

# Confirmation of identity of heptachlor residues by hydroxy derivatization on thin-layers of aluminium oxide

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von Personen aus dem Lebensmittelgewerbe werden ermittelt. Die Wirksamkeit antibakterieller Seifen wird geprüft. In einem letzten Versuch wird die Frage der bakteriellen Kontamination der Hände durch Geld erörtert.

#### *Literatur*

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#### *Resumé*

L'action du lavage sur l'élimination des microorganismes, des mains est étudiée. Les germes totaux, les coliformes, les staphylocoques, les levures et les moisissures sont dénombrés dans l'eau de savon ayant servi au lavage des mains, effectué dans des conditions définies. L'efficacité de savons bactéricides est examinée. Enfin, la contamination des mains par les bactéries de la monnaie est discutée.

#### *Short communication*

## Confirmation of identity of heptachlor residues by hydroxy derivatization on thin-layers of aluminium oxide

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The determination of heptachlor residues in foodstuffs is not seldom complicated by the presence of compounds having similar chromatographic characteristics as heptachlor. Not only some metabolites of other pesticides but also chlorinated biphenyls such as Aroclor 1242 may interfere with both gas and thin-layer chromatographic determination of heptachlor.

For this reason we have developed a simple identification procedure that can be applied to all sample extracts prepared following two widely used methods for multi-residue analyses (1, 2).

Several chlorinated pesticides are readily decomposed by heating them on thin-layers of neutral aluminium oxide (3). Under these conditions heptachlor is virtually quantitatively converted to 1-hydroxychlorde. This is a compound obtained by substitution of the chlorine atom at the allylic site of the heptachlor molecule by a hydroxyl group. The reaction occurs in basic hydroxylic media (4).

Identity was confirmed by comparison of chromatographic data with those of an authentic sample of 1-hydroxychlorde.

Further study revealed that the reaction proceeded only on aluminium oxide *thin-layers*. If the adsorbent was scraped off and heated with heptachlor, no dechloro-

mination occurred; nor was any reaction observed when aqueous suspension of aluminium oxide were refluxed with this insecticide.

During the last year we have used this remarkable phenomenon as a facile method for the confirmation of identity of heptachlor residues. The following procedure is recommended:

1. An aliquot of the cleaned up sample extract containing ca. 0,1 microgram of heptachlor is spotted on a layer of neutral aluminium oxide. Best results are obtained on strips of ready made plates such as alufoil Al<sub>2</sub>O<sub>3</sub>E, Merck, catalog number 5550/0025. It is essential that the extract does not contain waxy or fatty substances which interfere with the reaction. Besides the extracts a suitable quantity of heptachlor is applied as a reference substance.
2. The layer is heated during 45 minutes at 60 ° C in a stove.
3. Subsequently, the parts of the adsorbent where the sample and reference solution have been spotted are scraped off carefully and the powders thus obtained are brought small fritted glass filters, whereupon the reaction products are extracted with 1 ml of warm benzene.
4. Both benzene extracts are subjected to gas chromatography on a suitable column and the chromatograms are compared. The reference solution should yield a slightly tailing peak of hydroxychlordeane of which the relative retention time on different columns is given in the added table. If the derivatized sample solution shows the same peak with simultaneous disappearance of the originally observed heptachlor, the presence of this insecticide is beyond reasonable doubt.

Relative retention times of heptachlor, 1-hydroxychlordeane and related substances on three gas chromatographic columns.

Compound	1,5 % OV-17/1,95 QF-1 210 ° C, 40 ml/min	2 % DEGS + 0,5 % H <sub>3</sub> PO <sub>4</sub> 175 ° C, 60 ml/min	3 % OV-1 185 ° C, 50 ml/min
Aldrin (reference)	1,00	1,00	1,00
Heptachlor	0,81	1,00	0,78
1-Hydroxychlordeane	1,28	6,6	1,01
Heptachlor epoxide	1,48	2,86	1,28
gamma-Chlordane	1,67	2,86	1,47
alpha-Chlordane	1,84	3,03	1,67

The procedure can be used to confirm the presence of heptachlor at levels as low as 0,1 ppm in fats and 0,02 ppm in non fatty foods. The simultaneous presence of DDT, TDE, DDE, BHC, HCB, PCNB, aldrin, dieldrin, endrin, heptachlor epoxide and the chlordane isomers does not interfere with the derivatization or with the gas chromatography of 1-hydroxychlordeane. However, it may be

difficult to observe the derivative when large amounts of certain polychlorinated biphenyls or naphthalenes are present.

In that case, 1-hydroxychlorodene may be more easily detected by thin-layer chromatography and the thin-layer plate prepared and heated as described above, is directly developed in pentane-acetone 4 : 1, v/v as a mobile phase. Photochemical revelation with silver nitrate (5) indicates the presence of 1-hydroxychlorodene at  $R_F$  0,40. Other pesticides and polychlorinated compounds move close to the front. The limit of detection for 1-hydroxychlorodene is 25—50 nanograms.

#### *Acknowledgement*

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