REACTION OF 2,4-BIS-O-(TRIMETHYLSILYL)-5-FLUOROURACIL WITH SELENIUM TETRABROMIDE

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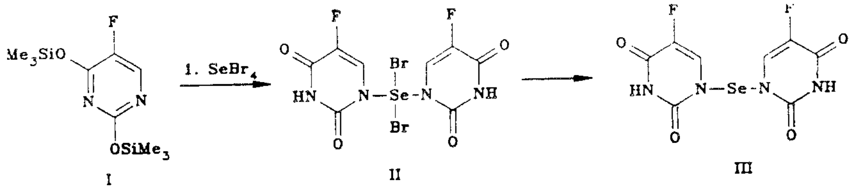
We have studied the reaction of selenium tetrabromide with 2,4-bis-O-(trimethylsilyl)-5-fluorouracil (I), which leads to the formation of bis(5-fluoro-1-uracilyl)selenium dibromide (II).

The reaction is regiospecific, and varying the mole ratio of (I:SeBr$_4$) from 1:1 to 2:1 leads only to a change in the yield of II.



The structure of compound II was adduced based on its PMR, IR, and UV spectral data. The PMR spectrum contains a signal for the 6-H proton in the 8.0-ppm region. The IR spectrum contains an intense band at 550 cm^{-1}, which is characteristic of a N–Se bond [1].

The selenium dibromide II can be debrominated using a standard procedure [2] to give bis(5-fluoro-1-uracilyl)selenide (III). As the pH of the medium is changed from neutral to basic, the UV spectra of compounds II and III do not exhibit a bathochromic shift of the main absorption maximum, which is characteristic of N$_{(1)}$-substituted uracils [3]. For compound II, λ_{max} in water is 290 nm; for selenide III, 267 nm.

Bis(5-fluoro-1-uracilyl)selenium Dibromide (II). To a solution of 2.2 g (8 mmoles) compound I in 20 ml chloroform was added dropwise to a solution of 1.6 g (4 mmoles) SeBr$_4$ ion 10 ml chloroform at 10°C under a nitrogen atmosphere. The mixture was stirred for 2 h. Absolute ethanol (15 ml) was added, and the resulting precipitate of 5-fluorouracil was removed by filtration and washed with chloroform (2 × 5 ml). The combined filtrates were evaporated and the residue recrystallized from a mixture of ethanol–hexane (1:3); 1.2 g (60%) of selenium dibromide II was isolated, mp 146-148°C (decomp.); R$_f$ 0.54 (acetic acid–benzene–ethanol, 10:9:6). PMR spectrum (C$_2$D$_5$OD): 8.0 ppm (1H, s, 6-H). IR spectrum: 550 (N–Se), 1680 (C=C), 1740 cm^{-1} (C=O).

Bis(5-fluoro-1-uracilyl)selenide (III). Compound II (1 g, 2 mmoles) was dissolved upon heating in 25 ml acetone and the solution was refluxed for 1 h. The solvent was evaporated until crystals appeared. Yield of selenide III, 0.34 g (50%). mp 120-122°C (decomp.); R$_f$ 0.48.

LITERATURE CITED

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