

Preliminary communication

On the Nitration of Retene

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In spite of several attempts to prepare a well defined nitro compound of retene (1-methyl-7-isopropyl-phenanthrene) by direct nitration, these have not been followed by success^{1, 2}.

However, we have succeeded in preparing a nitroretene in pure state by nitration of retene under mild conditions followed by chromatographic separation and molecular distillation of the reaction product. The nitro compound obtained has been identified as the 9-nitro compound.

Experimental. 75 ml of concentrated nitric acid (about 65%) was slowly added to a suspension of 20 g of retene in 200 ml *n*-butyric acid. The temperature was not allowed to rise above -6°C .

The clear orange coloured reaction mixture was poured into 1.5 l of water and the precipitated oil was dissolved in 600 ml of petroleum ether (b. p. 60—80°C). After the solution had been dried with sodium sulphate it was chromatographed on 150 g of aluminium oxide (activated by treatment with sodium hydroxide). Several zones were observed including a purple and a yellow one.

By elution of the yellow zone with benzene and dilution of the eluate with petroleum ether we obtained, on chilling the

solution in an ice box, 1.2 g of a yellow crystalline compound (m. p. 115—120°C, corr.).

The compound was then submitted to a molecular distillation at $5 \times 10^{-4}\text{mmHg}$. The distance between the heated and the cooled surface was 1 cm. At 62—67°C a yellow compound, which crystallized slowly on standing, was removed by distilling and at 72—77°C a yellow crystalline compound was collected. This compound was recrystallized from petroleum ether and melted at 123.5—125°C (corr.).

$\text{C}_{18}\text{H}_{17}\text{O}_2\text{N}$ (279.3)

Calc. N 5.02 Found N (Dumas) 5.08

A mixture of our compound and 9-nitroretene (m. p. 123—125°C), prepared by deaminating 9-nitro-3-aminoretene² melted at 123—125°C (corr.).

A detailed description will be published later.

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2. Karrman, K. J., and Sihlbom, L. *Svensk Kem. Tid.* 58 (1946) 189.

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