

2.4–1.9 [m, 4H, =C(CH₂-)₂], 1.6–1.3. [m, 6H, (-CH₂)₃-]. Mol.wt. 123 (MS).

3-Ethyl-6-methyl-4,5-heptadien-3-ol (IIb). Prepared as described for IIa from Ib (10.0 g; 0.039 mol) and LiAlH₄ (2.1 g; 0.055 mol) but refluxed for 15 min only. B.p. 70°/10 mmHg. Yield: 78 %. IR: 1960 cm⁻¹ (m, C=C=C). Mol.wt. 154 (MS).

5-Ethyl-2-methyl-2,4-heptadiene (IIIb). *Method 1.* Prepared as described for IIIa from IIb (5.0 g; 0.033 mol) and LiAlH₄ (1.7 g; 0.045 mol). B.p. 70°/35 mmHg. Yield 90 %. IR: 1610 cm⁻¹ (m, C=C), 845 cm⁻¹ (s, -CH=) NMR (neat): δ (ppm) = 5.7 (m, 2H, =CH-CH=), 2.3–1.8 [two superimposed q, 4H, =C(CH₂-)₂], 1.70 and 1.63 [two s, 6H, =C(CH₃)₂], 1.0 (t, 6H, -CH₂-CH₃), Mol.wt. 138 (MS).

Method 2. The procedure was analogous to that described for IIIa, *Method 2*, starting with Ib. (7.2 g; 0.028 mol) and LiAlH₄ (2.7 g; 0.074 mol). Yield: 70 %.

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The Hydrolysis of α-Sulphanuric Chloride, α-(NSOCl)₃

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According to Becke-Goehring¹ the hydrolysis of sulphanuric chloride (I) should lead to sulphanuric acid (II), but that this substance (II) is clearly very unstable and that even on careful acid hydrolysis imidosulphamide (IV) and sulphuric acid are formed immediately, the step from sulphanuric acid (II) to imidosulphamide (IV) presumably being *via* trisulphimide (III).

Hydrolysis of α-sulphanuric chloride with dilute silver nitrate solution yielded silver chloride and also colourless needle-shaped crystals which were identified by X-ray diffraction as the silver salt, Ag₃(NSO₂)₃·5H₂O, of trisulphimide (III). This shows that in the case of α-(NSOCl)₃ hydrolysis is possible with out breaking the ring and supports the view that trisulphimide is an intermediate in the hydrolysis of α-(NSOCl)₃.

A crystal structure determination² shows that the (NS)₃ ring in the trisulphimide ion retains the chair form which exists³ in α-sulphanuric chloride.

Experimental. The hydrolysis was carried out on a microscope slide using "bench" silver nitrate solution. The sulphanuric chloride was kindly provided by Dr. A. J. Banister.

X-Ray rotation and Weissenberg photographs were taken with CuKα radiation. Comparison of cell dimensions and intensities with those published by Fischer and Andress⁴ established the identity of the hydrolysis product as Ag₃(NSO₂)₃·3H₂O.

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