decomposition of these compounds does not involve carbenium ion formation. The behavior of **2c** was not investigated.

Since some of the ester **3a** might have been formed by the decomposition of the carboxyinversion compound, the solvent and washings from the decomposition of **1** in slurries were examined by means of IR in the carbonyl region as soon as possible, about 5 min into the run. Bands from the peroxide and the esters were seen, but none from carboxyinversion compounds. Experiments with slurries containing **2a** and **2b** showed that bands from **2a** could still be seen at 5 min (but not at 12 min) and that bands from **2b** could still be seen at 19 min (but not at 39 min).

About 77% of the carbenium ions are trapped as the esters **3a–c**. Those that escape reaction with the *m*-chlorobenzoate gegenion react with surface water or silanol groups. Alcohols **4a–c** were recovered from the silica in two parts, first, from the solvent plus ether washing of the silica, and, second, after refluxing the silica with wet ether to hydrolyze any surface silyl ethers. The total yield of alcohols, 20.4%, is much less than the 49% trapped nucleophilically by acetonitrile when **1** is decomposed in that solvent.[4]

## Experimental Section

**Cyclobutanecarbonyl *m*-chlorobenzoyl peroxide** was prepared by the method of Taylor.[4] It was crystallized several times from hexane at −20 to −18 °C. IR ($CH_2Cl_2$): C═O stretch at 1800 and 1770 $cm^{-1}$, C—O at 1230 $cm^{-1}$. Iodometric titer 99.1%.

**Silica.** The silica was Fisher S-679, similar to that described previously,[8] dried overnight at 130 °C.

**Cyclobutyl *m*-Chlorobenzoyl Carbonate and Cyclopropylmethyl *m*-Chlorobenzoyl Carbonate.** The carbonic ester anhydrides **2a,b** have been prepared and characterized previously by Taylor et al.[4] To avoid the use of phosgene, we used a modification of the method used by Michejda and Tarbell in their synthesis of *p*-nitrobenzoic *tert*-butylcarbonic anhydride.[9]

**Cyclobutyl *m*-Chlorobenzoyl Carbonate.** To 197 mg of NaH (50% assay), washed twice with low-boiling petroleum ether to remove oil, was added 8 mL of THF distilled over benzophenone ketyl. Then 0.296 g of cyclobutanol was added. After the evolution of $H_2$ subsided, dry $CO_2$ was bubbled in for 10–15 min. The solution became warm and then cooled to give a gelatinous mixture. To this was added dropwise 718 mg of *m*-chlorobenzoyl chloride in THF, with stirring for 10 min. The solvent was removed on a rotary evaporator and replaced with low-boiling petroleum ether; this solution was then washed with water and the solvent again removed. The residual oil was dissolved in about 40 mL of low-boiling petroleum ether and cooled to −20 °C, and a white precipitate (*m*-chlorobenzoic acid) was discarded. The solvent was again removed, leaving an oil. IR ($CH_2Cl_2$): carbonyl 1810 $cm^{-1}$ (strong), 1755 (weaker), C–H 2990 (center, weak to moderate), 1590 (weak), 1435 (weak), 1270 (weak), 1210 (strong), 1140 (weak), 1065 (weak). NMR ($CDCl_3$): δ 2.6–2.0 (m, 6 H), 5.1 (quintet, 1 H), 8.0–7.2 (m, 4 H).

Cyclopropylmethyl *m*-chlorobenzoyl carbonate was prepared similarly. IR ($CH_2Cl_2$): 1810 $cm^{-1}$ (strong), 1755 (weaker). NMR ($CDCl_3$): 0.7–0.3 (m, 4 H), 1.3 (m, 1 H), 4.2 (d, 2 H), 8–7.2 (m, 4 H).

**Kinetics.** The peroxide was decomposed both in slurries and on the silica after removal of the solvent.

Aliquots for a typical slurry run were prepared by using 5.0 g of the silica, to which were added 10 mL of $CCl_4$, and then, with stirring, 4 mL of a 0.038 M $CCl_4$ solution of peroxide **1**. The timer was started midway in the addition of the peroxide. After 5, 8, or 10 min, 4 mL of saturated KI and 50 mL of carbonated glacial acetic acid were added, and the mixture was stirred for 2 min and heated on a steam bath for 3 min while adding pellets of dry ice. Then 50 mL of carbonated water was added and the sample titrated with thiosulfate.

For investigation of the behavior of the peroxide on silica in the absence of solvent, pentane was used to put the peroxide on the silica. After stirring the slurry for 2 min, the pentane was removed as quickly as possible (8 min) by means of a rotary evaporator. The flask containing the sample was then evacuated by a mechanical pump for 3 min with shaking. The sample was then transferred to a weighing bottle, weighed, and titrated as in the slurry experiments. The amount of peroxide found immediately after this procedure was taken as the initial concentration. Up to four later points were taken from similar experiments.

**Product Isolation and Analysis.** Products were isolated from runs in which the peroxide was decomposed in a $CCl_4$ slurry and then extracted with ether. The mixture of reaction products, $CCl_4$, and ether was then analyzed with a GC/MS, using a Supelcowax-10 capillary column. Authentic samples were run under the same conditions for comparison.

Additional amounts of the alcohols were isolated from the silica by refluxing it with a mixture of 10 mL of $H_2O$ and 50 mL of ether for 12 h and then extracting with ether and drying the extract over $Na_2SO_4$.

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# Novel One-Step Syntheses of Cryptands and Bis(aza-12-crown-4 ethers)

Krzysztof E. Krakowiak* and Jerald S. Bradshaw*

*Department of Chemistry, Brigham Young University, Provo, Utah 84602*

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A novel and convenient one-step method to prepare cryptands (**1–5**, see Scheme I) from readily available α,ω-diiodides and α,ω-diamines is reported herein. The cryptands are excellent complexing agents[1,2] so that a more convenient method for their syntheses is very important. Bis(aza-12-crown-4) compounds **6–8** composed of two identical aza-crowns connected by a di- or trioxaalkane and formed by the reaction of one α,ω-diiodide with each primary amine function of the starting α,ω-diamine (see Scheme I) were isolated as byproducts in some cases. The most convenient syntheses of the cryptands are cyclization reactions from two linear reactants. These cyclic reactions can be 1:1,[3–7] 2:1,[8–13] or 3:2[7,14–19] cyclizations between

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**Scheme I. New Cryptands and Bis(aza-crowns)**

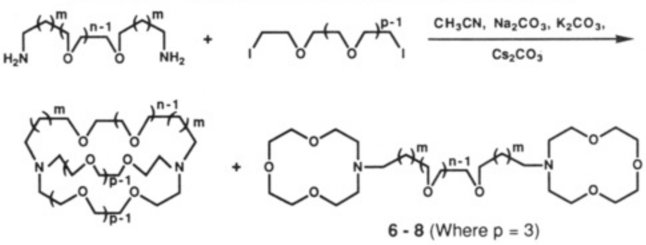

**Table I. New Cryptands and Bis(aza-crowns)**

| structure[a] | | | template | cryptand | bis-(aza-crown) | other experiments (% yield of |
|---|---|---|---|---|---|---|
| *m* | *n* | *p* | cation | (% yield) | (% yield) | cryptand) |
| 0 | 2 | 3 | $K^+$ | 1 (40) | 6 (17) | $Na^+$ (13)[b] |
| 1 | 2 | 2 | $Na^+$ | 2 (31) | not detected | $Na^+$ (50),[b] $K^+$ (42)[b] |
| 1 | 2 | 3 | $K^+$ | 3 (19) | 7 (16) | |
| 1 | 3 | 2 | $Na^+$ | 4 (34) | not detected | $Na^+$ (38),[b] $K^+$ (24) |
| 1 | 3 | 3 | $Cs^+$ | 5 (8) | 8 (26) | $K^+$ (6) |

[a] See Scheme I for structures. The values of *n* and *p* express the number of oxygen atoms in each bridge of the cryptand. [b] With use of high dilution techniques.

available linear starting materials. Recent 3:2 cyclizations[17-19] to form cryptands containing Schiff-bases are not universally viable because of the difficulty in reducing the Schiff-base moiety, especially where pyridine rings are part of the cryptand. Kulstad and Malmsten attempted to prepare cryptand[2.2.2] by a 2:1 cyclization of an aliphatic $\alpha,\omega$-diamine and an $\alpha,\omega$-diiodide but instead of [2.2.2], 4,13-diaza-18-crown-6 formed as the main product.[13,20]

There has been considerable success in the formation of cryptands containing rigid groups such as benzene or pyridine rings that help in the cyclization step. Cryptands containing these rigid units were formed in 20–35% yields but they were complexed with sodium or lithium iodide or bromide and required a decomplexation step to isolate the free cryptand.[7-10] Decomplexation was not straightforward and required transcomplexation by silver ions followed by treatment with hydrogen sulfide to precipitate silver sulfide and give the free cryptand.[8] A very attractive synthesis of a rigid cryptand containing a benzene subcyclic unit without complex formation was recently reported to occur in a 25% yield.[12]

Up to now, the most used method for the preparation of nonaromatic cryptands has been via the diaza-crown by a multistep process from diacid dichloride and diamine starting materials with overall yields of 20–25%.[21-23] We have found that cryptand[2.3.3] (**1**, see Scheme I) can be prepared in a good yield (see Table I) by the metal ion template cyclization reaction of two molecules of 1,11-diiodo-3,6,9-trioxaundecane and one molecule of 1,8-diamino-3,6-dioxaoctane. As reported,[13] a similar reaction with 1,8-diiodo-3,6-dioxaoctane to form cryptand[2.2.2] using sodium or potassium ions was not successful. Cryptand[2.2.2] forms strong internal complexes with sodium and potassium ions. Perhaps the large iodide groups interfere in the ring closure step of the forming metal ion–cryptand complex.

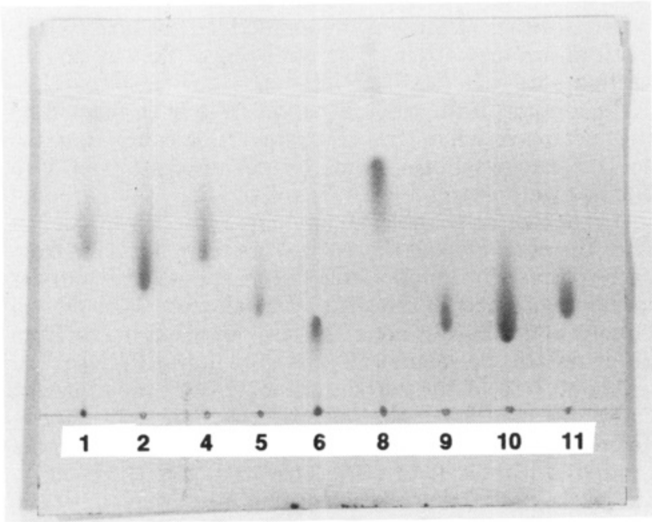

**Figure 1.** Silica gel TLC analysis of cryptands **1**, **2**, **4**, **5**, **9** ([2.2.1]), **10** ([2.2.2]), **11** (benzo[2.2.2]), and bis(aza-12-crown-4 ethers) **6** and **8** using methanol/30% aqueous ammonium hydroxide (5/1) as eluant.

As shown in Table I, the template-catalyzed 2:1 cyclization reaction is applicable for the preparation of a variety of cryptands. Thus, in addition to cryptand[2.3.3], other cryptands each containing two propylene bridges in one arm ($m = 1$, denoted by $Pr_2$) were prepared in the one-step process. These bispropylene-containing cryptands include [2.2.2$Pr_2$] (**2**), [2$Pr_2$.3.3] (**3**), [2.2.3$Pr_2$] (**4**), and [3.3.3$Pr_2$] (**5**). Where the diiodide derivative of tetraethylene glycol ($p = 3$) was used, bis(aza-12-crown-4) byproducts **6**–**8** were also isolated. This material was the major product when the large cavity cryptand [3$Pr_2$.3.3] (**5**) was prepared. These byproduct bis(aza-crowns) were not formed where $p = 2$ because of the difficulty in closing nine-membered rings.

The reactions were carried out in 25 mL of refluxing acetonitrile using 0.01 mol of diamine and 0.022 mol of diiodide in the presence of an excess of solid alkali metal carbonate for 48 h. We observed that under high dilution conditions, greater amounts of the bis(aza-12-crown-4) byproducts **6**–**8** were formed with a lesser amount of cryptands **1**, **3**, and **5**. Indeed, no cryptand **5** was observed in high dilution conditions. The products were isolated by alumina chromatography using mixtures of THF and ethanol as eluants. Cryptands **1**, **2**, and **4** were obtained in 38–50% yields, while the larger cryptands were obtained in lower yields. Metal template ions other than the alkali metal ions were not used.

The cryptands and bis(aza-crowns) have the same molecular formulas and cannot be distinguished by their molecular weight or their $^1H$ NMR and IR spectra. Bis(aza-crown) **8** was proved by its independent synthesis from two molecules of *N*-(3-hydroxypropyl)aza-12-crown-4 with one molecule of diethylene glycol ditosylate. The structure of cryptand **2** in the solid state was determined from an X-ray structure analysis.[24] The cryptands and bis(aza-crowns) can be distinguished by their $^{13}C$ NMR spectra. The peak attributable to the carbon of the propylene moiety next to the bridgehead nitrogen is at $\delta$ 51.8 ± 0.2 for cryptands **2**–**5**, whereas that peak shifts downfield

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**Table II. $^{13}C$ NMR Data for New Cryptands and Bis(aza-crowns)**

| compd | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| $NCH_2CH_2O$ | 55.0, 55.3 | 55.2 | 55.0 | 54.99 | 54.44 | 55.72, 56.16 | 55.37 | 55.3 |
| $NCH_2CH_2CH_2O$ | | 51.8 | 51.6 | 51.98 | 51.94 | | 53.92 | 54.0 |
| $NCH_2CH_2CH_2$ | | 28.1 | 28.2 | 27.97 | 27.90 | | 27.63 | 27.8 |

to δ 53.9 ± 0.1 for bis(aza-crowns) 7 and 8 (see Table II). Similar but much smaller shifts were observed for the peaks attributable to the carbons of the ethylene moieties next to the bridgehead nitrogen.

An interesting method to distinguish the two products is provided by silica gel TLC analysis (methanol/30% aqueous ammonium hydroxide = 5/1) after purification by alumina chromatography. The cryptand TLC spot has the unusual property of considerable fronting while the TLC spot for the bis(aza-crown) has the usual tailing (see Figure 1). The unusual TLC spot was also observed for commercially available cryptands [2.2.1], [2.2.2], and benzo[2.2.2] as shown in Figure 1. It is important to note that these unusual TLC properties are mainly observed where methanol/30% aqueous ammonium hydroxide is used as the eluant. It is likely that the cryptands are leaching trace metal ions from the silica gel and the resulting complex moves at a faster rate to produce fronting. Indeed, sodium and potassium complexes of the cryptands move faster on silica gel TLC under these conditions ($R_f$ = 0.95 for the complex vs 0.1 for the cryptand).

## Conclusion

This new synthetic method allows an inexpensive one-step synthesis of a variety of cryptands from commercially available α,ω-diamine and α,ω-diiodide starting materials. Bis(aza-12-crown-4) compounds, formed by a cyclization reaction on each primary amine group of the diamine starting material, are the major byproducts. These two products can be distinguished by their $^{13}C$ NMR spectra and by their TLC spots using silica gel TLC with methanol/30% aqueous ammonium hydroxide as the eluant.

## Experimental Section

Proton and carbon NMR spectra were obtained at 200 MHz in deuteriochloroform. Molecular weights were determined by electron-impact HRMS. Starting amines were used as received from Fluka, Aldrich, or Tokkyo-Kassei Chemical Cos. 1,11-Diiodo-3,6,9-trioxaundecane was prepared from the corresponding dichloride.[25] Silica gel $60_{F254}$ and alumina $69_{F254}$ (neutral, Type E) plates (Merck) were used for TLC. Similar material without the fluorescence indicator was used for column chromatography. Compound detection on the TLC plates was obtained by iodine vapor or 5% phosphomolybdic acid in ethanol followed by drying.

**General Procedure for the Preparation and Separation of Cryptands and Bis(aza-crowns).** The diiodocompound (10.5 mmol) was slowly added to a stirred mixture of 10 mmol of the appropriate diamine and 20 g of anhydrous metal carbonate (see Table I for the metal ion) in 300–350 mL of $CH_3CN$. The stirred mixture was refluxed for 24-36 h. The mixture was cooled, filtered, and evaporated, and the residue was dissolved in 20 mL of water. The aqueous phase was extracted three times with 20-mL portions of spectrograde $CH_2Cl_2$. The aqueous layer was saturated with NaCl before the third extraction. The combined $CH_2Cl_2$ solutions were evaporated and the resulting mixture was titurated three times with dry $CH_2Cl_2$. The combined $CH_2Cl_2$ solutions were filtered and evaporated and the solid residue was chromatographed on alumina (THF followed by THF/ethanol = 100/1) to give the products. When the products were not clearly separated or contained other impurities, they were chromatographed a second time in silica gel ($CH_3OH$/30% $NH_4OH$ = 20/1). The products and yields are listed in Table I. Selected carbon NMR data are given in Table II. Proton NMR spectral data and analyses for all products are as follows.

**Cryptand[2.3.3] (1):** $^1H$ NMR δ 2.85 (t, 12 H), 3.65 (m, 32 H). Anal. Calcd for $C_{22}H_{44}N_2O_8$: C, 56.91; H, 9.54. Found: C, 56.56; H, 9.62; MS m/e 464.

**Cryptand[2.2.2Pr$_2$] (2):**[24] mp 55–56 °C (isopropyl ether or acetonitrile/hexane); $^1H$ NMR δ 1.75 (m, 4 H), 2.65 (m, 12 H), 3.75 (m, 24 H); MS *m/e* 404.

**Cryptand[2Pr$_2$.3.3] (3):** $^1H$ NMR δ 1.75 (m, 4 H), 2.7 (m, 12 H), 3.6 (m, 32 H); MS *m/e* 492. Anal. Calcd for $C_{24}H_{48}N_2O_8$: C, 58.51; H, 9.82. Found: C, 58.36; H, 9.67.

**Cryptand[2.2.3Pr$_2$] (4):** $^1H$ NMR δ 1.7 (m, 4 H), 2.6 (t, 4 H), 2.7 (m, 8 H), 3.6 (m, 28 H); MS *m/e* 448. Anal. Calcd for $C_{22}H_{44}N_2O_7$: C, 58.90; H, 9.89. Found: C, 58.51; H, 9.64.

**Cryptand[3.3.3Pr$_2$] (5):** $^1H$ NMR δ 1.7 (m, 4 H), 1.9 (b, 2 H), 2.7 (m, 12 H), 3.6 (m, 36 H); MS *m/e* 536. Anal. Calcd for $C_{26}H_{52}N_2O_9 \cdot H_2O$: C, 56.30; H, 9.81. Found: C, 56.45, H, 9.56.

**Bis-crown 6:** $^1H$ NMR δ 2.8 (m, 12 H), 3.65 (m, 32 H); MS *m/e* 464. Anal. Calcd for $C_{22}H_{44}N_2O_8$: C, 56.91; H, 9.54. Found: C, 56.74; H, 9.63.

**Bis-crown 7:** $^1H$ NMR δ 1.8 (m, 4 H), 2.6 (t, 4 H), 2.7 (m, 8 H), 3.6 (m, 32 H); MS *m/e* 492. Anal. Calcd for $C_{24}H_{48}N_2O_8$: C, 58.51; H, 9.82. Found: C, 58.40; H, 9.64.

**Bis-crown 8:** $^1H$ NMR δ 1.75 (m, 4 H), 2.55 (t, 4 H), 2.7 (t, 8 H), 3.6 (m, 36 H); MS *m/e* 536. Anal. Calcd for $C_{26}H_{52}N_2O_9$: C, 58.18; H, 9.76. Found: C, 57.95; H, 9.55.

**Preparation of 1,13-Bis(*N*-monoaza-12-crown-4)-4,7,10-trioxatridecane (8).** Diethylene glycol ditosylate (0.9 g, 2.1 mmol) was added to a stirred mixture of 1.16 g (5 mmol) of *N*-1-(3-hydroxypropyl)monoaza-12-crown-4,[26] 10 mL of *t*-$C_4H_9OH$, 1 g (8.9 mmol) of KO-*t*-$C_4H_9$, and 8 mL of dioxane. The mixture was stirred at 70 °C for 16 h and evaporated and the residue mixed with 2 g of alumina. The mixed alumina was added to the top of a column of alumina, and the products were eluted with THF/$C_2H_5OH$ (50/1) to give 0.4 g (34%) of 8 which had identical spectral properties as reported previously for 8.

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# Ozone Epoxidation of Olefins Catalyzed by Highly Robust Manganese and Iron Porphyrin Complexes

Sandro Campestrini, A. Robert, and B. Meunier*

*Laboratoire de Chimie de Coordination du CNRS, 205 route de Narbonne, 31077 Toulouse cedex, France*

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## Introduction

During the last decade, many different oxygen atom donors (iodosylbenzene, hypochlorite, organic or inorganic peroxides) have been investigated for their ability to generate high-valent oxometalloporphyrin complexes, leading to various catalytic oxidation reactions.[1,2] This

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