SYNTHESIS OF SELENOPHENE-2,5-DICARBOXYLIC ACID

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Selenophene-2,5-dicarboxylic acid was obtained by liquid-phase oxidation of 2,5-dimethylselenophene with molecular oxygen.

Methods for the preparation of selenophene-2,5-dicarboxylic acid by oxidation of 2,5-diformylselenophene with moist silver oxide at 0-10°C [1] and by thermal disproportionation of selenophene-2-carboxylic acid [2] are known. The disadvantages of these methods are the low yields and the laboriousness of the preparation of 2,5-diformylselenophene.

We have obtained selenophene-2,5-dicarboxylic acid by oxidation of 2,5-dimethylselenophene with molecular oxygen in acetic acid at 80-98° and atmospheric pressure in the presence of a mixture of acetates of divalent cobalt ($5-7.5 \cdot 10^{-2}$ mole/liter) and manganese ($0.17-0.23 \cdot 10^{-4}$ mole/liter) as catalyst and 9,10-dibromoanthracene ($0.8-1.8 \cdot 10^{-2}$ mole/liter) as initiator by the method proposed for the oxidation of alkylaromatic hydrocarbons.

EXPERIMENTAL

A 1.5-g ($0.6 \cdot 10^{-2}$ mole) sample of cobalt acetate and 0.5 g ($1.5 \cdot 10^{-3}$ mole) of 9,10-dibromoanthracene in 60 ml of glacial acetic acid were heated to 80°C with stirring, and 2.19 g ($1.37 \cdot 10^{-2}$ mole) of 2,5-dimethylselenophene [bp 155-156.5° (746 mm), n_D^{20} 1.5498] and 0.003 g ($0.173 \cdot 10^{-4}$ mole) of manganese acetate, dissolved in 18 ml of acetic acid, were added. The mixture was then heated to 90-100° and stirred for 4 h while oxygen was bubbled in at 0.2 liter/min. At the end of the reaction, the mixture was cooled to room temperature, and the solid material was removed by filtration, washed with glacial acetic acid and cold water, and treated with 1 M sodium hydroxide solution. The mixture was filtered, and the filtrate was acidified with hydrochloric acid. The precipitated selenophene-2,5-dicarboxylic acid was washed with ice water and dried to give 1.1 g (37%) of a product with mp 310-311° (dec., from water). Found,%: C 33.0; H 1.8. $C_6H_4O_4Se$. Calculated,%: C 32.9; H 1.8.

LITERATURE CITED

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2. A. S. Angeloni and M. Tramontini, Boll. Sci. Fac. Chim. Ind. Bologna, 23, 301 (1965).

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