DOW CHEMICAL U.S.A.

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CHLOROBIPHENYLS IN ortho AND para-PHENYLPHENOL

The following is a brief description of the analytical procedure used to determine chlorinated biphenyls in ortho and para-phenylphenol.

Sample Preparation

Five grams of sample were weighed into a 4-oz bottle, 100 ml of ~0.5M of sodium hydroxide was added and shaken to dissolve the sample. When the sample was in solution, 10 ml of benzene were added and placed on a shaker and extracted for 30 minutes.

Ion exchange resin DOWEX* 21K, 50-100 mesh, chloride form, was converted to the hydroxide form as described by W. Blaser in report ML-AL 59-371. A column 1.3 x 30 cm was prepared with a 15 cm bed of resin.

The contents of the bottle were transferred to a 125-ml separatory funnel and the layers were allowed to separate. The lower aqueous layer was discarded and the benzene layer was drained off onto the column containing the resin. The benzene extract was drained to the top of the resin. After discarding the first 5 ml, a 7-dram vial was placed under the column. The walls of the column were washed with benzene and drained again to resin level; more benzene was added until 20-25 ml had been collected in the vial.

The effluent was evaporated just to dryness using a gentle air stream at room temperature. The residue was redissolved in exactly 1 ml of cyclohexane.

The sample was then analyzed for chlorinated biphenyls by gas chromatography with flame or electron capture detection.

GC Conditions

	I	II
Instrument	HP-5750 with FID	Pye-104 with ECD
Column	6' x 1/8" r.5% Bentone 34 + 3.0% DC-550 on 80/100 WHP	4'x 1/8" 4.5% Bentone 34 + 3.0% DC-550 on 80/100 WHP
Temperatures		
column	190°C	170°C
injection port	250°C	150°C
detector	250°C	300°C
Carrier gas	helium at 40 cc/min	nitrogen at 40 cc/min
Range	10^2	10^2
Attenuation	as needed	as needed
Sample size	2 µl	3 µl

GC-MS Conditions

Instrument	LKB 9000
Column	8' x 1/8" 5% OV-17 on 80/100 GCQ
Temperature	200° and 160°C
Carrier gas	helium at 50 cc/min

All the samples, from the starting material, to the final products: ortho-pehnylphenol were analyzed using conditions I (flame).

The final products: ortho-phenylphenol and para-phenylphenol were also analyzed using conditions II (electron capture).

The final products: o-phenylphenol and p-phenylphenol were analyzed for total chlorine by neutron activation analysis.

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