Research and Development

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Project Summary

Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846 Method 3510

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As part of an ongoing effort in a hierarchical scheme to demonstrate the applicability of present U.S. EPA analysis methods and/or to develop new methodology, the suitability of SW-846 Method 3510 for the determination of hazardous, organic compounds was evaluated. The compounds selected for this study included all semivolatile organic compounds on the borderline chemicals list, proposed Appendix IX list, Appendix VIII, and Michigan lists that were not priority pollutants and were shown to be amenable to gas chromatographic-mass spectrometric (GC-MS) analysis. After eliminating 13 compounds for various reasons, the extraction efficiency from water and seven-day aqueous stability of the remaining 153 compounds were determined.

Spiking mixtures were designed to eliminate coelutions and avoid compound interactions. Spiked distilled water samples and blanks were extracted using Method 3510 procedures except that a two stage liquid-liquid equilibration procedure (one equilibration with 300 mL methylene chloride under acid conditions and another equilibration with 300 mL methylene chloride under basic conditions) was substituted for the six 60-mL equilibrations specified in SW-846 Method 3510. The concentrated

extracts were analyzed by fused silica capillary column gas chromatography with flame ionization detection (GC-FID). Two separate GC-FID systems were used, one for acid/neutral mixtures and the other for base/neutral mixtures.

On the basis of extraction efficiency and seven-day aqueous stability results obtained at the 500 µg/L concentration level, 115 of the 153 compounds evaluated in this study are suitable for inclusion in the scope of SW-846 Method 3510 and in Method 8270 validation studies. Changes were recommended to the list of compounds to be included in Appendix IX as a result of this work.

This Project Summary was developed by EPA's Environmental Monitoring and Support 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 Resource Conservation and Recovery Act (RCRA) requires operators of land-based hazardous waste facilities to analyze ground water for approximately 375 hazardous constituents listed in Appendix VIII to 40 CFR Part 261. While some of the constituents are analytes included in EPA combined gas chromatographic-mass

spectrometric (GC-MS) screening procedures (Method 8240 for purgeable compounds and Method 8270 for semivolatile compounds), experience has shown that analysis is extremely difficult or impossible for many of the other constituents.

Another list of compounds, the Michigan list, was generated in response to a petition by the State of Michigan to expand Appendix VIII. EPA has proposed an amendment of Appendix VIII by the addition of over 100 Michigan list organic compounds (Federal Register, December 21, 1984).

A 1984 RCRA amendment requires EPA to take final action on all permits for land-disposal facilities by November, 1988. To facilitate meeting this deadline, EPA prepared a revised list representing EPA's current guidance on which Appendix VIII and Superfund hazardous substances constituents might realistically be determined by routine GC-MS screening procedures. As a result of this meeting, two lists of compounds to be considered in the current work assignment were proposed for testing groundwater for permit applications, Appendix IX, and a second, smaller list of borderline chemicals, compounds for which EPA has received conflicting data with regard to analytical feasibility (Federal Register, July 24, 1986).

In 1985 Battelle began experiments aimed at establishing a hierarchical approach to the development and validation of analytical methods for the determination of organic compounds in wastes. The first experiments, Work Assignment 1-04 of this contract, assessed the GC-MS suitability of RCRA Appendix VIII and Michigan list compounds. From these data, a third list of compounds to be considered in the current work assignment was identified, those semivolatile compounds from Appendix VIII and the Michigan list that were found suitable for GC-MS determination but were not included in the proposed Appendix IX list or the borderline chemicals list.

The objectives of this work assignment, 2-08, were 1) to assess the suitability of SW-846 Method 3510 for liquid-liquid extraction of semivolatile compounds that are on the proposed Appendix IX list, the borderline chemicals list, or list of compounds found suitable for GC-MS determination in Work Assignment 1-04, but are not priority pollutants and 2) to recommend which of these compounds to include on Appendix IX.

Experimental Approach

The compounds included in the original scope of Work Assignment 2-08 for Method 3510 performance testing were all compounds, other than priority pollutants, in Appendix VIII, the proposed Appendix IX, the list of borderline chemicals, and the Michigan list that had been determined in Work Assignment 1-04 to be amenable to GC-MS analysis or expected to be amenable to GC-MS analysis. After eliminating redundancies and eliminating 13 compounds for the reasons given in Table 1, 153 compounds remained for inclusion in the final scope of Work Assignment 2-08.

Stock solutions of each compound were prepared containing the compound at 2000 µg/mL. These stock solutions were combined to form 20 spiking mixtures, 100 µg/mL solutions, designed with the following considerations:

- Up to 15 compounds were included in each spiking mixture. This number of mixture components improved experimental efficiency by decreasing the number of required equilibrations, but did not result in extremely complicated GC-FID chromatograms.
- The compounds included in each mixture were selected to eliminate coelutions between compounds as well as between a compound and the internal standard.
- Acidic compounds, basic compounds, and organophosphate esters were segregated to avoid chemical interactions.
- Neutral compounds other than organophosphate esters were assigned to mixes containing acids, bases, or organophosphate esters in a manner to avoid compound coelutions.

For each spiking mixture, triplicate 1liter aliquots of distilled water were spiked with 5 mL of the mixture. A method blank, consisting of one liter of distilled water, was processed each day to detect interferences. The spiked water samples and method blanks were extracted according to Method 3510 except that during extraction under both basic and acidic conditions one 300-mL aliquot of methylene chloride was used instead of three 60-mL aliquots. Compounds were equilibrated by tumbling end-over-end for one hour in each extraction phase. This single exhaustive extraction with a larger volume of solvent provides equivalent compound recoveries with reduced

laboratory effort. The methylene chloric extracts obtained at acidic and basic μ were combined, concentrated to 5 m spiked with 250 μ g of internal standa (phenanthrene-DIO), and analyzed I GC-FID.

Two instruments, one for acidic/neuti mixtures and one for basic/neutr mixtures, were used to reduce tl possibility of compound interactions the injector or on the analytical column Both instruments were operated using the gas chromatographic operation conditions specified in SW-846 Meth-8270 and employed a 30-meter x 0. mm I.D. SPB-5 bonded-phas silicone-coated fused silica capilla column (0.25 µm film thickness) follow by flame ionization detection. Und these gas chromatographic operation conditions, dibenzo(a,e)-pyrene did n elute from the column.

Calibration standards, containing compounds at three concentration lever representing 100, 30, and 10% recover and the appropriate internal standard the 50 µg/mL concentration level, we prepared for each spiking mixture. The three calibration standards were analyzed before and after each set of wat extracts. Recoveries of each compour from water were calculated from the GC-FID data generated from wat extracts compared to the corresponding data from the calibration standards.

Aqueous stability experiments we conducted using spiked distilled wal samples that were prepared in triplica as described above, stored in 1 bottles at approximately 4°C and preceded from light for 7 days, a processed as detailed above. The samp bottles were thoroughly rinsed with the methylene chloride used for the initial extraction of the sample to minimic compound losses due to adsorption the walls of the bottle.

Results and Discussion

Evaluation of SW-846 Method 3510 Suitability.

An average recovery within the ran of 70 to 130 percent was considered indicator of SW-846 Method 35 suitability. Recoveries were within the range for 131 of the 153 compounds T 22 remaining compounds displaying average recoveries of less than percent are listed in Table 2.

Comments indicating possible reasc why compound recoveries were low than 70% are included in Table 2. L

recoveries for nine of the compounds can be at least partially attributed to unfavorable distribution coefficients (indicated by comment DC); compounds with suspected unfavorable distribution coefficients are 2,4-diaminoanisole sulfate, 2,4-diaminotoluene, ethyl carbamate, isosafrole, malononitrile, 1,2-phenylenediamine, 1,3-phenylenediamine, resorcinol, and thioacetamide. Low recoveries of most of the compounds can be partially or totally attributed to oxidation or hydrolysis during extraction (indicated by comments OE and HE, respectively).

Compound aqueous stability, defined as the recovery obtained Day 7 relative to that obtained on Day 0, of greater than 70% was considered acceptable. Acceptable aqueous stabilities were observed for 133 of the 153 compounds. The remaining 20 compounds, those displaying greater than 30 percent loss after storage for seven days, are listed in Table 3. Four of these compounds, benzenethiol, 4,4'-methylenebis(2-chloroaniline), pentachloroethane, and phthalic anhydride, also displayed unacceptable recoveries on Day 0 (see Table 3). Possible explanations for compound losses are given in the comments column of Table 3. In most cases, these compounds are expected to

hydrolyze or oxidize during storage. A compound's suitability for semivolatile analysis depends on its ability to be adequately stored, extracted, and analyzed. Table 4 lists compounds that produce both stability and Day 0 recovery values greater than 70%. While all 115 chemicals exhibited acceptable behavior, 13 compounds had poor reproducibility. Five compounds, 1,2:7,8-dibenzacridine, 3,3'-dimethoxybenzidine, p-dimethylaminoazobenzene, methyl parathion, and 4,4'oxydianiline, had RSD values of greater than 15 percent for recoveries on Day 0 and/or Day 7. Poor recovery reproducibility may result in a high method detection limit. Ten compounds, carbophenothion, 1,2:7,8-dibenzacridine, dichlorovos. 3.3'-dimethoxybenzidine. fenthion, 4,4'-methylenebis (N,Ndimethylaniline), N-nitrosopyrrolidine, 1,4-phenylenediamine, and sulfallate, had RSD values greater than 15 percent for GC-FID response factors from the highest level calibration standard (100 µg/mL). This factor becomes important in calculations of instrument and method ranges. These compounds may prove not to be suitable for semivolatile analysis when validation studies are performed.

Changes to Appendix IX

Fifty-four compounds on the originally proposed Appendix IX list should be included on the revised version of this list. Table 5A lists the 12 analytes from Appendix IX that were not judged suitable for Method 3510 extraction. Two of these compounds, malononitrile and pentachloroethane, have been shown to be suitable for further investigation under volatile analyte methodology and should not be excluded from Appendix IX without volatiles testing.

Recommended additions to Appendix IX come from the borderline chemicals and Appendix VIII lists. Only 3 compounds, diallate, 3-methylphenol, and o-toluidine, that did not also appear on the proposed list should be added to Appendix IX from the borderline chemicals list. Additionally, 10 compounds from Appendix VIII not included on proposed Appendix IX should now be added. The compounds recommended to be added from the borderline chemicals and Appendix VIII lists appear in Table 5B.

Conclusions and Recommendations

The following conclusions can be drawn from study results using analytes spiked into water at the 500 µg/L concentration level:

- Method 3510 provides acceptable extraction efficiency for 131 of the 153 compounds evaluated.
- Of the 153 compounds, 133 are stable in water for one week when protected from light and refrigerated.
- Hydrolysis, oxidation, adsorption, and/ or poor chromatographic performance prevent acceptable extraction efficiency or acceptable aqueous stability for the remaining compounds studied.
- On the basis of extraction efficiency and 7-day aqueous stability results, 115 of the 153 compounds evaluated in this study are suitable for inclusion in the scope of SW-846 Method 3510. These compounds derive from the following sources:

Proposed Appendix IX chemicals	54
Borderline chemicals	14
Michigan list chemicals	48
Appendix VIII chemicals not included on Appendix IX	<u>10</u>
Total citings	126
Dual Appendix IX/Borderline chemical citings	11
Total compounds found suitable	115

Based on Work Assignment 2-08 results, it is recommended that the 115 compounds with demonstrated acceptable extraction efficiency and 7-day aqueous stability should be included in future validation studies of SW-846 Method 8270. It would be valuable to conduct preliminary studies at concentrations less than 500 µg/L to detect potential concentration – related effects on compound recoveries and stability.

The following are recommendations for compounds to be included in Appendix IX:

- Of the 75 Appendix IX compounds studied, 10 should be removed from the list.
- Of the 14 compounds studied that are on the borderline list but not on the proposed Appendix IX list, 3 chemicals should be added to the Appendix IX list.
- Ten of the 22 Appendix VIII compounds studied that did not appear on the proposed Appendix IX list should be added to the Appendix IX list

Table 1. Compounds Eliminated from Method 3510 Performance Testing No. Compound Reason 1 1-Amino-2-methylanthraquinone Not available 2 Azinphos-ethyl Not available 3 Benzoic acid Derivatization required 1,2:5,6-Dibenzacridine Not available Excessively long elution time 5 Dibenzo(a,e)pyrene 6 Dibenzo(a,i)pyrene Not available Dibenzo(a,h)pyrene Not available 8 2,4-Dichlorophenoxyacetic acid Derivatization required a,a-Dimethylphenethylamine N,N-isomer inadvertently tested g 10 Dioxathion Standard on hand had decomposed 11 1,2-Diphenylhydrazine Not stable in water 12 Silvex Derivatization required 13 2,4,5-Trichlorophenoxyacetic acid Derivatization required

 Table 2.
 Compounds with Average Recoveries Less Than 70 Percent on Day 0

 Percent Recovery

			<u>on D</u>	ay 0	Comments
No.	Compound	List (a)	Avg	RSD	(b)
1	Benzenethiol	9 B	33	29	CP, OE, OS
2	p-Benzoquinone	9 B	0	0	OE
3	2,4-Diaminoanisole sulfate	M	60	08	AA, DC, OE
4	2,4-Diaminotoluene	М	42	07	AA, DC, OE
5	Dichlone	М	0	0	OE
6	Dimethoate	В	31	06	HE, HS
7	1,4-Dinitrobenzene	9	14	27	HE
8	Ethyl carbamate	8	28	02	DC
9	Isosafrole	9 B	46	05	AA, DC
10	Maleic anhydride	8	0	0	AA, HE
11	Malononitrile	9 B	09	11	CP, DC
12	4,4'-Methylenebis (2-chloroaniline)	9	33	19	AA, OE, OS
13	Monocrotophos	M	26	05	AA, HE
14	Nicotine	8	67	22	OE
15	Pentachloroethane	9	64	02	HE, HS
16	1,2-Phenylenediamine	8	32	11	DC, OE
17	1,3-Phenylenediamine	8	19	20	DC, OE
18	Phosphamidon	M	63	09	AA, HE
19	Phthalic anhydride	8	0	0	HE
20	Resorcinol	9 B	10	05	DC, OE
21	Thioacetamide	8	01	82	DC
22	Trimethyl phosphate	M	60	03	HE

- (a) 9 = Proposed Appendix IX to Part 264 as published in the Federal Register, 51, No. 142, July 24, 1986, pp. 26639-26642.
 - B = Borderline chemicals considered for additions to proposed Appendix IX to part 264 and published in the Federal Register, 51, No. 142, July 24, 1986, p. 26637.
 - M = Michigan list of chemicals proposed to be added to Appendix VIII to Part 261 and published in the Federal Register, 49, No. 247, December 21, 1984, p. 49793.
 - 8 = Appendix VIII to Part 261 as revised and published in the Federal Register, 51, No. 247, August 6, 1986, pp. 28305-28310. Compounds so designated are not present on proposed Appendix IX, borderline chemicals, or Michigan lists.
- (b) Comments:
 - AA = The internal standard coeluted with a mixture component/interference in both the samples and the calibration standards; compound recovery was calculated on the basis of average absolute area of the compound in the 100 µg/mL calibration standard.
- CP = Nonreproducible chromatographic performance.
 - DC = Unfavorable distribution coefficient.
 - HE = Hydrolysis during extraction accelerated by acidic or basic conditions.
 - HS = Hydrolysis during storage.
 - OE = Oxidation during extraction accelerated by basic conditions.
 - OS = Oxidation during storage.

Table 3. Compounds with Aqueous Stability Less Than 70 Percent

No.	Compound	Lis	t (a)	% Stab (b)	Comments (b)
1	Aramite (Isomers 1 and 2)	9		54,52	HS
2	Azınphos-methyl	N	,	62	HS
3	Benzenethiol	9 B		(c)	CP, OE, OS
4	Captafol	٨	1	55	AA, HS
5	Captan	N	1	40	AA, HS
6	Demeton-0	N	1	68	AA, HS
7	Diethylstilbestrol		8	67	AW, OS
8	Dihydrosafrole		8	10	HS
9	7,12-Dimethylbenz(a)anthracene	9		45	CP
10	Dinocap	N	1	28	CP,HS
11	Hexachlorophene	9		62	AW,CP
12	Malathion	N	1	5	HS
13	4,4'-Methylenebis(2-chloroaniline)	9		0	AA, OE, OS
14	Mexacarbate	N	1	68	HE, HS, RA
15	1-Naphthylamine	9		44	os
16	Pentachloroethane	9		4	HE, HS
17	Phosmet	N	1	15	HS
18	Phthalic anhydride		8	67	AA, CP, HE, HS
19	Strychnine		8	55	AW, OS
20	Toluene dusocyanate		8	6	AA, HE

- (a) 9 = Proposed Appendix IX to Part 264 as published in the Federal Register, 51, No. 142. July 24, 1986, pp. 26639-26642.
 - B = Borderline chemicals considered for additions to proposed Appendix IX to part 264 and published in the Federal Register, 51, No. 142, July 24, 1986, p. 26637.
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 - 8 = Appendix VIII to Part 261 as revised and published in the Federal Register, 51, No. 247, August 6, 1986, pp. 28305-28310. Compounds so designated are not present on proposed Appendix IX, borderline chemicals, or Michigan lists.
- (b) % Stab = Avg Recovery (Day 7) x 100/Avg Recovery (Day 0).
- (c) Compound not detected in Day 7 Sample extracts or calibration standards.
- (d) Comments:
 - AA = The internal standard coeluted with a mixture component/interference in both the samples and the calibration standards; compound recovery was calculated on the basis of average absolute area of the compound in the 100 μg/mL calibration standard.
 - AW = Adsorption to walls of glassware during extraction and storage.
 - CP = Nonreproducible chromatographic performance.
 - HE = Hydrolysis during extraction accelerated by acidic or basic conditions.
 - HS = Hydrolysis during storage.
 - OE = Oxidation during extraction accelerated by basic conditions.
 - OS = Oxidation during storage.
 - RA = A sample analyte was observed to coelute with the internal standard; compound quantification was calculated on the basis of peak area relative to the average internal standard peak area from the calibration standards.

Table 4 Compounds Tentatively Recommended for Inclusion in SW-846 Method 3510(a)

No.	3510(a) Compound	CAS No.	RCRA No.	List ((b)
1	Acetophenone	98-86-2	U004	9	
2	2-Acetylaminofluorene	53-96-3	U005	9 B	
3	2-Aminoanthraquinone	117-79-3	U264	M	
4	Aminoazobenzene	60-09-3	U257	M	
5	4-Aminobiphenyl	92-67-1	U274	9	
6	3-Amino-9-ethylcarbazole	132-32-1	U253	M	
7	Anilazine	101-05-3	U333	M	
8	Aniline	62-53-3		9 B	
g	o-Anisidine	90-04-0	U260	M	
10	Benzyl alcohol	100-51-6		9	
11	Bromoxynil	1689-84-5	U272	M	
12	2-sec-Butyl-4,6-dinitrophenol	88-85-7		9 B	
13	Carbaryl	63-25-2	U279	М	
14	Carbofuran	1563-66-2	U127	М	
15	Carbophenothion (d)	786-19-6	U148	М	
16	Chlorfenvinphos	470-90-6	P143	М	
17	4-Chloroaniline	106-47-8		9	
18	Chlorobenzilate	501-15-6		9	
19	5-Chloro-2-methylaniline	95-79-4	U329	М	
20	3-(Chloromethyl)pyridine hydrochloride	6959-48-4	U319	М	
21	4-Chloro-1,2-phenylenediamine	95-83-0	U306	M	
22	4-Chloro-1,3-phenylenediamine	5131-60-2	U305	М	
23	Coumaphos	56-72-4	P130	М	
24	p-Cresidine	120-71-8	U262	М	
25	Crotoxyphos	7700-17-6	U238	М	
26	2-Cyclohexyl-4,6-dinitrophenol	131-89-5	P034		8
27	Demeton-S	126-75-0		М	
28	Diallate	2303-16-4	U062	В	
29	Diazinon	333-41-5	U313	М	
30	1,2:7,8-Dibenzacridine (c,d)	224-42-0			8
31	Dibenzofuran	132-64-9		9	
32	1,2-Dibromo-3-chloropropane	96-12-8		9	
33	2,6-Dichlorophenol	87-65-0		9	
34	Dichlorovos (d)	62-73-7	P144	М	
35	Dicrotophos	141-66-2	P146	М	
36	3,3'-Dimethoxybenzidine (c,d)	119-90-4	U091	9	
37	p-Dimethylamınoazobenzene (c)	60-11-7	U093	9	
38	3,3-Dimethylbenzidine (d)	119-93-7	U095	ō	
39	1,2-Dinitrobenzene	528-29-0			8
40	1,3-Dinitrobenzene	99-65-0			8
41	Diphenylamine	122-39-4		9 B	
42	5,5-Diphenylhydantoin	57-41-0		М	
43	Disulfoton	298-04-4		9	
44	EPN	2104-64-5	P141	М	
45	Ethion	563-12-2	P154	М	
46	Ethyl methanesulfonate	62-50-0	U119		8
47	Ethyl parathion	56-38-2		9	
48	Famphur	52-85-7	P097	9	
49	Fensulfothion	115-90-2	P156	м	
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(Continued)

Table	4. (Continued).			
No.	Compound	CAS No.	RCRA No.	List (b)
51	Fluchloralin	33245-39-5	U330	М
52	Hexachloropropene	1888-71-7	U243	9 B
53	Hexomethyl phosphoramide	680-31-9	U312	М
54	Isodrin	465-73-6	P060	9 B
55	Kepone	143-50-0		9 B
56	Leptophos	21609-90-5	P140	M
57	Mestranol	72-33-3	U301	M
58	Methapyrilene	91-80-5	U155	9
59	Methoxychlor	72-43-5		9
60	3-Methylcholathrene	56-49-5	U157	9
61	4,4'-Methylenebis (N,N-dimethylaniline) (d)	101-61-1	U255	М
62	Methyl methanesulfonate	66-27-3	0200	9
63	2-Methylnaphthalene	91-57-6		9
64	2-Methyl-5-nitroaniline	99-55-8	U181	9
65	Methyl parathion(c)	298-00-0	0.0.	9
66	2-Methylphenol	95-48-7		9
67	3-Methylphenol	108-39-4		В
68	4-Methylphenol	106-44-5		9
69	2-Methylpyridine	109-06-8	U191	9 B
70	Mevinphos	7786-34-7	P131	M
70 71	Mirex	2385-85-5	U297	M
72	Naled	300-76-5	U309	M
72 73	1,4-Naphthoquinone	130-15-4	U166	9
73 74	2-Naphthylamine	91-59-8	U168	9
75	5-Nitroacenaphthene	602-87-9	U250	M
.76	2-Nitroanline	88-74-4	0230	9
, o 77	3-Nitroaniline	99-09-2		9
78	4-Nitroaniline	100-01-6		9
79	5-Nitro-o-Anisidine	99-59-2	U263	M
80	4-Nitrobiphenyl	92-93-3	U275	M
81	Nitrofen	1836-75-5	U288	M
82	4-Nitroquinoline-1-oxide	56-57-5	0200	8
83	N-Nitrosodi-n-butylamine	924-16-3		9
84	N-Nitrosodiethylamine	55-18-5		9
85	p-Nitrosodiethylamine	156-10-5	U287	M
86	N-Nitrosomethylethylamine	10595-95-6	0207	9 B
87	N-Nitrosomorpholine	59-89-2		9
88	N-Nitrosopiperidine	100-75-4	U176	9
89	N-Nitrosopyrrolidine(d)	930-55-2	0,,,0	9
90	4,4'-Oxydianiline(c)	101-80-4	U303	М
91	Pentuchlorobenzene	608-93-5	5550	9
92	Pentachloronitrobenzene	82-68-8		9
93	Phenacetin	62-44-2	U187	9
94	Phenobarbital	50-06-6	U268	M
95	1,4-Phenylenediamine(d)	106-50-3	0200	8
96	Phorate	298-02-2		9
97	Piperonyl sulfoxide	120-62-7	U270	M
98	Pronamide	23950-58-5	0210	9
30	r ronamo G	2330-36-3		(Continued)

(Continued)

Table	4. (Continued).			
No.	Compound	CAS No.	RCRA No.	List (b)
99	Safrole	94-59-7	U203	9
100	Sulfallate(d)	95-06-7	U277	М
101	Terbufos	13071-79-9	P149	М
102	1,2,4,5-Tetrachlorobenzene	95-94-3		9
103	2,3,4,6-Tetrachlorophenol	58-90-2		9
104	Tetrachlorvinphos	961-11-5	U308	M
105	Tetraethyl dithiopyrophosphate	3689-24-5	P109	9 B
106	Tetraethyl pyrophosphate	107-49-3		8
107	Thionazine	297-97-2	P040	9
108	o-Toluidine	95-53-4	U328	B M
109	2,4,5-Trichlorophenol	95-95-4		9
110	0,0,0-Triethyl phosphorothioate	126-68-1		8
111	Trifluralin	1582-09-8	U332	М
112	2,4,5-Trimethylaniline	137-17-7	U259	М
113	1,3,5-Trinitrobenzene	99-35-4	U234	8
114	Tri-p-tolyl phosphate	1330-78-5		М
115	Tris-(2,3-dibromopropyl) phosphate	126-72-7	U235	9 B

- (a) Based on a compound having demonstrated both extractability and aqueous stability values equal to or greater than 70%.
- (b) 9 = Proposed Appendix IX to Part 264 as published in the Federal Register, 51, No. 142, July 24, 1986, pp. 26639-26642.
 - B = Borderline chemicals considered for additions to proposed Appendix IX to part 264 and published in the Federal Register, 51, No. 142, July 24, 1986, p. 26637.
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 - 8 = Appendix VIII to Part 261 as revised and published in the Federal Register, 51, No. 247, August 6, 1986, pp. 28305-28310. Compounds so designated are not present on proposed Appendix IX, borderline chemicals, or Michigan lists.
- (c) Compounds exhibiting either Day 0 or Day 7 recoveries with relative standard deviations greater than 15 percent.
- (d) Compounds with calibration response factors having relative standard deviations greater than 15 percent.

Table 5. Recommended Changes for Semivolatile Organic Compounds on Proposed Appendix IX.

No.	Substance	List (a)		CAS No.	
5A.	Recommended Deletions				
1	Aramite (Isomers 1 and 2)	9		140-57-8	
2	Benzenethiol	9 B		108-98-5	
3	p-Benzoquinone	9 B		106-51-4	
4	7,12-Dimethylbenz(a)anthracene	9		57-97-6	
5	1,4-Dinitrobenzene	9		100-25-4	
6	Hexachlorophene	9		70-30-4	
7	Isosafrole	9 B		120-58-1	
8	Malononitrile	9 B		109-77-3	
9	4,4'-Methylenebis(2-Chloroaniline)	9		101-14-4	
10	1-Naphthylamine	9		134-32-7	
11	Pentachloroethane	9		76-01-7	
12	Resorcinol	9 B		108-46-3	
<u>5B.</u>	Recommended Additions				
1	2-Cyclohexyl-4,6-dinitrophenol		8	131-89-5	
2	Diallate	В		2303-16-4	
3	1,2:7,8-Dibenzacridine(b,c)		8	224-42-0	
4	1,2-Dinitrobenzene		8	528-29-0	
5	1,3-Dinitrobenzene		8	99-65-0	
6	Ethyl methanesulfonate		8	62-50-0	
7	3-Methylphenol	В		108-39-4	
8	4-Nitroquinoline-1-oxide		8	56-57-5	
9	1,4-Phenylenediamine(c)		8	106-50-3	
10	Tetraethyl pyrophosphate		8	107-49-3	
11	o-Toluidine	В		95-53-4	
12	0,0,0-Triethyl phosphorothioate		8	126-68-1	
13	1,3,5-Trinitrobenzene		8	99-35-4	

⁽a) 9 = Proposed Appendix IX to Part 264 as published in the Federal Register, 51, No. 142, July 24, 1986, pp. 26639-26642.

B = Borderline chemicals considered for additions to proposed Appendix IX to part 264 and published in the Federal Register, 51, No. 142, July 24, 1986, p. 26637.

^{8 =} Appendix VIII to Part 261 as revised and published in the Federal Register, 51, No. 247, August 6, 1986, pp. 28305-28310. Compounds so designated are not present on proposed Appendix IX, or borderline chemicals lists.

⁽b) 1,2:7,8-Dibenzacridine exhibits a Day 0 recovery with relative standard deviation

greater than 15%.
(c) Compounds with calibration response factors having relative standard deviations greater than 15%.

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James Longbottom is the EPA Project Officer (see below).

The complete report, entitled "Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846 Method 3510," (Order No. PB 88 161 559/AS; Cost: \$14.95, subject to change) will be available only from:

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5285 Port Royal Road

Springfield, VA 22161

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