# Original Article: Efficient Synthesis of Bis(3-Methylphenyl)Sulfoxide Aza Macrocycles from a Bis(3-Methylphenyl)Sulfoxide Diester and Diamine Derivatives

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# ABSTRACT

Some bis(3-methylphenyl)sulfoxide *aza* macrocycles (3–8) were synthesized in high yields based on the conventional methods of the reaction of corresponding bis(3-methylphenyl)sulfoxide diester and diamines in refluxing methanol. Bis(3-methylphenyl)sulfoxide diester was synthesized from the reaction of 2-[(2-hydroxy-5-methylphenyl)sulfinyl]-4-methylphenol and methyl chloroacetate. The structures of these compounds were confirmed using IR, $^1$H NMR, $^{13}$C NMR and Mass spectroscopy.

## Introduction

Macrocycles are probably the most widel y used family of host compounds in supramolecular chemistry. With the discovery of macrocyclic polyethylenes, Pedersen formed the basis of many studies on their ability to function as receptors for cationic, neutral and anionic species [1-3].

A rational design of macromolecular receptors is governed by a number of factors: nature, number, relative structure, spatial location of different connecting units, etc. A combination of these factors may induce an inherent equilibrium of non-covalent binding

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forces optimum for specificity in host-guest recognition [2-10]. The amide group, which is naturally used in a variety of antibiotic ionophores [7-11], has found a special place in the design of receptors because *(i)* it shows the character of a double bond (O or N and NH), *(ii)* it has a higher negative charge on oxygen than the ether and ester groups and *(iii)* has a geometric strength [12,13]. Amide-based macrocycles for the selective detection of metal cations [1,14], and organic molecules [1,15], typically adopt pre-organization of their binding sites through hydrogen bonding or configurational strength around the carbon-nitrogen amide bonds [13-18]

Macrocyclic *aza* crown compounds have gained a great attention due to their wide applications in chemistry, analysis, microanalysis, metal separation, sensoring, biology, biophysics and ecology [1,16]. Due to their high ability to selectively and effectively interact with various types of transition and heavy metals, there is a greater tendency to produce new crown ethers, including benzo-substituted macrocyclic diamides. It has been well established that the introduction of polar amide donors into a complexone plays an important role in the enhancement of the selectivity in cation binding [19-21].

Through N–C=O–metal interactions, the amide group in many hosts directs the specificity of the host toward softer alkaline earth cations, whereas ether macrocyclic ionophores show high selectivity in complex formation with alkali metal ions [22-24].

The amide group often plays a key role in the complexation of various guests in aza macrocycles. In previous studies, synthesis of dibenzosulfides and dibenzosulfoxides *aza* macrocycles as metal cations receptors has been reported [23,24]. As part of our ongoing program to develop efficient and robust methods for the preparation of heteroatom-containing organic compounds [25-28], we wish to report the synthesis of bis(3-methylphenyl)sulfoxide aza macrocycles derivatives (3-8) from a bis(3-methylphenyl)sulfoxide diester and an aliphathic diamine in methanol reflux with high yields (Figure 1).
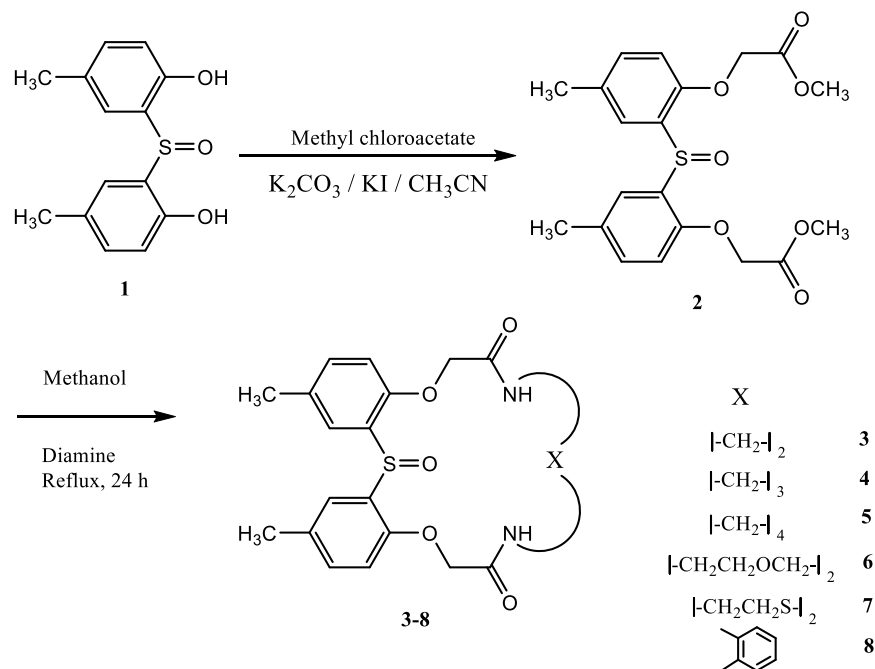


**Figure 1.** Synthesis of aza macrocycles (See Experimental Methods section for more details)

## Experimental Methods

Raw materials and solvents from Merck (Germany) and Fluka (Switzerland) were used without further refining. Then, Thin-layer chromatography (TLC) and Nuclear Magnetic Resonance (NMR) were used as characterization methods, which show no by-products. M.P. were measured on an Electrothermal 9100 device and has not been modified. Infrared (IR) spectra were measured on a Jasco 6300 FTIR spectrometer. $^1$H- and $^{13}$C-NMR spectra were measured (CDCl$_3$) with a *BRUKER DRX-500 AVANCE* spectrometer at 500 and 125 MHz, respectively. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. Flash chromatography columns were prepared with Merck silica gel powder.

### *Synthesis of methyl 2-(2-{[2-(2-methoxy-2-oxoethoxy)-5-methylphenyl]sulfinyl}-4-methylphenoxy)acetate (2)*

First, 3.35 mL (38 mmol) methylchloroacetate was added to a mixture of 4.98 g (19 mmol) of Compound 1 (Figure 1), 5.24 g (38 mmol) of potassium carbonate and catalytic potassium iodide in 100 mL of acetonitrile at room temperature. Then, the reaction product was refluxed for 24 h. After completion of the TLC, the mixture was cooled to room temperature, water was added to it and the mixture extracted with chloroform (3 × 50 mL), washed with 10% sodium hydroxide solution, dried, and recrystallized from ethanol to obtain pure diester 2 with 95% yield. mp. 127–128.3 ºC; IR (KBr): 2971, 2950, 1780, 1488, 1291, 1263, 1167, 1070, 980,423 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$): 2.23 (s, 6H, 2CH$_3$); 3.36 (s, 6H, 2OCH$_3$); 4.40 (s, 4H, 2(-CH$_2$O)); 6.81 (d, 2H, $^3J_{hh}$ = 8 Hz, H-Ar); 6.89 (s, 2H, H-Ar). 7.08 (d, 2H, $^3J_{hh}$ = 8 Hz, H-Ar).

### *General procedure for the preparation of bis(3-methylphenyl)sulfoxide aza macrocycle derivatives 3-8*

First, the appropriate diamine (2 mmol) at room temperature was added to 2 mmol of diester (2) in methanol (100 mL). Then, the resulting mixture was stirred for 20 minutes and refluxed for 24 h. After the completion of the TLC reaction, the mixture was cooled down to room temperature. Next, water was added and the resulting mixture extracted with chloroform (3 × 50 mL). The combined chloroform layers were dried and evaporated to obtain a precipitate. To purify the precipitate, column chromatography on silica gel along with appropriate eluent solvent has been used. The solvent was removed under reduced pressure and the products (3-8) were obtained. The characterization data of the derivatives are as follows:

### *2,17-Dimethyl-8,9,10,11-tetrahydro-19H-19λ⁴-dibenzo[b,e][1,7,4,10,13] dioxathiadiazacyclopentadecine-7,12,19(6H,13H)-trione (3)*

White crystals; mp: 297.1-298.4 ºC; Yield: 362 mg (90%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$):3397, 3328, 3101, 3030, 2977, 2964, 2880, 1688, 1671, 1598, 1540, 1487, 1452, 1292, 1280, 1248, 1212, 1154, 1074, 1044, 1036, 990, 825, 815, 802, 568 cm$^{-1}$. $^1$H NMR (500 MHz, CDCl$_3$): 2.23 (s, 6H, 2CH$_3$), 3.28 (s, 4H, 2CH$_2$ aliphatic), 4.56 (s, 4H, 2(-CH$_2$-O)), 6.65 (broad, 2H, 2NH), 6.83 (d, 2H, $^3J_{HH}$ = 8.02 Hz, H-Ar), 6.94 (s, 2H, H-Ar), 7.07 (d, 2H, $^3J_{HH}$ = 8.02 Hz, H-Ar). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.62 (2CH$_3$), 38.58 (2CH$_2$ aliphatic), 68.06 (2CH$_2$-O), 121.58, 129.63, 132.46 (6CH arom), 112.57, 132.69, 152.67 (6C arom), 168 (2C=O of amide group). Anal. calc. for C$_{20}$H$_{22}$N$_2$O$_5$S (402.46): C 59.69; H 5.51; N 6.96; Found: C 59.64; H 5.54; N 6.90%.

### *2,18-Dimethyl-9,10,11,12-tetrahydro-6H,20H-20λ⁴-dibenzo[b,e][1,7,4,10,14] dioxathiadiazacyclohexadecine-7,13,20(8H,14H)-trione (4)*

White crystals; mp: 225.6-226.8 ºC; Yield: 200 mg (48%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$): 3406, 3305, 3074, 3029, 2952, 2927, 2870, 1674, 1666, 1550, 1530, 1487, 1450, 1296, 1247, 1214, 1154, 1077, 1043, 825, 817, 798, 554 cm$^{-1}$. $^1$H

NMR (500 MHz, CDCl$_3$): 1.87-1.92 (m, 2H, CH$_2$), 2.23 (s, 6H, 2CH$_3$); 3.14 (t, 4H, 2CH$_2$ aliphatic), 4.55 (s, 4H, 2(-CH$_2$-O)), 6.60 (broad, 2H, 2NH), 6.82-7.09 (m, 6H, H-Ar). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.59 (2CH$_3$), 25.47, 36.70 (3CH$_2$ aliphatic), 67.65 (2CH$_2$-O), 121.67, 129.79, 132.66 (6CH arom), 112.33, 132.74, 152.80 (6C arom), 168.29 (2C=O of amide group). Anal. calc. for C$_{21}$H$_{24}$N$_2$O$_5$S (416.49): C, 60.56; H, 5.81; N, 6.73; Found: C, 60.60; H, 5.76; N, 6.76%.

### 2,19-Dimethyl-8,9,10,11,12,13-hexahydro-21H-21λ$^4$-dibenzo[b,e][1,7,4,10,15] dioxathiadiazacycloheptadecine-7,14,21(6H,15H)-trione (5)

White crystals; mp: 226-227 ℃; Yield: 232 mg (54%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$): 3397, 3349, 3027, 2930, 2855, 1679, 1660, 1547, 1528, 1493, 1435, 1291, 1256, 1157, 1081, 1064, 1043, 817, 583 cm$^{-1}$. $^1$H NMR (500 MHz, CDCl$_3$): 1.39 (s, 4H, 2CH$_2$), 2.22 (s, 6H, 2CH$_3$), 3.26 (t, 4H, 2CH$_2$ aliphatic), 4.55 (s, 4H, 2(-CH$_2$-O)), 6.22 (broad, 2H, 2NH), 6.80 (d, 2H, $^3J_{HH}$ = 8.2 Hz, H-Ar), 6.96 (s, 2H, H-Ar), 7.07 (d, 2H, $^3J_{HH}$ = 8.2 Hz, H-Ar). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.65 (2CH$_3$), 25.54, 37.78 (4CH$_2$ aliphatic), 67.65 (2CH$_2$-O), 121.20, 129.50, 132.28 (6CH arom), 112.10, 132.32, 153.13 (6C arom), 168.08 (2C=O of amide group). MS (EI, 70 eV): $m/z$ (%) = 431 (M$^+$, 2.79), 430 (37.06), 429 (76.92), 351 (37.06), 257 (29.37), 243 (30.76), 167 (100), 151 (32.86), 137 (41.95), 91 (11.88), 70 (32.86), 44 (4.19). Anal. calc. for C$_{22}$H$_{26}$N$_2$O$_5$S (430.52): C, 61.38; H, 6.09; N, 6.51; Found: C, 61.41; H, 6.13; N, 6.45%.

### 2,23-Dimethyl-9,10,12,13,16,17-hexahydro-6H,15H,25H-25λ$^4$-dibenzo[k,n][1,4,10,16,13,7,19] tetraoxathiadiazacyclohenicosine-7,18,25(8H,19H)-trione (6)

White crystals; mp: 158-159.2 ℃; Yield: 392 mg (80%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$): 3440, 3040, 2920, 2880, 1680, 1520, 1480, 1440, 1350, 1300, 1280, 1240, 1150, 1115, 1080, 1040, 895, 800, 545, 440 cm$^{-1}$. $^1$H NMR (500 MHz, CDCl$_3$): 2.23 (s, 6H, 2CH$_3$), 3.55 (t, 4H, 2CH$_2$), 3.62 (t, 4H, 2CH$_2$ aliphatic), 3.63 (s, 4H, 2CH$_2$), 4.56 (s, 4H, 2(-CH$_2$-O)), 6.83 (d, 2H, $^3J_{HH}$ = 7.92 Hz, H-Ar), 6.88 (s, 2H, H-Ar), 7.08 (d, 2H, $^3J_{HH}$ = 7.92 Hz, H-Ar), 7.43 (broad, 2H, 2NH). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.96 (2CH$_3$), 39.28, 68.64, 70.18, 70.75 (4CH$_2$ aliphatic), 123.10, 129.80, 132.78 (6CH arom), 113.35, 132.88, 153.94 (6C arom), 168.37 (2C=O of amide group). MS (EI, 70 eV): $m/z$ (%) = 491 (M$^+$, 16.54), 490 (43.16), 489 (89.92), 351 (27.33), 273 (39.56), 190 (38.12), 167 (100), 137 (69.78), 91 (32.37), 85 (33.81), 44 (39.56). Anal. calc. for C$_{24}$H$_{30}$N$_2$O$_7$S (490.57): C, 58.76; H, 6.16; N, 5.71; Found: C, 58.80; H, 6.13; N, 5.75 %.

### 2,21-Dimethyl-9,10,14,15-tetrahydro-6H,13H,23H-23λ$^4$-dibenzo[o,r][1,14,7,8,17,4,11] dioxatrithiadiazacyclononadecine-7,16,23(8H,17H)-trione (7)

White crystals; mp: 224.4-225.2 ℃; Yield: 168 mg (38%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$): 3410, 3321, 3063, 3044, 2939, 2886, 1692, 1669, 1537, 1501, 1440, 1301, 1260, 1085, 1055, 832, 813, 568 cm$^{-1}$. $^1$H NMR (500 MHz, CDCl$_3$): 2.23 (s, 6H, 2CH$_3$), 2.87 (t, 4H, $^3J_{hh}$ = 6.25 Hz, 2CH$_2$ aliphatic), 3.57 (t, 4H, $^3J_{hh}$ = 6.25 Hz, 2CH$_2$ aliphatic), 4.54 (s, 4H, 2(-CH$_2$-O)), 6.83 (d, 2H, $^3J_{hh}$ = 8.2 Hz, H-Ar), 6.90 (s, 2H, H-Ar), 7.09 (d, 2H, $^3J_{hh}$ = 8.2 Hz, H-Ar), 7.19 (broad, 2H, 2NH). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.635 (2CH$_3$), 38.42, 39.23 (4CH$_2$ aliphatic), 68.67 (2CH$_2$-O); 122.06, 129.67, 132.30 (6CH arom), 113.30, 132.88, 153.63 (6C arom), 168.29 (2C=O of amide group). Anal. calc. for C$_{22}$H$_{26}$N$_2$O$_5$S$_3$ (494.65): C, 53.42; H, 5.30; N, 5.66; Found: C, 52.47; H, 5.21; N 5.62 %.

### 2,19-Dimethyl-8,13-dihydro-21H-21λ$^4$-tribenzo[b,e,k][1,7,4,10,13] dioxathiadiazacyclopentadecine-7,14,21(6H,15H)-trione (8)

White crystals; mp: 275-276 ℃; Yield: 288 mg (64%); IR (KBr) ($\upsilon_{max}$, cm$^{-1}$): 3411, 3286, 3052, 3032, 2950, 2930, 1715, 1668, 1608, 1545, 1514, 1491, 1457, 1304, 1260, 1248, 1212, 1163, 1078, 1051, 817, 760 cm$^{-1}$. $^1$H NMR (500 MHz, CDCl$_3$): 2.25 (s, 6H, 2CH$_3$), 4.70 (s, 4H, 2(-CH$_2$-O)), 6.87-8.53 (m, 12H, H-Ar and H-amide). $^{13}$C NMR (125 MHz, CDCl$_3$): 20.62 (2CH$_3$), 69.44

(2CH$_2$-O), 122.73, 124.02, 125.94, 127.72, 133.08 (10CH arom), 113.96, 130.08, 133.28, 153.40 (8C arom), 166.78 (2C=O of amide group). Anal. calc. for C$_{24}$H$_{22}$N$_2$O$_5$S (450.51): C, 63.98; H, 4.92; N, 6.22; Found: C, 63.93; H, 4.95; N, 6.26 %.

## Results and Discussion

Bis(3-methylphenyl)sulfoxide *aza* macrocycles (3–8) were synthesized based on standard procedures shown in Figure 1. Compound 1 was synthesized based on procedures reported in [22-26]. The corresponding diester 2 was prepared by the reaction of 2-[(2-hydroxy-5-methylphenyl)sulfinyl]-4-methylphenol and methyl chloroacetate in the presence of potassium carbonate and potassium iodide in acetonitrile[31]. Bis(3-methylphenyl)sulfoxide *aza* macrocycles were prepared from the reaction of dister 2 and the appropriate diamine in dry methanol refluxed for more than 24 hours. Table one shows the products 3-8 and their corresponding yields. The reaction proceeded under mild conditions and no adverse reactions were observed. A mechanistic rationalization for this reaction is provided in Figure 2.

The structures of the products were deduced from their $^1$H NMR, $^{13}$C NMR, Mass and IR spectra. For example, the $^1$H NMR spectrum of derivative 3 consisted of one singlet for the 2 CH$_3$ ($\delta$=2.23 ppm), a singlet at $\delta$ = 3.28 ppm for 2 CH$_2$, a singlet for the 2(-CH$_2$-O) ($\delta$= 4.56 ppm), a board singlet for the 2NH ($\delta$= 6.65 ppm), a singlet at $\delta$ = 6.94 ppm for 2CH (aromatic) and two doublet for the aromatic protons ($\delta$= 6.83 and $\delta$= 7.07, $^3J_{HH}$ = 8.0 Hz). The aryl groups exhibited characteristic signals in the aromatic region of the spectrum. The $^1$H-decoupled $^{13}$C NMR spectrum of derivative 3 showed 10 distinct resonances, partial assignment of these resonances are given in the experimental section. The $^1$H and $^{13}$C NMR spectra of derivatives 4–8 were similar to those of derivative 3 except for the aromatic and aliphatic moieties, which exhibited characteristic signals with appropriate chemical shifts.

**Table 1.** Products resulting from synthesis of aza macrocycle and corespounding yields of the isolated products

| Derivative no. | X | % Yield [a] |
|:---:|:---:|:---:|
| 3 | (-CH$_2$-)$_2$ | 90 |
| 4 | (-CH$_2$-)$_3$ | 48 |
| 5 | (-CH$_2$-)$_4$ | 54 |
| 6 | (-CH$_2$CH$_2$OCH$_2$-)$_2$ | 80 |
| 7 | (-CH$_2$CH$_2$S-)$_2$ | 38 |
| 8 |  | 64 |
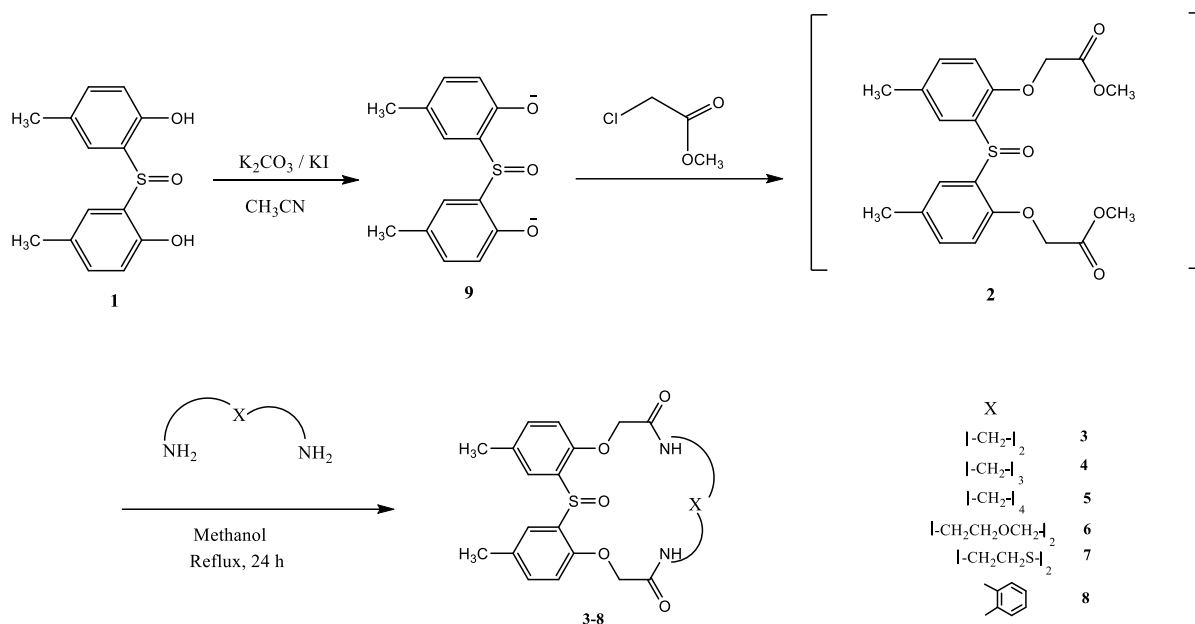
**Figure 2.** Proposed mechanism for the formation of aza macrocycle derivatives (3-8)

## Conclusion

We believe that the reported method provides a simple and efficient route to obtain products 3-8 of bis(3-methylphenyl)sulfoxide aza macrocycle from bis(3-methylphenyl)sulfoxide diester 2 and diamines in methanol reflux. Ease of operation, high efficiency, and relatively gentle reaction conditions make it a useful additive to modern synthetic methods.

## Acknowledgments

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