PRO EXPERIMENTIS

Polarographic Determination of DDT

Older methods of determination of DDT, which are still in use, make use of the partial or complete dehalogenation and subsequent determination of chlorine[1-7], which is disadvantageous on account of lack of specificity.

Colorimetric methods by SCHECHTER et al.[8-10] are based in essence on nitration of DDT and formation of a tetranitroderivative, which in alkaline medium gives a blue coloration. Another colorimetric method of STIFF and CASTILLO is used less frequently, in spite of its simplicity, since methoxychlor reacts in the same way[11].

Polarographically DDT can be determined in 85% ethyl alcohol in the presence of a quaternary base[12]. It gives a well developed wave at – 0.9 V. For determination of residues in biological material, this method cannot however be used on account of the high content of by-products, which frequently cause deformations of the wave, and with regard to its relatively low sensitivity.

In view of a series of favourable results obtained in polarographic determinations of nitration or nitrosation products respectively, of various substances having significance in food and pharmaceutical practice[13-15], it was attractive to utilize the formation of nitroderivatives of DDT for polarography also. The results obtained proved this method to be useful.

Nitration is carried out with a mixture of concentrated sulphuric acid and fuming nitric acid, and proceeds optimally on heating to 90–95°C for about 10 min. After cooling, the nitration mixture is diluted with water and methyl alcohol in a quantity to attain a final concentration of 50% minimum. In this strongly acidic medium the tetranitroderivative is reduced to a single well developed and readable wave (half wave potential in a 20% nitration acid mixture in 50% methyl alcohol – 0.13 V sat. calomel electrode).

The wave shows a diffuse character and is suitable for analytical evaluation (Figure).

The advantage of this newly elaborated polarographic determination of DDT compared with the colorimetric determination, lies in the possibility of using an acid medium directly. By its extraordinary high sensitivity and easy operation it surpasses all other methods, especially that of direct polarographic determination of DDT.

The principle of the present method, the behaviour of the pure tetranitroderivative, the precise working procedure of the nitration and its application in determinations of DDT residues in materials of biological character will be described elsewhere.

Zusammenfassung. Es wird eine schnelle, hochempfindliche polarographische Bestimmung von DDT beschrieben und das durch Nitrieren von DDT erhältliche Tetranitroderivat zur Polarographie verwendet.

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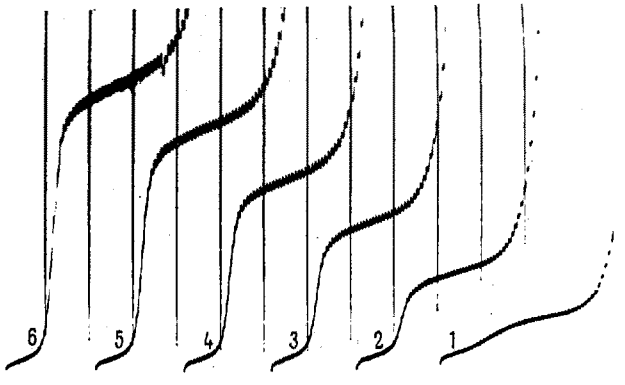

Calibration polarogram of DDT. Resulting concentration of the 20% nitration mixture in 50% methyl alcohol. Concentration of DDT: (1) 0; (2) 0.2; (3) 0.4; (4) 0.6; (5) 0.8; (6) $1 \cdot 10^{-4} M$. Mercurosulfate electrode, sensitivity 1:20; 198 mV/Absc. Performed according to the working procedure described in the text.

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Mikrobiologischer Nachweis von Antibiotika auf Dünnschichtchromatogrammen

Der Antibiotika-Fachmann ist insbesondere daran interessiert, festzustellen, wieviele und was für mikrobiologisch wirksame Fraktionen in einem natürlichen Gemisch vorliegen. Aus diesem Grund haben wir eine mikrobiologische Sichtbarmachung von auf dünnen Schichten von Silicagel G und Silicagur G (Merck AG) getrennten Antibiotika mit *Sarcina lutea* und *Bacillus subtilis* bei Gegenwart von Triphenyltetrazoliumsalzen entwickelt[1] welche hier kurz geschildert werden soll.

Die chromographierten Platten werden zu diesem Zweck in einen Halter aus Plexiglas eingebettet und mit einer Schicht Agar zugedeckt, welche mit dem geeigneten Mikroorganismus und einer kleinen Menge Triphenyltetrazoliumsalz eingeimpft ist. Nach beendeter Entwicklung in einem Brutkasten ist die Schicht dort wo der Mikroorganismus normal wachsen konnte intensiv rotbraun gefärbt, während dort wo das Antibiotikum vorhanden ist,

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