Efficient Synthesis of a Range of Benzosubstituted Macrocyclic Diamides

Sharghi, Hashem, Massah, Ahmad Reza and Niknam, Khodabakhsh*
Department of Chemistry, Shiraz University, Zip code 71454, Shiraz, I.R. Iran

ABSTRACT: *Some new macrocyclic dibenzotetraoxadiamides, tribenzotetraoxadiamides, tribenzopentaoxadiamide, tribenzohexaoxadiamide, and tetrabenzoheptaoxadiamide* ***15-22*** *have been prepared. These compounds were obtained in the macrocyclization step by reacting the diamines* ***6*** *and* ***7*** *with appropriate dicarboxylic acid dichlorides* ***8-14****. The cyclization does not require high dilution techniques or template effect and provides the expected dilactams in high yields ranging from 63% to 81%.*

KEY WORDS: *Macrocyclic diamides, Synthesis, Direct macrocyclization, Dicarboxylic acid dichloride*

INTRODUCTION

Macrocyclic ligands with amide units in the macro-ring are of interest because they often complex with metal ions where the metal ion has replaced the amide proton. Recently, the selectivity of macrocyclic diamides toward noble metals such as platinum (II), palladium (II), copper (II), nickel (II) and cobalt (II) is reported [1-3]. Up to now, only a few methods have been used to prepare these ligands [4-9]. *Vogtle* et al. [10,11] have synthesized some mixedheteroatom macrocyclic and macro-bicyclic molecules and prepared their complexes with various cations. Many macrocyclic aza-crown ethers and their corresponding amides find wide application in chemistry, biology, microanalysis, metal separation and molecular recognition [12-15]. However, they are very expensive owing to their difficult preparation, tedious purification, and in most cases very low yields [16-18].

In this paper synthetic routes towards new dilactam derivatives with 18-22 and 24 ring atoms is described. The target molecules were intended to include two, three and four benzo-condensed systems.

RESULT AND DISCUSION

The preparation of macrocyclic polylactam from diamines and activated diacids requires direct condensation reactions to form ring products in favour of polymers. In an attempt to achieve this goal, many different cyclization procedures have been developed. Early high-dilution techniques [19-22] have been complemented by various double-activation methods [23-26] and by a series of consecutive "zipper-type" reactions [27]. In most of these methods the macrocyclic lactams and polylactams are obtained by cyclization of a polyfunctional, linear precusor to a ring product.

* *To whom corresponding should be addressed.*
1021-9986/2000/1/6 7/$/2.70

The high dilution technique is, however, inconvenient as it requires a simultaneous addition of the diamine and diacid chloride to a large volume of solvent over an extended period of time.

An improvement of dilactam selectivity was observed in the synthesis of some polymethylene tetralactams using a diazasilolidine [28] derived from the diamine in order to take into account the template effect of the silicon atom. The yield of reaction seems to be sufficiently high for most practical purposes, and it appears to be slightly dependent on the size of the macrocycle. An inherent disadvantage of in situ syntheses is that a small excess of one or another of the organic reactants may lead to contamination of the required product with acyclic impurities.

Recently, we reported an efficient procedure to synthesize macrocyclic diamides by the reaction of 1,7-bis(2'-benzoyl chloride)-1,4,7- trioxaheptane with various diamines [29] and 1,5-bis(*o*-aminophenoxy)-3-oxapentane with different dicarboxylic acid dichlorides [30] in proper solvents under vigorous stirring with fast addition of starting materials.

To the study of the effect of ring size and conformation of the macrocycle on the cyclization step, macrocycles **15-22** are prepared by reacting the diamines **6,7** with appropirate dicarboxylic acid dichlorides **8-14** in proper solvent.

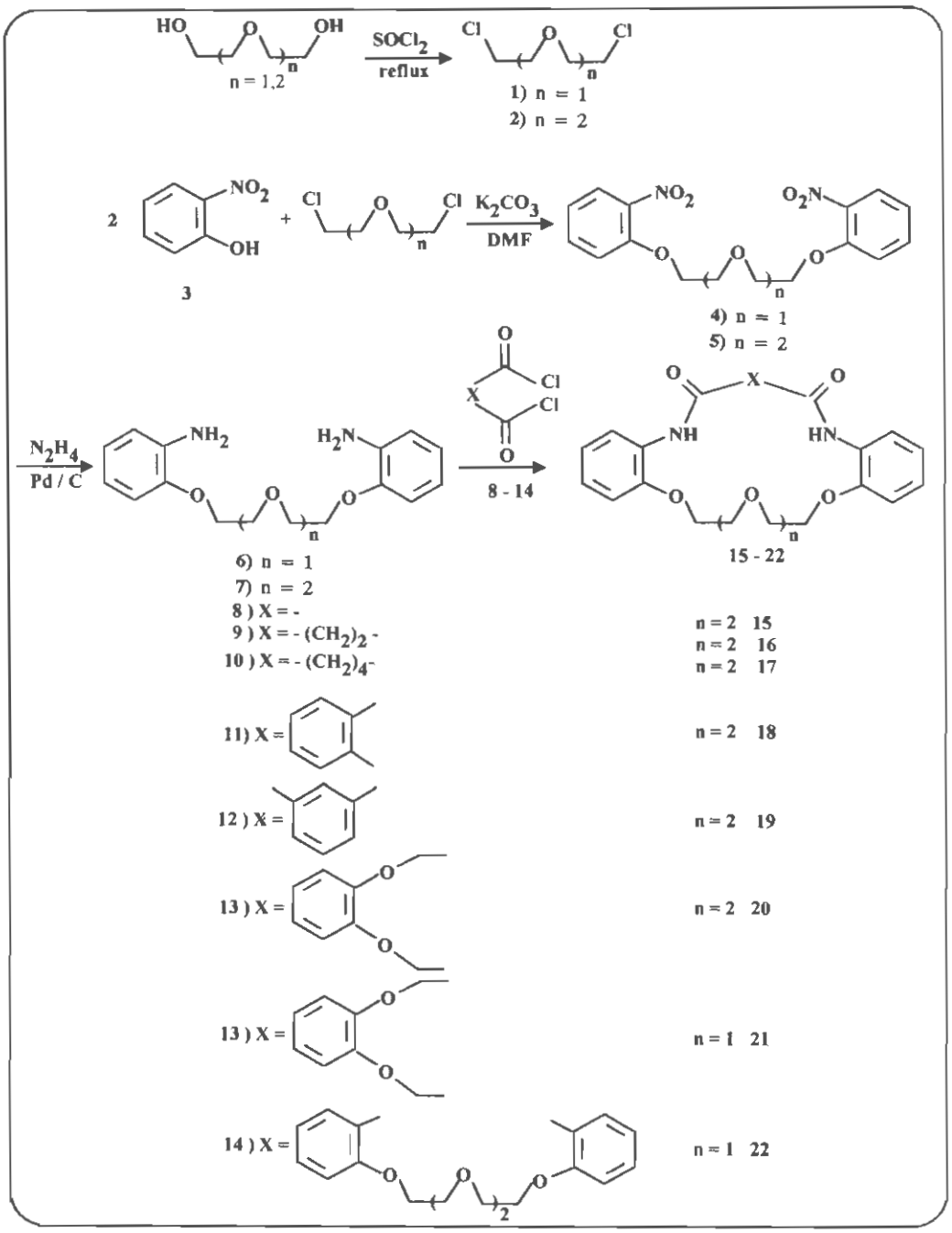
As shown in Scheme 1, *o*-nitrophenol was reacted with the dichlorides of olygoethylene glycols in the presence of potassium caronate in DMF to give the corresponding dinitro compounds **4** and **5** in 77% and 85% yields respectively. The dinitro compounds **4** and **5** were reduced by palladium on carbon with hydrazine into corresponding diamines **6** and **7** in 94% and 93% yields respectively.

The cyclization reactions between diamines **6** , **7** and dicarboxcylic acid dichlorides **8-14** were performed without the use of high-dilution techniques. The effect of a few solvents ($CHCl_3$, CH_3CN, CH_2Cl_2, C_6H_6 , acetone) on the yield of the macrocyclization reaction was investigated and CH_2Cl_2 was choosen as a suitable solvent for these macrocyclization reactions. In addition, cyclization reaction was carried out with fast addition of a mixture of diamine (2 mmol) in CH_2Cl_2 (10 mL) into a solution of dicarboxylic acid dichloride (2 mmol) in CH_2Cl_2 (10 mL) over 5 seconds with vigorous stirring at room temperature. The reaction mixture was stirred for further 20 min. to give dilactams **15-22** in high yields (Scheme 1). When this method was used at 0°C in the presence of triethylamine (4 mmol), the same result was obtained.

Compounds **15-20** were prepared from the reaction of diamino compound **7** with several types of aliphatic and aromatic dicarboxylic acid dichlorides **8-13** in high yields. The reaction of diamino compound **6** with 1,2-phenylene dioxydiacetyl chloride **13** [31] and 1,10-bis(2'-benzoyl chloride)-1,4,7,10-tetraoxadecane **14** [32] in dry CH_2Cl_2 gave macrocyclic diamides **21** and **22** in 81% and 69% yields respectively. It is important to mention that the reaction of *o*-phthaloyl dichloride **11** with diamine **7** in dry CH_2Cl_2 gave macrocyclic diamide **18**, but with diamino compound **6** gave a mixture of products and unreacted starting materials [30]. The structures proposed for the macrocyclic compounds are consistent with data derived from infrared and proton magnetic resonance spectra in addition to satisfactory combustion analysis and molecular weights determined by mass spectrometric analysis.

These results clearly indicate that the cyclization of different dicarboxylic acid dichlorides with diamines does not need high-dilution techniques or template effect and is independent on the size of macrocycles. In the present method, one question remains about the effect of vigorous stirring and fast addition of reactants on the macrocyclization step. This was addressed by performing reactions under normal stirring and/or low addition of reactants, the yield of cyclization reaction was decreased. All the cyclization reactions described in present study proceed efficiently by using fast addition method and the yields are improved by application of high speed stirring and fast addition of reactants, which is accompanied with evolution of hydrogen chloride.

The advantages of this method are as follows: (i) synthetic versatility; by this method even or odd membered dilactams, independent on the ring size could be prepared; (ii) high yields of cyclization without the need for high dilution technique or temp-



Scheme 1

late effects; (iii) ease of purification; (iv) low reaction time.

EXPERIMENTAL

NMR spectra were recorded on a Bruker Avance DPX-250 in $CDCl_3$. IR spectra were run on an Impact 400 D Nickolet FT-IR spectrophotometer. Mass spectra were recorded on a Shimadzu GCMS-QP 1000 EX instrument at 70 ev. UV spectra were recorded on a Philips PVB 700. Thin layer chromatography (TLC) were performed with silica gel polygram SILG/ UV 254 plates. Elemental analyses were performed at the

Research Center of National Oil Co. of Iran, Tehran. Column chromatography was carried out on short columns of silica gel 60 (230-400 mesh) in glass columns (2-3 cm diameter) using 15-30 g of silica gel per 1 g of crude mixture.Melting points were determined in open capillary tubes in a Buchi-510 circulating oil melting point apparatus and are uncorrected. Solvents, reagents and chemical materials were purchased from Merck and Fluka and were used without further purification. Compounds **13** [31] and **14** [32] were prepared according to their published procedures.

General Procedure for the Preparation of Dinitro Compounds 4,5

o-Nitrophenol (0.1 mol) and potassium carbonate (14 g) was mixed and stirred in DMF (100 mL) for 2 h. Diethyleneglycol or triethyleneglycol dichloride (0.05 mol) was added dropwise during 4 h and the mixture was refluxed for three days. After cooling, the mixture was poured into ice, and the precipitate was filtered off. The precipitate was washed by distilled water, and recrystallized from methanol to give corresponding dinitro compound.

1,5-Bis(o-nitrophenoxy)-3-oxapentane (4)

White solid, yield 77%, m.p.= 67-69°C (lit. [29], 69°C); IR(KBr): 3070(w), 1610(s), 1590(s) cm^{-1}; ^1H-NMR($CDCl_3$, 250 MHz): δ 3.98(t, 4H, J= 4.5 Hz), 4.26(t, 4H, J= 4.5 Hz), 7.01(m, 2H), 7.11(m, 2H), 7.49(m, 2H), 7.77(m, 2H).

1,8-Bis(o-nitrophenoxy)-3,6-dioxaoctane (5)

White cream solid, yield 85%, m.p.= 64-66°C; IR(KBr): 3070(m), 1610(s), 1590(s) cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz): δ 3.70(s, 4H), 3.87(t, 4H, J= 4.7 Hz), 4.22(t, 4H, 4.7 Hz), 6.98(m, 2H), 7.09(m, 2H), 7.45(m, 2H), 7.76(m, 2H).

General Procedure for the Preparation of Diamino Compounds 6,7

In a two necked round-bottom flask (250 mL) equipped with a reflux condenser and a dropping funnel, a suspension of dinitro compound 4 or **5** (0.012 mol), Palladium on carbon (5%, 0.4 g) and absolute ethanol (200 mL) was poured. The mixture was warmed and while being stirred magnetically, hydrazine hydrate 80% (10 mL) in ethanol (20 mL) was added dropwise over 90 min while maintaining the temperature at about 50°C. The reaction mixture was refluxed for 2 h and filtered hot. On cooling the filtrate gave the corresponding diamino compound after vacuum dried.

1,5-Bis(o-aminophenoxy)-3-oxapentane (6)

White crystals, yield 94%, m.p.= 65-66°C (lit. [29], 63-65°C); IR(KBr): 3450(s), 3360(s), 1605(s), 1510(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.58(s, 4H), 3.88(t, 4H, J= 4.6 Hz), 4.12(t, 4H, J= 4.6 Hz), 6.59-6.78(m, 8H).

1,8-Bis(o-aminophenoxy)-3,6-dioxaoctane (7)

White crystals, yield 93%, m.p= 48-50°C; IR(KBr): 3470(s), 3370(s), 1608(m), 1510(m) cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz): δ 3.67(s, 4H), 3.77(s, 4H), 3.85 (t, 4H, J= 4.5 Hz), 4.2(t, 4H, J= 4.5 Hz), 6.54-6.77 (m, 8H).

General Procedure for the Synthesis of Macrocyclic Diamides 15-22

A solution of diamine (2 mmol) in dry CH_2Cl_2 (50 mL) was added quickly to a vigorously stirring solution of diacid chloride (2 mmol) in dry CH_2Cl_2 (50 mL) at room temperature. The reaction mixture was stirred for a further 20 min. and then was washed with bicarbonate solution (2×50 mL) and water (2×50 mL). The organic layer was dried over $MgSO_4$ and the solvent was evaporated to give a solid product. The crude product was purified by column chromatography using petroleum ether (b.p.= 60-80°C)-ethyl acetate as eluent.

1,16-Diaza-2,3; 14,15- dibenzo- 4,7,10,13- tetraoxacyclooctadecane-17,18-dione (15)

White solid; yield 73%; m.p.= 163-165°C; R_f = 0.23(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 670(m), 745 (m), 860(m), 940(s), 1060(s), 1260(s), 1298(s), 1450 (s), 1528(m), 1602(m), 1690 (m), 1773(m), 2860 (m), 2955(m) 3070(w), 3340(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.65(s, 4H), 3.76(t, 4H, J= 4.5 Hz), 4.23(t, 4H, J= 4.5 Hz), 6.75-7.35(m, 8H), 8.37(b,

2H); UV(dioxane); $\lambda(\varepsilon_{max})$; 240.8(4450), 279(9050), 278 (9500). Anal. Calcd for $C_{20}H_{22}N_2O_6$: C, 62.18; H, 5.70; N, 7.25. Found: C, 62.2; H, 5.7; N, 7.2. MS; m/z(%): 387(M^++1, 7.9), 386(M^+, 37), 358(8.6), 343 (1.6), 298(2.0), 120(100), 109(66.6), 93(14.3).

1,16-Diaza- 2,3; 14,15- dibenzo-4,7,10,13- tetraoxa-cycloeicosane-17,20-dione(16)

White solid; yield 69%; m.p.= 183-185°C; R_f = 0.25(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 749(m), 860 (m), 940(s), 1060(s), 1118(s), 1260(s), 1454(s), 1528 (m), 1600(m), 1662(m), 2860 (m), 2925(m), 3070(w), 3295(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 2.69(s, 4H), 3.62(s, 4H), 3.75(t, 4H, J= 4.5 Hz), 4.06(t, 4H, J= 4.5 Hz), 6.72-6.98(m, 6H), 8.17(d, 2H, J= 7.5 Hz), 8.36(b, 2H); ^{13}C NMR($CDCl_3$, 62.9 MHz): δ 35.25, 69.36, 59.54, 69.80, 113.21, 121.18, 122.52, 124.29, 129.01, 147.69, 170.16; UV(dioxane); $\lambda(\varepsilon_{max})$; 239(2800), 257(8900), 286 (4400). Anal. Calcd for $C_{22}H_{26}N_2O_6$: C, 63.77; H, 6.28; N, 6.76. Found: C, 63.7; H, 6.2; N, 6.8. MS; m/z(%): 414(M^+, 48.5), 358(2.9), 326(9.9), 120(39.7), 109(51.1), 93(12), 55(100).

1,16-Diaza-2,3; 14,15- dibenzo-4,7,10,13- tetraoxa-cyclodicosane-17,22-dione (17)

White solid; yield 75%; m.p.= 183-185°C; R_f = 0.39(CH_2Cl_2 -CH_3OH/96-4); IR(KBr): 749(m), 940 (w), 1060(m), 1119(s), 1260(s), 1454(s), 1528(s), 1598 (m), 1663(s), 2860(m), 2925(m), 3070(w), 3294(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 1.85(t, 4H, J= 4.8 Hz), 2.35 (t, 4H, J= 4.8 Hz), 3.62(s, 4H), 3.75 (t, 4H, J= 4.5 Hz), 4.06(t, 4H, J= 4.5 Hz), 6.72-6.98 (m, 6H), 8.17 (d, 2H, J=7.5 Hz), 8.25(b, 2H); ^{13}C NMR ($CDCl_3$, 62.9 MHz): δ 24.92, 37.18, 69.84, 70.09, 70.78, 114.08, 120.80, 122.89, 124.03, 129.50, 147.45, 171.19; UV(dioxane); $\lambda(\varepsilon_{max})$; 263(21000), 276 (27000). Anal. Calcd for $C_{24}H_{30}N_2O_6$: C, 65.16; H, 6.79; N, 6.33. Found: C, 65.2; H, 6.8; N, 6.3. MS; m/z(%): 442(M^+, 31.9), 386(1.8), 354(9.5), 120(31.7), 109(43.2), 93(12.6), 55(100).

1,16-Diaza-2,3; 14,15; 18,19- tribenzo- 4,7,10,13-tetraoxacycloeicosane-17,20-dione (18)

White solid; yield 65%; m.p.= 197-199°C; R_f = 0.63(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 735 (m), 940 (s), 1060(s), 1110(s), 1225(w), 1260(s), 1450(s), 1522 (s), 1600(m), 1669(s), 2860(m), 2925(m), 3070(w), 3275(s) cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz): δ 3.65(s, 4H), 3.76(t, 4H, J= 4.5 Hz), 4.23(t, 4H, J= 4.5 Hz), 6.75 - 7.35(m, 10H), 7.76(m, 2H), 8.37(b, 2H); ^{13}C-NMR($CDCl_3$,62.9 MHz): δ 69.37,69.47, 69.73, 112.47, 122.25, 123.40, 124.53, 125.26, 128.70, 129.69, 132.23, 135.72, 149.06, 165.81; UV (dioxane); $\lambda(\varepsilon_{max})$; 255 (20500), 278.9(25000). Anal. Calcd for $C_{26}H_{26}N_2O_6$: C, 67.53; H, 5.63; N, 6.06. Found: C, 67.6; H, 5.6; N, 6.1. MS; m/z(%): 463(M^++1, 12.4), 462(M^+, 41.3), 104(100), 76(41.5).

1,16-Diaza- 2,3; 14,15; 18,20-tribenzo- 4,7,10,13-tetraoxacyclomono-eicosane-17,20-dione (19)

White solid; yield 63%; R_f = 0.63(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 750(s), 950(s), 1070(s), 1120 (s), 1220(w), 1260(s), 1455(s), 1490(m), 1540(s), 1600 (m), 1670(s), 2880 (m), 2960(m), 3070(w), 3300(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.65(s, 4H), 3.8 (t, 4H, J= 4.5 Hz), 4.13(t, 4H, J= 4.5 Hz), 6.5-7.15 (m, 7H), 7.5-8.5(m, 5H), 8.73(b, 2H); UV(dioxane): $\lambda(\varepsilon_{max})$; 235(2700), 255(10500), 294 (14700). Anal. Calcd for $C_{26}H_{26}N_2O_6$: C, 67.53; H, 5.63; N, 6.06. Found: C, 67.6; H, 5.6; N, 6.1. MS; m/z(%): 463(M^+ +1, 17.1), 462(M^+, 47.6), 419(9.8), 374(33), 104 (100), 76(77.5).

1,16-Diaza-2,3; 14,15; 20,21- tribenzo- 4,7,10,13, 19,22-hexaoxacyclotetraeicosane-17,24-dione (20)

White solid; yield 79%; m.p.= 155-157°C; R_f = 0.69(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 735(m), 1051 (m), 1132(m), 1214(m), 1259(s), 1457(m), 1485(m), 1535(s), 1559(m), 1689 (s), 2885(m), 2932(m), 3059 (w), 3267(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.49 (s, 4H), 3.65(t, 4H, J= 4.5 Hz), 4.05(t, 4H, J= 4.5 Hz), 4.60(s, 4H), 6.76(m, 2H), 6.87-6.99(m, 8H), 8.29(m, 2H), 9.07(b, 2H); ^{13}C NMR($CDCl_3$, 62.9 MHz): δ 69.90, 69.96, 71.12, 71.62, 112.71, 117.66, 120.87, 122.10, 124.28, 124.28, 124.87, 127.86, 148.09, 149.06, 166.87; UV(chloroform); $\lambda(\varepsilon_{max})$: 262(5619); Anal. Calcd for $C_{28}H_{30}N_2O_8$: C, 64.36; H, 5.79; N, 5.36. Found: C, 64.4; H, 5.8; N, 5.3. MS; m/z(%): 523 (M^++1, 9.9), 522(M^+, 25.3), 366(3.3), 156(24.2), 154

(47.1), 120(62.2), 109(27.5), 93(17.1), 45(100).

1,13-Diaza-2,3; 11, 12; 17,18- tribenzo-4,7,10,16, 19- pentaoxacyclouneicosane- 14,21-dione (21)

White solid; yield 81%; m.p.= 183-185°C; R_f= 0.77(CH_2Cl_2 - CH_3OH/96-4); IR(KBr): 744(m), 930 (w), 1033(w), 1050(w), 1112(m), 1133(w), 1210(w), 1253(m), 1287(w), 1337 (w), 1448(m), 1489(m), 1532 (s), 1600(m), 1682(s), 2883(m), 2910(m), 3030(w), 3382(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.65(t, 4H, J= 4.5 Hz), 3.98(t, 4H, J= 4.5 Hz), 4.53(s, 4H), 6.72(m, 2H), 6.80-6.90(m, 8H), 8.23(m, 2H), 9.02(b, 2H); ^{13}C NMR($CDCl_3$, 62.9 MHz): δ 69.52, 69.95, 70.29, 112.97, 116.35, 120.21, 122.21, 123.93, 124.71, 127.98, 147.63, 148.32, 166.59; UV(chloroform); $\lambda(\varepsilon_{max})$; 264(4907). Anal. Calcd for $C_{26}H_{26}N_2O_7$: C, 65.26; H, 5.48; N, 5.85. Found: C, 65.1; H, 5.5; N, 5.8. MS; m/z(%): 480(M^++2, 2.6), 479(M^++1, 10), 478 (M^+, 28.9), 435(12.6), 154(10.5), 148(75), 135(67.5), 120(100), 109(57.8), 93(11.5), 77(11.3).

14,23-Diaza-11,12; 15,16; 24,25; 28,29-tetrabenzo-1,4,7,10,20,23-hexaoxacyclononacosane-13,27-dione (22)

White solid; yield 69%; m.p.= 137-139°C; R_f = 0.75(CH_2Cl_2 - CH_3OH/95-5); IR(KBr): 749(m), 1051 (m), 1132(m), 1302(m), 1454 (m), 1598(s), 1655(s), 2871(s), 3083(w), 3328(s) cm^{-1}; ^1H NMR($CDCl_3$, 250 MHz): δ 3.53(s, 4H), 3.84(t, 4H, J= 4.7 Hz), 3.91(t, 4H, J= 4.7 Hz), 4.24(t, 4H, J= 4.7 Hz), 4.32(t, 4H, J= 4.7 Hz), 6.83-7.02(m, 8H), 7.13(t, 2H, J= 7.5 Hz), 7.43(t, 2H, J= 7.7 Hz), 8.27 (d, 2H, J= 7.7 Hz), 8.70 (d, 2H, J= 7.5 Hz), 10.22(s, 2H); ^{13}C NMR($CDCl_3$, 62.9 MHz): δ 68.77, 69.37, 69.98, 71.22, 112.56, 114.09, 122.06, 122.25, 123.53, 124.40, 129.12, 132.91, 133.34, 148.52, 156.84, 163.76. Anal. Calcd for $C_{36}H_{38}N_2O_9$: C, 67.28; H, 5.96; N, 4.36. Found: C, 67.2; H, 6.0; N, 4.4. MS; m/z(%): 643(M^++1, 2.4), 642(M^+, 6.2), 508(2.8), 238(22.5), 147(43.1), 121 (100), 104(62.6), 76(57.9).

ACKNOWLEDGMENT

We are grateful to the Research Council of Shiraz University for financial support, grant No. 76-SC-1038-614.

Received, 21st February 1999 ; Accepted, 3rd October 1999

REFERENCES

[1] Kimura, E., Kurogi, Y., Wada, S. and Shionoya, M., *Chem. Commun.,* 781(1989).

[2] Kimura, E., *Pure & Appl. Chem.,* **61,** 823(1989).

[3] Collins, T. J., Kostka, K. L., Munck, E. and Uffelman, E. S., *J. Am. Chem. Soc.,* **112,** 5637 (1990).

[4] Tabushi, I., Taniguchi, Y. and Kato, H., *Tetrahedron Letters,* 1049(1977).

[5] Tabushi, I., Okino, H. and Kuroda, Y., *Tetrahedron Letters,* 4339(1976).

[6] Dietrich, B., Lehn, J. M., Sauvage, J. P. and Blanzat, J., *Tetrahedron,* **29,** 1629(1973).

[7] Petranek, J. and Ryba, O., *Collect. Czech. Chem. Commun.,* **48,** 1945((1983).

[8] Krakowiak, K. E., Bradshaw, J. S., An, H. and Izatt, R. M., *J. Heterocyclic Chem.,* **27,** 2113 (1990).

[9] Bradshaw, J. S., Krakowiak, K. E. and Izatt, R. M., *J. Org. Chem.,* **55,** 3364(1990).

[10] Vogtle, F. and Neumann, P., *Tetrahedron,* **26,** 5847(1970).

[11] Vogtle, F., Oepen, G. and Frensch, K., *Justus Liebigs Ann. Chem.,* 858(1979).

[12] Krakowiak, K. E., Bradshaw, J. S. and Zamecka-Krakowiak, D., *J. Chem. Rev.,* **89,** 929(1989).

[13] Bradshaw, J. S., Krakowiak, K. E. and Izatt, R. M., *Tetrahedron,* **48,** 4475(1992).

[14] Dudler, V., Lindoy, L. F., Saltin, D. and Schlaepfer, C. W., *Aust. J. Chem.,* **40,** 1557(1987).

[15] Adam, K. R., Leong, A. J. and Lindoy, L. F., *J. Chem. Soc. Dalton Trans.,* 1733(1988).

[16] Biernat, J. F. and Luboch, E., *Tetrahedron,* **40,** 1927(1984).

[17] Krakowiak, K. E., Bradshaw, J. S. and Izatt, R. M., *J. Heterocyclic Chem.,* **27,** 1585(1990).

[18] Nagao, Y., Seno, K., Miyasaka, T. and Fujita, E., *Chemistry Letters,* 159(1980).

[19] Ruzicka, L., Salomon, G. and Meyer, K. E., *Helv. Chim. Acta,* **20,** 109(1937).

[20] Stetter, H., Marx, J., *Ann.,* **607,** 519(1957).

[21] Pellisard, D. and Louis, R., *Tetrahedron Letters,* 4589(1972).

[22] Overman, L. E., *J. Org. Chem.*, **37**, 4214(1972).
[23] Masamune, S., Hayase, Y., Schilling, W., Chan, W. K. and Bates, G. S., *J. Am. Chem. Soc.*, **99**, 6756 (1977).
[24] Collum, D. B., Chen, S. C. and Ganem, B., *J. Org. Chem.*, **43**, 4393(1978).
[25] Ogura, H. and Takeda, K., *Heterocycles*, **15**, 467 (1981).
[26] Fujita, E., *Pure Appl. Chem.*, **53**, 1141(1981).
[27] Kramer, U., Goggisberg, A., Hesce, M. and Schmid, H., *Angew. Chem., Int. Ed. Engl.*, **16**, 861 (1977).
[28] Schwartz, E., Gottlieb, H. E., Frolow, F. and Shanzer, A., *J. Org. Chem.*, **50**, 5469(1985).
[29] Sharghi, H. and Eshghi, H., *Tetrahedron*, **51**, 913(1995).
[30] Sharghi, H. and Eshghi, H., *J. Sci., I. R. Iran*, **9**, 238(1998).
[31] Wang, X., Wei, T., Chen, J. and Li, J., *Synth. Commun.*, **26**, 2765(1996).
[32] Sharghi, H., Massah, A. R., Eshghi, H. and Niknam, Kh., *J. Org. Chem.*, **63**, 1455(1998).