Chemistry of Heterocyclic Compounds, Vol. 42, No. 10, 2006

SYNTHESIS OF THE FIRST REPRESENTATIVES OF BENZANNELATED DIHYDROSELENOCHROMENES

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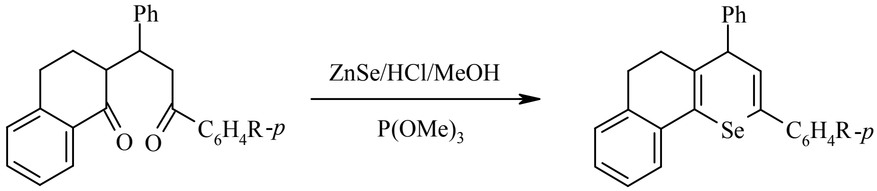
Keywords: 1,5-diketone, zinc selenide, 5,6-dihydrobenzo[*h*]selenochromene.

Benzannelated 5,6-dihydrothiochromenes became available relatively recently [1], but until now their selenium-containing analogs have been unknown.

We have for the first time realized the synthesis of 2,4-diaryl-5,6-dihydrobenzo[*h*]selenochromenes **1** and **2** by the reaction of 2-(1,3-diaryl-3-oxopropyl)tetrahydronaphthalen-1-ones **3** and **4** with hydrogen selenide *in situ* under the conditions of acid catalysis.



3, **4**

1, **2**

1, **3** R = H, **2**, **4** R = Cl

The reaction was conducted in the presence of trimethoxyphosphine, produced by the reaction of phosphorus(III) chloride with methanol. Here the necessary concentration of hydrogen chloride (7-8 M) was created, and the secondary disproportionation processes of the dihydrobenzoselenochromenes were suppressed [2]. The yields of the dihydrobenzoselenochromenes **1** and **2** amounted to 91 and 67% respectively.

Chromato-mass spectrometric investigations showed that thermal isomerization of the dihydrobenzoselenochromene **1** occurs under the conditions of GLC (EI 70 eV, 280°C) with the formation of four isomers (molecular ion *m*/*z* 400 for the Se^{80} isotope). In addition, the chromatogram contains signals for two products with molecular ions *m*/*z* 398 and 402 (for Se^{80}), which by analogy with 2,4-diphenyl-5,6-dihydrobenzo[*h*]thiochromene [1] probably result from the disproportionation of compound **1** with the participation of the heterocyclic and alicyclic fragments.

The ^1H NMR spectra were recorded in $CDCl_3$ on a Varian FT-80a instrument (80 MHz) with TMS as internal standard.

2,4-Diphenyl-5,6-dihydrobenzo[*h*]selenochromene (1). To methanol (20 ml), placed in a flask with the minimum amount of free space, we added with constant stirring and cooling PCl_3 (9.42 g, 68 mmol), compound **3** (1.42 g, 4 mmol), and ZnSe (0.72 g, 5 mmol). The mixture was stirred at room temperature for 7 h. The separated crystals were removed, washed with ethanol, and dried. We obtained 1.27 g of compound **1**. The

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mixture was extracted with diethyl ether (50 ml), the extract was washed with water to a neutral reaction and dried, and the solvent was evaporated. The oily product was passed through a column of Al_2O_3 (eluent 10:1 hexane–ether). A further 0.17 g of compound **1** was obtained (total yield 91%); mp 108-109°C (reprecipitated from diethyl ether with ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 6.87-7.49 (14H, m H_{Ar}); 6.30 (1H, d, *J* = 5.8, β-H); 4.37 (1H, d, *J* = 5.8, γ-H); 2.88-2.67 (2H, m, CH_2) 2.38-2.19 (2H, m, CH_2). Found %: C 74.95; H 5.27. $C_{25}H_{20}Se$. Calculated %: C 75.19; H 5.01.

2-(4-Chlorophenyl)-4-phenyl-5,6-dihydrobenzo[*h*]selenochromene (2). This compound was obtained similarly to compound **1**, and the reaction took 37 h. Yield 67%; mp 126-128°C (reprecipitated from diethyl ether with ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.05-7.49 (13H, m, H_{Ar}); 6.24 (1H, d, *J* = 5.4, β-H); 4.34 (1H, d, *J* = 5.9, γ-H), 2.86-2.49 (2H, m, CH_2); 2.35-1.97 (2H, m, CH_2). Found %: C 69.03; H 4.66. $C_{25}H_{19}ClSe$. Calculated %: C 69.20; H 4.38.

The work was carried out with financial support from the Russian Fundamental Research Fund (grant 06-03-3266a).

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