

Applications note

cat.# 59564

Extraction of PCBs using Resprep™-C18-47 SPE disks and Resprep™-6D

Use Resprep™-C18-47 SPE disks and Resprep™-6D to extract polychlorinated biphenyls (PCBs)

Sample Pretreatment

Allow 1 liter of deionized water to equilibrate to room temperature. Adjust sample pH to less than 2 with 6N HCl. Add 5mL of methanol and mix thoroughly. For QA/QC samples, spike with 1mL of a 5ug/mL solution for a final concentration in the water of 5ppb.

Apparatus Assembly

Assemble the Diskcover™-47mm with reservoir, making sure the Teflon® o-ring is in place. Also be sure to place the C18 disk in the Diskcover™-47 wrinkled side up. Assemble the Resprep™-6D as detailed in the instruction sheet, along with the vacuum pump, vacuum line, and vacuum trap.

Disk Precleaning

Add 10mL of methylene chloride to the disk's top surface and immediately draw through under vacuum at 15 in. Hg. Continue to draw vacuum at 15 in. Hg for 5 minutes, removing all solvent.

Disk Conditioning

Add 10mL of methanol to the disk's top surface and let stand for a few minutes. Draw the methanol through the disk until the methanol's top surface is just above the disk. Do not allow any air to pass through the disk or reach the top surface of the disk. Note: It is preferable to leave extra liquid above the disk rather than allow any air to contact the disk surface.

Sample Addition

Pour the sample into the Diskcover™-47 reservoir, adding it directly to the film of methanol left on the disk from the conditioning step. Adjust the vacuum to approximately 25 in. Hg.

Disk Drying

After the entire sample has been processed, draw air through the disk under vacuum to remove any residual water from the disk.

Analyte Elution

Release system vacuum. Insert the collection rack and vessels into the glass chamber, making sure to label each vessel with the corresponding Diskcover™-47. Reassemble the Resprep™-6D. Add 5mL methylene chloride directly to the sample bottle, and gently swirl to rinse all inner surfaces of the bottle. Transfer the methylene chloride to the disk using a

glass pipette and rinse the reservoir sides in the process. Let the solvent stand for three minutes. Draw the solvent through the disk at 5 in. Hg. Repeat the bottle rinse and disk elution once more with a fresh aliquot of methylene chloride, combining all eluates in the collection tube.

Final Analysis

Remove water from sample eluate by passing through anhydrous sodium sulfate. Concentrate to 1mL, and analyze.

For more information
call technical service:

(800) 356-1688,

ext. 4

Table 1

Accuracy/precision data from seven determinations of PCBs in distilled water at 5µg/L, using the Resprep™-C18-47 glass fiber extraction disk.

Aroclor®	peak #	congener	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	AVG	SD
1016	1	8	97.6	95.6	91.1	91.9	91.4	93.4	85.2	92.3	3.6
	2	18	99.0	100.4	92.4	97.0	101.5	106.0	95.2	98.8	4.1
	3	31	94.7	85.3	82.8	88.4	88.5	98.6	95.4	90.5	5.4
	4	52,69	97.3	85.9	86.2	96.1	91.3	100.5	102.0	94.2	6.0
	5	44	93.0	89.8	92.7	97.2	93.6	103.7	102.7	96.1	4.9
1242	1	18	112.0	109.3	113.6	94.0	96.8	102.8	89.0	102.5	8.8
	2	8	103.6	104.8	106.0	87.4	90.8	97.5	81.6	96.0	8.8
	3	31	100.6	98.7	99.9	78.7	83.0	95.0	76.3	90.3	9.8
	4	44	101.7	103.5	103.0	87.1	90.0	98.2	81.6	95.0	8.1
	5	56,92	93.5	97.3	95.6	76.4	83.0	91.7	73.8	87.3	8.8
1248	1	31	81.7	92.0	86.3	88.3	81.9	79.2	82.7	84.6	4.1
	2	48	79.7	89.3	87.4	96.2	83.5	85.9	82.1	86.3	5.0
	3	41	81.1	88.6	84.8	88.5	81.9	80.3	82.9	84.0	3.2
	4	70	4.1	90.3	87.6	92.8	87.7	84.4	85.0	87.4	3.0
	5	56,92	82.4	90.3	86.5	91.5	86.2	84.9	84.6	86.6	3.0
	6	110,77,154	84.8	90.9	84.5	94.0	78.8	72.4	81.0	83.8	6.7
1254	1	52,69	99.8	90.9	79.4	96.4	100.9	87.0	98.3	93.2	7.3
	2	95,66	95.2	89.2	79.9	91.9	99.2	80.0	52.1	83.9	14.6
	3	110,77,154	93.6	98.5	88.4	95.9	110.2	79.4	45.2	87.3	19.3
	4	118	90.1	83.2	73.2	81.6	96.0	64.2	82.4	81.5	9.7
	5	138	87.1	89.2	77.3	85.7	95.6	64.6	109.5	87.0	13.0
1260	1	149	87.2	97.9	91.3	85.6	79.0	82.5	88.9	87.5	5.7
	2	141	79.6	69.9	78.6	82.8	76.3	78.4	90.64	79.5	5.8
	3	187	87.2	76.1	85.9	90.3	82.3	83.3	95.4	85.8	5.7
	4	180	103.4	94.2	108.1	113.4	102.5	104.6	102.2	104.1	5.4
	5	170	79.1	73.0	84.6	88.1	78.6	81.9	67.7	79.0	6.4

Trademarks:

Resprep™ and Diskcover™ are trademarks of Restek Corporation. Teflon® is a trademark of E.I. du Pont de Nemours & Company, Inc.

For permission to reproduce any portion of this application note, please contact Restek's publications/graphics department by phone (ext. 2128) or FAX (814) 353-9278.



© Copyright 1997,
Restek Corporation