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Organic Selenium Compounds

V. Diselenolylium Salts

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By oxidation of dithiomalonamide, salts of the 3,5-diaminodithiolyl cation or 3,5-diaminodithiolylium ion * have been obtained. 4,2 When diselenomalonamide be-

came available in this laboratory,³ we tried to prepare the corresponding 3,5-diselenolylium salts, but our first attempts, using hydrogen peroxide as oxidant, were unsuccessful.³ However, when iron(III) chloride or iodine were used as oxidants, 3,5-diaminodiselenolylium chloride or iodide (1) were obtained without difficulty.

The properties of these diselenolylium salts are very similar to those of the corresponding dithiolylium salts and their infrared spectra are very similar. The infrared bands of the chloride (in KBr) were 3400m, 3250s, 3100s, 1630m, 1604s, 1510—1520vs, 1355m, 1272m, 1140w, br, 800m, 785w. As in the case of the dithiolylium salts the iodide showed a shift of some of the bands to lower frequencies (1610m, 1590s, 1330m), probably due to a lattice effect (for discussion of the infrared spectra, see the paper on the dithiolylium salts 1).

Experimental. 3,5-Diaminodiselenolylium chloride. Diselenomalonamide 3 (1.14 g; 5 mmol) was dissolved in ethanol by heating at 40°C. The solution was cooled and filtered from some selenium that had separated, and one equivalent of iron(III) chloride, dissolved in ethanol, was added with stirring. After $\frac{1}{2}$ h, pentane was added, which induced the precipitation of a yellow crystalline solid. Yield 0.90 g (76 %). It was recrystallized from aqueous methanol. On heating it decomposed above 200°C without melting. (Found: C 13.79; H 2.02; N 10.61. Calc. for $\rm C_3H_5Se_2Cl$: C 13.73; H 1.92; N 10.68).

3,5-Diaminodiselenolylium iodide. An ethanolic solution of diselenomalonamide (0.456 g) was oxidized with one equivalent of iodine (0.508 g) dissolved in ethanol. The solution was stirred for ½ h at room temperature and the resulting salt isolated as above. Yield 0.45 g (68 %) of a yellow crystalline substance, which decomposed on heating above 200°C without melting. It was recrys-

^{*} In the literature, the dithiolyl cation is often called the "dithiolium" ion. As was pointed out earlier, this name is misleading because stoichiometrically the dithiolyl cation is a didehydrodithiolium ion. According to the IUPAC rules for organic chemical nomenclature, cations formed by loss of an electron from an -yl radical may be denoted by the ending -ylium (this ending being considered as an entity and not to imply that the ion is formed by addition of a proton to the radical). Accordingly, the dithiolyl and diselenolyl cations may be called dithiolylium and diselenolylium ions.

tallized from ethanol with addition of a little dimethylformamide. (Found: C 10.41; H 1.50; N 7.89. Calc. for $\mathrm{C_3H_5Se_2I}$: C 10.18; H 1.42; N 7.92).

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