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HANDBOOK OF ENVIRONMENTAL ANALYSIS

THIRD EDITION

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Other contaminants seen in the semi-volatiles lab arise from the solvents[176] that are used in the extraction process. Methylene chloride and other chlorinated solvents are subject to free-radical degradation initiated by ultraviolet light. Degradation products include hydrochloric acid, phosgene, and a variety of hydrocarbons and chlorinated hydrocarbons of increasing chain-length. Free-radical scavengers used to stabilize chlorinated solvents include amylene and ethanol (up to 1%). Phosgene is of particular interest, not only because it is toxic (it was employed as one of the first chemical warfare agents during World War I) but also because it is extremely reactive toward alcohols, phenols and amines that originally may be in the sample. Reaction with phosgene can transform target analytes into unrecognizable by-products.

Ether solvents are prone to formation of hydroperoxides, initiated by exposure of the solvent to air and ultraviolet light. Not only are hydroperoxides famous for their instability, old opened bottles of ether and tetrahydrofuran have been known to spontaneously explode, but the peroxides are effective oxidizing agents of target analytes. Ether solvents are stabilized by addition of either BHT or ethanol, and by packaging the solvent in a light-proof container under a nitrogen atmosphere. Bottles or cans of ether solvents should be purchased in the minimum size needed for weekly analysis use. Any excess should be disposed of properly. Containers that have been opened and stored more than 30 days should be suspected as having peroxides present. The presence of peroxides can be checked by shaking a 5 mL portion of the solvent with 1 mL of a 10% potassium iodide solution. Any formation of a yellow to brown or purple color in the solvent layer is interpreted as indicating that peroxides are present.

Ketone solvents such as acetone or methylethyl ketone (MEK) can form peroxides; however, the more frequently noted mode of degradation is condensation of two or more molecules of the solvent in the aldol reaction. 4-Hydroxy-4-methyl-2-pentanone (diacetone alcohol) is found as a contaminant in almost every instance when acetone has been used in the glassware washing process. Acetone and MEK can also condense with anilines and other aromatic amines to form very stable imines ($Ph\text{-}N{=}C[CH_3]_2$) that change the retention time and mass spectum of the target analyte.

Table 1-54. Phthalates and other contaminants found in common laboratory items. Amounts are in ng/uL injected into the GC-MS from 1.0 mL final volume of extract

Item and number	Contaminant found	Amount
sodium sulfate	di-*n*-butyl phthalate	5-100
1 g glass wool	di-*n*-octylphthalate	2
1 sheet of paper towel roll	di-*n*-butyl phthalate	36.5
	N,N-dimethyl-9-octadecenamide	-
	4,4'-butylidenebis[2-(1,1-dimethylethyl)-5-methyl] phenol	-
	pentatriacontane	-
#4 filter paper	di-*n*-butyl phthalate	139
	butylbenzyl phthalate	0.8
	Bis(2-ethylhexyl) phthalate	1.1
	di-*n*-octyl phthalate	0.2
	decamthylcyclopentasiloxane	-

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176 Seaver, C., J. Przybytek, and N. Roelofs, 1995. "Solvent Selection, Part III - Solvent Life and Degradation." *LC-GC*, 13(11). pp. 860-864. November, 1995.

Table 1-54. Phthalates and other contaminants found in common laboratory items. Amounts are in ng/uL injected into the GC-MS from 1.0 mL final volume of extract, *continued*

Item and number	Contaminant found	Amount
Acrodisk PTFE 0.45 mm filter disk (15)	di-*n*-butyl phthalate	3.8
Safeskin glove	di-*n*-butyl phthalate	1.1
	Bis(2-ethylhexyl) phthalate	12.2
	butylbenzyl phthalate	2.4
	N,N-bis(2-hydroxyethoxy)-dodecamide	-
	octadecadienal	-
	octadecene	-
	hydrocarbon oils	-
Triclean glove	Bis(2-ethylhexyl) phthalate	4.3
	butylbenzyl phthalate	11.4
	4-chloro-3-methyl phenol	6.5
	N,N-bis(2-hydroxyethoxy)-dodecamide	-
	Hexadecanoic acid	-
	4,4'-butylidene bis[2-(1,1-dimethylethyl)-5-methyl]phenol	-
	octadecadienal	-
N-Dex nitrile glove	di-*n*-butyl phthalate	6.3
	benzyl alcohol	56.8
	Bis-(2-ethylhexyl) phthalate	4.6
	3,3'-imino bis propanenitrile	-
	2-mercaptobenzothiazole	-
	hydrocarbon oils	-
	4,4'-butylidene bis[2-(1,1-dimethylethyl)-5-methyl]phenol	-
Chemsolve (1 g)	hexamethylcyclotrisiloxane	-
	pentamethyldisiloxane	-
	octamethylcyclotetrasiloxane	-
	2-[2-[4-(1,1,3,3-tetramethylbutyl)phenoxy]ethoxy]ethanol	-
	di-*n*-octylphthalate	1.4
HDPE container (500 mL)	2,5,8,11,14-pentaoxahexadecan-16-ol and other alcohols	-
Rubber suction bulb (large)	large amounts of hydrocarbon oils	-
Latex suction bulb (3)	4 unknown compounds	-
1 Alumina PrepSep column	(none detected)	
Tygon R-3603 tubing (41 inches)	Bis(2-ethylhexyl)phthalate	44,000
Fisher disposable Pasteur pipets (12)	(none detected)	
Soxhlet extraction thimble	di-*n*-butyl phthalate	9.3
	unidentified phthalate	-
	unidentified adipate	-
Silastic tubing (27")	diethyl phthalate	6.0
	phenanthrene	2
	anthracene	1.9
	di-*n*-butyl phthalate	5.1
	Bis(2-ethylhexyl) phthalate	15.5
	ethanol, 2-(2-butoxyethoxy)-acetate	-
	unidentified siloxane polymer series	-
Aluminum foil	(none detected)	
Baker C18 SPE	Bis(2-ethylhexyl) phthalate	2.7
	unidentified siloxane polymer series	-
McDonald's fingers	C_{12}-C_{18} Fatty acid series	varies