METHOD FOR THE GENERATION OF THE PHENYLETHYNESELENOLATE ANION FROM METALLIC SELENIUM AND PHENYLACETYLENE UNDER PHASE TRANSFER CATALYSIS CONDITIONS

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A method has been reported for the preparation of alkyl(phenylethynyl)selenides, whose formation proceeds through the phenylethyneselenolate anion generated from selenium and phenylacetylene in the KOH/HMPTA superbase system [1].

We have developed a method for the generation of the phenylethyneselenolate anion in a three-phase system containing metallic selenium, aqueous KOH, and a solution of phenylacetylene in toluene in the presence of a phase transfer catalyst. The yield of the phenylethyneselenolate anion was determined relative to the product of its alkylation by butyl bromide and is 50% relative to the selenium taken.

$$\mathrm{Se} + \mathrm{PhC{\equiv}CH} + \mathrm{KOH} \xrightarrow[85-90^\circ]{\mathrm{MeR_3N^+MeSO_4^-}} \mathrm{PhC{\equiv}CSe^-} \xrightarrow{\mathrm{BuBr}} \mathrm{PhC{\equiv}CSeBu}$$
$$\mathrm{R} = \mathrm{C_7H_{15}} - \mathrm{C_9H_{19}}$$

Under analogous conditions, the yield of butyl(phenylethynyl)selenide in the absence of the phase transfer catalyst is only 1%. Despite the formation of selenide anions in aqueous alkali from metallic selenium [2], the products of the addition of these anions to phenylacetylene were not detected under the reaction conditions.

Butyl(phenylethynyl)selenide, bp 156°C (1 mm), n_D^{20} 1.6013. IR spectrum (ν, cm^{-1}): 2145 ($C{\equiv}C$). PMR spectrum (δ, ppm, in $CDCl_3$): 7.22 m (C_6H_5), 2.80 t ($SeCH_2$), 1.77 m ($SeCH_2CH_2$), 1.44 m (CH_2CH_3), 0.95 t (CH_3). Mass spectrum (m/z): M^+ 238. Found: C 60.50; H 5.98; Se 34.17%. Calculated for $C_{12}H_{14}Se$: C 60.79; H 5.90; Se 33.30%.

LITERATURE CITED

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2. V. A. Potapov, N. K. Gusarova, S. V. Amosova, et al., Zh. Org. Khim., 22, 276 (1986).

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