CHROM. 4768

A note on the use of Florisil adsorbent for the separation of polychlorobiphenyls from chlorinated pesticides*

The analytical procedures for various types of materials for chlorinated pesticide residues by gas chromatography usually require some form of clean-up prior to the application of the sample to the gas chromatograph. The adsorbent Florisil[1] is commonly used in the clean-up step, and based on BURKE AND MALONE's report in 1966[2], it was hopefully assumed that a modified product, Florisil PR (pesticide residue grade), had become available with consistent and reliable adsorbent properties. However, MILLS' work[3] in 1968 indicated that soluble sulfates, reported as Na_2SO_4, varied from 0.15 to 2.44% in twelve batches of the Florisil PR grade, and that this variation would affect the adsorptive characteristics of the Florisil to the detriment of pesticide recoveries from sample materials. It has been recommended to either pretest the adsorbent with a lauric acid procedure[3] or with a known group of chlorinated pesticides[4].

A new problem, which may now exist in pesticide residue analysis, is the possible presence of polychlorobiphenyls (PCB's) in the sample material[5–8]. The chromatographic behavior of the PCB's is so similar to that of some of the chlorinated pesticides that if they are not separated from the pesticides prior to analysis by gas chromatography, the end result will be a misinterpretation of the analytical data.

REYNOLDS[5] recently reported a method which included the use of Florisil, presumably the regular grade and not the PR grade, which separated the PCB's from some of the chlorinated pesticides including DDT. With this procedure, the PCB's were eluted from the column with hexane, and DDT and other pesticides were eluted

TABLE I

PER CENT RECOVERIES OF TECHNICAL GRADE DDT FROM FLORISIL COLUMNS USING THE REYNOLDS PROCEDURE[5]

Adsorbent	*Solvent*	*Eluting agents*[a]	
		200 ml hexane	*250 ml 20% diethyl ether in hexane*
Florisil PR grade	Petroleum ether[b]	97	0
	Hexane	97	0
Florisil regular grade	Petroleum ether	90	7
	Hexane	57	43

[a] The column was first eluted with 200 ml hexane, followed immediately by elution of the same column with 250 ml 20% diethyl ether–hexane solution.

[b] The DDT (10 μg) was placed on the column with either 50 ml of petroleum ether or 50 ml of hexane.

* Journal Series No. 1186 of the Hawaii Agricultural Experiment Station.

TABLE II

PER CENT RECOVERIES OF *p,p'*-DDT[a] FROM FLORISIL COLUMNS USING THE REYNOLDS PROCEDURE[5]

Adsorbent	*Eluting agents*				
	Hexane				*250 ml 20% diethyl ether in hexane*
	1st fraction (50 ml)	*2nd fraction (50 ml)*	*3rd fraction (50 ml)*	*4th fraction (50 ml)*	
Florisil PR grade	0	35	51	5	0
Florisil regular grade	0	0	0	23	70

[a] The *p,p'*-DDT (10 μg) was placed on the column with a minimum amount (2 ml) of hexane.

TABLE III

PER CENT RECOVERIES OF CHLORINATED PESTICIDES FROM FLORISIL COLUMNS USING THE REYNOLDS PROCEDURE[5]

Adsorbent	*Eluting agent*	*Lindane*	*Hepta-chlor*	*Aldrin*	*Hepta-chlor epoxide*	*DDE*	*DDD*	*p,p'-DDT*	*Dieldrin*
Florisil PR grade[a]	200 ml hexane	89	91	91	0	87	83	88	0
Florisil regular grade		7	94	95	0	93	0	100	0
Florisil PR grade	250 ml 20% diethyl ether in hexane	6	0	0	96	0	0	0	97
Florisil regular grade		92	0	0	67	0	94	0	94

[a] The columns were packed with PR and regular grades of Florisil which had been desiccated 24 h after treatment for 16 h at 130°. All other columns described in Tables I–III were packed with hot Florisil immediately after its removal from a 130° oven.

TABLE IV

PER CENT RECOVERIES OF CHLORINATED PESTICIDES FROM FLORISIL COLUMNS USING THE FDA PROCEDURE[4]

Adsorbent	*Eluting agents*							
	200 ml 6% diethyl ether in petroleum ether							*200 ml 15% diethyl ether in petroleum ether*
	Lindane	*Hepta-chlor*	*Aldrin*	*Hepta-chlor epoxide*	*DDE*	*DDD*	*p,p'-DDT*	*Dieldrin*
Florisil PR grade	89	96	99	100	125[a]	98	100	39
Florisil regular grade	91	88	96	93	92	93	93	95

[a] DDE and dieldrin superimposed on gas chromatograph chart under the conditions used; therefore the recovery estimate is based on DDE calculation.

with a subsequent mixture of diethyl ether–hexane. Using both the regular grade and PR grade of Florisil, we were unable to repeat the work of REYNOLDS.

As noted in Tables I–III, all or part of the DDT was eluted from Florisil columns of either grade with the hexane solution, depending upon the conditions used. Table IV shows that our supply of Florisil regular grade performed satisfactorily as prescribed by the official procedure of the Food and Drug Administration (FDA)[4]. However, our supply of Florisil PR grade did not differentiate wholly the dieldrin fraction.

It is apparent that the procedure recommended by REYNOLDS[5] for the separation of PCB's from pesticides will require a preliminary examination of the pesticide elution characteristics of the Florisil, regular or PR grade, prior to the use of the adsorbent for this purpose.

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Received April 6th, 1970