Pesticides

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Lectures held at the
IUPAC Third International Congress of
Pesticide Chemistry, Helsinki
3–9 July 1974

Scientific Chairman: Pekka Koivistoinen

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List of Contributors

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Automation of the Storherr Sweep Co-Distillation Method and Associated Techniques

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Residues analysis on animal tissues is generally divided into four main steps.

1. Extraction of fat from the tissues.
2. Clean-up or extraction of the various pesticides from the extracted fat.
3. Removal of solvent from the recovered pesticide.
4. Quantitation by Gas-Liquid Chromatography.

Apparatus and Method

Extraction of Fats from Tissues

In case where the tissue contains sufficient fat, heat treatment is an efficient means of extraction. In cases where insufficient fat is present, treatment of the residues with a solvent such as petroleum ether or hexane may have to be resorted to. Butter may be melted and filtered through a glass wool pledget contained in the filter funnel. Cream may be separated from cows milk by a separator or a centrifuge, made into butter by shaking and finally melting. Rancid fats, which are quite troublesome by any method of clean-up, can be treated by a prolonged heating at ~ 90–100° thus driving off the troublesome volatiles. For toxicological purposes where great accuracy is not required, the method will give satisfactory results.

Apparatus

Apparatus consists of an aluminium casting clad with stainless steel (Fig. 1 and 2). The casting is fitted with 4 x 50 watt heating elements, each of which is controlled by a "Sunvic" control switch. The aluminium block runs at a temperature of ~ 100–150°. The apparatus is built to take 4 x 3″ diameter pyrex conical funnels each of which has a pledget of pyrex glass wool washed with petroleum ether and dried. The melted fat is retained in 2″ x 1″ stoppered specimen tubes in which it is kept in the deep freeze until ready for analysis.

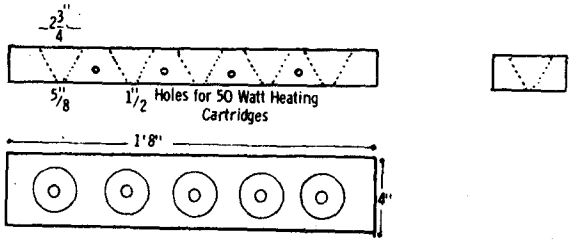

Fig. 1 Cast Aluminium Heating Block for Treatment of Fats

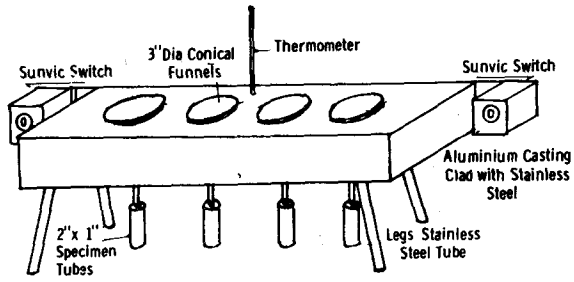

Fig. 2 Apparatus for Heat Extraction of Fats from Animal Tissues

Advantages of Heat Treatment for Extraction of Fatty Tissues

1. Results can be reported on a fat basis which is used in the F.D.A. tolerances.
2. A uniform and completely hexane-soluble sample is obtained.
3. Losses are minimal as the fat is removed from the system as soon as it melts.

Losses of Pesticides with Heat Treatment

That little loss occurs in the organic phosphates such as Delnav, Dursban, and Ethion is demonstrated by Fig. 3. 20 g of fat "spiked" with 1 ppm each of Delnav, Dursban, and Ethion heated in a beaker sample for analysis at 5 min intervals for a period of 1 hour, maintaining the temperature in the mean time at 160°. In actual practice, the fat is heated in a funnel and drips out as soon as it melts, its temperature seldom exceeds 60–70°. It can be confidently assumed that losses of pesticide from this melting are negligible. [1] Organic chlorine losses by heat rendering have been shown to be negligible also, fats have been "spiked" with 0.1 ppm HCB, gamma BHC, Aldrin, DDE, DDD, and DDT. [2]

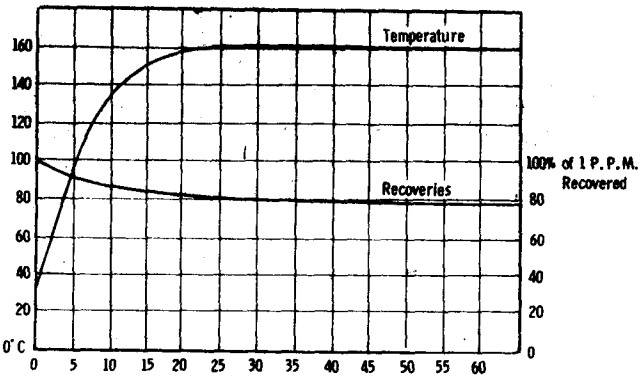

Fig. 3 Showing Ave. Loss of O.Ps (Delnav Dursban Ethion) Pesticides from Heat-Treated Fat

Clean-up or Extraction of Pesticides from Fat (The Automated Storherr Technique)

The principle of the method is demonstrated in Fig. 4 [3, 4, 5].

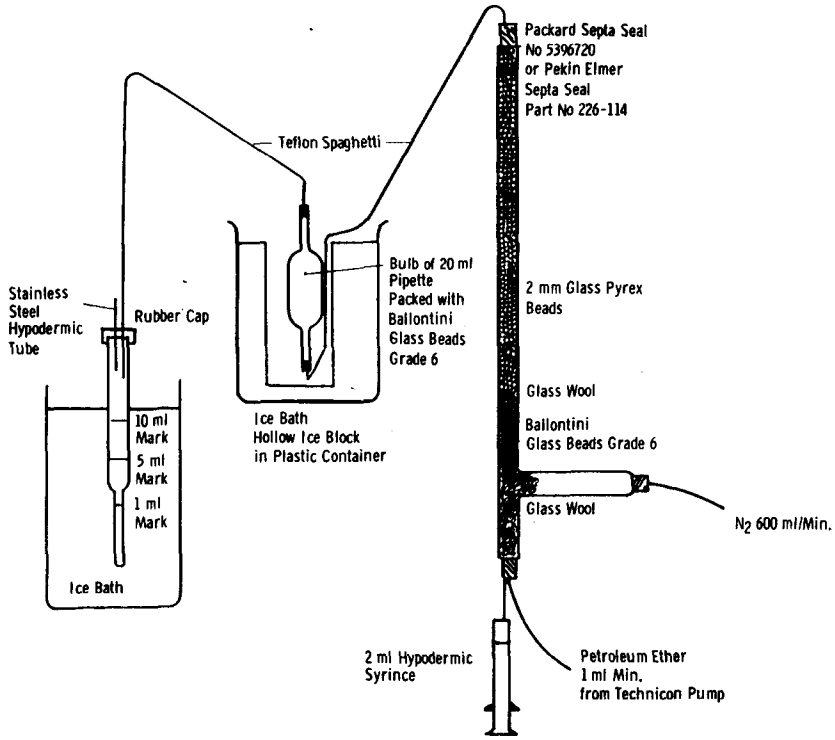

Fig. 4 Schematic Diagram of Storherr Method

The advantages of the Storherr method may be outlined as follows:

1. The Storherr method will "clean-up" any pesticide that can be gas-chromatographed.
2. There are no poisonous or objectionable solvents involved in the process.
3. With the automated version, only one solvent is necessary, thus eliminating possible interference from the use of several solvents.
4. The method is ideal for organic phosphates as all that is needed for a Storherr treatment is a reduction of 15–20 ml of highly volatile petroleum ether to 1 ml and the same is ready for direct injection, into the Gas Chromatograph. "Clean-up" is good enough to allow ~ 5,000 G.L.C. injections before the column needs replacing.
5. Only a very minor florisil "clean-up" is required for the electron-capture detector to enable the estimation of the chlorinated hydrocarbons.
6. The automated Storherr apparatus can complete four batches of eight in one day operated by three people. This includes the G.L.C. estimation of organic phosphate and the "clean-up" for G.L.C. of the chlorinated hydrocarbons and recording results. Standard recoveries are continuously checked.

Reagent

Commercial grade petroleum ether under the trade name of Shell X4 is treated as follows:

Shake 2.25 l + 100 ml concentrated sulphuric acid in 2.5 l winchester for one hour. Pour