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Synthesis of New Dibenzosulfide Macrocyclic Diamides

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Abstract

Some new dibenzosulfide macrocyclic diamides (**6**–**12**) are synthesized. The macrocyclization process is performed by reaction of the dimethyl ester (**5**) and diacid chloride (**4**) with appropriate diamines by two methods. Also the results of two methods are discussed and compared.

Introduction

Macrocyclic diamides and corresponding aza crown compounds have gained a great deal of attention due to their wide applications in chemistry, biology, analysis and microanalysis, metal separation and transport, molecular recognition, medical and industrial uses, biophysics, catalysis, enzyme mimics, sensoring and switching agriculture and ecology [1]. Due to their high capability in selective and effective complexation with variety of transition and heavy metals, molecular ions and neutral molecules, there is an increasing interest in the preparation of them [2, 3]. Macrocyclic diamides are valuable intermediates for the preparation of aza crown compounds and more complicated ligands such as cryptands and cryptohemispherands that can be functionalized by additional ligating centers including chromogenic and proton ionizable groups [4].

The methods for the preparation of macrocyclic diamides and corresponding aza crown compounds have been extensively reviewed [5]. Among these methods, the high dilution-techniques [6], the route based on the template effect [7], and the high pressure approach [8] are frequently used. Diesters such as α,γ-diesters, di-*t*-butyl and diethyl diesters, especially dimethyl diesters have been used for the preparation of them under different conditions in good yields [9]. Using of diesters allow the reaction take place under normal conditions without using of high dilution-techniques. Activated carboxylic acid derivatives have also been used for the preparation of macrocyclic diamides in excellent yields under normal reaction conditions reaction [10]. Bradshaw and Krakowiak have developed a method based on bis (α-chloro amides) for the preparation of them and corresponding aza crown compounds in good yields in ''crab-like'' cyclization [11]. Recently Sharghi and co-workers have reported a method in dry media (SiO_2/MnO_2) in moderate yields and a method without using high dilution-techniques called ''high-concentration'' method for the preparation of macrocyclic diamides that take place under vigorous and high speed stirring with fast addition reactants consisted of diacid chloride and diamine in proper solvent together [12].

This work is in continuation of our previous researches toward the synthesis of new dibenzosulfide and dibenzosulfoxide macrocyclic compounds [13]. Herein we report the synthesis of set of new macrocyclic bisamides containing dibenzosulfide moiety obtained via the cyclization reactions of terminal dimethyl ester and diacid chlorides with terminal diamines.

The dibenzosulfide **1** was synthesized based on reported procedure in 50% yield. Treatment of **1** with the chloroacetonitrile and methylchloroacetate in the presence of K_2CO_3 and KI at refluxed dry acetone gaved dinitrile **2** in 100% yield and dimethyl ester **5** in 98% yield respectively. Basic hydrolysis of dinitrile **2** at refluxed alchoholic aqueous solution followed by acidification afforded the diacid **3** in 90% yield. The diacid **3** was converted to diacid chloride **4** in 95% yield by reaction with oxalyl chloride in the presence of DMF (cat.) in CH_2Cl_2 and used without purification. All the reactions were carried out based on standard procedures [14] (Scheme 1, 2).

Experimental

All the materials purchased from Merck, Fluka and Aldrich chemical companies and applied without further purification. The melting points (uncorrected) were measured with a Electrothermal engineering LTD 9100 apparatus. IR spectra were measured on a Perkin–Elmer model 543, the ^1H NMR and ^{13}C NMR spectra were

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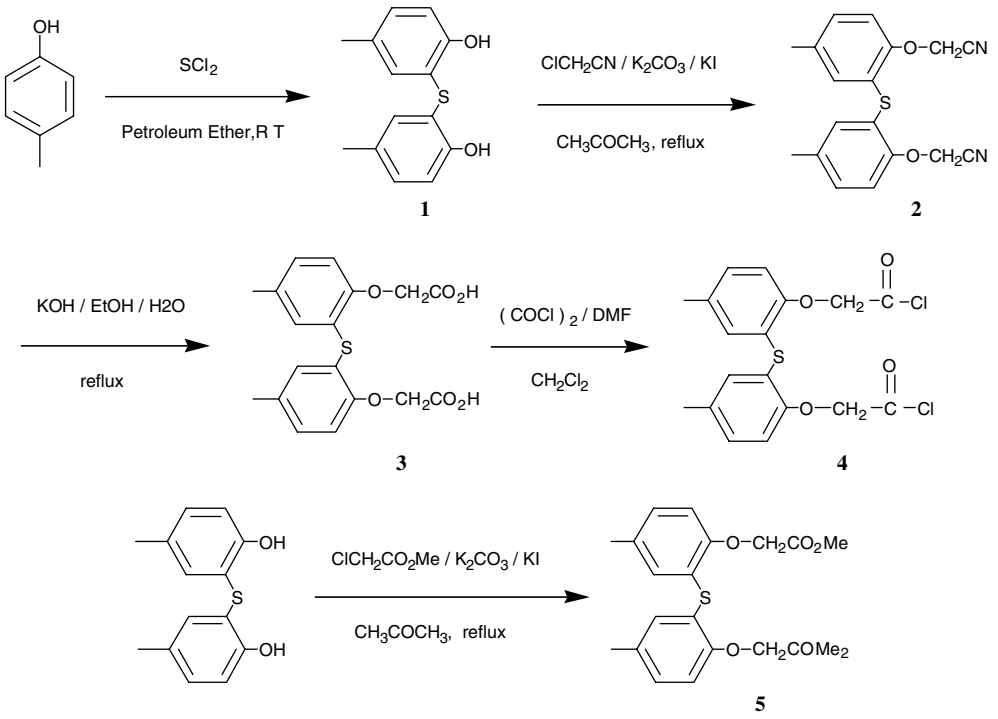


Scheme 1, 2.

obtained using BRUKER AVANCE DRX 500 apparatus and mass spectra were obtained with Shimadzu GC-MS-QP 1100 EX model. CH_2Cl_2 was dried over P_2O_5 and then distilled from CaH_2. CH_3CN and MeOH were dried over CaH_2 and then distilled.

General procedure for the method A

A solution of diamine (0.002 mol) and triethylamine (0.004 mol) in an appropriate dried solvent (50 ml, CH_3CN or CH_2Cl_2) was added quickly (5 s) to a vigorously stirred solution of diacid dichloride (0.002 mol) in the same solvent (50 ml) at 0 °C. The reaction mixture was stirred at room temperature for 30 min. The precipitate was filtered off and the filtrate was washed with water (2×50 ml), 10% aqueous NaOH solution (50 ml) and then with water (100 ml). The organic layer was dried (Na_2SO_4) and evaporated to afford solid products which was purified by recrystallization or column chromatography. In the absence of Et_3N the reaction took place at room temperature and for the entry **6** the reaction took place in the excess of Et_3N (10 times in mole).

General procedure for the method B

A mixture of diamine (0.01 mol) and dimethyl esters (0.01 mol, 0.78 gr) in dry methanol (80 ml) was refluxed for 18–24 h. Then the solvent was evaporated under reduced pressure and the crude product was purified by column chromathography on silica gel and/or recrystallization. Reaction in the presence of Cs_2CO_3 in the same conditions lead to higher yields and in the presence of Et_3N and NaOMe didn't give good yields for **6**.

*7,14-Diaza-1,10,11-trithia-4,17-dioxa-21,25-dimethyl-2,3;18,19-dibenzo-cyclonona-decane-6,15-dione (**6**)*

This compound was purified by column chromatography on silica gel using EtOAc as eluent and then recrystallized from $CHCl_3$/*n*-Hexane to afford white powder, m.p. 204–206 °C; IR (KBr) 3410, 3321, 3063, 3044, 2939, 2886, 1692, 1669, 1537, 1501, 1440, 1301, 1260, 1085, 1055, 832, 813, 568 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.23 (s, 6H), 2.86–2.89 (m: distorted "ddd", 4H), 3.56–3.6 (m: distorted "ddd", 4H), 4.54 (s, 4H), 6.82–6.84 (d, $j = 8.2$ Hz, 2H), 6.9 (s, 2H), 7.09–7.1 (d, $j = 8.2$ Hz, 2H), 7.19 (broad, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.29, 153.63, 132.88, 132.3, 129.67, 122.06, 113.3, 68.67, 39.23, 38.42, 20.63 ppm; MS (EI) m/z 478 $(M)^+$, 479 $(M+1)^+$, 480 $(M+2)^+$, 481 $(M+3)^+$, 420, 403, 363, 316, 303, 257, 246, 241, 228, 206, 190, 184, 151, 121, 108, 91, 77, 56.

7,12-Diaza-1-thia-4,15-dioxa-19,23-dimethyl-2,3;16,17-dibenzo-cycloheptadecane-6,13-dione (7)

This compound was purified by column chromatography on silica gel using EtOAc/ MeOH (20:1) as eluent and then recrystallized from $CHCl_3$/n-Hexane to afford white fluffy solid, m.p. 206–208 °C; IR (KBr) 3397, 3349, 3027, 2930, 2855, 1679, 1660, 1547, 1528, 1493, 1435, 1291, 1256, 1157, 1081, 1064, 1043, 817, 583 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.39 (s, 4H) , 2.22 (s, 6H), 3.26 (s, 4H), 4.55 (s, 4H), 6.22 (broad, 2H), 6.79–6.81 (d, $j = 8.2$ Hz, 2H), 6.96 (s, 2H), 7.06–7.08 (d,

$j = 8.2$ Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.32, 153.41, 132.55, 132.51, 129.76, 121.49, 112.41, 67.94, 38.04, 25.84, 20.86 ppm; MS (EI) m/z 414 $(M)^+$, 415 $(M+1)^+$, 416 $(M+2)^+$, 417 $(M+3)^+$, 303, 259, 257, 247, 241, 228, 195, 184, 152, 151, 121, 70.

*7,11-Diaza-1-thia-4,14-dioxa-18,22-dimethyl-2,3;15,16-dibenzo-cyclohexadecane-6,12-dione (***8***)*

This compound was purified by column chromatography on silica gel using EtOAc /MeOH (20:1) as eluent and then recrystallized from $CHCl_3$/*n*-Hexane to afford colorless fine crystals, m.p. 207–209 °C; IR (KBr) 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, $CDCl_3$) δ 1.87–1.92 (m: distored "ddd", 2H), 2.23 (s, 6H), 3.13–3.17 (m: distored "ddd", 4H), 4.55 (s, 4H), 6.6 (broad, 2H), 6.82–6.83 (d, $j = 8.3$ Hz, 2H), 6.975–6.977 (d, $j = 1$ Hz, 2H), 7.07–7.09 (dd, $j_1 = 8.3$, $j_2 = 1$ Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.29, 152.8, 132.74, 132.66, 129.79, 121.67, 112.33, 67.65, 36.7, 25.47, 20.59 ppm; MS (EI) m/z 400 $(M)^+$, 401 $(M+1)^+$, 402 $(M+2)^+$, 403 $(M+3)^+$, 404 $(M+4)^+$, 303, 257, 241, 233, 228, 195, 184, 152, 151, 141, 121, 91, 56.

*7,10-Diaza-1-thia-4,13-dioxa-17,21-dimethyl-2,3;14,15-dibenzo-cyclopentadecane-6,11-dione (***9***)*

This compound was purified by column chromatography on silica gel using EtOAc as eluent and then recrystallized from $CHCl_3$/*n*-Hexane to afford colorless fine crystals, m.p. 277–279 °C; IR (KBr) 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, $CDCl_3$) δ 2.23 (s, 6H), 3.28–3.29 (m, 4H), 4.56 (s, 4H), 6.65 (broad, 2H), 6.82–6.84 (d, $j = 8.3$ Hz, 2H), 6.95 (d, $j = 0.52$, 2H), 7.06–7.08 (dd, $j_1 = 8.3$, $j_2 = 0.52$ Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168, 152.67, 132.69, 132.46, 129.63, 121.58, 112.57, 68.06, 38.58, 20.62 ppm; MS (EI) m/z 386 $(M)^+$, 388 $(M+2)^+$, 389 $(M+3)^+$, 257, 241, 228, 219,195, 184, 151, 121, 91, 43.

*7,10-Diaza-1-thia-4,13-dioxa-17,21-dimethyl-2,3;8,9;14,15-tribenzo-cyclopentadec-ane-6,11-dione (***10***)*

This compound was purified by column chromatography on silica gel using Petroleum Ether/EtOAc (1:1) as eluent and then recrystallized from $CHCl_3$ or $CHCl_3$/ *n*-Hexane to afford white fluffy solid, m.p. 255–257 °C; IR (KBr) 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, $CDCl_3$) δ 2.25 (s, 6H), 4.7 (s, 4H), 6.87, 6.98, 7.08, 7.18 (m, 8H), 7.98 (s, 2H), 8.5 (s, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.78, 153.4, 133.28, 133.08, 130.08, 127.72, 125.94, 124.02, 122.73, 113.96, 69.44, 20.62 ppm; MS (EI) m/z 434 $(M)^+$, 435 $(M+1)^+$, 436 $(M+2)^+$, 437 $(M+3)^+$, 408, 257, 254, 241, 228, 215, 205, 195, 184, 161, 151, 147, 121, 108, 91, 77, 52.

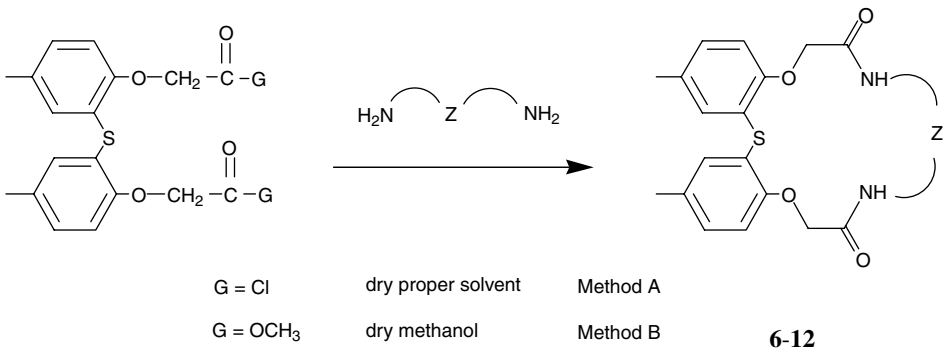
Results and discussion

We used two methods for the macrocyclization process (Scheme 3). The results are shown in Table 1.

Table 1. Synthesized macrocycles (**6**–**12**) by two methods

Compd	Z	Method A yield (%)	Method B yield (%)	Proper solvent	Et_3N
6	$-(CH_2)_2SS(CH_2)_2-$	85	40	CN_3CN	Excess
7	$-(CH_2)_4-$	48	54	CH_2Cl_2	+
8	$-(CH_2)_3-$	46	52	CH_2Cl_2	+
9	$-(CH_2)_2-$	42	84	CH_2Cl_2	+
10	$-O-C_6H_4-$	72	N. R.	EtOAc	–
		64		CN_3CN	
11	$-(CH_2)_2NH(CH_2)_2-$[a]	45	85	CH_2Cl_2	+
12	$-(CH_2)_2O(CH_2)_2O(CH_2)_2-$[a]	58	75	CH_2Cl_2	+

[a] The macrocycles [11, 12] are reported in the ref. [13 a].



Scheme 3.

We concluded that the method B gave the macrocycles in good yields. The effects of solvent and the presence or absence of Et_3N in the yield of macrocycliztion process in the method A was investigated. Identical results were obtained in the presence or absence of Et_3N in all entries but good yields obtained in the absence of Et_3N for the entries that only took place by the method A.

Method A: In this method the cyclization reaction between diacid chloride and diamines were performed based on "high-concentration" method, with fast addition of a solution of diamine (0.002 mol) and Et_3N (0.004 mol, if it was necessary) in dry proper solvent (50 ml, CH_2Cl_2 or CH_3CN) into a solution of diacid chloride (0.002 mol) in the same solvent (50 ml) under vigorous stirring at 0 °C. In the absence of Et_3N the reaction took place at room temperature [12].

Method B: In this method the cyclization reaction between dimethyl ester (0.01 mol) and diamines (0.01 mol) was taken place in dry methanol (80 ml) at reflux for 18–24 h under ambient and usual conditions without using the high dilution-techniques (as it is mentioned in the text), (Scheme 3).

The results are summarized as following:

(1) The method B is a very simple, convenient and proper technique for the preparation of macrocyclic diamides in moderate to very good yields and can be taken place in quantitative scales (for example 1–10 gr).
(2) The method A is a very simple, convenient procedure for the preparation of macrocyclic diamides in moderate to good yields (in proper solvent). Both the very short time reaction (20–30 min) and the reaction with low reactive diamines such as aromatic diamines are as advantages for this method. The high yields can be resulted in the proper solvent but the low scale reaction (0.5 gr) is the disadvantage of this method.
(3) In the both methods, the more dry the solvents, the higher yields obtained.
(4) Using the method B leads to high yields for **9**, **11**, **12** and moderate yields for **6**, **7**, **8** while the method A give good yields for **6** and moderate yields for **7**, **8**, **9**, **11**, **12** and relatively good yields for **10** in applied solvent.
(5) In addition, the effect of salt and template effect are investigated for macro-cyclization reaction in the method B. In the presence of Na_2CO_3, K_2CO_3 and especially Cs_2CO_3 the yields were better and by-products were little. More investigations are being done about the "salt", "solvent" and "template" effects in our laboratoary.
(6) Based on ^1H NMR analysis of these macrocycles compared to dibenzosulfoxide macrocycles series in our previous work [13b], these series of macrocycles are more flexible. ^1H NMR analysis of these series proved that they are also relatively more flexible compared to smaller macrocycles. As a general result, the smaller the cavity, the more reduction in flexibility was observed. The important point found in this analysis is the rigidity and flexibility affect on the diastereotopic character of the protons in the cavity. The more rigid the cavity, the more diastereotopic the protons are. For example there isn't any diastereotopicity for the protons in the cavity of macrocycle **7**.

References

1. (a) J.S. Bradshaw, K.E. Krakowiak, and R.M. Izatt: *Tetrahedron* **48**, 4475 (1992); (b) K.J. Jankowski and D. Parker: In B.A. Murer (ed.), *Advances in Metals in Medicines*, Tai Press (1992); (c) D. Parker: *Chem. Soc. Rev.* **19**, 271 (1990); (d) J.S. Bradshaw, K.E. Krakowiak, and R.M. Izatt: *Aza-Crown Macrocycles in the Chemistry of Heterocyclic Compounds*, vol. 51, Wiley, New York (1993); (e) L.F. Lindoy: *Chemistry of Macrocyclic Ligand Complexes*, Cambridge Univ. Press, Cambridge (1989); (f) R.D. Hancock and A.E. Martell: *Chem. Rev.* **89**, 1875 (1989); (g) M.W. Hosseini, J.M. Lehn, S.R. Duff, K.Gu, and M.P. Mertes: *J. Org. Chem.* **52**, 1662 (1987); (h) X.X. Zhang, J.S. Bradshaw, and R.M. Izatt: *Chem. Rev.* **97**, 3313 (1997); (i) N. Voyer and B. Guerin: *Chem. Commun.* 2329 (1997); (j) N. Comatsu: *Org. Biomol. Chem.* **1**, 204 (2003); (k) B. Jousselme, P. Blanchard, E. Levillain, J. Delaunay, M. Allain, P. Richomme, D. Rondeau, N. Gallego-Planas, and J. Roncali: *J. Am. Chem. Soc.* **125**, 1363 (2003); (l) P. Tongraung, N. Chantarasiri, and T. Tuntulani: *Terahedron Lett.* **44**, 29 (2003); (m) K. Goto, Y. Yano, E. Okada, C.-W. Liu, K. Yamamoto, and R. Ueoka: *J. Org. Chem.* **68**, 865 (2003); (n) K.R. Adam, A.J. Leong, and L.F. Lindoy: *J. Chem. Soc. Dalton Trans.* **1**, 733 (1988).
2. (a) T.J. Collins, K.L. Kostka, E. Munck, and E.S. Uffelman: *J. Am. Chem. Soc.* **112**, 5637 (1990); (b) R.M. Izatt, J.S. Bradshaw, S.A. Nielsen, J.D. Lamb, J.J. Christensen, and D. Sen: *Chem. Rev.* **85**, 271 (1985); (c) J.S. Bradshaw, K.E. Krakowiak, B.J. Tarbet, R.L. Bruening, J.F. Biernat, M. Bochenska, R.M. Izatt, and J.J. Christensen: *Pure Appl. Chem.* **61**, 1619 (1989).
3. (a) I.O. Sutherland: *Chem. Soc. Rev.* **15**, 63 (1986); (b) R.M. Izatt, J.S. Bradshaw, K. Pawlak, R.L. Bruening, and B.J. Tarbet: *Chem. Rev.* **92**, 1261 (1992); (c) M.W. Hosseini and J.M. Lehn: : *Helv. Chim. Acta* **70**, 1312 (1987); (d) M.S. Goodman and S.D. Rose: *J. Org. Chem.* **57**, 3268 (1992).
4. (a) J.S. Bradshaw, K.E. Krakowiak, H. An, and R.M. Izatt: *J. Heterocyclic Chem.* **27**, 2113 (1990); (b) K.E. Krakowiak, J.S. Bradshw, and R.M. Izatt: *J. Org. Chem.* **55**, 3364 (1990); (c) J. Jurczak and D.T. Gryko: R. Van Edlik and C.D. Hubbard (eds.), *Organic Synthesis at High Pressure. In Chemistry under Extreme or Non Classical Conditions*, John Wiley & Sons, Inc. and Spektrum Akademisher Verlag, New York, Heidelberg, pp. 163–188 (1997); (d) R.A. Schultz, B.D. White, D.M. Dishong, K.A. Arnold, and G.W. Gokel: *J. Am. Chem. Soc.* **107**, 6659 (1985); (e) E. Graf and J.M. Lehn: *Helv. Chim. Acta* **64**, 1038 (1981); (f) E. Weber: *Kontakte* (Merck) 26 (1984).
5. (a) K.E. Krakowiak, J.S. Bradshaw, and D.J. Zamecka–Krakowiak: *Chem. Rev.* **89**, 929 (1989); (b) J. Jurczak and R. Ostaszewski: *J. Coord. Chem.* **27**, 201 (1992).
6. (a) J.S. Bradshaw, K.E. Krakowiak, and R.M. Izatt: *In Aza Crown Macrocycles*, John Wiley & Sons, New York (1993); (b) see ref. [1d]; (c) P. Knops, N. Sendhoff, H.B. Mekelburger, and F. Vogtle: *Top. Curr. Chem.* **161**, 1 (1992).
7. (a) E. Schwrtz, H.E. Gottlieb, F. Frolow, and A. Shanzer: *J. Org. Chem.* **50**, 5469 (1985); (b) A. Ninagawa, T. Maeda, and H. Matsuda: *Chem. Lett.* 1985 (1984); (c) N. Leygue, L. Cazaux, C. Picard, and P. Tisnes: *Terahedron Lett.* **28**, 4049 (1987).
8. (a) J. Jurczak and M. Pietraszkiewicz: *Top. Curr. Chem.* **130**, 183 (1985); (b) see ref. [4d].
9. (a) J. Jurczak, S. Kasprzyk, P. Salanski, and T. Stankiewicz: *High. Press. Res.* **11**, 139 (1992); (b) I. Tabushi, Y. Taniguchi, and H. Kato: *Terahedron Lett.* 1049 (1977); (c) D.T. Gryko, P. Piatek, and J. Jurczak: *Terahedron* **53**, 7957 (1997); (d) D.T. Gryko, P. Piatek,

P. Salanski, and J. Jurczak: *Terahedron Asymmetry* **9**, 1771 (1998); (e) J. Jurczak, S. Kasprzyk, P. Salanski, T. Stankiewicz: *J. Chem. Soc. Chem. Commun*. 956 (1991).

10. (a) Y. Nagao, T. Miyasaka, K. Seno, and E. Fujita: *Heterocycles* **15**, 1037 (1981); (b) Y. Nagao, K. Seno, T. Miyasaka, and E. Fujita: *Chem. Lett*. 159 (1980); (c) R.A. Bartsch, D.A. Babb, and B.E. Knudsen: *J. Incl. Phenom*. **5**, 515 (1987).
11. K.E. Krakowiak, J.S. Bradshaw, and R.M. Izatt: *Synlett* 611 (1992).
12. (a) H. Sharghi, Kh. Niknam, and A.R. Massah: *J. Heterocyclic Chem*. **36**, 601 (1999); (b) H. Sharghi, A.R. Massah, H. Eshghi, and Kh. Niknam: *J. Org. Chem*. **63**, 1455 (1998); (c) H. Sharghi and H. Eshghi: *Terahedron* **51**, 913 (1995).
13. (a) A. Shockravi, S. Bavili T., E. Rostami, A. Yousefi, A. Dehjurian, and R. Tohidi: *J. Incl. Phenom. Macrocyclic Chem.* **49**(1–2), 163 (2004); (b) A. Shockravi, E. Rostami, A. Dehjurian, R. Tohidi and S. Bavili T.: *Phosphorus, Sulfur and Silicon and The Related Elements*: **179**, 535 (2004); (c) A. Shockravi, S. Bavili T., *XXth International Conference on Organometallic Chemistry*, July 7–12, 2002, Corfu, Greece.
14. (a) A. Shockravi, R. Alizadeh, H. Aghabozorg, A. Moghimi, E. Rostami, and S. Bavili T.: *Phosphorus, Sulfur and Silicon and The Related Elements*: **178**, 2519 (2003); (b) E.M. Collins, M.A. McKerevey, E. Madigan, M.B. Moran, and M. Harries: *J. Chem. Soc. Perkin Trans*. **1**, 3137 (1991); (c) J. Byron, G.W. Gray, and R.C. Wilson: *J. Chem. Soc*. (C) 841 (1996); (d) S.E. Diamond, B. Grant, G.M. Tom, and H. Taube: *Tetrahedron Lett*. 4025 (1974); (e) P. Tecilla, S. Chang, A.D. Hamilton: *J. Am. Chem. Soc*. **112**, 9586 (1990); (f) A.S. Vogel: *Textbook of Practical Organic Chemistry,* Longman, New York (1986).