GAS PHASE SYNTHESIS OF SELENOPHENE FROM DIMETHYL SELENIDES AND ACETYLENE

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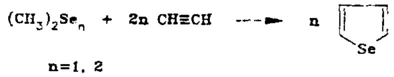
The reaction of acetylene with elemental selenium at 450°C is the most important method for the preparation of selenophene [1, 2]. The method has been subjected to repeated improvements with the object of increasing the contact surface of the reactants [3, 4]. However, this heterogeneous reaction is of low efficiency on account of the need to pass acetylene at high rates to prevent selenium being carried out of the reactor. Another drawback of the known method is its low rate.

We have developed a continuous method for the preparation of selenophene based on the gas phase reaction of acetylene with dimethyl selenide or -diselenide:



Selenophene is formed from dimethyl selenide at 420-470°C, or from the diselenide at 400-420°C, in yields of 84-96 and 78-85% respectively. The conversion of $(CH_3)_2Se_n$ amounts to 80-96%. Side products of the reaction are aromatic hydrocarbons (benzene, toluene, xylene, naphthalene, diphenyl), methyl selenophene, and elemental selenium. When n = 2 $(CH_3)_2Se$ is also formed and the amount of Se separating is increased.

A quartz tube 25 × 630 mm was heated to the required temperature and $(CH_3)_2Se_n$ passed through at a rate of 10-20 ml/h in a current of acetylene (5-12 liters/h). The condensate was collected in a trap cooled to −40°C and examined by GLC using an LKhM-8MD-2 chromatograph with XE-60 liquid phase on Chezasorb N-AW-HMDS, column 2 m × 3 mm. Selenophene was separated by fractionation of the condensate from the trap, bp 105-110°C (purity, 98% of the basic component). The structure of all the reaction products was confirmed by GLC-mass spectrometry (MAT-12 instrument, liquid phase SE-54, ionization potential 70 eV), NMR, and IR spectroscopy.

LITERATURE CITED

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