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New Czechoslovak sorbent, Ekosorb

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ABSTRACT

A new sorbent, Ekosorb, has been developed and tested. It is prepared on the basis of a modified silica gel and warrants perfect purification of food extracts from fats and other co-extract substances. The pure eluate is suitable for trace analysis of polychlorinated aromatic compounds (polychlorinated biphenyls, hexachlorobenzene), chlorinated pesticides etc. Recovery of residues of chlorinated compounds is higher than 96%, allowing a quantitative determination and detection by gas chromatography. Ekosorb is an equivalent to Florisil.

INTRODUCTION

Polychlorinated aromatic compounds [polychlorinated biphenyls (PCBs), benzenes and other derivatives] and chlorinated pesticides are hazardous, persistent chemicals which have been detected in all environmental compartment including the food chain. These chemicals are more soluble in fat than in aqueous systems. Levels of these compounds must be monitored periodically so that trends and levels of exposures can be assessed.

Quantitative determination of chemical residues in biological samples requires use of accurate and reliable methods. Sample extraction and clean-up are two of the most important steps for ensuring the reliability of the procedure [1]. Florisil is an effective sorbent used for the clean-up of animal fats and biological sample extracts for the analysis of pesticides, PCBs and other non-polar contaminants [1–3].

A new sorbent, Ekosorb, has been developed and tested in Czechoslovakia [4]. This paper presents a comparison of results obtained using Florisil and Ekosorb.

EXPERIMENTAL

Solvents and chemicals

All solvents used were glass-distilled and free from interfering residues as tested by gas chromatography (GC). Anhydrous Na_2SO_4 and glass wool were pre-washed with hexane and petroleum ether.

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The standards used were 1,1-dichloro-2,2-bis(4-chlorophenyl)ethene (*p,p'*-DDE) and hexachlorobenzene (HBC) from Supelco (U.S.A.) and PCBs (as Delor 106, an equivalent to Aroclor 1260) from Chemko (Strážské, Czechoslovakia). All standards were dissolved in hexane.

Florisil (60–100 mesh), PR grade, was obtained from Serva (U.S.A.).

Ekosorb (60–120 mesh) was prepared from silica gel by modifying its surface with a thin layer of magnesium silicate (Czechoslovak patent application [4]).

Fat preparation and fortification

Raw chick fat was homogenized with hexane. The hexane layer was filtered through anhydrous N_2SO_4 and evaporated to dryness under vacuum on a water-bath at 30°C. Dried fat was free from *p,p'*-DDE, HCB and PCBs at the limit of detection (0.010 mg/kg) as tested by GC. This fat was dissolved in hexane and fortified by addition of an appropriate volume of the respective standard solution. The fortification levels are shown in Table I. The hexane phase was evaporated to dryness under vacuum on a water-bath at 30°C.

Clean-up recovery

Clean-up of all fortified fat samples was carried out in six parallel determinations by the following procedure: 14 g of activated Florisil or Ekosorb (see Table I) in glass columns was pre-washed with 20 ml petroleum ether. Then, 0.1 g of fat was dissolved in a small volume of petroleum ether and added to the column. The residues were eluted with 75 ml of 6% ethyl ether in petroleum ether. The eluate was evaporated to dryness under vacuum on a water-bath at 30°C. The residues were dissolved in hexane for GC analysis.

Gass chromatography

A Varian Vista 6000 gas chromatograph equipped with a ^{63}Ni electron-capture detector was used.

Packed column. A glass column, 250 cm × 2 mm I.D., packed with Chromosorb W HP (80–100 mesh) coated with 5% OV-101, was used. The GC conditions were as follows: nitrogen carrier gas flow-rate, 30 ml/min; injector temperature, 235°C; oven temperature, 21°C; detector temperature, 300°C. On-column injection was used; the sample size injected was 2 μl.

Capillary column. A 12 m × 0.32 mm I.D. silica capillary column coated with SE-30 (0.5 μm film) was used.The GC conditions were as follows: hydrogen carrier gas flow-rate, 2.5 ml/min; nitrogen make-up gas flow-rate, 30 ml/min; temperature programme, 100°C for 2 min, then to 210°C at 20°C/min and then held for 12.5 min; injector temperature, 220°C, detector temperature, 300°C. The sample size injected was 1 μl; the injection was splittless for 75 s.

RESULTS AND DISCUSSION

Table I shows the comparison of recoveries of the chlorinated compounds under study after clean-up of the fortified fat on Ekosorb and Florisil. Conditions conforming with those given in official analytical methods [1–3] were used for the activation of the sorbents, *i.e.*, 24 h at 130°C, 4 h at 400°C and 4 h at 650°C. HCB was

TABLE I

RECOVERIES OF PCBs, HCB AND *p,p'*-DDE FROM FAT USING EKOSORB AND FLORISOL

Activated	Added (mg/kg)	Recovery (mean ± S.D., $n=6$) (%)	
		Florisil	Ekosorb
PCBs			
130°C, 24 h	0.860	98.8 ± 2.6	99.6 ± 2.6
400°C, 4 h	0.860	95.4 ± 3.2	98.9 ± 2.8
650°C, 4 h	0.860	82.8 ± 4.8	96.3 ± 3.1
HCB			
130°C, 24 h	0.260	95.2 ± 3.8	96.8 ± 3.6
400°C, 4 h	0.260	87.4 ± 5.7	92.6 ± 4.0
p,p'-DDE			
130°C, 24 h	0.600	96.8 ± 2.2	97.2 ± 2.3
400°C, 4 h	0.600	94.6 ± 2.9	96.3 ± 2.7

included among the compounds under study, because it shows greater retention with Florisil than with other chlorinated aromatic compounds. As demonstrated in Table I, recoveries of all test compounds are higher on Ekosorb than Florisil. The differences became more pronouced when the activation temperatures were increased.

The purity of eluates from Ekosorb and Florisil is shown on chromatograms of PCBs separated from the fortified fat samples (Fig. 1). Ekosorb warrants perfect purification of food extracts from fat and other co-extract substances, which are

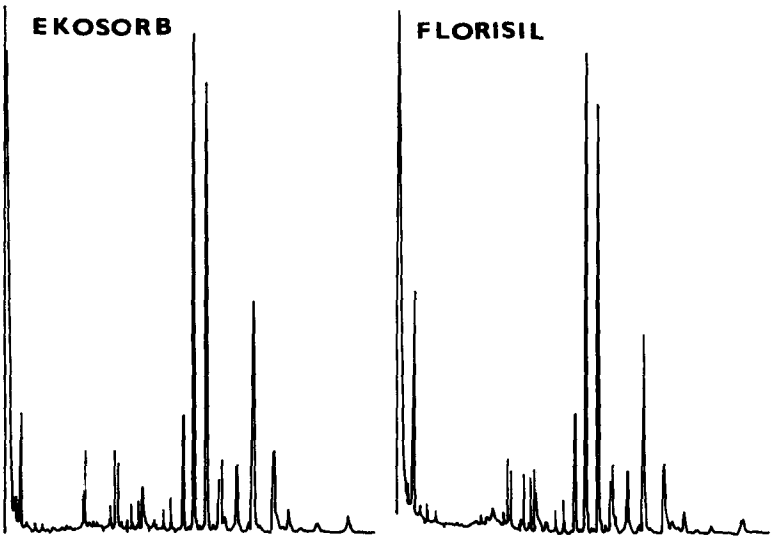

Fig. 1. Electron-capture chromatograms of Ekosorb and Florisil eluates of PCB-fortified fat on the SE-30 silica capillary column.

quantitatively retained on the sorbent column. The pure extract is suitable for trace analysis of polychlorinated aromatic compounds (PCBs, benzenes), chlorinated pesticides, etc.

Ekosorb can be used repeatedly after washing out the retained co-extract substances with acetone and water, drying and combustion of organic impurities at 650°C.

Twelve batches of Ekosorb, prepared under laboratory conditions, and three batches from pilot production were tested. Commercial production has already been launched by Glass-works Kavalier (Votice, Czechoslovakia). Constant-quality, reproducible recoveries of all test compounds and high purity of eluates were confirmed in all batches checked.

Compared with Florisil, Ekosorb possesses further advantages. Ekosorb is a synthetic sorbent, its preparation is quick, simple and inexpensive. It is prepared from silica gel for column chromatography (60–120 mesh) by modifying its surface with a thin layer of magnesium silicate. The composition of the batches tested was 97.0% SiO_2 and 2.7% MgO. The activity and sorption capacity of Ekosorb can be changed to comply with the compostion of the compound under study and the character of the sample.

Confirmatory examinations of the use of Ekosorb for the determination of pesticides and hazardous industrial polychlorinated aromatic compounds in food, feedstuffs and other biological materials and environmental compartments are ongoing.

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