Synthesis of Crown Ethers on the Basis of 4-isopropyl-1,2-cyclohexanediene Dioximes

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Macrocyclic polyethers have extraordinary chemical features that include ability to form coordinative compounds with metal cations in crystalline form and in solution. Macrocyclic compounds are characterized by high resistance and catalytic activity and are extractant of different metals. They are successfully applied in analytic chemistry, thin organic chemistry, phase transfer catalysis, ecology, medicine, electrochemistry, metallurgy and agronomy. There is no information about synthesis of crown ethers on the basis of cyclic dioximes in the literature. Recently we have synthesized nitrogen-containing crown ethers based on aliphatic and alicyclic dioximes. [1-3] Continuing the research in this direction we developed the method of obtaining new crown ethers by polycondensation reaction of 4-isopropyl-1,2-cyclohexanediene dioximes (4-isopropyl-noxime) with ethylene oxide or choloroethanol in the presence of alkali. The reaction is held in benzene sphere at 25-30°C and at a constant addition of an excess of ethylene oxide or chororoethanol. The length of the reaction is 10–12 hours. The polycondensation reaction of 4-isopropyl-noxime with ethylene oxide or choloroethanol in presence of alkali proceeds in accordance with the scheme stated below:

where, n=4-16.

The polycondensation reaction is held until the end allocation of water that is collected in a trap of Din Stark through an azeotrope mixture of benzene – water. The solvent is removed on a rotary evaporator and the rest is acidified with 5 ml of concentrated HCl (until the acid reaction by universal indicator), it is washed, filtered, and then it is washed with acetone and dried in an oven.

Developed method of preparation of crown ethers helps to avoid side reactions of oligomerization and isomerization of the starting compounds and to achieve quantitative yield of target products of 558 – 76 %. The structure of 4-isopropyl-cyclohexane-1,2-diae-18-crown-6-ethers is proved by NMR ¹¹N, IC and mass – spectroscopy. IC spectrum: the presence of stripes in the field of 1135–1138 sm⁻¹ is characteristic of crown ether's fragment, for the polyether chains 1440-1500 sm⁻¹, 1450, 1350 (V₁), 1120 (V₂) sm⁻¹. NMR spectrum ¹H, o.m.d.: ~ 8.2–7.3 m.d., 3.56 (s.), 4.42 (s.), 7.32 (m.) m.d., proton signals of crown ethers appear as multiplets in the field of 4.2–2.6 m.d.

Preliminary tests of the synthesized nitrogencontaining macrocyclic compounds showed the expressed ability of the latter to complexformation and highly selective attenuation while extraction of metals in various fields.

REFERENCES

