ethanol containing a catalytic quantity (0.5 mL) of triethylamine. The reaction mixture was refluxed for 15 h before being condensed to ~30 mL. This solution was added slowly to 200 mL of anhydrous ether at which time the crude product precipitated from solution as a pale yellow solid (5.6 g, 97% yield). Recrystallization from an acetonitrile–diethyl ether solvent mixture provided 5.2 g (90% yield) of the purified product, mp 216–218 °C; MS, *m/e* 444 (FDMS); ^{1}H NMR (CD_2Cl_2) δ 4.32 (s, 2 H), 3.72 (s, 3 H), 3.36 (s, 1 H), 6.70–8.22 (m, 20 H, Ar). Anal. Calcd for $C_{31}H_{26}NO_2BF_4$: C, 70.07; H, 4.93; N, 2.64. Found: C, 70.2; H, 5.0; N, 2.7.

***N*-[2-(Bromomethyl)phenyl]-2-(4-methoxyphenyl)-4,6-diphenylpyridinium Tetrafluoroborate (6).** *N*-[2-(Hydroxymethyl)phenyl]-2-[4-methoxyphenyl]-4,6-diphenylpyridinium tetrafluoroborate (4.4 mmol) was dissolved in 25 mL of thionyl bromide and stirred at room temperature for 15 h. The reaction mixture was poured into 200 mL of water at which time the crude product precipitated from solution. The crude product was collected by suction filtration and air-dried before being dissolved in 100 mL of methylene chloride. This solution was filtered to remove inorganic salts and flash evaporated. Recrystallization from acetonitrile–diethyl ether provided 2.16 g (95% yield) of purified product, mp 254 °C dec; MS, *m/e* 507 (FDMS); ^{1}H NMR (CD_3CN) δ 4.12 (s, 2 H), 3.84 (s, 3 H), 6.95–8.32 (m, 20 H, Ar). Anal. Calcd for $C_{31}H_{25}NOBr{\cdot}BF_4$: C, 62.66; H, 4.24; N, 2.36. Found: C, 62.7; H, 4.3; N, 2.4.

1-[4-Methoxyphenyl]-5,7-diphenyl-2,3-benzindolizine (7) and 1,7-Diphenyl-5-(4-methoxyphenyl)-2,3-benzindolizine (8). *N*-[2-(Bromomethyl)phenyl]-2-(4-methoxyphenyl)-4,6-diphenylpyridinium tetrafluoroborate (**6**) (100 mg, 0.2 mmol) was dissolved in 10 mL of pyridine and refluxed for 2 h. The reaction mixture was then poured into 100 mL of water and extracted with three 100-mL portions of diethyl ether. The combined ether extract was then washed with 10% HCl and dried over $MgSO_4$ before flash evaporation. The mixture of benzindolizines (75 mg, 0.17 mmol) was isolated by chromatographic separation on silica gel by using cyclohexane as eluant. ^{1}H NMR in CD_2Cl_2 showed the presence of two distinct methoxy groups, δ 4.00 (s, 3 H) and 4.05 (s, 3 H) in a 1:1 ratio; MS, *m/e* 425. Anal. Calcd for $C_{31}H_{23}NO$: C, 87.50; H, 5.45; N, 3.29. Found: C, 88.0; H, 5.6; N, 3.4.

Registry No. **2**, 113404-16-3; **3**, 113404-17-4; **4**, 113404-18-5; **5**, 113404-20-9; **6**, 113404-22-1; **7**, 113404-23-2; **8**, 113430-70-9; *o*-$H_2NC_6H_4CH_2OH$, 5344-90-1; 2,4,6-triphenylpyrylium chloride, 40836-01-9; *N*-[2-(hydroxymethyl)phenyl]-2,4,6-triphenylpyridinium chloride, 113404-15-2; 4-[4-(dimethylamino)phenyl]-2,6-diphenylpyrylium perchlorate, 2970-29-8; 2-(4-methoxyphenyl)-4,6-diphenylpyrylium tetrafluoroborate, 2907-13-3; pyridine, 110-86-1.

Supplementary Material Available: X-ray crystallographic data for **3**: crystal data, numbering scheme, positional parameters, displacement parameters (β), bond lengths and angles, and least-square planes (8 pages). Ordering information is given on any current masthead page.

Thermal Cycloelimination of Bis(dialkylamino)cyclopropanes to Amidines and Cycloalkenes

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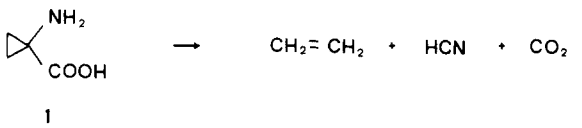
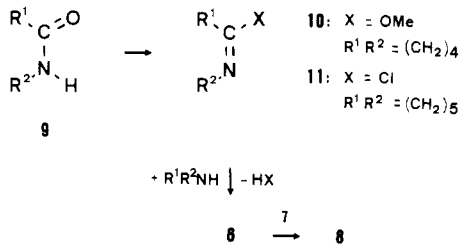
Aminocyclopropanecarboxylic acid (**1**) was shown to be the precursor of the phytohormone ethylene in nature.[1] Investigations[1,2] on the mechanism of ethylene formation from **1** led to an increase of interest in cycloelimination reactions of aminocyclopropane derivatives (Scheme I). So far alkene generation from aminocyclopropanes has required a primary amino function,[1-3] which was shown to be oxidized in the initial step.

Scheme I



1

Scheme II



9 **8**

We have found a non oxidatively induced cycloelimination of an aminocyclopropane derivative by flash vacuum pyrolysis of diaminobicyclo[*n*.1.0]alkanes **2**. Thus decomposition of dimorpholinobicyclo[4.1.0]heptane (**2a**) at 700 °C (10^{-5} Torr) gave heterocycle **6a** and cyclohexene (**3a**) in 70% and 93% yield, respectively, indicating a clean cleavage of the cyclopropane unit (Scheme II). The structure of **6a** is established unequivocally by ^{1}H NMR [δ 2.70–2.76 (2 H), 3.21–3.29 (4 H), 3.59–3.75 (10 H), 3 AA'XX' systems], ^{13}C NMR [δ 70.7, 66.9, 65.9 (OCH_2), 52.3, 46.9 (NCH_2), 32.4 (all t), 167.8 (s)], and IR spectroscopy [$\nu_{C=N}$ 1620 cm^{-1}]. Cyclohexene (**3a**) was identified by its ^{1}H NMR spectrum in carbon tetrachloride solution.

Similarily the pyrrolidino and the piperidino compounds **2c** and **2d** were cleaved by flash pyrolysis conditions to give amidines **6c** and **6d**. In these cases small amounts of impurities present could not be removed by distillation, therefore the oily reaction products were isolated and characterized as picrates (yields: **8c**, 88%; **8d**, 84%).

For structural clarification amidines **6c**/**8c** and **6d**/**8d** additionally were prepared by standard procedures[4] from lactams **9** via imidate **10** or imidoyl chloride **11** (Scheme III). The picrates **8c** and **8d** thus obtained and the corresponding picrates resulting from the thermolysis gave identical melting points and IR and ^{1}H NMR spectra.

The bicyclododecane derivatives **2b** and **12** were used to study the stereoselectivity of the cycloelimination. The separation of amidine **6** and cycloalkene was performed by an acidic ion-exchange resin. Cycloundecene was isolable in 89% and 92% yield from the corresponding aminals **2b** and **12**, respectively. Especially the IR and ^{1}H NMR spectra demonstrate the generation of *cis*-cycloundecene (**3b**) from cis aminal **2b** and of *trans*-cycloundecene (**13**) from trans aminal **12** as the main products (Scheme IV). GC–MS investigations[5] showed *cis*-cyclo-

(1) Vilsmaier, E. *The Chemistry of the Cyclopropyl Group*; Rappoport, Z., Ed.; Wiley: London, 1987; Part 2, p 1341 and references therein.

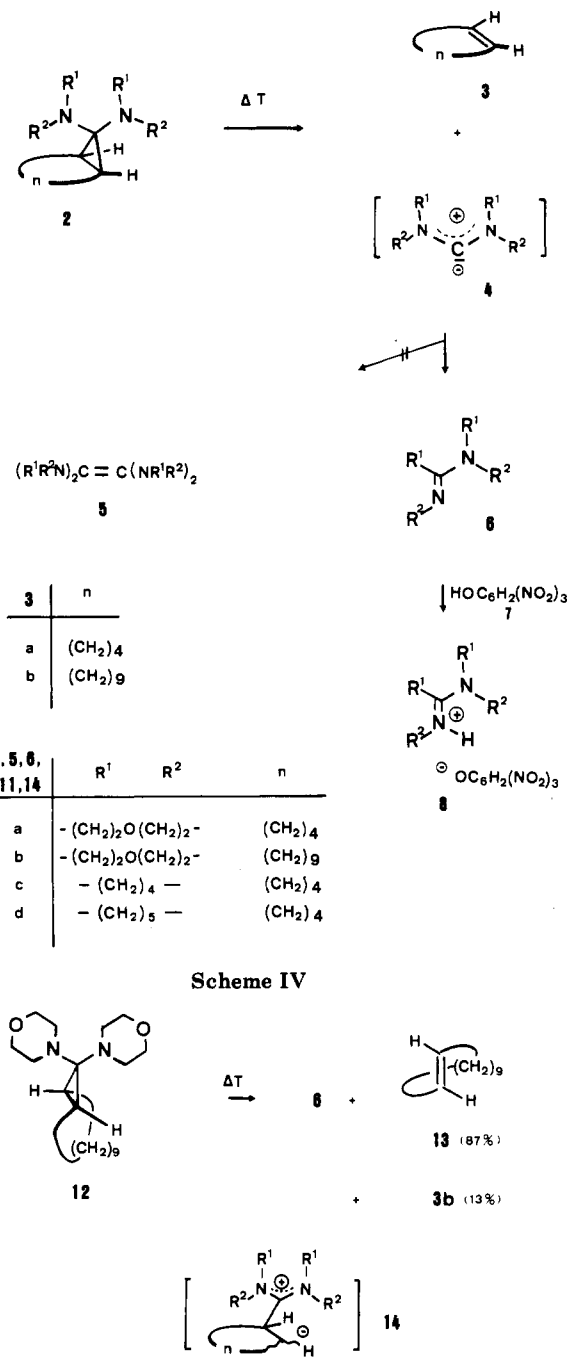
(2) Pirrung, M. C. *J. Am. Chem. Soc.* **1983**, *105*, 7207. Adlington, R. M.; Baldwin, J. E.; Rawlings, B. J. *J. Chem. Soc., Chem. Commun.* **1983**, 290. Baldwin, J. E.; Jackson, D. A.; Adlington, R. M.; Rawlings, B. J. *J. Chem. Soc., Chem. Commun.* **1985**, 206. Peiser, G. D.; Wang, T. T.; Hoffman, N. E.; Yang, S. F.; Liu, H.; Walsh, C. T. *Proc. Natl. Acad. Sci. U.S.A.* **1984**, *81*, 3059. Pirrung, M. C.; McGeehan, G. M. *J. Am. Chem. Soc.* **1986**, *108*, 5647.

(3) Hiyama, T.; Koide, H.; Nozaki, H. *Tetrahedron Lett.* **1973**, 2143; *Bull. Chem. Soc. Jpn.* **1975**, *48*, 2918.

(4) Körösi, J.; Lay, A.; Szabo, G. (E. GY. T. Gyogyszervegyeszeti Gyar). DOS 2 119 163, 1971; *Chem. Abstr.* **1972**, *76*, 25121.

0022-3263/88/1953-1806$01.50/0

Scheme III



3	n
a	$(CH_2)_4$
b	$(CH_2)_9$

2,4,5,6, 8-11,14	R^1 R^2	n
a	$-(CH_2)_2O(CH_2)_2-$	$(CH_2)_4$
b	$-(CH_2)_2O(CH_2)_2-$	$(CH_2)_9$
c	$-(CH_2)_4-$	$(CH_2)_4$
d	$-(CH_2)_5-$	$(CH_2)_4$

Scheme IV

undecene (**3b**) from **2b** to be contaminated by 7% *trans*-**13** and *trans*-cycloundecene (**13**) from **12** to contain 13% *cis*-alkene **3b**. Thus decomposition of aminals **2b** and **12** proved to be highly stereoselective. The reason for the partial isomerization could not be established; it could be the consequence of a configurational instability of **2b**/**12** (see ref 6) or **3b**/**13** (see ref 7) under the special FVP conditions as well as participation of an intermediate, **14**.

Difluorocyclopropanes have been decomposed upon heating into an alkene and difluorocarbene. The stabilization of the carbene by the fluorine substituents was proposed to be essential for the cycloelimination.[8] Analogously a diaminocarbene **4** should accompany cycloalkene formation in the pyrolysis of **2** or **12**. Obviously **4** is stabilized by an insertion reaction into the heterocyclic system. Insertion into a vicinal bond—well-known for carbenes[9,10]—has not been observed thus far for diaminocarbenes. Diaminocarbenes have been discussed as intermediates in various reactions.[11,12] Products **5** resulting from a dimerization reaction of **4** were not obtained under the low-pressure conditions. In spite of a long-standing interest in diaminocarbenes, a method for the generation of these electron-rich carbenes in the gas phase has been lacking.[13,14] Therefore, the clear pyrolysis of diaminocyclopropanes reported here should be a starting point for further investigations of diaminocarbenes.

Experimental Section

For vacuum flash pyrolysis an electrically heated quartz tube (30 cm × 1.5 cm; length of tube heated, 20 cm) was used; the starting materials were evaporated at 10^{-4}–10^{-5} Torr in a small flask (10 mL) connected directly with the quartz tube. The pyrolysis products immediately were trapped on a cold finger, which was cooled by liquid nitrogen.

5-Morpholino-2,3,6,7-tetrahydro-1,4-oxazepine (6a). 7,7-Dimorpholinobicycloheptane (**2a**)[15] (1.22 g, 4.58 mmol) was vaporized at 90 °C within 7 h and pyrolyzed at 700 °C. During the pyrolysis tetrachloromethane (total amount 8.50 g) in small portions (intervals of 30 min) was brought to the cold finger via an inlet. After warming on room temperature, the tetrachloromethane–cyclohexene mixture was found at the bottom of the flask, while oxazepine **6a** remained at the top of the trap. The volatile liquid was distilled at 20 °C (10^{-4} Torr) and completely collected (8.85 g) in a cooling trap. The ^1H NMR spectrum of the resulting distillate exclusively showed the signals of cyclohexene (**3a**) (δ 5.55 (mc, 2 H), 1.92 (mc, 4 H), 1.56 (mc, 4 H) lit.[16]); the solution contained 0.35 g (93%) cyclohexene as determined by addition of benzene (0.40 g). The oxazepine residue was removed from the trap by solving in dichloromethane (10 mL). Recrystallization from *n*-hexane gave colorless crystals of **6a** (0.59 g, 70%): mp 84–86 °C. Anal. Calcd for $C_9H_{16}N_2O_2$: C, 58.68; H, 8.77; N, 15.21. Found: C, 58.7; H, 8.77; N, 15.4.

2,3,4,5-Tetrahydro-6-pyrrolidinopyridinium Picrate (8c). 7,7-Dipyrrolidinobicycloheptane (**2c**)[15] (1.05 g, 4.48 mmol) was vaporized at 55 °C within 6 h and pyrolyzed at 650 °C. The pyrolysate was dissolved in pentane (20 mL); removal of the volatile components gave a colorless oil (0.61 g). Addition of ethanol (5 mL) and of a saturated ethanolic solution of picric acid (10 mL), refluxing for 2 min, and cooling to 0 °C yielded pale yellow crystals (1.5 g; 88%): mp 109 °C; IR (KBr, cm^{-1}) 1620 (C=N), 1580, 1330 (NO_2); ^1H NMR ($CDCl_3$) δ 8.87 (s, 2 H), 3.78–3.36 (m, 6 H), 2.77–2.48 (m, 2 H), 2.22–1.72 (m, 10 H). Anal. Calcd for $C_{15}H_{19}N_5O_7$: C, 47.25; H, 15.02; N, 18.36. Found: C, 47.2; H, 15.00; N, 18.5.

3,4,5,6-Tetrahydro-7-piperidino-2*H*-azepinium Picrate (8d). 7,7-Dipiperidinobicycloheptane (**2d**)[15] (1.5 g, 5.7 mmol) was vaporized at 75 °C within 2 h and pyrolyzed at 650 °C. Analogous working up as for **8c** gave pale yellow crystals of **8d** (1.95 g, 83.6%): mp 118 °C; IR (KBr, cm^{-1}) 1670 (C=N), 1570, 1320 (NO_2); ^1H

(5) The GC–MS analyses were performed at a Perkin-Elmer Σ 1, 20-m glass capillary column SE 30, 2 mL of He/min, 120 °C column temperature; retention times: **3b**, 11.91 min; **13**; 11.14 min.

(6) Vilsmaier, E.; Schwaben, B.; Joerg, K. *Chem. Ber.* **1984**, *117*, 2900.
(7) Cope, A. C.; Moore, P. T.; Moore, W. R. *J. Am. Chem. Soc.* **1960**, *82*, 1744.
(8) Hoffman, R. W. *Angew. Chem., Int. Ed. Engl.* **1971**, *10*, 529.
(9) Kirmse, W. *Carbene Chemistry*; Blomquist, A. T., Wasserman, H. H., Eds.; Academic: New York, 1971; Vol. 1, p 457.
(10) For monoaminocarbenes, insertion reactions combined with dimerization and other reactions were described: ter Borg, A. P.; Razenberg, E.; Kloosterziel, H. *Recl. Trav. Chim. Pays-Bas* **1966**, *85*, 776. Meriem, A.; Majoral, J. P.; Revel, M.; Navech, J. *Tetrahedron Lett.* **1983**, *24*, 1975. Hoffman, R. W.; Barth, W. *Chem. Ber.* **1985**, *118*, 634. Zoch, H. G.; Kinzel, E.; Szeimies, G. *Chem. Ber.* **1981**, *114*, 968.
(11) Wiberg, N. *Angew. Chem., Int. Ed. Engl.* **1968**, *7*, 754.
(12) Krasuski, W.; Nikolaus, D.; Regitz, M. *Liebigs Ann. Chem.* **1982**, 1451 and references cited therein.
(13) Hoffmann, R. W. *Acc. Chem. Res.* **1985**, *18*, 248.
(14) Hoffmann, R. W.; Barth, W.; Schüttler, R.; Mayer, B. *Chem. Ber.* **1986**, *119*, 3297.
(15) Vilsmaier, E.; Tröger, W.; Haag, G. *Chem. Ber.* **1981**, *114*, 67.
(16) Wiberg, K. B.; Nist, B. J. *J. Am. Chem. Soc.* **1961**, *83*, 1226.

NMR ($CDCl_3$) δ 8.82 (s, 2 H), 3.88–3.48 (m, 6 H), 2.94–2.67 (m, 2 H), 2.0–1.53 (m, 12 H). Anal. Calcd for $C_{17}H_{23}N_5O_7$: C, 49.88; H, 5.66; N, 17.11. Found: C, 49.8; H, 5.63; N, 17.1.

***cis*-Cycloundecene (3b).** 12,12-Dimorpholino-*cis*-bicyclo-[9.1.0]dodecane[17] (**2b**) (0.36 g, 1.07 mmol) was vaporized at 120 °C within 2 h and pyrolyzed at 650 °C. The condensate was dissolved in pentane (10 mL) and treated with acid ion-exchange resin (0.4 g, Type I, Fa. Merck). Filtration, evaporation of the solvent and distillation in a Kugelrohr apparatus gave **3b** as colorless oil (0.145 g, 89%): bp 25–30 °C (0.01 Torr) [lit.[18] bp 100 °C (12 Torr)]; IR (film, cm^{-1}) 710 (=C—H) (identical with IR spectrum in ref 18); ^1H NMR ($CDCl_3$) δ 5.35 (mc, 2 H), 2.27–2.14 (m, 4 H), 1.55–1.25 (m, 14 H).

***trans*-Cycloundecene (13).** Analogously, 12,12-Dimorpholino-*trans*-bicyclo[9.1.0]dodecane (**12**)[17] (0.96 g, 2.85 mmol; vaporization at 150 °C within 3 h; pyrolysis at 650 °C) yielded **13** (0.40 g, 92%): bp 25–30 °C (0.01 Torr) [lit.[18] bp 88–90 °C (15 Torr)]; IR (film, cm^{-1}) 980 (=C—H) (identical with IR spectrum in ref 19); ^1H NMR ($CDCl_3$) δ 5.45(mc, 2 H), 2.12–1.98 (m, 4 H), 1.50–1.12 (m, 12 H).

2,3,4,5-Tetrahydro-6-pyrrolidinopyridine (6c). A mixture of 6-methoxy-2,3,4,5-tetrahydropyridine (**10**)[20] (4.0 g, 35 mmol) and pyrrolidine (4.75 g, 70 mmol) was stirred for 2 days at 20 °C. Removal of the volatile compounds at 15 Torr and distillation of the residue at 0.01 Torr gave pure **6c** (5.0 g, 94%): bp 60–63 °C (0.01 Torr); IR (film, cm^{-1}) 1620 (C=N); ^1H NMR ($CDCl_3$) δ 3.62–3.19 (m, 6 H), 2.41–2.12 (m, 2 H), 1.98–1.39 (m, 10 H). Anal. Calcd for $C_9H_{16}N_2$: C, 71.00; H, 10.59; N, 18.40. Found: C, 71.4; H, 10.67; N, 18.2. Picrate **8c**: mp 109 °C; for IR and ^1H NMR data, see above.

3,4,5,6-Tetrahydro-7-piperidino-2*H*-azepine (6d). According to a general procedure,[4] caprolactam **9d** (23.0 g, 0.2 mol) was transferred into 7-chloro-3,4,5,6-tetrahydro-2*H*-azepine (**11**) by interaction with phosphoryl chloride (31.0 g, 0.2 mol) in benzene (140 mL, 20 °C, 2 h). Piperidine (17.03 g, 0.2 mol) was added directly to the reaction mixture at 0 °C; after refluxing for 8 h, the usual workup, and distillation at 0.01 Torr pure **6d** (17.7 g, 49%) was obtained. **6d**: bp 56–58 °C (0.01 Torr) [lit.[4] bp 133–134 °C (5 Torr)]; IR (film, cm^{-1}) 1635 (C=N); ^1H NMR ($CDCl_3$) δ 3.53–3.37 (m, 2 H), 3.30–3.09 (m, 4 H), 2.53–2.38 (m, 2 H), 1.88–1.27 (m, 12 H). Anal. Calcd for $C_{11}H_{20}N_2$: C, 73.28; H, 11.18; N, 15.54. Found: C, 73.7; H, 11.34; N, 15.4. Picrate **8d**: mp 118 °C; for IR and ^1H NMR data, see above.

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(17) Vilsmaier, E.; Klein, C. M.; Tröger, W.; Dausmann, D. *Synthesis* **1981**, 724.
(18) Prelog, V.; Boarland, V. *Helv. Chim. Acta* **1955**, *38*, 1776.
(19) Ziegler, K.; Sauer, H.; Bruns, L.; Froitzheim-Kühlhorn, H.; Schneider, J. *Justus Liebigs Ann. Chem.* **1954**, *589*, 122.
(20) Ralls, J. W. *J. Org. Chem.* **1961**, *26*, 66.

A Simple and Convenient Method for the Preparation of *N,N'*-Dibenzyldiaza-crown Compounds

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There has been considerable recent interest in the synthesis of nitrogen-containing crown compounds.[1-8] The aza crown compounds, in some cases, have anion complexation properties that are similar to complexation in certain biological systems.[1,9,10] The diaza-crown compounds have enhanced complexing abilities for ammonium cations in comparison with the all-oxygen crown compounds.[5] In addition, the diaza-crown compounds are important intermediates for the synthesis of the cryptands.[11,12] We now report a convenient two-step synthesis of *N,N'*-dibenzyldiaza-15-crown-5, 18-crown-6, and 21-crown-7.

Many of the previous syntheses of the diaza-crowns have used the reaction of a diamine and a diacid chloride for ring closure, followed by reduction of the resulting diamide to form the diaza-crowns.[11,12] The more recent reports have used the reaction of certain diamines with dihalides, particularly the less available diiodides.[2-4] Okahara and his co-workers[6,7,13] and Oda and his co-workers[14] have used one-step methods to prepare the aza crowns from previously prepared oligoethylene oxide containing diamines. The open-chain diamines, in general, are important intermediates for the synthesis of the diaza-crown compounds.

We have found that *N,N'*-dibenzyldiazaoligoethylene glycols (**1–2** and **6**, Scheme I) can easily be converted in high yields into the *N,N'*-dibenzyldiaza-crown compounds (**7–9**). The diazaoligoethylene glycols were readily prepared by reacting dichlorides **11** or **12** with *N*-benzylethanolamine (or the *N*-ethyl isomer) (see Scheme I). Okahara and his co-workers[6] and Ishidate, Sakurai, and Maruyama[15] have reported similar mono and diaza glycols, but the yields of their reactions were only 20–50%. Diaza compound **1** was also prepared by reacting 1,14-dihydroxy-3,12-diaza-6,9-dioxatetradecane[16] with benzyl bromide.

Secondary diamines **4** and **5** were obtained in good yields by reacting a fourfold excess of the amine with the dichloride in toluene (Scheme I). Generally, these types of diamines have been prepared by various methods, including the Gabriel synthesis,[12,17] the reaction of primary amines with diacid chlorides followed by the reduction of the diamide,[3,18] or the reduction of diazides formed from the reaction of a dichloride with sodium azide.[19] Diamine **4** was also reported by Gokel and co-workers[2] using 1,8-diiodo-3,6-dioxaoctane.

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(1) Hosseini, M. W.; Lehn, J. M.; Duff, S. R.; Gu, K.; Mertes, M. P. *J. Org. Chem.* **1987**, *52*, 1662.
(2) Gatto, V. J.; Arnold, K. A.; Viscariello, A. M.; Miller, S. R.; Morgan, C. R.; Gokel, G. W. *J. Org. Chem.* **1986**, *51*, 5373.
(3) Gatto, V. J.; Arnold, K. A.; Viscariello, A. M.; Miller, S. R.; Gokel, G. W. *Tetrahedron Lett.* **1986**, *27*, 327.
(4) Gatto, V. J.; Gokel, G. W., *J. Am. Chem. Soc.* **1984**, *106*, 8240.
(5) Pietraszkiewicz, M.; Jurczak, J. *J. Chem. Soc., Chem. Commun.* **1983**, 132.
(6) Kuo, P.-L.; Miki, M.; Ikeda, I.; Okahara, M. *J. Am. Oil Chem. Soc.* **1980**, 227.
(7) Maeda, H.; Furuyoshi, S.; Nakatsuji, Y.; Okahara, M. *Bull. Chem. Soc. Jpn.* **1983**, *56*, 3073.
(8) Biernat, J.; Luboch, E. *Tetrahedron* **1984**, *40*, 1927.
(9) Lehn, J. M. *Science (Washington, D.C.)* **1985**, *227*, 849.
(10) Yohannes, P. G.; Mertes, M. P.; Mertes, K. B. *J. Am. Chem. Soc.* **1985**, *107*, 8288.
(11) Dietrich, B.; Lehn, J. M.; Sauvage, J. P. *Tetrahedron Lett.* **1969**, 2885.
(12) Dietrich, B.; Lehn, J. M.; Sauvage, J. P. *Tetrahedron* **1973**, *29*, 1629.
(13) Kikui, T.; Maeda, H.; Nakatsuji, Y.; Okahara, M. *Synthesis* **1984**, 74.
(14) Kawaguchi, M.; Ohashi, J.; Kawakami, Y.; Yamamoto, Y.; Oda, J. *Synthesis* **1985**, 701.
(15) Ishidate, M.; Sakurai, Y.; Maruyama, K. *Chem. Pharm. Bull.* **1958**, *6*, 164.
(16) King, A.; Krespan, C. *J. Org. Chem.* **1974**, *39*, 1315.
(17) Krakowiak, K.; Kotelko, B. *Pol. J. Chem.* **1982**, *56*, 1145.
(18) Krakowiak, K.; Kotelko, B. *Acta Polon. Pharm.* **1983**, 313.
(19) Kurlstad, S.; Malmsten, L. A. *Acta Chem. Scand., Ser. B* **1979**, *B33*, 469.

0022-3263/88/1953-1808$01.50/0