Facile Synthesis of Benzene-bridged Azaoxamacrobicyclic Ligands

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Dedicated to Professor Gottfried Heinisch, Leopold-Franzens-Universität, Innsbruck, Austria, on the occasion of his 60th birthday

Five new barrel-shaped macrobicyclic compounds capable of complexation with anions have been prepared. Intermediate triamines were prepared in one-step using a triple Mitsonobu reaction, thereby shortening the overall synthetic sequence. A previously prepared benzene-bridged hexaazamacrocycle was prepared in only four-steps.

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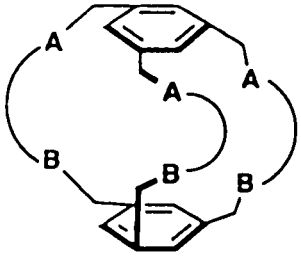
There is great interest in macrocyclic host ligands capable of selective recognition of metal cations, anions and neutral molecules [1]. After Pedersen published his data on complexation of cations by the crown ethers [2,3], many research centers began to prepare macrocyclic ligands for selective affinity for cations [4-6]. Although complexation of anions by macrocyclic compounds was recognized from nearly the same time, the main research effort has been devoted to complexation of cations by the macrocyclic ligating agents.

More recently, there has been great interest in macrocyclic host ligands capable of selective recognition of anions. A number of reviews and books have been published concerning this topic [7-16]. Compared to cations, anions are large and have a variety of geometries, such as spherical, linear, planar, tetrahedral, and octahedral. The size and geometry of the anions dictate the size and shape of the binding sites in ligands. For this reason, a number of rigid three-dimensional cyclophane molecules (see structure below) have been synthesized [17,18], but specific synthetic details for these ligands were not published. The aromatic rings on each end of the barrel-like compounds provide rigidity and, thereby, improve the preorganization of the binding sites for cooperative binding of guest anions. The molecular architecture of these anion binding systems, which require ligating heteroatoms in specific sites, is a particular challenge for synthetic chemists.



A, B = oxygen atoms

or

A,B = nitrogen atoms

A general route for the production of these molecules has been achieved from two readily accessible trifunctionalized parts [19,20]. The benzene-bridged macrocyclic compounds containing either nitrogen or oxygen donor atoms have been prepared in many steps [17]. Occasionally, similar cyclophanes have been prepared by one-step macrocyclizations which required six or more bonds to be formed during the reaction. However, only a few macrocycles containing additional benzene rings were prepared in this way and in low yields [21]. We have reported the synthesis of macrobicyclic polyethers using a five-step process from 1,3,5-benzenetricarboxylate [22] and more recently, the synthesis of hexaazamacrobicyclic ligands [21].

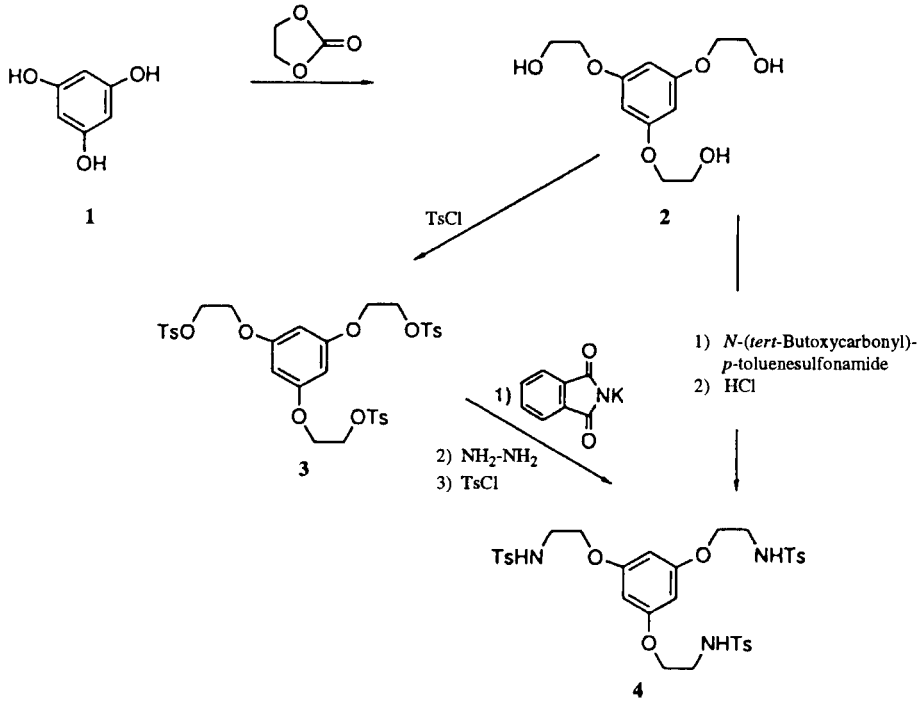
We now report the synthesis of nitrogen- and oxygen-containing benzene-bridged macrobicyclic systems. Our preparation of these mixed oxygen-nitrogen donor bicyclic systems uses a triple Mitsonobu reaction for the preparation of the intermediate triamines [4,8]. This process provides a shortcut in the overall synthesis by allowing a tripod connection strategy involving the formation of only three bonds in the final ring closure step. A modified four-step synthesis of a benzene-bridged hexaazamacrobicyclic compound, which was obtained previously by an eight-step process [17], is also reported.

Results and Discussion.

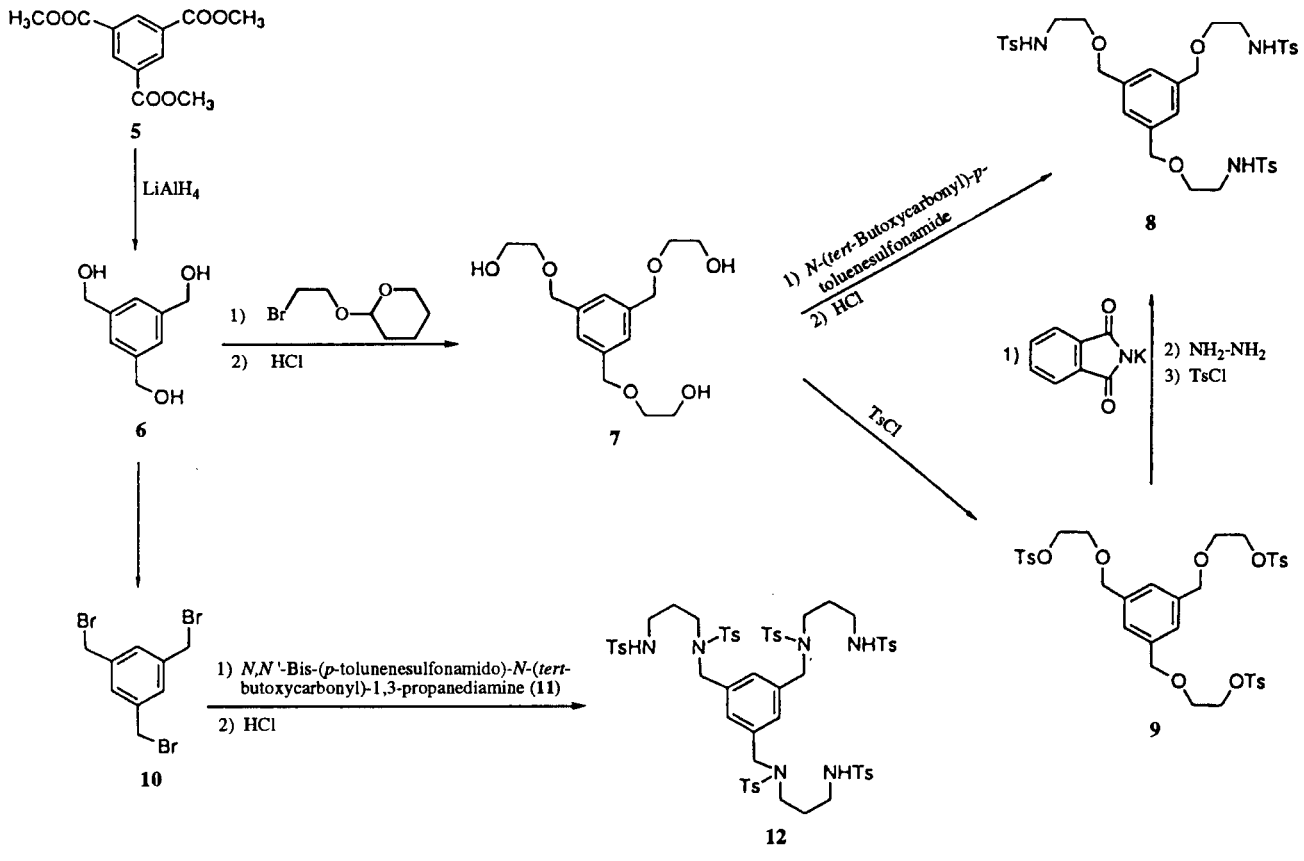
Schemes 1-3 show the preparation of trifunctionalized compounds **3**, **4**, **8**, **9**, and **16** which were used for the final ring closure steps in the preparation of the macrobicyclic ligands. Starting triol **2** (Scheme 1) was obtained after treatment of phloroglucinol **1** with ethylene carbonate in the presence of tetraethylammonium bromide in dimethylformamide [23,24]. Up to now, only a short communication concerning **2** has been reported [24]. Other attempts to prepare **2**, such as treating phloroglucinol with tetrahydropyran protected bromoethanol, were unsuccessful. Compound **4** was prepared from **2** after four-steps *via* a Gabriel reaction to obtain the amino groups from the triol as shown in Scheme 1. We discovered a shorter, two-step transformation using the Mitsonobu reaction and commercially available *N*-(*tert*-butoxycarbonyl)-*p*-toluenesulfonamide. This method was used by us for a similar transformation from a tribromide to a trisaminotosyl compound [21]. The Mitsonobu reaction with *N*-(*tert*-butoxycarbonyl)-*p*-toluenesulfonamide has previously been used to introduce one amine function [25-27]. The triple Mitsonobu reaction was carried out in tetrahydrofuran in the presence of triphenylphosphine and diethyl azodicarboxylate to give a yield of about 60%. Separation of the desired product from the diethyl azodicarboxylate and minor by-products was done on a silica gel column.

Intermediate **8** was prepared from 1,3,5-tris(hydroxymethyl)benzene (**6**) in a similar way using a triple Mitsonobu transformation in the second step (Scheme 2). Starting triol **6** was prepared as reported from trimethyl 1,3,5-benzenetricarboxylate (**5**) [28-30]. Pertosylated hexaaza intermediate **12** was prepared from **6** *via* 1,3,5-tris(bromomethyl)benzene (**10**). Compound **10** was treated with **11** [21] to give **12** in a 61% yield. New synthon **11** allows the synthesis of macrobicycle **25** in only four rather than the eight-steps previously reported for this compound [17]. The *tert*-butoxycarbonyl protecting group was removed in hydrochloric acid or thermally in dimethylformamide at 120° [31].

Scheme 1

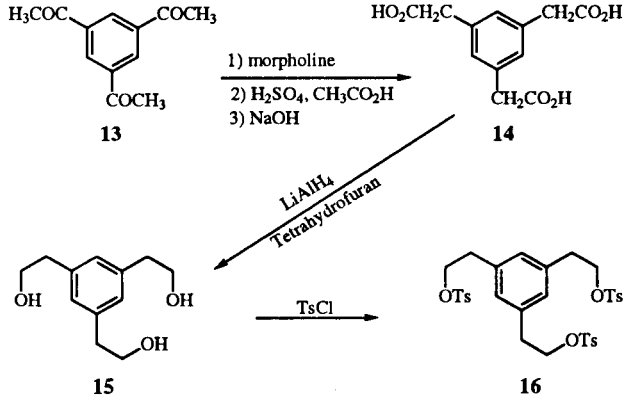


Scheme 2



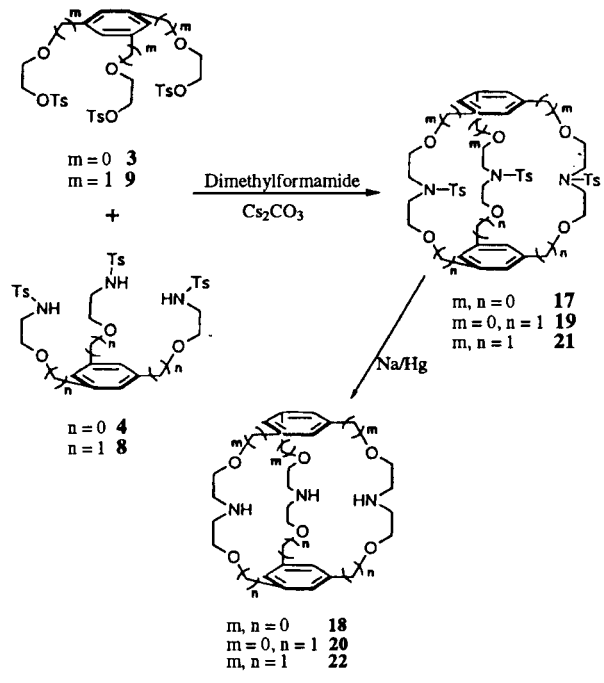
1,3,5-Tris(2-tosyloxymethyl)benzene (**16**) was prepared from 1,3,5-triacetylbenzene [32,33] by means of the Kindler modification of the Willgerodt reaction (Scheme 3). 1,3,5-Benzenetriacetic acid (**14**) was reduced to 1,3,5-tris(2-hydroxyethyl)benzene (**15**) in a 45% yield. The reported reduction of the triester analog of **14**, triethyl benzene-1,3,5-triacetate, gave a higher yield but required an additional step to prepare the triester [32]. Tritosylate **16** was prepared from **15** by tosylation in a tetrahydrofuran-water solution.

Scheme 3



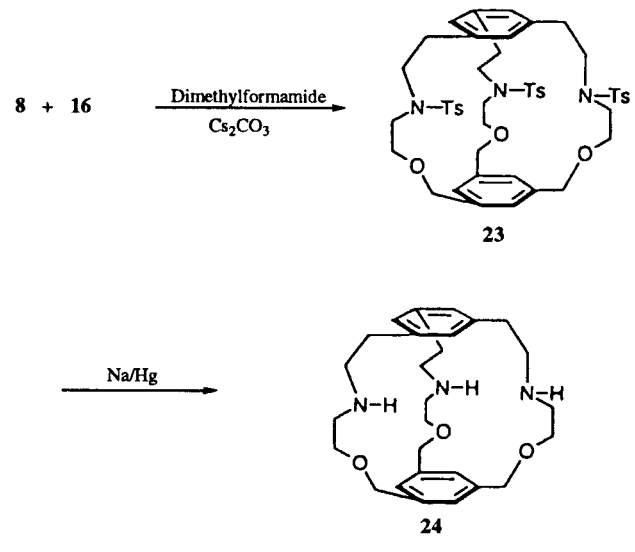
The above five trifunctional intermediates **3**, **4**, **8**, **9**, and **16** were used for the preparation of the benzene-bridged molecules as shown in Schemes 4-6. The

Scheme 4

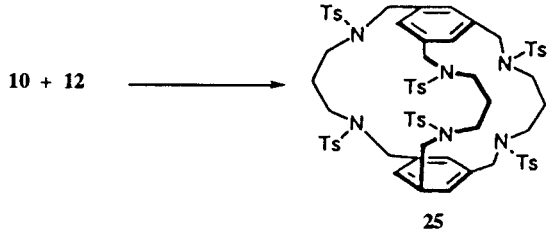


reactions were carried out in dimethylformamide in the presence of cesium carbonate using high dilution techniques (syringe pumps) or without high dilution (see Experimental). Removal of the *p*-toluenesulfonyl protecting groups from **17**, **19**, **21**, and **23** was achieved using commercially available sodium amalgamate. This method was superior to other methods such as hydrobromic acid in acetic acid or lithium aluminum hydride reduction for removal of the *p*-toluenesulfonyl groups from these benzene-bridged macrobicyclic compounds.

Scheme 5



Scheme 6



EXPERIMENTAL

Proton and carbon nmr spectra were obtained at 200 MHz in deuteriochloroform. Molecular weights were determined by electron-impact high resolution ms. Starting compounds were purchased from Aldrich, Fluka, Lancaster, Janssen, TCI and Anachemia Chemical Companies. Other starting materials were prepared as reported: 2-bromoethyl 2'-tetrahydropyranyl ether [34], 1,3,5-tris(hydroxymethyl)benzene (**6**) [28-30], 1,3,5-tris-(bromomethyl)benzene (**10**) [29], 1,3,5-tris(4-hydroxy-2-oxa-1-butyl)benzene (**7**) [22], and 1,3,5-tris[(tosyloxy)-2-oxa-1-butyl]-benzene (**9**) [22]. Elemental analyses were not carried out on new intermediates **2**, **3**, **4**, **8**, **12**, **15**, and **16**. However, satisfactory elemental analyses were obtained for all new macrobicyclic ligands made from these intermediates.

1,3,5-Tris(2-hydroxyethoxy)benzene (**2**) (Scheme 1).

Phloroglucinol (12.6 g, 0.1 mole), 35 g (0.39 mole) of ethylene carbonate and 8 g of tetraethylammonium bromide were added to 15 ml of dimethylformamide and mixed at 150° during 14 hours. After evaporation of the solvent, the residue was chromatographed on silica gel using methylene chloride/acetonitrile: 1/1, 1/2, 1/3 and 1/4 as eluants to give 10 g of **2** (39%), mp 102-104°; ^{1}H nmr (perdeuterated acetone): δ 2.9 (m, 3H), 3.9 (m, 6H), 4.0 (m, 6H), 6.15 (s, 3H); ^{1}H nmr (perdeuterated dimethyl sulfoxide-deuteriochloroform): δ 3.85 (m, 9H), 4.05 (m, 6H), 6.15 (s, 3H).

1,3,5-Tris(3'-tosyloxy-1'-oxapropyl)benzene (**3**) (Scheme 1).

Triol **2** (4.5 g, 0.075 mole) was dissolved in 50 ml of pyridine. Into this solution was added dropwise at 0°, 16 g (0.084 mole) of *p*-toluensulfonyl chloride in 40 ml of pyridine. The resulting mixture was stirred for 5 hours at 0-3°. The solution was poured into a mixture of water, ice and 92 ml of concentrated hydrochloric acid. After stirring for a short time, the solution was extracted three times by methylene chloride. The organic layers were dried using anhydrous magnesium sulfate and the solvent was evaporated. Chloroform and ethyl acetate in a ratio of 50:1 were added the residue and the material was thoroughly mixed. The mixture was filtered and the filtrate dried. The solvent was evaporated to give 8.8 g (70%) of **3**, mp 138°; ^{1}H nmr: δ 2.4 (s, 9H), 4.05 (t, 6H), 4.35 (t, 6H), 5.9 (s, 3H), 7.35 (d, 6H), 7.8 (d, 6H).

1,3,5-Tris[4'-(*p*-toluenesulfonyl)-1'-oxa-4'-azabutyl]benzene (Scheme 1) (**4**) and 1,3,5-Tris[5'-(*p*-toluenesulfonyl)-2'-oxa-5-azapentyl]benzene (**8**) (Scheme 2).

N-(*tert*-Butoxycarbonyl)-*p*-toluenesulfanamide (12.2 g, 0.045 mole) and 23.6 g (0.09 mole) of triphenylphosphine were added to the 100 ml of tetrahydrofuran under nitrogen. To the stirred solution was added 2.58 g (0.01 mole) of **2**, or 3 g (0.01 mole) of **7** and 12.9 g (0.074 mole) of diethyl azodicarboxylate in 20 ml of tetrahydrofuran. After 18 hours, the solvent was evaporated and the residue was chromatographed on a silica column (1500 g) using toluene/ethyl acetate: 10/1 as eluant. The resulting compound (R_f ~0.6 in toluene/ethanol: 5/1) containing some impurity was added to a mixture of 200 ml of isopropyl alcohol saturated with hydrochloric acid and 100 ml of methylene chloride. After stirring 48 hours, the solution was evaporated and the residue was crystallized from methanol to give 5.3 g (69%) of **4**, mp 163-164°; ^{1}H nmr: δ 2.4 (s, 9H), 3.2 (m, 6H), 3.9 (m, 6H), 5.95 (s, 3H), 6.5 (b, 3H) 7.3 (d, 6H), 7.8 (d, 6H) or 5.1 g of **8** (63%); ^{1}H nmr: δ 2.4 (s, 9H), 3.15 (m, 6H), 3.55 (m, 6H), 4.45 (s, 6H), 5.1 (t, 3H), 7.15 (s, 3H), 7.3 (d, 6H), 7.7 (d, 6H).

1,3,5-Tris[2,6-di(*p*-toluenesulfonyl)-2,6-diazahexyl]benzene (**12**) (Scheme 2).

To 2.65 g (0.0075 mole) of **10** in 400 ml of dimethylformamide containing 20 g of potassium carbonate was added 11.1 g (0.023 mole) of **11**. The mixture was stirred at room temperature for 48 hours. The solvent was evaporated and 400 ml of methyl-

ene chloride and 200 ml of water were added. The mixture was extracted 3 times with additional portions of methylene chloride. The combined organic layers were dried using anhydrous magnesium sulfate. The filtrate was evaporated and the residue was chromatographed on 700 g of silica gel using toluene/tetrahydrofuran/methylene chloride: 10/1/1 as eluants. The crude product was added to a mixture of 60 ml of methanol and 30 ml of chloroform containing 20 ml of hydrochloric acid and the resulting mixture was refluxed for 24-48 hours. After cooling, the solvents were evaporated to dryness. The residue was purified on silica gel using toluene/tetrahydrofuran/methylene chloride: 10/1/1 and 5/1/1 as eluants to give 6 g (61%) of **12**; ^{1}H nmr: δ 1.5 (m, 6H), 2.40 (s, 9H), 2.45 (s, 9H), 2.7 (m, 6H), 3.1 (m, 6H), 4.2 (s, 6H), 5.4 (t, 3H), 7.2 (m, 15H), 7.70 (m, 12H).

1,3,5-Tris(2'-hydroxyethyl)benzene (**15**) (Scheme 3).

A mixture of 12.5 g (0.05 mole) of **14** and 100 ml of tetrahydrofuran was added to 600 ml of tetrahydrofuran containing 10 g (0.26 mole) of lithium aluminum hydride under nitrogen. The mixture was refluxed for 24 hours. After cooling, 10 ml of water was slowly added and then 10 ml of 15% sodium hydroxide and 30 ml of water. The mixture was filtered and the residue was washed with hot tetrahydrofuran. The solid was mixed with a new portion of hot tetrahydrofuran and filtered. The tetrahydrofuran solution was dried using anhydrous magnesium sulfate and the solvent was evaporated. The residue was chromatographed on silica gel using acetonitrile to give 4.5 g (42%) of **15**, which crystallized from ethyl acetate. The physical properties of **15** were the same as those reported [29].

1,3,5-Tris[(2'-tosyloxy)ethyl]benzene (**16**) (Scheme 3).

To 50 ml of pyridine containing 4.4 g (0.021 mole) of **15** was added dropwise, 20 g (0.11 mole) of *p*-toluensulfonyl chloride in 40 ml of pyridine at 0-5°. After 5 hours, the mixture was poured into 92 ml of hydrochloric acid and ice. The solution was extracted 3 times with methylene chloride. The combined organic layers were dried using anhydrous magnesium sulfate. The solvent was evaporated and the residue chromatographed on silica gel using chloroform/ethyl acetate: 50/1 and 20/1 as eluants to give 12 g (85%) of **16**, mp 70-72°; ^{1}H nmr: δ 2.4 (s, 9H), 2.8 (t, 6H), 4.15 (t, 6H), 6.7 (s, 3H), 7.3 (d, 6H), 7.7 (d, 6H).

Preparation of Macrobiocyclic Compounds **17**, **19**, **21**, **23** and **25** (Schemes 4-6).

The appropriate intermediate, **3**, **9**, **16** or **10** (0.01 mole) was dissolved in 50 ml of dimethylformamide and placed in a syringe. To a second syringe was added 0.01 mole of **4**, **8** or **12** in 50 ml of dimethylformamide. The two mixtures were simultaneously added to 350 ml of dimethylformamide containing 50 g of cesium carbonate at 70° over a 36-hour period. The resulting solution was stirred at 75-80° for 48 hours. The dimethylformamide was evaporated, water and methylene chloride or chloroform were added and the material was thoroughly mixed and separated. The aqueous layer was extracted three times and the organic layers were dried using anhydrous magnesium sulfate. After removing the solvent, the residue was chromatographed on silica gel using methylene chloride/ethyl acetate: 100/1 or 50/1 or chloroform/ethyl acetate 10/1 (in the case of **21**) as eluants to give the crude *N*-tosylated products as follows. Satisfactory elemental analyses were obtained for all final macrobicyclic ligands (**18**, **20**, **22**, and **24**) prepared from tosylated macrobicycles **17**, **19**, **21** and **23**.

Product **17** was obtained in 50% yield; ^{1}H nmr: δ 2.45 (s, 9H), 3.50 (t, 12H), 4.05 (t, 12H), 5.65 (s, 6H), 7.35 (d, 6H), 7.75 (d, 6H).

Product **19** was obtained in 59% yield; ^{1}H nmr: δ 2.4 (s, 9H), 3.4 (m, 12H), 3.7 (m, 6H), 4.1 (m, 6H), 4.4 (s, 6H), 5.75 (s, 3H), 6.8 (s, 3H), 7.4 (d, 6H), 7.8 (d, 6H).

Product **21** was obtained in 41% yield; ^{1}H nmr: δ 2.4 (s, 9H), 3.3 (t, 12H), 3.65 (t, 12H), 4.2 (s, 12H), 7.0 (s, 6H), 7.3 (d, 6H), 7.7 (d, 6H); ms: (low voltage) m/z 544.

Product **23** was obtained in 22% yield; ^{1}H nmr: δ 2.4 (s, 9H), 2.75 (t, 6H), 3.2 (m, 18H), 4.3 (s, 6H), 6.5 (s, 3H), 7.0 (s, 3H), 7.3 (d, 6H), 7.75 (d, 6H).

Product **25** was obtained in 66% yield, mp (foam) 158-160°; ^{1}H nmr: δ 1.4 (m, 6H), 2.4 (s, 18H), 3.0 (t, 12H), 4.1 (s, 12H), 7.25 (s, 6H), 7.35 (d, 12H), 7.7 (d, 12H).

Anal. Calcd. for $C_{69}H_{78}N_6O_{12}S_6$: C, 60.24; H, 5.71. Found: C, 60.44; H, 5.90.

Preparation of Macrobicyclic Ligands **18**, **20**, **22** and **24** (Schemes 4-6).

Compound **17**, **19**, **21** or **23** (0.005 mole) was added to a mixture of 200 ml of dioxane and 20 ml of methanol containing 20 mg of anthracene and 10 g of sodium dihydrogen phosphate. While stirring, 60 g of crushed 6% sodium amalgam was added and the mixture was refluxed for 72 hours. After the reaction solution was decanted, the mercury was disposed. The solution was evaporated and methylene chloride was added to the residue. After mixing, the material was filtered and evaporated. The residue, dissolved in methanol and added to the top of the column, was chromatographed on a small amount of silica gel using methanol/ammonium hydroxide: 40/1, 20/1 then 10/1 as eluants to give the products as follows.

Product **18** had mp 173°; ^{1}H nmr δ 1.7 (m, 3H), 3.0 (t, 12H), 4.0 (t, 12H), 5.85 (s, 6H); ms: (CI) m/z 460.

Anal. Calcd. for $C_{24}H_{33}N_3O_6$: C, 62.72, H, 7.24. Found: C, 62.40, H, 7.62.

Product **20** was obtained in 90% yield, mp 101-102°; ^{1}H nmr: δ 1.8 (s, 3H), 2.85 (t, 6H), 2.95 (t, 6H), 3.65 (t, 6H), 4.0 (t, 6H), 4.4 (s, 6H), 5.95 (s, 3H), 7.05 (s, 3H); ^{13}C nmr: δ 48.3, 48.4, 67.1, 70.0, 72.4, 94.9, 124.6, 138.6, 160.5.

Anal. Calcd. for $C_{27}H_{39}N_3O_6$: C, 64.64; H, 7.84. Found: C, 64.70; H, 7.93.

Product **22** was obtained in 76% yield, mp 78.5-80°; ^{1}H nmr: δ 2.9 (t, 12H), 3.55 (t, 12H), 4.55 (s, 12H), 4.75 (s, 3H), 7.25 (s, 6H).

Anal. Calcd. for $C_{30}H_{45}N_3O_6$: C, 66.27; H, 8.34. Found: C, 66.39; H, 8.43.

Product **24** was obtained in 44% yield, mp 114-115°; ^{1}H nmr: δ 1.5 (b, 3H), 2.55 (s, 12H), 2.7 (t, 6H), 3.55 (t, 6H), 4.35 (s, 6H), 6.7 (s, 3H), 7.1 (s, 3H); ^{13}C nmr: δ 35.4, 48.5, 49.3, 68.7, 72.9, 127.0, 127.8, 138.6, 139.7; ms: (CI) m/z 455.

Anal. Calcd. for $C_{27}H_{39}N_3O_3$: C, 71.49; H, 8.66. Found: C, 71.26; H, 8.53.

REFERENCES AND NOTES

[1] R. M. Izatt, K. Pawlak, J. S. Bradshaw and R. L. Bruening, *Chem. Rev.*, **95**, 2529 (1995).

[2] C. J. Pedersen, *J. Am. Chem. Soc.*, **89**, 7017 (1967).

[3] C. J. Pedersen, *Aldrichimica Acta*, 1 (1971).

[4] B. Dietrich, J-M. Lehn and J. P. Sauvage, *Tetrahedron Letters*, **10**, 2885 (1969).

[5] J. S. Bradshaw and R. M. Izatt, *Acc. Chem. Res.*, **30**, 338 (1997).

[6] Y. Inoue and G. W. Gokel, Cation Binding by Macrocycles, Marcel Dekker, NY, 1991.

[7] A. Bianchi, K. Bowman-Jones and E. Garcia-Espana, Supramolecular Chemistry of Anions, Wiley-VCH, New York, 1997.

[8] F. Schmidtchen and M. Berger, *Chem. Rev.*, **97**, 1609 (1997).

[9] J. Scheerder, J. F. J. Engbersen and D. N. Reinhoudt, *Rec. Trav. Chim. Pays-Bas.*, **115**, 307 (1996).

[10] J. L. Atwood, K. T. Holman and J. W. Steed, *J. Chem. Soc., Chem. Commun.*, 1601 (1996).

[11] J-M. Lehn, Supramolecular Chemistry, VCH, Weinbeim, Chapter 3, 1995.

[12] C. Seel, A. Galan and J. de Mendoza, *Top. Curr. Chem.*, **175**, 101 (1995).

[13] B. Dietrich, *Pure Appl. Chem.*, **65**, 1457 (1993).

[14] C. Seel and J. de Mendoza, in Comprehensive Supramolecular Chemistry, Vol **2**, Chapter 17, From Chloride Katapinates to Trinucleotide Complexes: Development in Molecular Recognition of Anionic Species, F. Vögtle, ed, Elsevier Science, Inc, Oxford, UK, 1996, p 519.

[15] J. Hodacova, *Chem. Listy*, **88**, 99 (1996).

[16] J. L. Sessler, M. Cyr, H. Furuta, V. Kral, T. Mooy, T. Morrishima and S. Weghorn, *Pure Appl. Chem.*, **65**, 393 (1993).

[17] D. Heyer and J-M. Lehn, *Tetrahedron Letters*, **27**, 5869 (1986).

[18] R. A. Pascal, J. Spergel and D. van Engen, *Tetrahedron Letters*, **27**, 4099 (1986).

[19] V. Boekelheide and R. A. Hollins, *J. Am. Chem. Soc.*, **92**, 3512 (1990).

[20] F. Vögtle, *Liebigs Ann. Chem.*, **735**, 193 (1970).

[21] K. E. Krakowiak and J. S. Bradshaw, *J. Heterocyclic Chem.*, **33**, 1 (1996).

[22] H-Y. An, J. S. Bradshaw, K. E. Krakowiak, B. J. Tarbet, N. K. Dalley, X. L. Kou, C. Y. Zhu and R. M. Izatt, *J. Org. Chem.*, **58**, 7694 (1993).

[23] M. G. Dolson and J. S. Swenton, *J. Am. Chem. Soc.*, **103**, 2361 (1981).

[24] R. Lapouyade and J.-P. Morand, *J. Chem. Soc., Chem. Commun.*, 223 (1987).

[25] H. Yamada, M. Socheoka and M. Shibasaki, *J. Org. Chem.*, **56**, 4569 (1991).

[26] M. L. Edwards, D. M. Stemerick and J. R. McCarthy, *Tetrahedron*, **50**, 5579 (1994).

[27] J. R. Henry, L. R. Marcin, M. C. McIntosh, P. M. Scola, G. D. Harris, Jr. and S. M. Weinrieb, *Tetrahedron Letters*, **30**, 5709 (1989).

[28] J. Houk and G. M. Whitesides, *J.* Am. *Chem. Soc.*, **109**, 6825 (1987).

[29] M. Safar, Z. Kafka, V. Galik and S. Lauda, *Collect. Czech. Chem. Commun*, **39**, 3390 (1974).

[30] M. Nakazaki, K. Yamamoto and T. Toya, *J. Org. Chem.*, **46**, 1611 (1981).

[31] K. E. Krakowiak and J. S. Bradshaw, *Synth. Commun.*, **26**, 3999 (1996).

[32] W. P. Cochrane, P. L. Pauson and T. S. Stevens, *J. Chem. Soc.(C)*, 630 (1968).

[33] M. S. Newman and H. S. Lowrie, *J. Am. Chem. Soc.*, **76**, 6196 (1954).

[34] A. Anantanarayan, P. J. Dutton, T. M. Fyles and M. J. Pitre, *J. Org. Chem.*, **51**, 752 (1986).