Gas Chromatographic Analysis of Ecdysone

Moulting and metamorphosis in insects is controlled by steroid hormones of the ecdysones group. One of these, α-ecdysone, is the principal moulting hormone in insects[1]. Following the isolation, elucidation of its structure[2] and synthesis, this hormone is now commercially available.

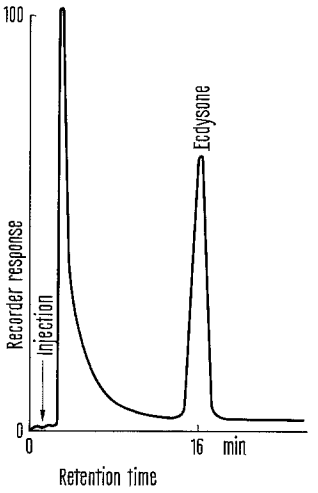
The level of ecdysone in insects has until now been determined by bioassays with *Calliphora erythrocephala*[3], *Musca domestica*[4] and other insects. Although, these assays are very sensitive, their evaluation is based on the percentage of puparium formation in treated animals. An attempt was made to develop a method which would permit a quantitative analysis of α-ecdysone. For this purpose gas-liquid chromatography was used.

α-ecdysone was analyzed as a derivative of *Bis* (Trimethylsilyl) Acetamide (Applied Sci., Pa. USA). Up to 1 mg of α-ecdysone (Hoffmann-La Roche, Basel[5]) was converted into the above derivative using 0.5 ml *Bis* (Trymethylsilyl) Acetamide with 0.1 ml pyridine. The solution was heated to 80 °C for 1 min and directly injected with the reagent into the gas chromatograph.

The sample was analyzed on a Packard gas chromatograph equipped with a flame ionization detector, using an all glass column (3′ × 1/8″) packed with 3% SE-30 on gas-Chrom Q (Applied Sci.). Operating temperatures for the inlet, column and detector were 290, 250, and 280 °C, respectively. Nitrogen was used as a carrier gas at a flow rate of 100 ml/min.

The ecdysone peak appeared 16 min after injection, as shown in the Figure. All other steroids including cholesterol and phytosterols came out with the peak of the solvent and did not interfere with the analysis. Using this procedure 50 ng of α-ecdysone could be detected.

Further investigations are in progress to determine the concentration of this hormone in insects.



Gas chromatogram of 100 ng α-ecdysone.

Résumé. Nous avons développé une méthode d'analyse micro-quantitative de l'ecdysone (hormone de la mue des insectes) par chromatographie gazeuse. Elle permettera l'analyse chimique de l'ecdysone, qui a été dosée jusqu'à présent par des méthodes biologiques.

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An Apparatus for Investigation of Heterogeneous Reactions in a Flow System

Devices of different types have been reported for the study of heterogeneous reactions; a 'one shot' micro reactor[1], a one batch reaction device[2], a pulse reactor[3] and a flow system reactor[4]. Of these, assemblies of the last type are especially useful in the study of catalytic reactions[5].

The apparatus described here is a simple and versatile device for the study of reactions in a continuous flow system at temperatures up to 700 °C. It can be used for liquids, gases or a mixture of the two in the presence of a solid catalyst or an inert support, with or without dilution with suitable carrier. The reactants may be fed separately into the reaction chamber at various flow rates so that mixing takes place only in the reaction chamber. The reaction time can be predetermined and the liquid hourly space velocity (LHSV)[6] can be measured.

Method. The apparatus is composed of 3 main parts (Figure). 1. A feeding device, 2. a reaction chamber and 3. a collector.

1. The feeding device is composed of a Bird Kymograph (A) (Phipps and Bird Inc., Richmond, Virginia, USA, Cat. No. 70-060), in which the shaft is replaced by a threaded one (B) connected to a platform which can thus be raised or lowered. A syringe (c) is located between the platform and the upper shaft holder so that the raising of the platform depresses the plunger and the sample in the syringe is introduced into the reaction chamber (M) at a predetermined velocity through the capillary (D) and hypodermic needle (F). If a carrier gas is used, it is introduced through a second capillary whose terminus (G) is placed about 20 mm above the outlet of the sample needle in order to prevent the con-

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