



and the peaks at 12.15, 12.32, 12.55 and 12.65 μ must be due to the presence of the Se-O groups. (IR-spectra of selenoxides have not been described in the literature.) A comparison of this peak system with the characteristic absorption peaks for S-O in the *trans*- and *cis*-form of thianthren-5,10-dioxide suggests that the selenoxide is not a pure *trans*- form but rather a *cis*-form or a mixture of both or perhaps a dioxide with planar configuration ⁶.

Experimental: *Selenanthren-5,5-10,10-tetrachloride (II)*. To a solution of 1.55 g of selenanthrene in 100 ml of dry benzene, 5.0 g (excess) of SO_2Cl_2 in 50 ml of benzene was added. The mixture was boiled under reflux for 30 min. The tetrachloride separated as a yellow fine crystalline solid. About 70 ml of benzene was then removed by distillation, and from the cooled reaction mixture the tetrachloride was obtained by filtration. It was washed with dry benzene and then dried in vacuum, m. p. 208–209.5° (decomp.). Yield 2.10 g (89 %). (Found: Se 34.3; 34.7; Cl 31.3. Calc. for $\text{C}_{12}\text{H}_8\text{Se}_2\text{Cl}_4$: Se 34.9; Cl 31.4).

Selenanthren-5,10-dioxide dihydrochloride (III). 2.00 g of selenanthren tetrachloride was hydrolysed with 100 ml of boiling water. After filtration and evaporation the dihydrochloride was obtained as a white solid in quantitative yield, m. p. 228–229° (decomp.). (Found: Se 38.1; Cl 17.1. Calc. for $\text{C}_{12}\text{H}_8\text{O}_2\text{Se}_4 \cdot 2\text{HCl}$: Se 38.4; Cl 17.1).

Selenanthren-5,10-dioxide (IV). 1.25 g of selenanthren-5,10-dioxide dihydrochloride was dissolved in 100 ml of water. 0.56 g of NaHCO_3 was added and the solution evaporated on a water bath. The residue was extracted with 100 ml of boiling chloroform and after evaporation the crude dioxide (m. p. 256–258°) was obtained in quantitative yield. This product was washed with 50 ml of ether and 15 ml of water and then recrystallized from water. The dioxide separated on cooling as a hydrate which

was dried to constant weight at 120°. In this way selenanthren-5,10-dioxide with m. p. 264–265° (decomp.) was obtained. Yield 0.68 g (66 %). (Found: Se 46.3. Calc. for $\text{C}_{12}\text{H}_8\text{O}_2\text{Se}_4$: Se 46.2). The selenium analyses have been carried out according to Fredga ⁷.

Thanks are due to Mr. Stig Bergwall for technical assistance with the IR-spectra. A grant from the *Swedish Natural Science Research Council* is gratefully acknowledged.

1. Campbell, T. W., Walker, H. G. and Copping, G. M. *Chem. Revs.* **50** (1952) 335.
2. Gould, E. S. and McCullough, J. D. *J. Am. Chem. Soc.* **73** (1951) 1105.
3. Gould, E. S. and Burlant, Wm. *J. Am. Chem. Soc.* **78** (1956) 5825.
4. Krafft, F. and Kaschau, A. *Ber.* **29** (1896) 443.
5. Cullinane, N. M. *J. Chem. Soc.* **1951** 237.
6. Bergson, G. *To be published.*
7. Fredga, A. *Diss. Uppsala Universitets Årsskrift* 1935:5, p. 16.

Received March 20, 1957.

Preparation of Lithium Azide

NIELS HOFMAN-BANG

Chemistry Department A, Technical University of Denmark, Copenhagen, Denmark

The most common methods for the preparation of lithium azide, LiN_3 , are interaction of lithium sulfate and barium azide, and, especially, neutralization of aqueous hydrazoic acid with lithium hydroxide. Considering that preparation and handling of hydrazoic acid require care, it occurred to me that sodium azide and

