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A IONIC HYDROGENATION OF ARYL-SUBSTITUTED 4H-SELENOPYRANS

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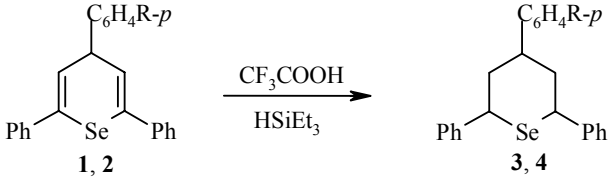
Aryl-substituted 4H-thiopyrans readily undergo catalytic and ionic hydrogenation [1]. Their seleno analogs readily eliminate elemental selenium in attempts to perform catalytic and other traditional methods of hydrogenation [2]. Aryl-substituted selenacyclohexanes were obtained and characterized upon the disproportionation of 4H-selenopyrans under acid catalysis conditions [3].

We are the first to report the ionic hydrogenation of 2,4,6-triaryl-4H-selenopyrans **1** and **2** to give the corresponding 2,4,6-triarylselenacyclohexanes.



1, **2** **3**, **4**

1, **3** R = H; **2**, **4** R = OMe

GC/MS analysis of the reaction mixture indicated that the isomer content of the sample of 2,4,6-triphenylselenacyclohexane (**3**) obtained by ionic hydrogenation was identical to the isomer content of the product obtained by disproportionation [3] and contained more than 97% of one isomer, while the second isomer was present as an impurity. (*p*-Methoxyphenyl)-2,6-diphenyl-4-selenacyclohexane (**4**) was obtained as a mixture of four isomers; the content of the major isomer was about 70%. Selenacyclohexane **4** obtained by disproportionation contained more than 90% major isomer.

The GC/MS analysis of the samples was carried out on an HP5890/5972 unit equipped with an HP-5MS capillary column. The injector temperature was 200°C. The onset time was 3 min. The onset temperature was 50°C. The final temperature was 280°C, ΔT = 10°C/min. Helium served as the gas carrier, v = 1 ml/min. The ^{1}H NMR spectra were obtained at 30°C on a Varian FT 80A spectrometer at 80 MHz in $CDCl_3$ using TMS as the internal standard.

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Preparation of Selenacyclohexanes 3 and 4 (General Method). Triethylsilane (0.3 ml, 2.1 mmol) was added to selenopyran (1 mmol) and then trifluoroacetic acid (0.37 ml) was added with stirring. After the reaction mixture became a homogeneous liquid phase, it was cooled to room temperature. The crystalline precipitate was filtered off, analyzed by GC/MS, and recrystallized from ethanol to give compound **3** and compound **4** as single isomers.

2,4,6-Triphenylselenacyclohexane (3) was obtained in 79% yield (0.297 g), mp 110-112°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.37-2.50 (4H, m, β-H); 2.51-2.84 (1H, m, γ-H); 4.48 (2H, dd, *J* = 3.9, *J* = 11.8, α-H); 7.14-7.39 (15H, m, C_6H_5).

4-(*p*-Methoxyphenyl)-2,6-diphenylselenacyclohexane was obtained in 85% yield (0.343 g) (38% after recrystallization), mp 116-118°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.32-2.72 (5H, m, β-H + γ-H); 3.71 (3H, s, OCH_3); 4.35 (2H, dd, *J* = 4.3, *J* = 9.6, α-H); 6.71 (2H, d, *J* = 8.8, arom); 7.05-7.33 (12H, m, arom).

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