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MACROHETEROCYCLES.

38.* SIMPLE SYNTHESIS OF AN UNSYMMETRICAL CYLINDRICAL TRICYCLIC CRYPTAND

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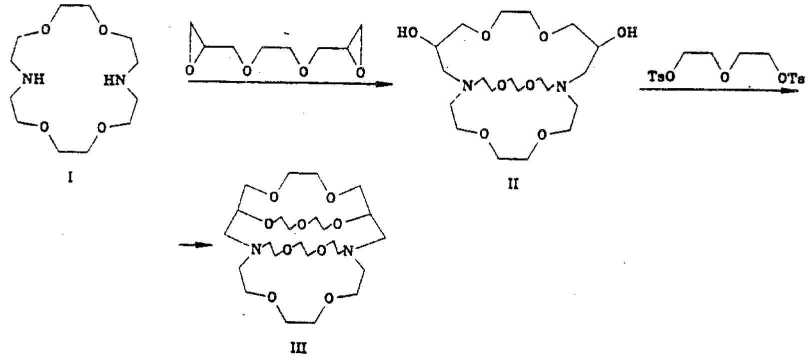
UDC 547.898.07

Cylindrical cryptands composed of two different crown ether rings as substructures are capable of the selective formation of binuclear inclusion complexes (cryptates) with two different metal ions or with organic dications; this opens up broad possibilities in terms of practical applications. Unfortunately, however, the synthesis of these types of cryptands is often multi-stage and complex [1, 2].

Using the synthesis of cryptand III as an example, we have demonstrated the feasibility of a relatively simple procedure for the preparation of cylindrical tricyclic cryptands, in which two different crown ether rings are bound via a methylene group.

Treatment of equimolar amounts of the diglycidic ether of ethylene glycol [3] and diaza-18-crown-6 [4] in a refluxing mixture of dry ethanol and tetrahydrofuran (1:1) resulted in the formation of the dihydroxycryptand II, which was isolated by column chromatography on neutral alumina. Condensation of dihydrocryptand II with diethylene glycol ditosylate in refluxing dioxane in the presence of sodium hydride gave the cylindrical tricyclic cryptand III.



3,10-Dihydroxy-5,8,15,18,23,26-hexaoxa-1,12-diazabicyclo[10.8.8]octacosane (II). Yield 69%, mp 82-83°C. Mass spectrum: m/e 436 (M^+). PMR spectrum ($CDCl_3$): 2.63 (m, 12H, NCH_2); 3.55 ppm (m, 28H, OCH_2, CH–O, OH).

4,7,13,16,19,24,27,31,34-Nonaoxa-1,10-diazatricyclo[18,17.1.$1^{10,12}$]pentatriacontane (III). Yield 19%, oil. Mass spectrum: m/e 506 (M^+). PMR spectrum ($CDCl_3$): 2.62 (m, 12H, NCH_2); 3.67 ppm (m, 34H, OCH_2, OCH).

The results of elemental analysis of compounds II and III corresponded to the calculated values.

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*For Communication 37, see [5].

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ALKYLATION OF 2-PHENYLBENZOTELLURAZOLE

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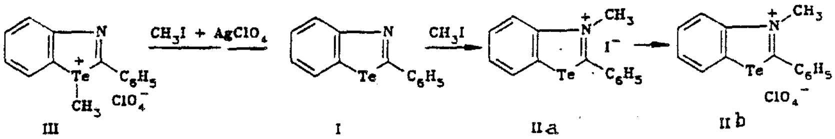
UDC 547.789.9

Benzotellurazoles have two potential nucleophilic centers, their nitrogen and tellurium atoms. We have found that the direction of alkylation of these compounds depends on the reaction conditions. For instance, heating 2-phenylbenzotellurazole (I) with excess methyl iodide in a sealed ampoule at 100°C results in the formation of the immonium salt IIa in 90% yield. Its PMR spectrum in CF_3COOH solution contains a methyl proton signal at 3.81 ppm, which is almost superimposable with the chemical shift values of the N-methyl group in 1-methyl-2-phenylbenzoxazolium and 1-methyl-2-phenylbenzothiazolium iodides (3.95 and 3.91 ppm, respectively). In contrast, however, treatment of compound I with methyl iodide in the presence of an equivalent amount of silver perchlorate in acetonitrile gives the telluronium salt III in 95% yield. Its PMR spectrum in $DMSO-D_6$ solution shows the methyl group signal at 2.28 ppm (the methyl group signals in diarylmethyltelluronium halides generally occur in the 2.7-3.0 ppm region [1], while the chemical shift value for the Te-methyl protons in 3,5,7-trimethyl-10-ethylphenotelluroniumazine is 2.55 ppm [2]).

Reaction of iodide IIa with an equimolar amount of silver perchlorate in acetonitrile gives an almost quantitative yield of perchlorate IIb, in which the N-methyl proton signal (in CF_3COOH solution) appears at 3.88 ppm.



The results of elemental analysis and IR spectra of compounds II and III were consistent with the proposed structures.

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