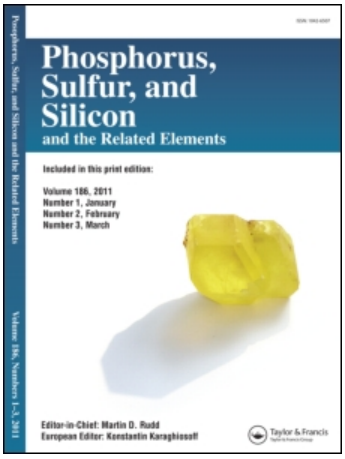

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SYNTHESIS OF NEW MULTIBENZO OXYGEN–SULFUR DONOR MACROCYCLES CONTAINING LACTAMS AT ROOM TEMPERATURE

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Some new oxygen–sulfur, multibenzo macrocyclic ligands containing amide groups have been prepared using the macrocyclization process with the reaction of 2,2′-thiobis-[4-methyl(2-aminophenoxy)phenyl ether] as a symmetrical diamine with appropriate dicarboxylicacid dichlorides in moderate yields. This macrocyclization led to the formation of di- and tetramide macrocycles. These reactions were routinely carried out at ambient temperature in CH_2Cl_2 as solvent in high dilution without template effect conditions. It is found that sulfur the atom affects the rigidity of the macrocycles and diastereotopicity of nuclei in the ring of these series of macrocyclic compounds.

Keywords Diamide; diamine; dicarboxylic acid dichloride; lactam; macrocycle

INTRODUCTION

The increasing interest in the molecular recognition properties of naturally occurring macrocycles has attracted much attention in the design and synthesis of new cyclic polyaza- and polyoxa macrocycles.[1] Among others, macrocyclic amides are used as host molecules. They act as hydrogen-bond donors and hydrogen-bond acceptors, and can form complexes with neutral molecules of biological interest.[2] Macrocyclic diamides and corresponding aza crown compounds have gained a great deal of attention due to their wide applications in chemistry, analysis and microanalysis, metal separation and transport, molecular recognition, medical and industrial uses, biophysics, catalysis, enzyme mimics, sensoring and switching agriculture, and ecology.[3] There have been recent developments in the use of particular macrocyclic amides for metal ion discrimination. For example, diamide-containing macrocycles have been reported to exhibit selectivity for lithium which is superior to that reported for 14-crown-4. In the other study, a 14-membered ligand ring containing two amides and two sulfur donor groups was shown to be selective for Pd(II) and Pt(II) over

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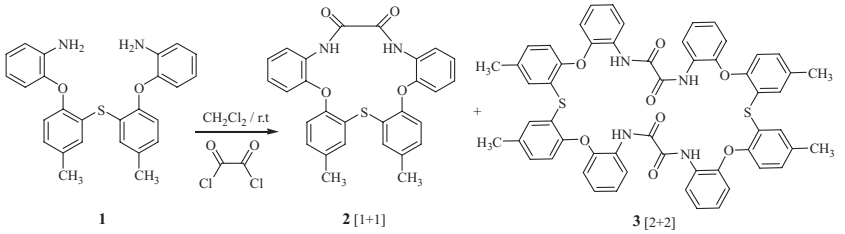
Co(II), Ni(II), and Cu(II).[4] In particular, the use of thia-containing macrocycles as building blocks in such systems yields the potential of binding soft metals in either the ligand's endo- or exocyclic modes; in particular exo-coordination has now been well documented for such sulfur-containing ligand systems.[5] Macrocyclic diamides are also valuable intermediates for the preparation of azacrown compounds and more complicated ligands such as cryptands and cryptohemispherands, which can be functionalized by additional ligating centers including chromogenic and proton ionizable groups.[6]

There are a variety of synthesis methods for macrocyclic diamides such as high dilution techniques,[7] the route based on the template effect,[8] and high pressure approach.[9] In addition, they are prepared by various procedures including reaction of α, γ-diester and different diester derivatives,[10] dicarboxylic acid diesters, diacid dichloride,[11,12] bis(α-chloroamide) compounds,[13] and activated carboxylic acids[14] with various diamines.

In our previous work, we have described the synthesis of new dibenzosulfide and dibenzosulfoxide amidic macrocycle compounds using dicarboxylic acid diester and diacid dichloride methods.[15,16] In this article, we report the synthesis of series of new multibenzooxathia macrocyclic amides. For this purpose, 2,2′-thiobis-[4-methyl(2-aminophenoxy)phenyl ether] (**1**)[17] was reacted with dicarboxylic acid dichlorides in dry CH_2Cl_2 solvent at ambient conditions, which afforded new multibenzo oxygen-sulfur donor macrocycles containing lactams. It should be noted that the diamine **1** was synthesized by our research team previously and was introduced as a novel complexing agent for Cd^{2+} and Co^{2+} with no significant interference from other ions such as Zn^{2+} being observed.[17] This selectivity evidence in the complexation behavior of diamine **1** encouraged us to synthesize the corresponding amidic macrocyclic derivatives. The complexation of these macrocycles with different metal ions is under study in our laboratory.

RESULTS AND DISCUSSION

The reactions of dicarbonyl compounds with diamines are much more complicated and can produce a wide spectrum of products. As the model reaction, we initially examined the reaction of bisamine **1** with oxalyl chloride in dry CH_2Cl_2 as solvent in high dilution conditions in the presence of triethyl amine at room temperature. Thin layer-chromatography (TLC) indicated that a period of at least 20 h was needed for the reaction to be completed. TLC also showed that the mixture consisted of two products. These products were separated by column chromatography on silica gel using hexane/EtOAc (4:1) as eluent to afford tetrabenzodioxathiadiamide **2** and octabenzotetraoxadithiatetramide **3** with 40% and 38% yield, respectively (Scheme 1).

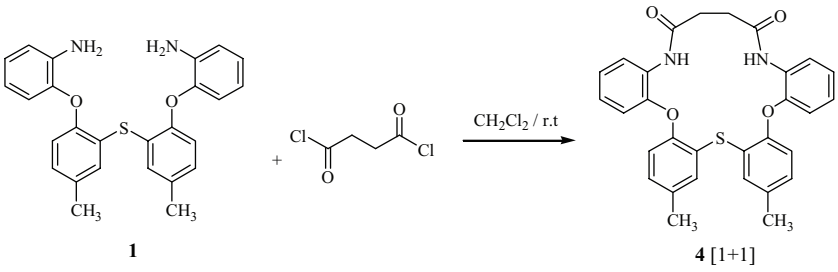


1 **2** [1+1] **3** [2+2]

Scheme 1 Synthesis of [1+1] and [2+2]-condensation products **2** and **3**.

Identification of compounds **2** and **3** was made by elemental analysis, mass spectra, and NMR spectroscopy. The ^{1}H NMR spectra of compounds **2** and **3** showed a sharp singlet for the methyl groups at δ 2.1 and 2.3 ppm, and amide protons appeared as singlet signal at about δ 9.0 and 9.6 ppm, respectively. The other protons showed in the aromatic regions. The ^{13}C NMR spectra were identified for the compounds **2** and **3**. The IR spectra revealed an amide absorption bond at about 1680 cm^{-1}. FAB mass spectra confirmed the mass and proposed structures of compounds **2** and **3** as 15-membered macrocyclic diamide and 30-membered macrocyclic tetramide, respectively. These compounds were obtained from [1+1] and [2+2]-condensation of diamine and oxalyl chloride via regular macrocyclization and dimerization, respectively.

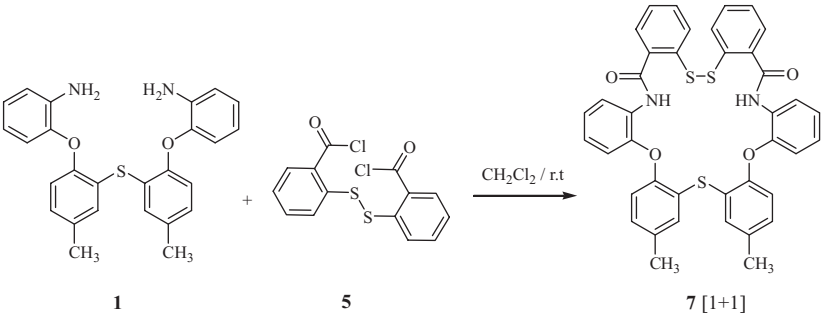
We also examined condensation of symmetrical diamine **1** with succinyl chloride in the same reaction conditions. In this reaction, [1+1] condensation macrocyclic diamide **4** was obtained in 40% yield. The ^{1}H NMR spectrum showed two singlet signals in the aliphatic regions for methyl and methylene protons at δ 2.29 and 2.48 ppm, respectively, and a sharp singlet signal appeared at δ 8.07 ppm for NH. The ^{13}C NMR spectrum revealed two signals for above methyl and methylene carbon at δ 20.83 and 32.52 ppm, respectively. Mass spectrum showed the molecular ion peak at *m/z* 510, which also is in agreement with the proposed structure (Scheme 2).



Scheme 2 [1+1]-Macrocyclization of diamine **1** and succinyl chloride.

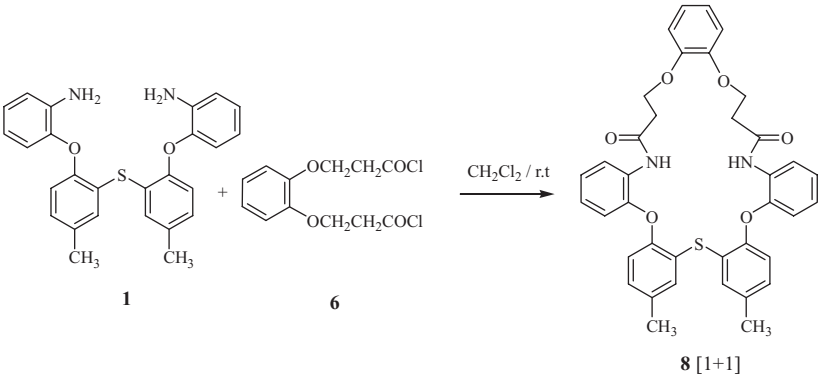
In the macrocyclization reaction of diamine **1** with 2,2′-dithiodibenzoicacid dichloride (**5**), only regular macrocyclization ([1+1]) occurred. In this reaction, a 21-membered macrocyclic diamide **7** (hexabenzodioxatrithiadiamide) was isolated in 60% yield as the main product (Scheme 3). The interesting point in compound **7** was the absorption of two types of methyl signals at δ 2.16 and 2.26 ppm in the ^{1}H NMR spectrum. The extent of rigidity of these macrocycles affects the methyl absorption regions. However, the macrocycles are twisted due to the repulsion effects of nonbonding electron pairs on sulfur, nitrogen, oxygen atoms and carbonyl groups in the cavity, and the intramolecular H-bondings cause rigidity in the macrocycles. Also, the repulsion between hydrogens of phenyl groups on the crown help the deformation in this part of the molecule. As an example, the rigidity of macrocycle **7** was confirmed by the absorption of two methyl groups at δ 20.56 and 20.70 ppm in the ^{13}C NMR and 38 types of carbons in aromatic regions. Observation of a molecular ion peak in the mass spectrum confirmed the molecular structure.

In another experiment, macrocyclization of diamine **1** with 1,2-bis(2-chlorocarbonyl ethoxy)benzene (**6**) was investigated under the same reaction conditions. This reaction led to the formation of [1+1] condensation macrocycle **8** (pentabenzotetraoxathiadiamide) in 65% yield as the only product (Scheme 4).



1 **5** **7** [1+1]

Scheme 3 Synthesis of [1+1]-macrocycle **7**.



1 **6** **8** [1+1]

Scheme 4 Cyclocondensation of diamine **1** with 1,2-bis(2-chlorocarbonyl ethoxy)benzene **6**.

It should be noted that the *meta* and *para* isomers of compound **6** were examined for a similar macrocyclization process, but the corresponding macrocycles were not obtained. Identification of compound **8** was carried out on the basis of spectroscopic data. The ^{1}H NMR spectra of compound **8** show a sharp singlet for two methyl groups at about δ 2.2 ppm. These observations indicated that the rigidity is reduced in this macrocycle compared to macrocycle **7**. This behavior may be because of reduction of the intramolecular H-bonding density and more flexibility in the cavity of this macrocycle compared to macrocycle **7**. Methylene protons revealed two triplets at about δ 2.7 and 4.2 ppm, and NH protons showed a singlet signal at about δ 8.6 ppm (Figure 1). Again, the observation of a molecular ion peak confirmed the molecular structure.

In conclusion, we have described the synthesis of a new multibenzo macrocyclic containing sulfur, oxygen, and lactam functional groups. In this process, the reaction of diamine **1** with oxalyl chloride give [1+1] and [2+2] condensations, with succinyl chloride, 2,2′-dithiodibenzoicacid dichloride (**5**), and 1,2-bis(2-chlorocarbonyl ethoxy)benzene (**6**) afford [1+1] condensation macrocyclic diamides, respectively. The flexibility and rigidity of this series of macrocycles are confirmed by spectroscopic means in this work. The repulsion between the hydrogens of phenyl groups in the crown part of the ring and the tetrahedral character of sulfide atoms affects the rigidity of the cavities significantly.

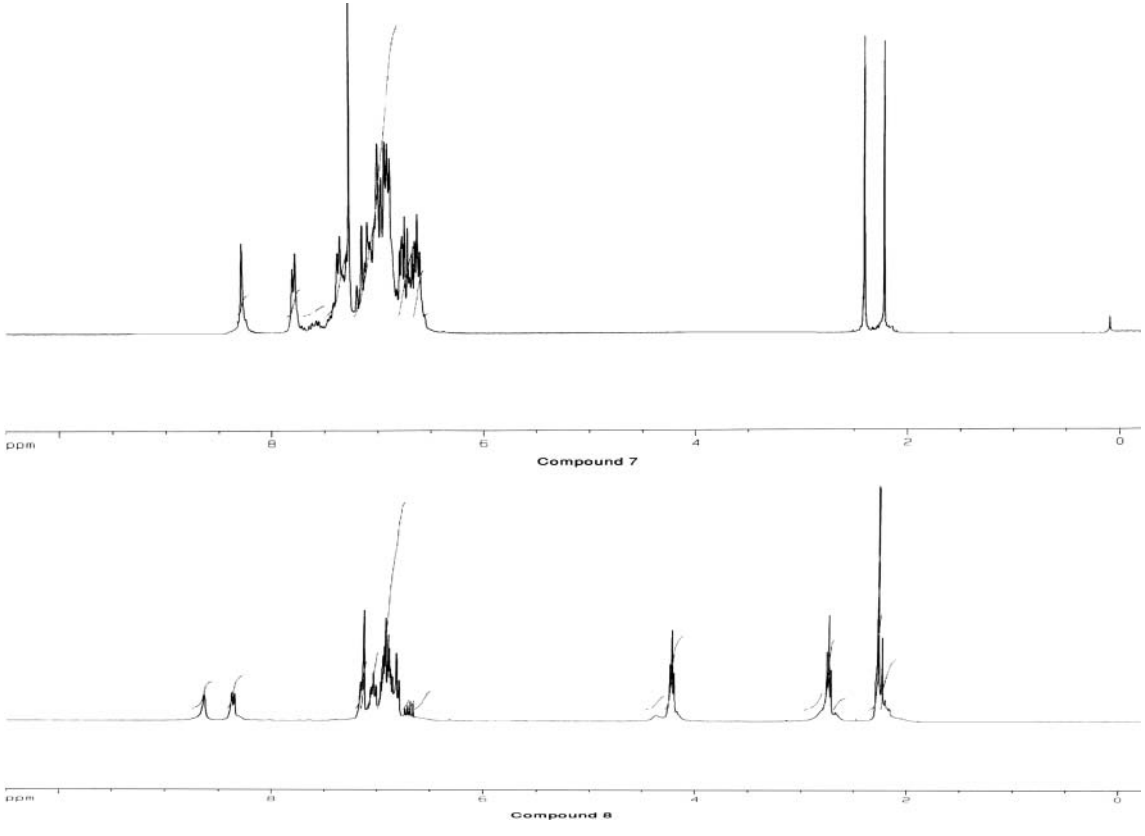


Figure 1 Comparison ^{1}H NMR spectra of compounds **7** in $CDCl_3$ and **8** in acetone-d_6.

EXPERIMENTAL

All commercially available chemicals and reagents were used without further purification. Melting points (uncorrected) were determined by an Electrothermal Engineering LTD 9100 apparatus. IR spectra were recorded on a Perkin-Elmer model 543, the ^{1}H and ^{13}C NMR spectra were obtained using a Bruker Avance DRX 300 apparatus at 298 K. Chemical shifts (δ) are reported in ppm and are referenced to the NMR solvent peak. Elemental analyses were carried out by a CHN-O-Rapid Heraeus elemental analyzer (Wellesley, MA). Mass spectra of the products were obtained with an HP (Agilent technologies) 5937 Mass Selective Detector. Also the FAB-mass spectrum of compound **3** was obtained by electron ionization Varian Incos 50 and JEOLJMS-700 and MULDI spectra Bruker Biflexα. CH_2Cl_2 was dried over P_2O_5 and then distilled from CaH_2. 2,2′-Dithiodibenzoic acid dichloride (**5**) was obtained by the reaction of 2,2′-dithiodibenzoic acid and oxalyl chloride in the presence of catalytic amounts of DMF in CH_2Cl_2[18] and identified with spectroscopic analysis. Compound **6** was obtained by the reaction of 1,2-bis(2-carboxyethoxy)benzene[19] with oxalyl chloride in the presence of catalytic amounts of DMF in CH_2Cl_2 and identified with spectroscopic analysis.

General Procedure for the Preparation of Macrocyclic Diamides 2–4, 7–8

A solution of diacid dichloride (2 mmol) in dry dichloromethane (50 mL) was added dropwise to a vigorously stirred solution of diamine **1** (2 mmol) in the same solvent (50 mL) containing triethylamine (4 mmol) for 30 min at 0–5°C. The mixture was stirred for 20 h at room temperature. The solvent was then removed under reduced pressure to give a crude product, which was purified by column chromatography on silica gel 60 using the proper eluent solvent.

2,21-Dimethyltetrabenzo[b,e,h,n][1,7]dioxa[4]thia[10,13]diazacyclopentadecin-11,12-(10H,13H)-dione (2). This compound was purified by column chromatography on silica gel using hexane:EtOAc (4:1) as eluent to afford a white solid product, mp 254–255°C; IR (potassium bromide): 3332, 3294, 3066, 2929, 1678, 1602, 1511, 1440, 1446, 1251, 1226, 746 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.12 (s, 6H, CH_3), 6.81–7.65 (m, 14H, Ar-H), 9.00 (s, 2H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.23, 152.44, 148.29, 134.12, 132.88, 128.92, 127.53, 126.98, 125.50, 124.46, 124.07, 119.54, 117.53, 20.55 ppm; ms: m/z 482 $[M]^+$, 483 $[M+1]^+$, 484 $[M+2]^+$, 485 $[M+3]^+$, 227, 197, 152, 91; *Anal.* Calcd. For $C_{28}H_{22}N_2O_4S$: C, 69.71; H, 4.56; N, 5.81. Found: C, 69.65; H, 4.60; N, 5.80.

2,21,25,44-Tetramethyloctabenzo[b,e,h,n,q,t,w,c_1][1,7,16,22]tetraoxa[4,19]dithia[10,13,25,28]tetraazacyclotriacontin-11,12,26,27-(10H,13H,25H,28H)-tetraone (3). This compound was purified by column chromatography on silica gel using hexane:EtOAc (4:1) as eluent to afford a white solid product, mp 350–353°C; IR (potassium bromide): 3360, 3322, 3025, 2927, 1689, 1602, 1524, 1469, 1446, 1250, 1228, 751 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.33 (s, 12H, CH_3), 6.61–8.38 (m, 28H, Ar-H) 9.62 (s, 4H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.57, 152.30, 147.23, 134.84, 133.81, 129.65, 127.82, 126.25, 124.73, 123.30, 121.02, 120.57, 116.51, 20.81 ppm; MS-FAB: m/z 964 $[M]^+$, 965 $[M+1]^+$, 966 $[M+2]^+$, 967 $[M+3]^+$, 937, 533, 482, 460, 455, 307, 228, 197; *Anal.* Calcd. For $C_{56}H_{44}N_4O_8S_2$: C, 69.71; H, 4.56; N, 5.81. Found: C, 69.68; H, 4.52; N, 5.84.

2,23-Dimethyl-12,13-dihydrotetrabenzo[b,e,h,p][1,7]dioxa[4]thia[10,15]diazacycloheptadecin-11,14-(10H,15H)-dione (4). This compound was purified by column chromatography on silica gel using hexane:EtOAc (5:1) as eluent to afford a white solid product, mp 242–244°C; IR (potassium bromide): 3418, 3349, 2944, 2926, 1682, 1600, 1520, 1473, 1442, 1244, 1225, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 2.29 (s, 6H, CH_3), 2.48 (s, 4H, CH_2), 6.66–7.27 and 8.28 (m, 14H, Ar-H), 8.07 (s, 2H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.43, 152.32, 146.08, 134.90, 133.28, 129.82, 129.27, 126.90, 125.70, 123.72, 121.23, 119.91, 116.81, 32.52, 20.83 ppm; ms: m/z 510 $[M]^+$, 511 $[M+1]^+$, 512 $[M+2]^+$, 513 $[M+3]^+$, 214, 197, 77, 55; Anal. Calcd. For $C_{30}H_{26}N_2O_4S$: C, 70.58; H, 5.10; N, 5.49. Found: C, 70.52; H, 5.14; N, 5.44.

2,31-Dimethylhexabenzo[b,e,h,l,p,t][1,7]dioxa[4,14,15]trithia[10,19]diazacyclohenicosin-11,118-(10H,19H)-dione (7). This compound was purified by column chromatography on silica gel using hexane:EtOAc (3:1) as eluent to afford a white solid product, mp 170–171°C; IR (potassium bromide): 3425, 3350, 3058, 1669, 1604, 1519, 1479, 1444, 1324, 1229, 745 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 2.16 (s, 3H, CH_3), 2.26 (s, 3H, CH_3), 6.62–7.35 (m, 22H, Ar-H), 8.50 (br, 2H, NH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.35, 165.20, 153.52, 151.80, 146.22, 139.19, 138.31, 138.21, 135.27, 133.39, 133.14, 133.02, 132.80, 132.57, 131.66, 130.07, 129.86, 129.28, 128.50, 127.33, 127.05, 126.90, 126.02, 124.64, 124.28, 123.47, 120.79, 120.50, 120.46, 120.02, 119.61, 118.54, 118.32, 116.90, 116.41, 116.30, 115.82, 115.65, 20.70, 20.56 ppm; ms: m/z 698 $[M]^+$, 699 $[M+1]^+$, 700 $[M+2]^+$, 701 $[M+3]^+$, 562, 471, 428, 349, 228, 214, 197, 137; *Anal.* Calcd. For $C_{40}H_{30}N_2O_4S_3$: C, 68.76; H, 4.29; N, 4.01. Found: C, 68.80; H, 4.22; N, 4.07.

2,31-Dimethyl-12,13,18,19-tetrahydropentabenzo[b,e,h,o,v][1,7,14,17]-tetraoxa[4]thia[10,21]diazacyclotricasin-11,20-(10H,21H)-dione (8). This compound was purified by column chromatography on silica gel using hexane:EtOAc (3:1) as eluent to afford a white solid product, mp 130–132°C; IR (potassium bromide): 3420, 3348, 3063, 2925, 1692, 1603, 1526, 1479, 1449, 1254, 747 cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): δ 2.27 (s, 6H, CH_3), 2.72–2.76 (t, 4H, $J = 5.8$ Hz, CH_2), 4.20–4.24 (t, 4H, $J = 5.8$ Hz, CH_2), 6.80–8.38 (m, 18H, Ar-H), 8.64 (s, 2H, NH); ^{13}C NMR (75 MHz, acetone-d_6): δ 169.76, 153.48, 150.24, 135.59, 133.75, 130.71, 125.64, 124.53, 124.34, 123.53, 122.02, 120.25, 118.36, 117.88, 117.51, 116.05, 66.56, 38.12, 20.65 ppm; ms: m/z 646 $[M]^+$, 647 $[M+1]^+$, 648 $[M+2]^+$, 649 $[M+3]^+$, 536, 482, 391, 319, 252, 228, 214, 197, 146, 110, 55; *Anal.* Calcd. For $C_{38}H_{34}N_2O_6S$: C, 70.58; H, 5.26; N, 4.33. Found: C, 70.52; H, 5.25; N, 4.36.

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