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## 1,8-BIS(9'-CARBAZOLYL)-3,6-DIOXAUCTANE: SYNTHESIS AND STRUCTURE

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The most common phase transfer catalysts are known to be crown-esters, cryptands, quaternary ammonia salts and podands. The podand kind of phase transfer catalysts is the most efficient in chemical technology of organic substances. The aim of this investigation is the syntheses of new podand containing two carbazole cycles and triethylene glycol chain. The method of the synthesis of 1,8-bis(9'-carbazolyl)-3,6-dioxauctane has been developed. This podand containing two carbazolyl groups is stable in alkaline media. The reaction of the synthesis is carried out in the 1,4-dioxane solution. Carbazole reacts with an excess of potassium hydroxide in the boiling solvent in the presence of tetrabutyl ammonium bromide. Then the hot solution of potassium salt of carbazole is filtered off. Subsequently, the reaction of potassium salt of carbazole with 1,8-dibromo-3,6-dioxauctane is carried out in 1,4-dioxane solution. After boiling the reaction mixture is cooled. The obtaining potassium bromide is filtered off. The 1,4-dioxane was removed by vacuum. The residue is washed with water and dried at a reduced pressure. The product is extracted by boiling carbon tetrachloride. After evaporating the solvent the crude product is obtained with a yield of 77%. The solid residue is crystallized from benzene or ethanol yielding the pure product. The additional quantity of the product can be obtained by column chromatography of the remaining filtrate. The overall yield of pure 1,8-bis(9'-carbazolyl)-3,6-dioxauctane is 54%. The structure of the obtained compound has been confirmed by NMR <sup>1</sup>H spectra, NMR <sup>13</sup>C spectra, mass-spectra and XRD study.

**Keywords:** podands, phase transfer catalysts, carbazol, 1,8-bis(9'-carbazolyl)-3,6-dioxauctane, structure.

### Introduction

The main kinds of phase transfer catalyses (PTC) are quaternary ammonia salts, crown-esters, cryptands and podands [1–4]. The PTC of mixed type, hybrides of podands and quaternary ammonia salts, were obtained then [5–10]. It was found that 3,8-bis(trialkylammonium)-3,6-dioxauctane salts were an effective PTC for the trialkylorthoformates syntheses in the hetero phases systems [6,7,9,10]. Also it was found that 3,8-bis(trialkylammonium)-3,6-dioxauctane salts were an effective inhibitors of iron corrosion [11,12]. But the quaternary ammonia salts slowly decompose in alkali media at the heating.

Then it was shown that 3,8-bis(dialkylamino)-3,6-dioxauctanes are also effective PTC for the synthesis of trialkylorthoformates from primary alcohols, trichloromethane and alkali hydroxides [10,13]. These podands are stable in the alkali

solutions at the high temperatures.

The aim if our investigation is synthesis of 1,8-bis(9'-carbazolyl)-3,6-dioxauctane, which may be not only new podand of mixed type but also the potential luminophore.

### Experimental

300 MHz <sup>1</sup>H and 75 MHz <sup>13</sup>C NMR spectra were recorded on a VARIAN VXR-300 spectrometer with Me<sub>4</sub>Si as an internal standard. Mass spectrum was recorded on VG 770-70EQ spectrometer in FAB regime. 1,4-Dioxane was dried by the boiling and distillation over KOH. Benzene was dried by the boiling and distillation over Na.

X-ray diffraction data were collected at 298 K an Xcalibur 3 diffractometer (graphite-monochromated MoK<sub>a</sub> radiation, CCD detector "Sapphire3", 2 $\theta$ / $\theta$  scans, 2 $\theta$ <sub>max</sub>=60.6°). The structures were solved dual-space method with SHELXD

program [14] and it was refined by full-matrix least-squares procedure against  $F^2$  with SHELXL program [14].

#### 1,8-Bis(9'-carbazolyl)-3,6-dioxaoctane

The mixture of the solution of 3.344 g (20.00 mmol) carbazole and 1.30 g (4.04 mmol) tetrabutylammonium bromide in 60 ml of 1,4-dioxane and 4.50 g (80.36 mmol) KOH was boiled with stirring during 1 h, then the hot reaction solution was filtered. The precipitate was washed by 20 ml of boiling dioxane. The solution of 2.760 g (10 mmol) 1,8-dibromo-3,6-dioxaoctane in 10 ml dioxane was added to combined filtrate. Then the reaction mixture was boiled at the stirring during 10 h, and then it was cooled. The KBr precipitate was filtered off. The filtrate was concentrated *in vacuo* (50 torr) by removing of 39 ml of dioxane yielding the further reaction solution.

The mixture of the solution of 3.344 g (20.00 mmol) carbazole, 1.30 g (4.04 mmol) tetrabutylammonium bromide in 45 ml 1,4-dioxane and 1.20 g (21.43 mmol) KOH was boiled at stirring during 1 h, then the precipitate was filtered off at 80°C. The filtrate was combined with the obtained previously reaction solution, this reaction mixture was boiled at the stirring during 5 h, then it was cooled. The precipitate was filtered off, and then it was washed by 20 ml of dioxane. The combined filtrates were evaporated *in vacuo*. The obtained residue was washed by 3 portion of water (15 ml), dried *in vacuo* 2 torr. Then it was extracted by 50 ml of boiling  $\text{CCl}_4$ , the obtained extract was evaporated *in vacuo* (20 torr), yielding 3.450 g (76.9%) crude 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**, colourless crystals, mp. 120–121°C. The additional crystallization from boiling benzene yields 1.779 g (39.7%) pure 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**, colourless crystals with mp. 128–129°C. Column chromatography of crystallization residues ( $\text{Al}_2\text{O}_3$ , benzene) additionally yields 0.644 g (14.3%) 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**, colourless crystals, mp. 127–128°C.  $^1\text{H}$  NMR (300 MHz,

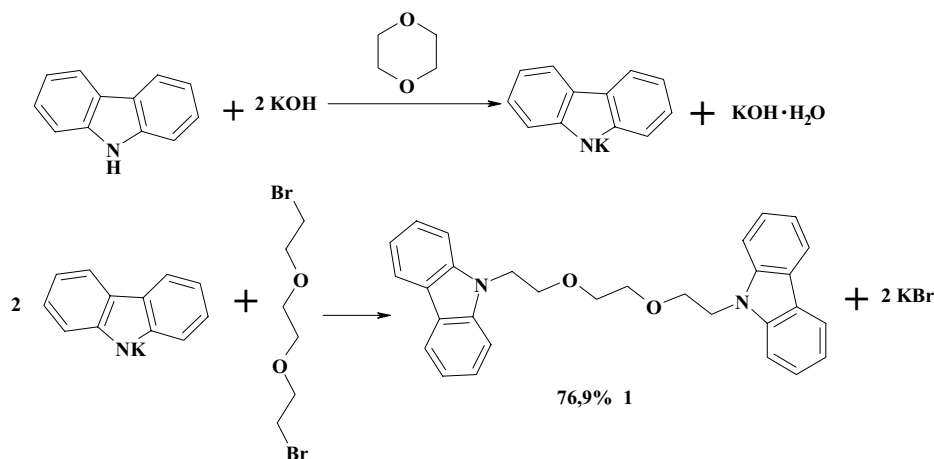
$(\text{CD}_3)_2\text{SO}$ ,  $\delta$ , ppm): 3.27 (4H, s,  $\text{OCH}_2\text{CH}_2\text{O}$ ), 3.57 (4H, t,  $\text{N}_{\text{Het}}\text{CH}_2\text{CH}_2\text{O}$ ,  $^3J=5.1$  Hz), 4.35 (4H, t,  $\text{N}_{\text{Ar}}\text{CH}_2\text{CH}_2\text{O}$ ,  $^3J=5.1$  Hz), 7.18 (4H, t,  $\text{C}_{\text{Het}}(3,3',6,6')$ H,  $^3J=7.35$  Hz), 7.39 (4H, t,  $\text{C}_{\text{Het}}(2,2',7,7')$ H,  $^3J=7.65$  Hz), 7.48 (4H, d,  $\text{C}_{\text{Het}}(1,1',8,8')$ H,  $^3J=8.4$  Hz), 8.13 (4H, d,  $\text{C}_{\text{Het}}(4,4',5,5')$ H,  $^3J=8.4$  Hz).  $^{13}\text{C}$  NMR(75 MHz,  $\text{CDCl}_3$ , APT regime, ppm): a)  $\text{C}_{\text{second}}$ ,  $\text{C}_{\text{quater}}$ : 42.4 ( $\text{NCH}_2$ ), 68.6 ( $\text{N}_{\text{Het}}\text{CH}_2\text{CH}_2\text{O}$ ), 69.8 ( $\text{OCH}_2\text{CH}_2\text{O}$ ), 121.9  $\text{C}_{\text{Het}}(4a,4a',5a,5a')$ , 140.1  $\text{C}_{\text{Het}}(1a,1a',8a,8a')$ ; b)  $\text{C}_{\text{primar}}$ : 109.2  $\text{C}_{\text{Het}}(2,2',7,7')$ , 118.4  $\text{C}_{\text{Het}}(2,2',7,7')$ , 119.7  $\text{C}_{\text{Het}}(4,4',5,5')$ , 125.2  $\text{C}_{\text{Het}}(1,1',8,8')$ . MS (FAB)  $m/z$  ( $I_{\text{rel}}(\%)$ ): 449  $[\text{M}+\text{H}]^+$  (32), 448  $\text{M}^+$ (51), 194 (64), 130 (100).

Crystals of compound **1** were grown from  $\text{CH}_2\text{Cl}_2$ ,  $\text{C}_{30}\text{H}_{28}\text{N}_2\text{O}_2$ ,  $M_r=448.54$ , at 298 K orthorhombic,  $a=17.3238(6)$  Å,  $b=18.0608(5)$  Å,  $c=7.7852(3)$  Å,  $V=2435.84(15)$  Å<sup>3</sup>, space group Pbcn,  $Z=4$ ,  $d_{\text{calc}}=1.223$  g/cm<sup>3</sup>,  $\mu(\text{MoK}\alpha)=0.077$  mm<sup>-1</sup>,  $F(000)=952$ . Refinement against  $F^2$  in an anisotropic approximation (the hydrogen atoms isotropic in the riding model) by a full matrix least-squares method for 2111 reflections was carried out to  $wR_2=0.132$  ( $R_1=0.037$  for 2105 reflections with  $I>2\sigma$  (I),  $S=1.03$ ). Atomic coordinates, bond lengths and bond angles have been deposited at the Cambridge Crystallographic Data Centre (CCDC) the reference number 1055947.

#### Results and discussion

It was found that carbazole interaction with excess of KOH in boiling dioxane yielded the potassium salt of carbazole which dissolved in the boiling dioxane. The excess of KOH facilitate the formation of the potassium salt of carbazole by bonding of formed water as hydrate. 1,8-Dibromo-3,6-dioxaoctane easy reacts with the potassium salt of carbazole at the reaction conditions yielding 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I** (scheme 1).

The further purification of compounds **I** the traces of carbazole carries on by the crystallization from benzene. The application of ethanol is possible for this aim too.



Scheme 1. The synthesis of 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**

The structure of compound **I** was proved by data of  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and mass spectra.

$^{13}\text{C}$  NMR spectra were recorded in APT regime and correspond to structure of compound **I**. In the field of  $\text{CH}_2$  and  $\text{C}_{\text{quater}}$  atoms the one  $\text{NCH}_2$ -group and two  $\text{OCH}_2$ -group signals (42.4, 68.6 and 69.8 ppm), and also two quaternary carbon atoms of carbazolyl moiety (121.9 and 140.1 ppm) are observed. In the field of  $\text{CH}$ -carbon atom the four signal of  $\text{CH}$ -carbazolyl atom are observed (109.2, 118.4, 119.7 and 125.2 ppm)

In the recorded in FAB regime mass spectra (figure), the peaks of the ions  $[\text{M}+\text{H}]^+(\text{m/z } 449)$  and  $\text{M}^+(\text{m/z } 448)$  are observed (scheme 2). The further fragmentation on these ions yields ions with  $\text{m/z } 194$  and  $\text{m/z } 180$ .

Finally the compound **I** structure was proved by XRD data (figure, tables 1,2)

In the crystal the molecule **I** is located in a special position at  $2_1$  screw axis, which passes through the central  $\text{C}-\text{C}$  bond of polyester chain. The angle between the carbazolyl moieties plains is  $76.8^\circ$ . These carbazolyl groups are oriented towards each other

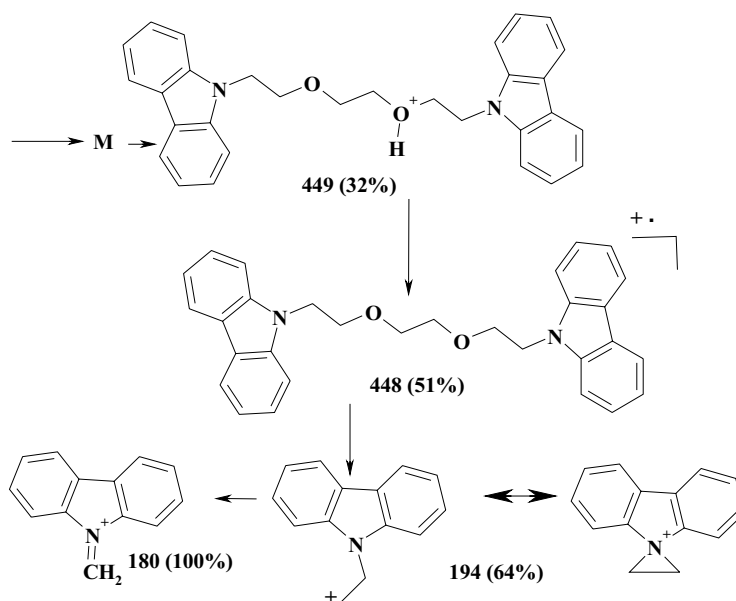
with H11 hydrogen atoms, this orientation results in the formation of the short intramolecular contact H11...H11 2.24 Å (van der Waals radii sum is 2.32 Å [15]). In the crystal molecules **I** are linked into infinite along  $c$  axis due to formation of weak  $\text{C14}-\text{H14}\dots\text{O1}^i$  [ $i: x, 2-y, -1/2+z$ ] hydrogen bonds between the polyester chains.

Table 1

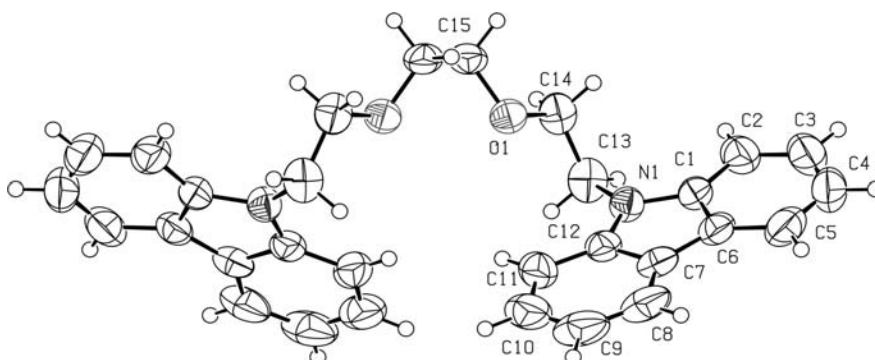
Bond lengths in 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**

Bond	Length, Å	Bond	Length, Å
O1-C14	1.4141(16)	C5-C6	1.399(2)
O1-C15	1.4216(15)	C6-C7	1.437(2)
N1-C1	1.3798(16)	C7-C8	1.391(2)
N1-C12	1.3805(16)	C7-C12	1.4044(19)
N1-C13	1.4528(17)	C8-C9	1.374(3)
C1-C2	1.3897(19)	C9-C10	1.389(3)
C1-C6	1.4030(19)	C10-C11	1.372(2)
C2-C3	1.365(2)	C11-C12	1.3871(19)
C3-C4	1.372(3)	C13-C14	1.494(2)
C4-C5	1.372(3)	C15-C15 <sup>1</sup>	1.488(3)

Note: <sup>1</sup> -  $X, +Y, 1/2-Z$



Scheme 2. MS fragmentation of 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I**



The 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane **I** structure according XRD data

Table 2  
Bond angles in 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane 1  
(in E)

C14–O1–C15	111.72(10)
C1–N1–C12	108.54(11)
C1–N1–C13	125.18(12)
C12–N1–C13	126.04(11)
N1–C1–C2	129.03(13)
N1–C1–C6	109.23(11)
C2–C1–C6	121.74(13)
C3–C2–C1	117.67(17)
C2–C3–C4	121.65(17)
C3–C4–C5	121.58(16)
C4–C5–C6	118.62(17)
C1–C6–C7	106.50(12)
C5–C6–C1	118.69(14)
C5–C6–C7	134.77(15)
C8–C7–C6	134.16(15)
C8–C7–C12	119.05(15)
C12–C7–C6	106.79(12)
C9–C8–C7	119.00(17)
C8–C9–C10	120.92(16)
C11–C10–C9	121.66(17)
C10–C11–C12	117.34(17)
N1–C12–C7	108.94(12)
N1–C12–C11	129.03(14)
C11–C12–C7	122.02(13)
N1–C13–C14	114.26(11)
O1–C14–C13	109.33(11)
O1–C15–C15 <sup>1</sup>	110.41(9)

Note: <sup>1</sup> – X,+Y,1/2– Z

### Conclusion

1,8-Bis(9'-carbazolyl)-3,6-dioxaoctane has been synthesized and XRD-study of its structure has been done.

### Acknowledgements

We are grateful to Department of Education and Science of Ukraine for financial support, grant No 0115U003159.

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Received 12.06.2015

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**Keywords:** podands; phase transfer catalysts; carbazole; 1,8-bis(9'-carbazolyl)-3,6-dioxaoctane; structure.

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