X - 0.82	SR = 0.104X + 1.07	SR = 0.107X + 3.79
OVERALL PRECISION	S = 0.187X - 0.84	S = 0.209X - 1.43	S = 0.199X + 1.99
MEAN RECOVERY	X = 0.911C + 1.52	X = 0.925C - 7.75	X = 1.003C - 14.34
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.075X + 0.25	SR = 0.063X + 1.20	SR = 0.104X + 1.64
OVERALL PRECISION	S = 0.166X - 0.07	S = 0.171X - 0.05	S = 0.329X - 6.21
MEAN RECOVERY	X = 0.936C + 1.99	X = 0.766C - 0.02	X = 1.146C - 14.00
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.056X + 0.55	SR = 0.074X + 0.71	SR = 0.069X + 4.26
OVERALL PRECISION	S = 0.201X - 0.65	S = 0.232X + 0.06	S = 0.173X + 5.02
MEAN RECOVERY	X = 0.958C - 0.04	X = 1.050C - 8.80	X = 1.076C - 13.69
WATER TYPE	TRIS(2,3-DIBROMOPROPYL) P BENZYL ALCOHOL	DINHYDROPROK	1,3-DINITROBENZENE
APPLICABLE CONC. RANGE	(48.00 - 480.00)	(20.00 - 480.00)	(48.00 - 480.00)
REAGENT WATER			
SINGLE-ANALYST PRECISION	SR = 0.182X + 1.60	SR = 0.053X + 2.70	SR = 0.092X + 3.72
OVERALL PRECISION	S = 0.330X + 8.13	S = 0.099X + 3.49	S = 0.129X + 4.96
MEAN RECOVERY	X = 1.044C - 36.30	X = 0.730C - 2.24	X = 0.900C - 7.87
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.053X + 9.92	SR = 0.089X - 0.22	SR = 0.124X - 2.51
OVERALL PRECISION	S = 0.501X + 1.75	S = 0.127X + 0.02	S = 0.170X - 2.13
MEAN RECOVERY	X = 0.741C - 16.44	X = 0.744C + 1.38	X = 0.817C - 0.05
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.134X + 1.48	SR = 0.088X - 0.04	SR = 0.050X + 2.56
OVERALL PRECISION	S = 0.737X - 9.22	S = 0.245X + 0.10	S = 0.200X + 1.11
MEAN RECOVERY	X = 1.611C - 61.48	X = 0.800C - 3.63	X = 0.999C - 11.38
X = MEAN RECOVERY			
C = TRUE VALUE FOR THE CONCENTRATION			

Table 1. (Continued)

WATER TYPE	CHLOROBENZILATE	KEPONE	2-METHYLPHENOL	ACETOPHENONE
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(20.00 - 480.00)	(20.00 - 480.00)	(9.60 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.169X + 0.76	SR = 0.086X + 1.31	SR = 0.093X - 0.14	SR = 0.113X - 0.51
OVERALL PRECISION	S = 0.797X + 0.46	S = 0.129X + 0.99	S = 0.184X + 1.26	S = 0.152X + 0.56
MEAN RECOVERY	X = 0.270C - 0.06	X = 0.861C - 5.28	X = 0.771C - 1.82	X = 0.835C - 0.13
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.088X + 0.43	SR = 0.234X + 0.96	SR = 0.103X - 0.01	SR = 0.093X - 0.22
OVERALL PRECISION	S = 0.656X + 0.00	S = 0.354X + 1.21	S = 0.119X + 2.12	S = 0.148X + 1.90
MEAN RECOVERY	X = 0.354C - 1.08	X = 0.457C - 3.46	X = 0.761C + 4.99	X = 0.806C + 2.07
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.255X + 0.01	SR = 0.143X + 0.42	SR = 0.051X + 2.36	SR = 0.072X - 0.04
OVERALL PRECISION	S = 0.569X + 0.08	S = 0.152X + 2.68	S = 0.149X + 1.06	S = 0.120X + 0.49
MEAN RECOVERY	X = 0.211C - 0.55	X = 0.829C - 5.55	X = 0.729C - 1.39	X = 0.788C + 0.03
WATER TYPE	M-NITROPIPERINE	HEXACHLOROPROPENE	SAFROLE	2,4,5-TRICHLOROPHENOL
APPLICABLE CONC. RANGE	(9.60 - 480.00)	(9.60 - 480.00)	(20.00 - 480.00)	(46.00 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.070X - 0.04	SR = 0.127X - 0.05	SR = 0.109X - 0.55	SR = 0.053X + 0.27
OVERALL PRECISION	S = 0.130X - 0.18	S = 0.300X + 0.62	S = 0.158X + 0.64	S = 0.122X + 0.25
MEAN RECOVERY	X = 0.926C - 0.72	X = 0.625C - 1.51	X = 0.812C - 0.14	X = 0.924C - 9.27
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.136X + 0.14	SR = 0.051X + 1.30	SR = 0.142X - 1.21	SR = 0.089X - 2.87
OVERALL PRECISION	S = 0.133X + 2.33	S = 0.243X + 0.72	S = 0.218X - 0.78	S = 0.162X - 4.12
MEAN RECOVERY	X = 0.863C + 3.41	X = 0.647C - 2.48	X = 0.740C + 2.20	X = 0.872C + 0.06
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.116X - 0.08	SR = 0.078X + 0.35	SR = 0.061X + 0.82	SR = 0.060X + 1.02
OVERALL PRECISION	S = 0.142X + 0.38	S = 0.224X + 0.76	S = 0.161X + 1.51	S = 0.108X + 4.25
MEAN RECOVERY	X = 0.840C - 0.25	X = 0.585C - 1.40	X = 0.756C + 2.13	X = 0.647C - 2.05
WATER TYPE	1,4-DINITROBENZENE	PENTACHLOROBENZENE	2,3,4,6-TETRACHLOROPHENOL	DIPHENYLAMINE
APPLICABLE CONC. RANGE	(46.00 - 480.00)	(9.60 - 480.00)	(46.00 - 480.00)	(9.60 - 480.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.065X - 1.00	SR = 0.052X - 0.07	SR = 0.049X + 2.46	SR = 0.109X - 0.51
OVERALL PRECISION	S = 0.102X + 2.81	S = 0.078X + 1.02	S = 0.087X + 1.92	S = 0.133X + 0.74
MEAN RECOVERY	X = 0.929C - 10.01	X = 0.802C + 0.24	X = 0.888C - 7.55	X = 0.896C - 0.86
GROUND WATER				
SINGLE-ANALYST PRECISION	SR = 0.112X - 2.99	SR = 0.083X - 0.30	SR = 0.068X + 1.39	SR = 0.163X - 1.31
OVERALL PRECISION	S = 0.110X - 0.25	S = 0.119X + 0.87	S = 0.164X + 4.67	S = 0.227X - 0.89
MEAN RECOVERY	X = 0.874C - 4.95	X = 0.790C + 0.19	X = 0.845C - 4.77	X = 0.880C + 0.51
LEACHATE				
SINGLE-ANALYST PRECISION	SR = 0.066X + 3.20	SR = 0.054X + 0.32	SR = 0.074X + 2.02	SR = 0.065X + 0.86
OVERALL PRECISION	S = 0.091X + 4.14	S = 0.116X + 1.30	S = 0.112X + 4.06	S = 0.141X + 0.85
MEAN RECOVERY	X = 0.914C - 7.18	X = 0.740C + 0.83	X = 0.802C - 1.83	X = 0.894C + 0.55

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

Table 1. (Continued)

PENTACHLOROMITROBENZENE		2-SEC-BUTYL-4,6-DINITRO 7,12-DIMETHYLBENZ(A)ANTHR 3-METHYCHOLANTHRENE	
WATER TYPE	(46.00 - 480.00)	(46.00 - 480.00)	(20.00 - 480.00)
REAGENT WATER	SR = 0.132X - 4.79	SR = 0.117X - 0.64	SR = 0.254X - 1.71
SINGLE-ANALYST PRECISION	S = 0.092X + 4.15	S = 0.151X + 6.36	S = 0.483X + 5.74
OVERALL PRECISION	X = 0.886C - 5.41	X = 0.975C - 18.61	X = 0.524C - 0.71
MEAN RECOVERY			X = 8.244C - 58.86
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.176X - 3.92	SR = 0.135X - 1.09	SR = 0.221X + 0.40
OVERALL PRECISION	S = 0.205X - 3.30	S = 0.203X - 1.93	S = 0.533X + 2.68
MEAN RECOVERY	X = 0.817C - 2.50	X = 0.974C - 16.31	X = 0.398C - 2.05
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.067X + 2.26	SR = 0.162X + 1.56	SR = 0.302X - 1.37
OVERALL PRECISION	S = 0.071X + 4.50	S = 0.219X + 1.81	S = 0.482X - 0.14
MEAN RECOVERY	X = 0.821C + 0.75	X = 1.030C - 23.00	X = 0.621C - 4.76
DIBENZO(A,E)PYRENE		ETHYL METHANE SULFONATE	1,2-DIBROMO-3-CHLOROPROPA 1,2-DINITROBENZENE
WATER TYPE	(46.00 - 480.00)	(9.60 - 480.00)	(20.00 - 480.00)
REAGENT WATER	SR = 0.129X + 6.01	SR = 0.097X - 0.40	SR = 0.124X + 0.20
SINGLE-ANALYST PRECISION	S = 0.418X + 9.30	S = 0.121X + 0.51	S = 0.196X + 1.00
OVERALL PRECISION	X = 1.094C - 16.84	X = 0.803C + 0.88	X = 0.703C - 7.62
MEAN RECOVERY			SR = 0.054X - 0.17
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.357X - 4.16	SR = 0.098X - 0.07	S = 0.214X + 0.84
OVERALL PRECISION	S = 0.367X + 0.88	S = 0.137X + 0.84	X = 0.632C - 6.78
MEAN RECOVERY	X = 0.951C - 17.70	X = 0.899C + 0.65	SR = 0.145X - 0.66
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.487X - 5.48	SR = 0.059X + 0.34	S = 0.176X + 0.03
OVERALL PRECISION	S = 0.678X + 1.92	S = 0.068X + 0.88	X = 0.645C - 6.64
MEAN RECOVERY	X = 1.899C - 54.63	X = 0.797C + 0.17	SR = 0.059X + 1.30
1,3,5-TRINITROBENZENE		DIALATE	4-NITROBUTANOLINE-N-OXIDE
WATER TYPE	(46.00 - 480.00)	(9.60 - 480.00)	(46.00 - 480.00)
REAGENT WATER	SR = 0.256X - 5.39	SR = 0.102X - 0.50	SR = 0.172X - 0.33
SINGLE-ANALYST PRECISION	S = 0.288X + 3.82	S = 0.164X + 0.42	S = 0.286X + 5.47
OVERALL PRECISION	X = 0.637C - 9.21	X = 0.793C + 0.09	X = 0.950C - 30.73
MEAN RECOVERY			
GROUND WATER			
SINGLE-ANALYST PRECISION	SR = 0.222X - 0.89	SR = 0.177X - 1.22	SR = 0.269X - 2.44
OVERALL PRECISION	S = 0.383X + 0.83	S = 0.183X - 0.83	S = 0.301X + 2.21
MEAN RECOVERY	X = 0.521C - 11.52	X = 0.823C + 0.11	X = 0.905C - 27.46
LEACHATE			
SINGLE-ANALYST PRECISION	SR = 0.155X + 4.67	SR = 0.073X - 0.31	SR = 0.129X + 2.00
OVERALL PRECISION	S = 0.440X + 2.17	S = 0.166X - 0.46	S = 0.220X + 7.69
MEAN RECOVERY	X = 0.576C - 8.78	X = 0.856C + 0.23	X = 0.927C - 29.15

X = MEAN RECOVERY
 C = TRUE VALUE FOR THE CONCENTRATION
 MESSAGE SUMMARY: MESSAGE NUMBER - COUNT

Table 2. RCRA Appendix IX Analytes not Included in Method 8270	
<i>Compound</i>	<i>Reason for Exclusion from Study</i>
<i>p</i> -Benzoquinone	No recovery at 20 and 600 ppb.
Benzenethiol	Unstable in methanol or acetone
Pentachloroethane	Decomposes to tetrachloroethene with less than 2% recovery at 600 ppb.
Resorcinol	No recovery at 20 and 600 ppb.
Hexachlorocyclopentadiene	Unstable by thermal decomposition and solvent reactivity
<i>p</i> -Naphthoquinone	No recovery at 20 and 600 ppb.
Aramite	No recovery at 50 ppb. 6 % recovery at 600 ppb. Four peaks are produced from 96% pure material.
Hexachlorophene	No recovery at 50 ppb. 18% recovery at 600 ppb.
Dibenzo (a,h) pyrene	Insoluble in spiking solvents and commercially unavailable.
Dibenzo (a,i) pyrene	No recovery at 50 ppb. Detection limit approximately 150 ppb.
<i>n</i> -Nitrosodimethylamine	Compound coelutes with solvent 30% recovery at 10 ppb.
1-Naphthylamine	20% recovery at 10 ppb.
Phthalic anhydride	No recovery at 100 ppb.

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The complete report, entitled "USEPA Method Study 36 SW-846 Methods 8270/3510 GC/MS Method for Semivolatile Organics: Capillary Column Technique; Separatory Funnel Liquid-Liquid Extraction," (Order No. PB 89-190 581/AS; Cost: \$28.95, subject to change) will be available only from:

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