Monatshefte für Chemie 112, 1103—1104 (1981)

Monatshefte für Chemie

A Microsynthesis of Fluoranthene

Short Communication

Ernest Malý*

Institute of Preventive Medicine, Bratislava, Czechoslovakia

(Received 21 April 1981. Accepted 4 May 1981)

A simple synthesis of fluoranthene based on the distillation of benzanthrone with zinc dust is described, in addition to those cited in *Clar*'s monograph[1]. It may be useful for small scale preparations of this polycyclic hydrocarbon. The pure product was obtained by chromatography.

(Keywords: Polycyclic hydrocarbon; Zinc dust distillation)

Eine Synthese von Fluoranthen (Kurze Mitteilung)

Die Darstellung von Fluoranthen durch Zinkstaubdestillation von Benzanthron und die chromatographische Reinigung des Produktes werden beschrieben.

Benzanthrone (0.15 g) and zinc dust (5.0 g) were mixed thoroughly. The mixture was divided into fifteen portions, and each placed on the bottom of a test tube of high melting glass (8 mm in diameter and 20 cm in length). Each portion of the mixture was covered by four times its depth of pure zinc dust, and the walls of the tubes above the zinc dust were cleaned with cotton wool. The test tubes were then heated at a height of about 2.5 cm and drawn into capillaries about 2 mm in diameter and bent over at about 120° to make microretorts. The glass above the zinc dust was heated until red with a small flame and the flame was shifted slowly towards the bottom. The microretorts were finally cut off at the taper and the capillaries were divided into shorter segments which were extracted five times with cold benzene. The united benzene extracts were concentrated to a small volume; the concentrate was then applied to the whole start line, 1.5 cm from the edge on Whatman 17 paper, impregnated with 10% liquid paraffin petrol ether solution and chromatographed as described earlier[2].

* New address: 90101 Malacky 902, Czechoslovakia.

0026-9247/81/0112/1103/$ 01.00

The skyblue fluorescent zone (R_F 0.46) was cut out and extracted 6 times with cold benzene. The combined extracts after complete evaporation of the benzene, were dissolved in a minute volume of distilled hexane and applied to an alumina column (activity II, 63 × 2.6 cm). The column was washed with hexane until the liquid paraffin was eluted, then the outside of the outlet was washed and elution was continued. Three zones were visible in UV: I, intensely violet fluorescence. After elution colourless plates, m.p. 148 °C, were obtained. II, skyblue fluorescence; after elution and evaporation of the hexane colourless crystals of fluoranthene were obtained (11 mg), m.p. 110 °C. If a benzene solution of this product was chromatographed as described earlier[2], either alone or mixed with authentic fluoranthene, only one spot with blue fluorescence (R_F 0.46) was obtained. Found H 4.8%, C 95.1%. $C_{16}H_{10}$ requires 5.0% H, 95.0% C. III, lemon yellow-green fluorescence zone, was not isolated.

References

[1] *Clar E.*, Polycyclic Hydrocarbons. London-New York: Academic Press. 1964.
[2] *Malý E.*, Mikrochim. Acta **1971**, 800; ibid. **1977**, 429.

Eigentümer: Österreichische Akademie der Wissenschaften, Dr. Ignaz Seipel-Platz 2, A-1010 Wien. — Herausgeber: Österreichische Akademie der Wissenschaften, Dr. Ignaz Seipel-Platz 2, A-1010 Wien, und Verein Österreichischer Chemiker, Eschenbachgasse 9, A-1010 Wien. — Verlag: Springer-Verlag, Mölkerbastei 5, A-1011 Wien. — Für den Textteil verantwortlich: Doz. Dr. Otmar Hofer, Währinger Straße 38, A-1090 Wien. — Für den Anzeigenteil verantwortlich: Mag. Bruno Schweder, Mölkerbastei 5, A-1011 Wien. — Druck: Adolf Holzhausens Nachfolger, Kandlgasse 19-21, A-1070 Wien.
Printed in Austria