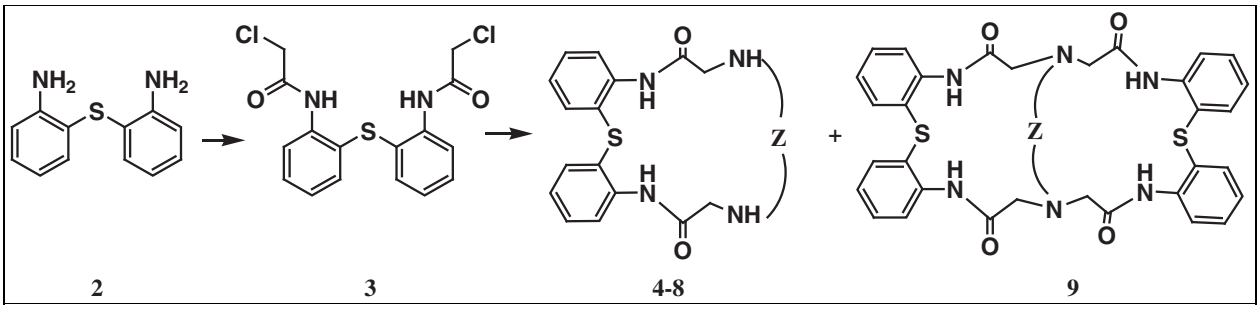
Synthesis of New Aza Macrocyclic Diamides 2,2′-Diaminodiphenyl Sulfide Using Crab-Like Method

Abbas Shockravi* and Mahmood Kamali

Faculty of Chemistry, Tarbiat Moallem University, 15614 Tehran, Iran
*E-mail: abbas_shockravi@yahoo.co.uk

Received July 4, 2010
DOI 10.1002/jhet.775
View this article online at wileyonlinelibrary.com.



Six new aza crown ethers **(4–9)** were synthesized based on the conventional route crab-like method with the reaction of corresponding bis-α-chloroacetamidediphenylsulfide (BCADPS) **(3)** and aliphathic diamines **(a–e)** in refluxing acetonitrile in good yields. BCADPS **(3)** was synthesized with the reaction of 2,2′-diaminodiphenyl sulfide **(2)** and chloroacetyl chloride. Interestingly, only the macrocyclization of BCADPS **(3)** with diamine **(e)** was led to the cryptand **(9)** in which methylene hydrogens were found as diastereotopic nucleis which is attributed to the rigidity of the cryptand (**9**). The formation of this cryptand (**9**) may be related the template effect of potassium ion. The structures of all compounds were confirmed using IR, ^{1}H-NMR, ^{13}C-NMR, mass spectroscopies, and elemental analysis.

J. Heterocyclic Chem., **49**, 499 (2012).

INTRODUCTION

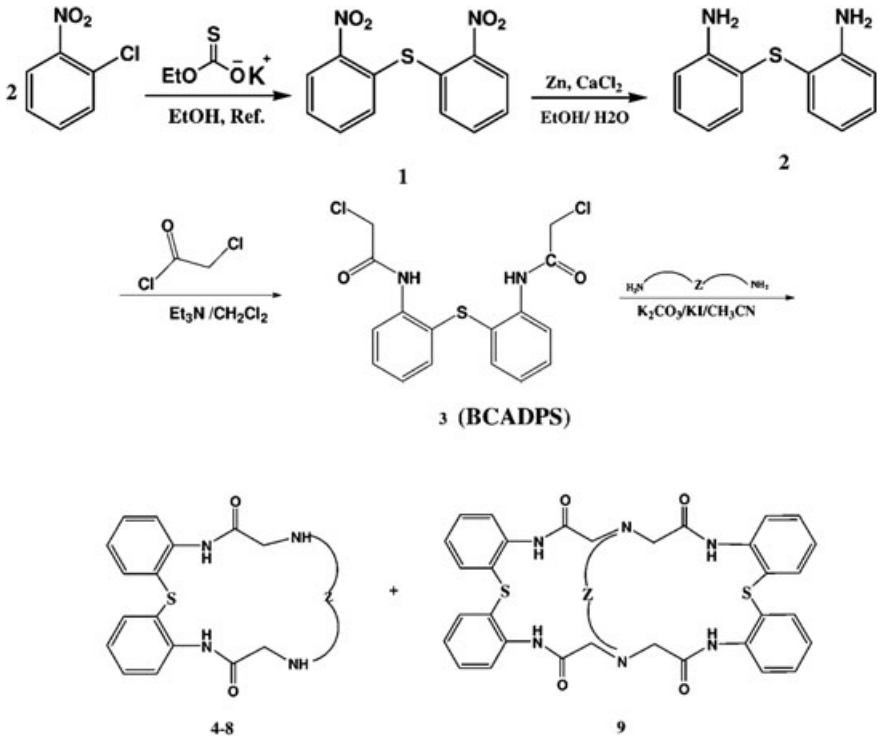
Since Pedersen's discovery [1], crown ethers have been intensively investigated macrocyclic compounds due to their facile synthesis and almost infinite possibilities of chemical modifications as host compounds. Aza crown macrocyclic compounds have gained a great deal of attention due to their wide applications in chemistry, metal separation, sensoring, analysis, biology, microanalysis, biophysics, and ecology [2,3].

A rational design of macromolecular receptors is governed by a number of factors (the nature, the number, the relative structural, and spatial placement of various ligating units, etc.), and the combination of these factors may induce an intrinsic balance of noncovalent binding forces optimum for specificity in host-guest recognition [4]. The amide group, so generously used by nature in a variety of antibiotic ionophores [5], has acquired a special status in the design of receptors because it displays dual (O or N and NH) ligating character, higher negative charge on oxygen than for ether and ester groups, and geometrical rigidity [6]. The averaged effects of these factors were found also in our researches on the complexation of aza-thia macrocyclic diamides with varieties of transition metal ions [7]. Amide-based macrocycles for selective recognition of metal cations [2,8] and organic molecules [2,3] typically adopt preorganization of their binding sites through hydrogen bonding or configurational rigidity around the amide carbon-nitrogen bond.

The method for the preparation of macrocylic diamides and corresponding aza crown compounds have been extensively reviewed [9]. Among these methods, the high dilution-techniques [10], the route base template effect [11], and the high pressure approach [12] are frequently used. Krakowiak *et al*. have developed a method based on the using bis(α-chloroamides) for preparation of corresponding aza crown compounds known as "crab-like" cyclization method [13] in good yield. Izatt and coworkers have used their methodology with appropriate dithiols in the presence of sodium or cesium carbonate to prepare the macrocycles in yields of about 40% [14]. These macrocycles were also synthesized by forming two C—N bonds in the ring closure step in which diacylation of diamines using diacid dichlorids [15,16a,b, diisocyanates [17], or diesters [16b,c,18 were used as diacylation agents. Aza thia macrocycles were also prepared by the interaction of disodium salts of *N*,*N*′-ditosyl-bis-(2-aminoethyl)sulfide with polyethylene glycol ditosylates [19] or a per-*N*-tosyl di-thia tetramine with 1,2-ethanediyl ditosylate using cesium carbonate in DMF [20]. Other suitable intramolecular cyclization method was used by Okahara to synthesis substituted sulfur containing macrocycles [21].

As, generally in the crab like synthetic method the high dilution technique is not required, the use of bis(α-chloroacetamide) is an important step in macrocyclization process [15,22]. Also, because the aromatic amine nitrogens are moderately active nucleophiles, generally the

Scheme 1



azamacrocycles were prepared by corresponding bis(α-chloroactamide)s substrats, which are converted to electrophilic moieties with relatively good leaving group.

The crab like cyclization using the bis-α-chloroacetamidediphenylsulfide (BCADPS) **(3)** was used to synthesis the aza macrocycles **(4–9)**. The formation of two C—N bonds in the ring closure step was performed by diacylation of diamines **(2)** using α-chloroacetylchloride.

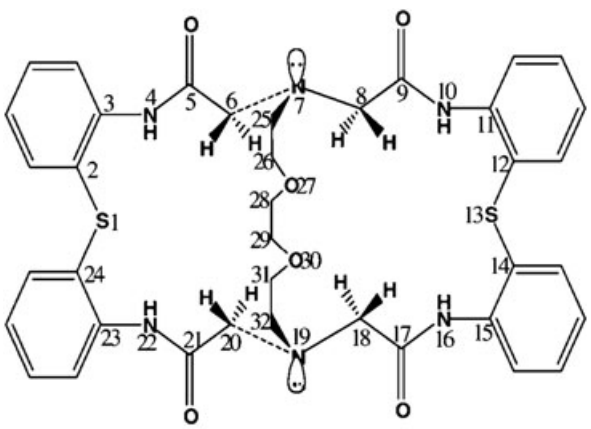
We have previously prepared a series of macrocyclic compounds based on dibenzosulfide, dinaphthosulfide containing hydroxyl phenolic groups and studied their metal ion complexations [7,16]. In this work, we wish to report the synthesis of six aza-thia and oxa macrocycles **(4–8)** using the reaction of BCADPS **(3)** as crab-like condensing reagent with different diamines **(a–e)** (Scheme 2) and the synthesis of cryptand **(9)** as the only [1 + 2] condensation product.

RESULTS AND DISCUSSION

In this work, diamine diphenyl sulfide (**2**) [23] was obtained from the reduction of corresponding dinitro compound (**1**) which its reaction with chloroacetyl chloride at room temperature led to BCADPS (**3**) in 4 h. High dilution technique and template effect of potassium ion were most effective conditions for the synthesis of macrocycles (**4–9**) in moderate to good yields and short time (5 h). (Scheme 1 and Table 1)

All products were identified using IR, ^{1}H-NMR, ^{13}C-NMR, and mass spectroscopies. All macrocycles were obtained as [1 + 1] condensation products except when diamine (**e**) was used in which both [1 + 1] and [1 + 2] products were synthesized. The formation of [1 + 2] product as cryptand (**9**) may be attributed to the template of effect of potassium ion (K^{+}) which apparently its ionic radius was more proper for such macrocyclization reaction conditions.

Scheme 2



9

Table 1

Macrocycles (**4–9**) and cryptand (**9**).

	Z	Products
a	—$(CH_2)_2$—	**4**
b	—CH_2—(*CH)CH_3—	**5**
c	—$(CH_2)_3$—	**6**
d	—$(CH_2)_4$—	**7**
e	—$(CH_2)_2$ O$(CH_2)_2$ O$(CH_2)_2$—	**8, 9**

Interestingly, the diastereotopicity character was found in the cryptand **(9)** which could be because of the rigidity of the cryptand structure. The dioxy triethylene bridge has also decreased the flexibility of the cryptand significantly which in turn participates in the diastereotopicity character. It seems that potassium ion's (K^+) template effect was effective in the formation of the cryptand especially due to oxygen donor atoms in the bridge between two nitrogen atoms. Identification of this cryptand was made by different spectroscopic means.

The ^{1}H-NMR spectrum of cryptand **(9)** showed one triplet at δ 2.63 ppm (4H) for two methylenes (25, 32) which are linked with nitrogen atoms. Two diasterotopic methylene hydrogens (6, 8, 18, 20) are appeared as two doublets at 3.17–3.23 ppm (4H, *J*= 17.3 Hz) and 3.42–3.48 ppm (4H, *J*= 17.3 Hz), respectively. One triplet appeared at δ 3.31ppm (4H) for two methylene hydrogens (26, 31) which are linked with oxygen atoms. One sharp singlet is observed at δ 3.38 ppm for four isochronous methylene hydrogens (28, 29) and 12 hydrogen atoms aromatic at 6.97–7.75 ppm (Scheme 2). Such diastereotopic evidences were not found in other macrocycles **(4–8)** which is due to their flexible cavities. For example, ^{1}H-NMR macrocycle **(4)** showed one sharp singlet for two methylenes **(8, 9)** linked with NH amines (δ 2.75 ppm, 4H) and one sharp singlet for two methylenes **(6, 11)** linked with carbonyl groups (δ 3.28 ppm, 4H) and 8H aromatic (δ 7.05–8.25 ppm, 8H) (Scheme 3).

EXPERIMENTAL

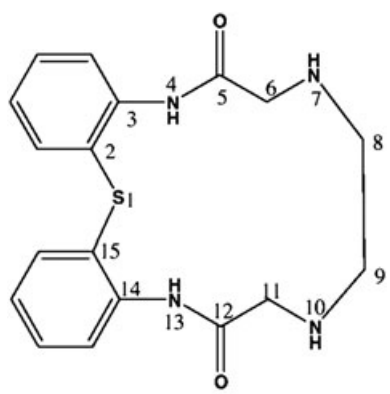
Chemicals and apparatus. All reactions were carried out in an efficient hood. The starting materials were purchased from Merck and fluka chemical companies. Column chromatography was performed on silica gel 60 (0.04–0.063 mm). Melting points were determined with a Branstead Electrothermal model 9200 apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer RX 1 Fourier transform infrared spectrometer. The ^{1}H- and ^{13}C-NMR spectra were recorded in DMSO-d_6 and $CDCl_3$ on Bruker Avance 300 MHz spectrometers. Elemental analyses were carried out by a Perkin Elmer 2400 series II CHN/O analyzer. Mass spectra were obtained by an electron ionization Varian Incos 50 and JOELJMS-700 and the MuLDI spectra BRUKER Biflex.

Synthesis of 2,2′-sulfanyl-bis(-α-chloroacetanilide) (3). To a solution of the appropriate 2,2′-daminodiphenyl sulfide (10 mmol, 2.16 g) and triethylamine (22 mmol, 3 mL) in CH_2Cl_2 (80 mL), chloroacetylchloride (1.75 mL, 22 mmol) in 1 h was added and stirred 3 h at room temperature. After completion of the reaction (TLC), the mixture washed with solution of HCl 10% (3× 40) and organic layer evaporated to afford a precipitate which was recrystallized from ethanol to give pure diamine **(2)** in the quantitative yield. Mp 152–153°C; IR (KBr): 3925, 3263, 3176, 1669, 1590, 1579, 1537, 1436, 768, 659 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$); δ 4.17 (s, 4H), 7.09–7.15 (t, 2H, *J* = 7.8 Hz), 7.23–7.26 (d, 2H, *J* = 9.0 Hz), 7.33–7.38 (t, 2H, *J* = 7.8 Hz), 8.13–8.16 (d, 2H, *J* = 9.0 Hz), 8.98 (s, 2H, exchanged with D_2O) ppm; ^{13}C-NMR (300 MHz, $CDCl_3$); δ 43.08, 122.67, 124.06, 126.15, 129.45, 132.69, 136.42, 164.24 ppm; Anal. Calcd. for $C_{16}H_{14}Cl_2N_2O_2S$: C, 52.04; H, 3.82; N, 7.59. Found: C, 52.31; H, 3.84; N, 7.53.

General procedure for the synthesis of bis acetamide-diphenylsulfide aza macrocycles. To 2 mmol of compound **(2)** in acetonitrile (100 mL), K_2CO_3 (2 mmol, 0.276 g), KI (0.1 mmol, 0.017 g), and the appropriate diamine (2 mmol) were added, then mixture was refluxed for 5 h. On completion of the reaction (TLC), the reaction mixture was allowed to cool to room temperature, and precipitate isolated with filtering, then the filtrate was evaporated under reduced pressure and respective crown was isolated by column chromatography on silica gel 60 using solvent mixed ethyl acetate and methanol (90:10).

Synthesis of 4,7,10,13-tetraaza-1-thia-2,3;14,15-dibenzo cyclopentadecane-5,12-dione (4). This macrocycle was synthesized based on the general procedure involving the reaction of compound **3** (2 mmol, 0.92 g), and ethylenediamine (2 mmol, 0.14 mL) in refluxing acetonitrile (100 mL). The solid product was purified using column chromatography to afford **4** as a white solid in 76% yield, mp 211–213°C; IR (KBr): 3325, 3296, 3232, 3164, 1671, 1585, 1572, 1517, 758 cm^{-1}; ^{1}H-NMR (300 MHz, CD_3CN); δ 2.1 (s, 2H, exchanged with D_2O), 2.75 (s, 4H), 3.28 (s, 4H), 7.05–7.10 (t, 2H, *J* = 7.8 Hz), 7.31–7.36 (t, 2H, *J* = 8.1 Hz), 7.42–7.45 (d, 2H, *J* = 7.8 Hz), 8.21–8.25 (d, 2H, *J* = 8.1 Hz), 10.57 (s, 2H, exchanged with D_2O, NH) ppm; ^{13}C-NMR (300 MHz, CD_3CN); δ 49.73, 53.23, 117.24, 120.92, 124.19, 129.39, 134.24, 138.16, 170.50 ppm; ms (electron impact) *m/z*: (M^+, molecular ion) 356, 261, 243, 216, 199, 161, 140, 124, 85, 44.

Scheme 3



4

Synthesis of 4,7,10,13-tetraaza-1-thia-8-methyl-2,3;14,15-dibenzo cyclopenta decane-5,13-dione (5). This macrocycle was synthesized based on the reaction of compound **3** (2 mmol, 0.92 g), and 1-methyl-1,2-diaminopropane (optical active) (2 mmol, 0.18 mL) in refluxing acetonitrile (100 mL). The solid product was purified using column chromatography to afford **5** as a white solid in 70% yield, mp 267–269°C; IR (KBr): 3435, 3354, 3304, 3232, 3155, 1671, 1586, 1573, 1514, 773, 757 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6); δ 0.99–1.01 (d, 3H, J = 6.1 Hz), 2.58 (d, 2H, J = 6.1 Hz), 2.67 (m, 1H), 3.07–3.09 (d, 1H, J = 7.7 Hz), 3.13–3.15 (d, 1H, J = 7.7 Hz), 3.31–3.32 (d, 1 H, J = 17.4 Hz), 3.35–3.38 (d, 1H, J = 17.4 Hz), 3.33 (s, 2H, exchanged with D_2O, NH), 7.08–7.13 (tb, 2H), 7.32–7.38 (2t, 2H), 7.36–7.38(d, 1H, J = 7.8 Hz), 7.42–7.45 (d, H, J = 8.1 Hz), 8.08–8.10 (d, 1H, J = 8.1 Hz), 8.18–8.20 (d, 1H, J = 8.1 Hz), 10.64 (s, exchange with D_2O) ppm; ^{13}C-NMR (300 MHz, DMSO-d_6); δ 17.86, 50.67, 53.36, 53.84, 55.98, 120.66, 121.56, 123.06, 123.84, 124.33, 124.62, 129.37, 129.61, 134.03, 134.46, 137.77, 137.96, 170.62, 171.04 ppm; ms (electron impact) m/z: (M^+, molecular ion) 371, 216, 199, 183, 154, 124, 93, 56.

Synthesis 4,7,11,14-tetraaza-1-thia-2,3;15,16-dibenzo cyclohexadecane-5,14-dione (6). This macrocycle was synthesized based on the reaction of compound **3** (2 mmol, 0.92 g), and 1,3-diaminopropane (2 mmol, 0.17 mL) in refluxing acetonitrile (100 mL). The solid product was purified using column chromatography to afford **6** as a white solid in 69% yield, mp 192–195°C; IR (KBr): 3357, 3332, 3291, 3250, 1663, 1584, 1571, 1507, 763 cm^{-1}; ^{1}H-NMR (300 MHz, CD_3CN); δ 1.66–1.70 (m, 2H), 2.5 (s, 2H, exchange with D_2O, NH), 2.81–2.85 (t, 4H, J = 6.0 Hz), 3.25 (s, 4H), 7.06–7.11 (t, 2H, J = 7.6 Hz), 7.28–7.34 (m, 4H), 7.97–8.00 (2d, 2H, J = 8.7 Hz), 9.96 (s, exchanged with D_2O) ppm; ^{13}C-NMR (300 MHz, CD_3CN); δ 29.36, 46.69, 60.91, 117.32, 122.05, 125.23, 129.80, 133.35, 137.68, 170.81 ppm; ms (electron impact) m/z (relative intensity %): (M^+, molecular ion) 371, 243, 216, 199, 183, 154, 124, 106, 85, 44.

Synthesis 4,7,12,15-tetraaza-1-thia-2,3;15,16-dibenzo cycloheptadecane-5,15-dione (7). This macrocycle was synthesized based on the reaction of compound **3** (2 mmol, 0.92 g), and 1,4-diaminobutane (2 mmol, 0.2 mL) in refluxing acetonitrile (100 mL). The solid product was purified using column chromatography to afford **7** as a white solid in 69% yield, mp 206–207°C; IR (KBr): 3441, 3354, 3243, 3189, 1690, 1674, 1586, 1575, 1519, 761 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6); δ 1.61 (t, 4H, J = 8.5 Hz), 2.54 (t, 4H, J = 8.5 Hz), 3.20 (s, 4H), 3.32 (2H, exchanged with D_2O), 7.03–7.08 (t, 2H, J = 7.7 Hz), 7.10–7.13 (d, 2H, J = 7.7 Hz), 7.33–7.38 (t, 2H, J = 8.2 Hz), 8.33–8.35 (d, 2H, J = 8.2 Hz), 10.27 (s, exchanged with D_2O) ppm; ^{13}C-NMR (300 MHz, DMSO-d_6); δ 27.54, 49.63, 53.32, 121.33, 121.92, 125.44, 129.33, 131.62, 137.06, 171.55 ppm; ms (electron impact) m/z: (M^+, molecular ion) 385, 243, 199, 128, 84, 43.

Synthesis (8) and (9). These macrocycles were synthesized based on the reaction of compound **3** (2 mmol, 0.92 g), and 3,6-dioxa-1,8-diamino octane (2 mmol, 0.3 mL) in refluxing acetonitrile (100 mL). The solid product was purified using column chromatography to afford **9** as a white solid in 66% yield, with mp 276–280°C and white solid **10** yield 15%, with mp 169–170°C. These macrocycles were characterized as following.

Macrocycle 4,7,16,19-tetraaza-10,13-oxa-1-thia-2,3;20,21-dibenzo cyclodocosane-5,18-dione (8). IR (KBr): 3357, 3224, 3057, 1679,1576, 1512, 763 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6); δ 2.61–2.64 (t, 4H, J = 4.5 Hz), 3.41–3.44 (t, 4H, J = 4.5 Hz), 3.47 (s, 4H), 3.95 (s, 4H), 7.01–7.04 (d, 2H, J = 7.5 Hz), 7.07–7.12 (t, 2H, J = 7.2 Hz), 7.29–7.34 (t, 2H, J = 7.8 Hz), 7.86–7.89 (d, 1H, J = 8.0 Hz), 7.91–7.94 (d, 1H, J = 8.0 Hz), 9.98 (s, exchanged with D_2O) ppm; ^{13}C-NMR (300 MHz, DMSO-d_6); δ 49.09, 52.59, 70.14, 70.41, 123.62, 125.75, 126.43, 129.47, 132.37, 137.54, 171.06 ppm; ms (electron impact) m/z: (M^+, molecular ion) 445, 199, 93, 42.

Macrocycle 4,7,10,16,20,22-hexaaza-10,13-dithia-27,30-dioxa-3,2;11,12;17,18;-20,21-tetrabenzo bicycle[11,11,8] triacontane-5,9,17,21-tetraone (9). IR (KBr): 3460, 3248, 3057, 1735, 1686, 1576, 1508, 752 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6); δ 2.63 (tb, 4H), 3.17–3.23 (d, 4H, J = 17.3 Hz), 3.31 (tb, 4H), 3.38 (s, 4H), 3.42–3.48 (d, 4H, J = 17.3 Hz), 6.97–7.00 (d, 4H, J = 7.5 Hz), 7.07–7.12 (t, 4H, J = 7.5 Hz), 7.25–7.30 (t, 4H, J = 7.8 Hz), 7.73–7.75 (d, 2H, J = 7.8 Hz), 9.82 (s, 4H, exchanged with D_2O) ppm; ^{13}C-NMR (300 MHz, DMSO-d_6); δ 55.46, 59.35, 69.41, 70.42, 124.87, 126.55, 126.74, 129.18, 131.87, 137.20, 170.62 ppm; ms (electron impact) m/z: (M^+, molecular ion) 741, 216, 199, 173, 136, 120, 93, 74, 56.

Acknowledgment. We thank the research council of the Tarbiat Moallem University for financial support.

REFERENCES AND NOTES

[1] Pedersen, C. J. J Am Chem Soc 1967, 89, 7017.

[2] Izatt, R. M.; Pawlak, K.; Bradshaw, J. S.; Bruening, R. L. Chem Rev 1995, 95, 2529.

[3] Jacobsen, F. E.; Lewis, J. A.; Cohen, S. M. J Am Chem Soc 2006, 128, 3156.

[4] (a) Ibrahim, R.; Tsuchiya, S.; Ogawa, S. J Am Chem Soc 2000, 122, 12174; (b) Bricks, J. L.; Kovalchuk, A.; Trieflinger, C.; Nofz, M.; Buschel, M.; Tolmachev, A. I.; Daub, J.; Rurack, K. J Am Chem Soc 2005, 127, 13522; (c) Chartres, J. D.; Lindoy, L. F.; Meehan, G. V. Tetrahedron 2006, 62, 4173; (d) Szczygelska Tao, J.; Biernat, J. F. Polym J Chem 2002, 76, 931.

[5] (a) Le Grel, P.; Salaun, A.; Potel, M.; Le Grel, B.; Lassagne, F. J Org Chem 2006, 71, 5638; (b) Meinke, P. T.; Arison, B.; Culberson, J. C.; Fisher, M. H.; Mrozik, H. J Org Chem 1998, 63, 2591.

[6] Novak, I.; Potts, A. W. J Org Chem 1996, 61, 786.

[7] (a) Shockravi, A.; Shamsipur, M.; Fattahi, H.; Taghdiri, M.; Heidaryan, D.; Alizadeh, K.; Rostami, E.; Abbaszadeh, A.; Yousefi, A. J Inclusion Phenom Macrocycl Chem 2008, 61, 153; (b) Shockravi, A.; Chaloosi, M.; Zakeri, M.; Mozaffari, S.; Rostami, E.; Abouzari-Lot, E. Phosphorus Sulfur Silicon Relat Elem 2006, 181, 2321; (c) Mashhadizadeh, M. H.; Khani, H.; Shockravi, A. J Inclusion Phenom Macrocycl Chem 2010, 68, 219.

[8] Freiria, A.; Bastida, R.; del Carmen Fernandez-Fernandez, M.; Macias, A.; Valencia, L.; Vicente, M. Inorg Chem 2005, 44, 930.

[9] (a) Krakowiak, K. E.; Bradshaw, J. S.; Zamecka-Krakowiak, D. J. Chem Rev 1989, 89, 929; (b) Jurczak, J.; Ostaaszewski, R. J Coord Chem 1992, 27, 201.

[10] (a) Krakowiak, K. E.; Bradshaw, J. S.; Izatt, R. M. In Aza Crown Macrocycles; Wiley: New York, 1993; (b) Knops, P.; Sendhoff, N.; Mekeelburger, H. B.; Vogtel, F. Top Curr Chem 1992, 161, 1.

[11] (a) Schwrtz, E.; Gottlieb, H. E.; Frolow, F.; Shanzer, A. J Org Chem 1985, 50, 5469; (b) Ninagawa, A.; Maeda, T.; Matsuda, H.

Chem Lett 1984, 1985; (c) Leygue, N.; Cazaux, L.; Picard, C.; Tisnes, T. Tetrahedron Lett 1987, 28, 4049.

[12] (a) Jurzack, J.; Pietraszkiewicz, M. Top Curr Chem 1985, 130, 183; (b) see ref. 4d.

[13] Krakowiak, K. E.; Bradshaw, J. S.; Izatt, R. M. Synlett 1992, 611.

[14] (a) Krakowiak, K. E.; Bradshaw, J. S.; Izatt, R. M. J Heterocycl Chem 1990, 27, 1585; (b) Bradshaw, J. S.; Krakowiak, K. E.; An, H. Y.; Izatt, R. M. J Heterocycl Chem 1990, 27, 2113.

[15] Pelissard, D.; Louis, R. Tetrahedron Lett 1972, 4589.

[16] (a) Shockravi, A.; Tabrizi, S. B.; Rostami, E.; Yousefi, A.; Dehjurian, A.; Tohidi, R. J Inclusion Phenom Macrocycl Chem 2004, 49, 163; (b) Shockravi, A.; Tabrizi, S. B. J Inclusion Phenom Macrocycl Chem 2005, 52, 223; (c) Rostami, A.; Shockravi, A.; Fattahi, H.; Heydarian, D.; Shbhbanzadeh, S.; Ghorbani, S.; Javadi, A.; Mehdipoure Ataei, S. Phosphorus Sulfur Silicon Relat Elem 2009, 184, 2066.

[17] Ishii, F.; Usagawa, Y.; Sakamoto, H. Chem Abstr 1989, 111, 15275e.

[18] (a) Tabushi, I.; Okino, H.; Kuroda, Y. Tetrahedron Lett 1976, 4339; (b) Kiamwa, E.; Machinda, R.; Kodama, M. J Am Chem Soc 1984, 106, 5497; (c) Kodama, M.; Koike, T.; Hoshinga, N.; Machida, R.; Kimwa, E. J Chem Soc Dalton Trans 1984, 673.

[19] Hart, S. M.; Boeyens, J. C. A.; Michael, J. P.; Hancock, J Chem Soc Dalton Trans 1983, 1601.

[20] Craig, A. S.; Kataky, R.; Parka, D.; Adams, H.; Bailey, N.; Schneider, J Chem Soc Chem Commun 1989, 1870.

[21] Nakatsuji, Y.; Mizuno, T.; Okahara, M. J Heterocycl Chem 1982, 19, 733.

[22] Krakowiak, K. E.; Bradshaw, J. S.; Izatt, R. M. Tetrahedron Lett 1988, 29, 3521.

[23] (a) Price, C. C.; Stacy, G. W. Org Synth Coll 1955, 3, 86; (b) Allinger, N. L.; Youngdale, G. A. J Am Chem Soc 1962, 84, 1020.