stabilization of the anion, then the difference between ΔH_I and ΔH_{II} will be assigned to this stabilization energy. However, the fact that there is an energy difference does not provide evidence that the initial assumption is correct. Because of Hess' law, ΔH_{II} is greater than ΔH_I by the amount of the difference in acidity between acetic acid and isopropyl alcohol, regardless of the reason for this difference.

Once we recognize that both initial- and final-state effects influence the acidities of carboxylic acids,[9] then we need a model that can account for both effects. The use of isodesmic reactions does *not* provide information that can separate the two effects. Such separation can come only from comparisons of experimental and/or theoretical results that are sensitive in different ways to initial- and final-state effects.

However, consideration of a series of isodesmic reactions in which carboxylic acids and alcohols are compared with compounds having substituents of different electronegativities does provide some insight into the interaction between a carbonyl oxygen and a hydroxyl group attached to the same carbon atom. Such a configuration appears to be about 35 kJ/mol more stable than would be expected from electronegativity considerations alone. This result suggests that there is some special stabilization of the carboxylic acid because of this interaction.

Acknowledgment. We would like to thank Professor Otto Exner for sharing his thoughts on isodesmic reactions with us prior to publication. This work was supported by the National Science Foundation.

(9) The idea that the initial-state effect is important is not new, but was well recognized in early treatments of the subject. (See, for instance: Wheland, G. W. *The Theory of Resonance and Its Application to Organic Chemistry*; Wiley: New York, 1944; pp 167–172. Also see: *Resonance in Organic Chemistry*; Wiley: New York, 1955; pp 340–345.) It is only more recently that the greater acidity of carboxylic acids has been attributed almost exclusively to resonance stabilization of the anion (ref 2).

Tristrimethylsilylation of Phenol and Related Reactions

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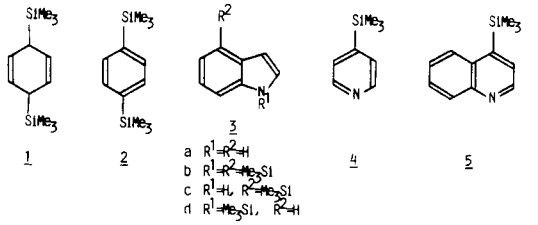
Received September 28, 1987

In 1962 Weyenberg and Toporcer reported that benzene was reduced by the use of lithium and chlorotrimethylsilane in THF to produce 3,6-bis(trimethylsilyl)-1,4-cyclohexadiene (**1**) (40%) and 1,4-bis(trimethylsilyl)-benzene (**2**) (15%).[1] This silyl Birch reduction chemistry was extended to toluene and anisole to produce the corresponding 1,4-cyclohexadiene systems (30–50%). Laguerre et al. subsequently optimized the preparation of **1** (85%), observed that air oxidation was particularly effective in the aromatization of **1**, and extended the chemistry to a series of alkylbenzene derivatives.[2] Recently we have utilized this methodology for the conversion of indole (**3a**) into 1,4-bis(trimethylsilyl)indole (**3b**) and subsequently 4-(trimethylsilyl)indole (**3c**). Thus reaction of **3a** or **3d** with lithium metal and chlorotrimethylsilane under ultrasonication gave **3b** (50%, 55% respectively) on oxidation of the intermediate dihydroindole derivative with *p*-benzoquinone.[3] Subsequent methanolysis of **3b** gave **3c** (98%). 4-(Trimethylsilyl)indole (**3c**) was found to be useful in the preparation of 4-acylindoles via ipso Friedel–Crafts acylation. In addition to indole (**3a**), both pyridine and quinoline were converted into the corresponding C-4 trimethylsilyl derivatives **4** (42%) and **5** (35%).



Herein we report the extension of the silyl Birch reduction to phenol and its derivatives. Reduction of phenol by use of lithium in the presence of chlorotrimethylsilane proceeded efficiently providing that the reaction mixture was ultrasonicated.[4] Without ultrasonication the reaction was much slower and proceeded in poor yield. The 1,4-cyclohexadiene product was not isolated. Direct oxidation by reflux in air gave the trisilyl product **6a** (72%). In the same way *m*- and *p*-cresol were converted into **7a** (39%) and **8a** (58%). Since aerobic oxidation of the intermediate **9a** was very slow, *p*-benzoquinone was used to restore aromaticity. Anisole was converted into **9b** by use of the silyl Birch reduction. Again this material was readily air oxidized to produce **6b** (63% overall). Finally 4-methoxytoluene was converted into **8b** (64%). The trisilyl derivatives **6a–8a** were smoothly and cleanly monodesilylated by reaction with tetrabutylammonium fluoride in THF to provide the corresponding phenols **6c**, **7b**, and **8c** (87–92%). It is clear from these experiments that simple oxygenated benzene derivatives may be readily converted into the *p*-bis(trimethylsilyl) aromatic systems with retention of the oxygen substituent. This is in contrast to the naphthalene derivatives **10a** and **10b** which have been reported to be converted respectively into **10c** and **10d** on silyl Birch reduction.[5] Additionally it is clear that the regioselectivity of the reaction follows directly the known mechanism of the Birch reduction.[6]

We have briefly examined the reaction of **6b** with electrophiles.[7] Bromination of **6b** with *N*-bromosuccinimide (−30 °C) or bromine (25 °C), respectively, gave **11** (82%) and **12** (84%). In contrast to these reactions **6b** gave **13** (67%) on Friedel–Crafts acylation. Presumably the electrophile in this case is too bulky to ipso substitute the

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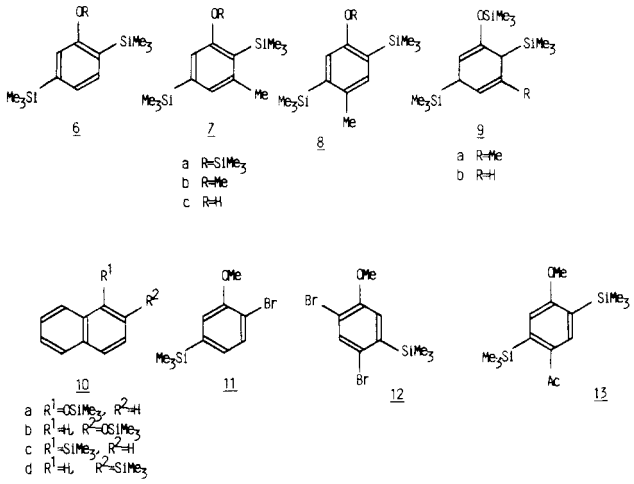

ortho trimethylsilyl group. In conclusion the silyl Birch reduction of phenol and its derivatives provides an experimentally simple procedure to prepare bis- and mono-(trimethylsilyl)-substituted aromatic substances.[8]

Experimental Section

Melting points were determined on a Kofler hot stage apparatus and are uncorrected. Reactions were carried out under dry argon or nitrogen. All solvents and reagents were purified and dried before use. Ultrasonication was carried out in a PUL55 Kerry QH ultrasonic bath (Kerry Ultrasonics Ltd., England). Organic extracts were concentrated by rotary evaporation under reduced pressure at ≤40 °C. Unless stated to the contrary, chromatography refers to flash chromatography on Merck Kieselgel H.

1,4-Bis(trimethylsilyl)-2-[(trimethylsilyl)oxy]benzene (6a). To phenol (0.94 g) at 0 °C in dry THF (30 mL) under argon was added Me_3SiCl (5.43 g) followed by lithium metal (0.35 g) cut into ca. 20 pieces. The mixture was agitated in an ultrasonic bath at 0–5 °C for 3 h and then at 40 °C for 12 h. The THF was evaporated under reduced pressure and the residue was triturated with hexane (4 × 40 mL). The combined extracts were filtered through Celite and refluxed in air for 72 h. The solvent was evaported under reduced pressure and the residue chromatographed (silica gel, hexane) to give **6a** (2.72 g, 72%) as a colorless oil: IR (film) 3034, 2955, 2898, 1583, 1521, 1405, 1368, 1252, 1231, 1116, 966, 838 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.30 (s, 18 H), 0.38 (s, 9 H), 6.95 (s, 1 H), 7.12 (d, 1 H, J = 7.7 Hz), 7.41 (d, 1 H, J = 7.7 Hz); mass spectrum, m/e 310 ($M^{\cdot+}$), 295 (M^+ – Me). Anal. Calcd for $C_{15}H_{30}OSi_3$: C, 57.97; H, 9.74. Found: C, 58.23; H, 10.00.

3-Methyl-2,5-bis(trimethylsilyl)-1-[(trimethylsilyl)oxy]benzene (7a). To m-cresol (1.08 g) in dry THF (30 mL) at 0 °C under argon was added Me_3SiCl (5.43 g) followed by lithium metal (0.35 g) freshly cut into ca. 20 pieces. The resulting mixture was agitated in an ultrasonic bath at 0 °C for 6 h and then at 40 °C for 18 h. The THF was evaporated under reduced pressure and the residue was triturated with hexane (4 × 40 mL). The combined organic phase was filtered through Celite and the solvent evaporated under reduced pressure. The residue was redissolved in dry CH_2Cl_2 (20 mL) and p-benzoquinone (1.62 g) was added. The resulting solution was stirred at 25 °C for 72 h, the solvent was evaporated under reduced pressure, and the residue was triturated with hexane (3 × 30 mL). The solvent was evaporated under reduced pressure and the residue chromatographed (silica gel, hexane) to give **7a** (1.26 g, 39%) as a colorless oil: IR (film) 2955, 2897, 1579, 1518, 1405, 1371, 1273, 1251, 1046, 911, 842 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 0.30 (s, 9 H), 0.35 (s, 18 H), 2.45 (s, 3 H), 6.85 (s, 1 H), 6.95 (s, 1 H); mass spectrum, m/e 324 ($M^{\cdot+}$).

5-Methyl-1,4-bis(trimethylsilyl)-2-[(trimethylsilyl)oxy]benzene (8a). To p-cresol (1.08 g) at 0 °C in dry THF (30 mL) under argon was added Me_3SiCl (5.43 g) followed by lithium metal (0.35 g) cut into ca. 20 pieces. The mixture was agitated in an ultrasonic bath at 0–5 °C for 6 h and at 40 °C for 16 h. The THF was evaporated under reduced pressure and the residue triturated with hexane (4 × 40 mL). The combined organic phase was filtered through Celite and then refluxed in air for 48 h. The solvent was evaporated under reduced pressure and the residue chromatographed (silica gel, hexane) to give **8a** (1.88 g, 58%) as a colorless oil: IR (film) 2955, 2898, 1465, 1337, 1251, 1237, 1116, 891, 837 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.27 (s, 9 H), 0.33 (s, 18 H), 2.40 (s, 3 H), 6.91 (s, 1 H), 7.15 (s, 1 H); mass spectrum, m/e 324 (M^+). Anal. Calcd for $C_{16}H_{32}OSi_3$: C, 59.17; H, 9.94. Found: C, 59.06; H, 10.18.

1-Methoxy-2,5-bis(trimethylsilyl)benzene (6b). To PhOMe (1.08 g) in dry THF (30 mL) under argon at 0 °C was added Me_3SiCl (5.43 g) followed by lithium (0.347 g) cut into ca. 50 pieces. The mixture was agitated in an ultrasonic bath at 5–10 °C for 3 h and subsequently overnight at 45 °C. The solvent was evaporated under reduced pressure and the residue triturated with hexane (4 × 40 mL). The combined extracts were filtered through Celite and the solvent evaporated under reduced pressure to leave crude **9b**: ^1H NMR (60 MHz, $CDCl_3$) δ 0.10 (2 s, 18 H), 2.20 (m, 2 H), 3.55 (s, 3 H), 4.30 (m, 1 H), 5.45 (m, 2 H). The crude intermediate **9b** was allowed to air-oxidize by standing at room temperature for 72 h. Recrystallization gave **6b**[1] (1.55 g, 63%) as white crystals: mp 49–51 °C (from MeOH at –20 °C); IR (CH_2Cl_2) 2950, 2910, 2860, 1590, 1450, 1230, 1180, 1120, 1045, 850 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.22 (s, 9 H), 0.23 (s, 9 H), 3.79 (s, 3 H), 6.95 (s, 1 H), 7.15 (d, 1 H, J = 7.6 Hz), 7.40 (d, 1 H, J = 7.6 Hz); mass spectrum, m/e 252 ($M^{\cdot+}$). Anal. Calcd for $C_{13}H_{24}OSi_2$: C, 61.82; H, 9.59. Found: C, 61.87; H, 9.71.

1-Methoxy-4-methyl-2,5-bis(trimethylsilyl)-4-methylbenzene (8b). To 4-methylanisole (1.22 g) in THF (30 mL) at 0 °C under argon was added Me_3SiCl (5.43 g) followed by lithium metal (0.35 g) freshly cut into ca. 20 pieces. The mixture was agitated in an ultrasonic bath at 0 °C for 4 h and then at 40 °C for 16 h. The THF was evaporated under reduced pressure and the residue triturated with hexane (4 × 40 mL). The combined extracts were filtered through Celite and refluxed in air for 72 h. The solvent was evaporated under reduced pressure and the residue recrystallized from MeOH at –20 °C to give **8b** (1.70 g, 64%) as a white crystalline solid: mp 76–78 °C (from MeOH at –20 °C); IR (CH_2Cl_2) 2950, 2915, 2860, 1590, 1430, 1230, 1170, 1040, 840 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 0.15 (s, 9 H), 0.25 (s, 9 H), 2.32 (s, 3 H), 3.71 (s, 3 H), 6.90 (s, 1 H), 7.13 (s, 1 H); mass spectrum, m/e 266 ($M^{\cdot+}$). Anal. Calcd for $C_{14}H_{26}OSi_2$: C, 63.07; H, 9.84. Found: C, 62.90; H, 10.01.

2,5-Bis(trimethylsilyl)phenol (6c). To **6a** (0.90 g) in THF (10 mL) was added tetrabutylammonium fluoride in THF (1.0 M; 2.9 mL). The solution was stirred for 1 h at room temperature, poured into saturated $NaHCO_3$ solution (10 mL), and extracted with Et_2O (3 × 20 mL). The combined extracts were dried, the solvent was evaporated under reduced pressure, and the residue was chromatographed (silica gel, Et_2O/hexane 1:49) to give **6c** (0.64 g, 92%) as a white solid: mp 70–73 °C (from MeOH at –60 °C); IR (CH_2Cl_2) 3574, 2949, 2897, 1592, 1530, 1491, 1375, 1225, 1190, 1144, 1112, 1060, 828 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.25 (s, 9 H), 0.27 (s, 9 H), 4.75 (s, 1 H), 6.78 (s, 1 H), 7.06 (d, 1 H, J = 7.5 Hz), 7.38 (d, 1 H, J = 7.5 Hz); mass spectrum, m/e 238 ($M^{\cdot+}$), 223. Anal. Calcd for $C_{12}H_{22}OSi_2$: C, 60.42; H, 9.30. Found: C, 60.52; H, 9.59.

3-Methyl-2,5-bis(trimethylsilyl)phenol (7c). Desilylation of **7a** (0.324g) using Bu_4NF and chromatography (silica gel, hexane) gave **7c** (0.22 g, 87%) as a white solid: IR (CH_2Cl_2) 3590, 3530, 2954, 2897, 1588, 1526, 1443, 1369, 1248, 1159, 908, 836, and 755 cm^{-1}; ^1H NMR (60 MHz; $CDCl_3$) δ 0.20 (s, 9 H), 0.30 (s, 9 H), 2.40 (s, 3 H), 4.85 (s, 1 H), 6.65 (s, 1 H), 6.90 (s, 1 H); mass spectrum, m/e 252 ($M^{\cdot+}$). Anal. Calcd for $C_{13}H_{24}OSi_2$: C, 61.82; H, 9.59. Found: C, 61.54; H, 9.58.

4-Methyl-2,5-bis(trimethylsilyl)phenol (8c). Using an identical procedure desilylation of **8a** (0.32 g) and chromatography (silica gel, Et_2O/hexane 3:97) gave **8c** (0.229 g, 92%) as a white

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solid: mp 69–70 °C; IR (CH_2Cl_2) 3580, 2952, 2898, 1589, 1446, 1379, 1353, 1228, 1180, 1113, 1032, 839 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.30 (s, 18 H), 2.40 (s, 3 H), 4.61 (s, 1 H), 6.78 (s, 1 H), 7.12 (s, 1 H); mass spectrum, m/e 252 ($M^{\cdot+}$). Anal. Calcd for $C_{13}H_{24}OSi_2$: C, 61.82; H, 9.59. Found: C, 61.76; H, 9.65.

1-Bromo-2-methoxy-4-(trimethylsilyl)benzene (11). To **6b** (0.252 g) in dry CH_2Cl_2 (10 mL) under argon at −30 °C was added *N*-bromosuccinimide (0.179 g) in one portion. The solution was stirred at −30 °C for 20 min an then warmed to room temperature. The solvent was evaporated under reduced pressure and the residue triturated with hexane (3 × 20 mL). The combined extracts were washed with brine (10 mL) and dried. The solvent was evaporated under reduced pressure and the residue chromatographed (silica gel, hexane) to give **11** (0.218 g, 82%) as a colorless oil: IR (film) 3004, 2954, 2845, 1570, 1481, 1461, 1371, 1247, 1185, 1106, 1044, 885, 837, 754 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.30 (s, 9 H), 3.75 (s, 3 H), 6.85 (s, 1 H), 7.05 (d, 1 H, J = 7.0 Hz), 7.30 (d, 1 H, J = 7.0 Hz); mass spectrum, m/e 260, 258 ($M^{\cdot+}$). Anal. Calcd for $C_{10}H_{15}BrOSi$: C, 46.31; H, 5.84. Found: C, 46.52; H, 5.95.

1,5-Dibromo-2-methoxy-4-(trimethylsilyl)benzene (12). To **6b** (0.126 g) in dry CH_2Cl_2 (5 mL) was added bromine (0.160 g) dropwise. The resulting solution was stirred for 1 h at room temperature and washed with saturated aqueous sodium thiosulfate (2 mL) and brine (2 mL). The solvent was evaporated under reduced pressure and the residue chromatographed (silica gel, hexane) to give **12** (0.141 g, 84%) as white crystals: mp 91–93 °C (from hexane); IR (KBr) 2960, 2910, 2840, 1560, 1455, 1330, 1245, 1050, 840, 755 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.40 (s, 9 H), 3.90 (s, 3 H), 6.93 (s, 1 H), 7.69 (s, 1 H); mass spectrum, m/e 335 ($M^+ - 1$). Anal. Calcd for $C_{10}H_{14}Br_2OSi$: C, 35.50; H, 4.18. Found: C, 35.80; H, 4.05.

4-Methoxy-2,5-bis(trimethylsilyl)acetophenone (13). To $AlCl_3$ (0.667 g) in CH_2Cl_2 (10 mL) under argon was added AcCl (0.39 g). When all the $AlCl_3$ had dissolved, the solution was cooled to −78 °C and a solution of **6b** (1.23 g) in dry CH_2Cl_2 (15 mL) was added rapidly. The resulting solution was stirred at −78 °C for 30 min, allowed to warm up to 0 °C, and poured into saturated aqueous $NaHCO_3$ (15 mL). The aqueous phase was extracted with CH_2Cl_2 (3 × 15 mL), the combined organic phase was dried, and the solvent was evaporated under reduced pressure. The residue was chromatographed (silica gel, Et_2O/hexane 1:4) to give **13** (0.98 g, 67%) as a white solid: mp 101–102 °C; IR (CH_2Cl_2) 2947, 2899, 1671, 1576, 1511, 1456, 1358, 1335, 1227, 1121, 1041 cm^{-1}; NMR (90 MHz, $CDCl_3$) δ 0.27 (s, 18 H), 2.59 (s, 3 H), 3.88 (s, 3 H), 7.18 (s, 1 H), 7.95 (s, 1 H); mass spectrum, m/e 279 (M^+ − Me). Anal. Calcd for $C_{15}H_{26}O_2Si_2$: C, 61.15; H, 8.90. Found: C, 61.10; H, 8.95.

Registry No. **6a**, 113353-56-3; **6b**, 18405-84-0; **6c**, 113353-60-9; **7a**, 113353-57-4; **7c**, 113353-61-0; **8a**, 113353-58-5; **8b**, 113353-59-6; **8c**, 113353-62-1; **9b**, 113378-71-5; **11**, 113353-63-2; **12**, 113353-64-3; **13**, 113353-65-4; PhOMe, 100-66-3; *m*-cresol, 108-39-4; *p*-cresol, 106-44-5; 4-methylanisole, 104-93-8; phenol, 108-95-2.

A New Strategy for the Synthesis of Polyazamacrocyclic Compounds: Use of a Removable Protecting and Rigid Group

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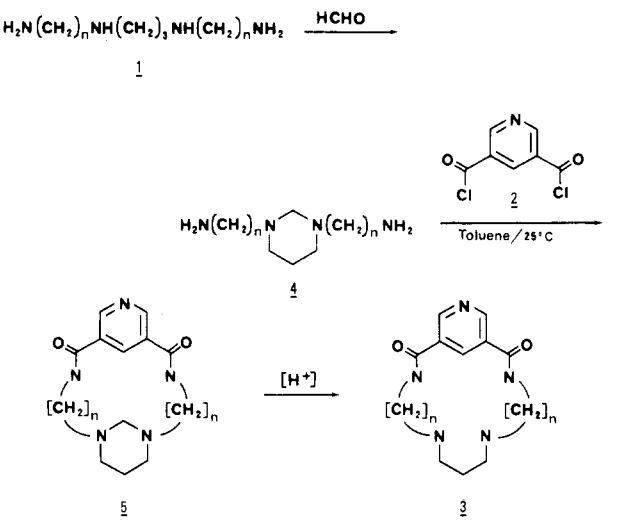
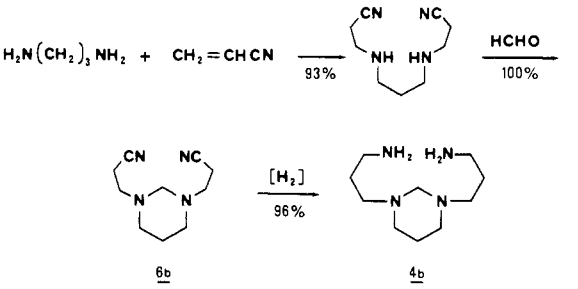
Received June 23, 1987

Within the past decade, the chemistry of macrocyclic ring systems has developed rapidly. Among the general strategies for ring closure reactions,[1] the high dilution principle[2] and the template effect[3] proved to be most profitable in that the formation of oligo- or polycondensation products is suppressed or minimized. The rigid group principle[4] has also been used to restrict the rotational possibilities by having a number of atoms composing the open-chain precursor held in the form of a rigid group.

Scheme I



Scheme II



The syntheses of pyridyl-containing polyazamacrocycles are mainly concerned with the template and high dilution methods. Our interest for compounds **3** incorporating a 3,5-disubstituted pyridino group prompted us to develop a convenient synthetic route for polyazamacrocycles incorporating available secondary amino groups. The general procedure for the preparation of polyazamacrocycles **3a–c** involves the reaction of the 3,5-pyridinedicarbonyl dichloride (**2**) with a twofold excess of the corresponding tetraamine **1a–c**. Although high dilution conditions were used, in all cases the overall yields were poor. We now report an efficient new procedure for the synthesis of macrocycles involving the temporary chemical modification of the linear precursor. This principle has been applied to linear tetraamines **1a–c** which are used in the preparation of polyazamacrocycles **3a–c**. The synthetic process is summarized in Scheme I.

Addition of an equimolar amount of formaldehyde (37% in water) to tetraamines **1a** and **1c** resulted in the formation of the six-membered aminals **4a** and **4c** (Scheme I). The reaction was very selective and only the six-membered rings were formed. Reaction of **1b** with formaldehyde always led to mixtures of variable amounts of differently substituted derivatives, probably due to the

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