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Registry No. 1, 63439-10-1; 2, 63372-77-0; 3, 96502-52-2; 4, 96502-53-3; (OEP)RhCH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>, 57650-51-8; (OEP)RhBr, 63372-78-1; PhCH=CH<sub>2</sub>, 100-42-5; C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>Br, 100-39-0.

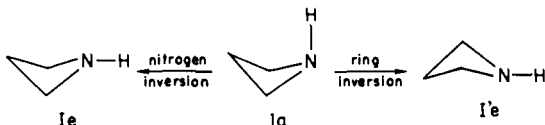
## On the Reported High Barrier to Nitrogen Inversion in Azetidine (Trimethylenimine)

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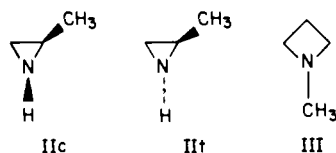
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A paper by Friedman, Chauvel, and True (FCT) states that two separate (chemically shifted) NH resonances with unequal areas (ca. 3:1) are observed in the <sup>1</sup>H NMR spectrum of azetidine (I) at room temperature and that the free energy barrier to nitrogen inversion is about 17.9 kcal/mol.<sup>1</sup> This would require that both ring inversion and nitrogen inversion in nonplanar I be slow on the dynamic NMR time scale under these conditions, since either of the above processes is sufficient to cause exchange of the NH proton between the quasi-axial and quasi-equatorial sites, as can be seen from the structures Ia, Ie, and I'e.<sup>2</sup> Thus, both



free energy barriers must be more than 17 kcal/mol. However, it is known from other investigations quoted by FCT that the barrier to ring inversion in azetidine is at best only a few kilocalories per mole. Furthermore, the methylene chemical shifts of I in CCl<sub>4</sub> are known<sup>3</sup> and are quite different from those reported by FCT.

A consideration of the data reported by FCT shows that the compound that they studied must be 2-methylaziridine (II). The



250-MHz <sup>1</sup>H NMR spectrum of this compound in CCl<sub>4</sub> has been measured and analyzed previously.<sup>4</sup> The chemical shifts and integration are consistent with those given by FCT, except for the NH proton signals whose chemical shifts are about 1-1.5 ppm to lower fields in CCl<sub>4</sub> than are those in the gas phase. These

(1) Friedman, B. R.; Chauvel, J. P., Jr.; True, N. S. *J. Am. Chem. Soc.* 1984, 106, 7638-7639.

(2) FCT correctly point out that both fast ring and nitrogen inversions are required for the  $\beta$ -CH<sub>2</sub> protons in Ia and Ie to give a single averaged chemical shift. However, the four lines that they ascribe to the  $\beta$ -protons at room temperature collapse to two lines and not to one line at high temperatures. Thus, their conclusion that there is "complete exchange" of these protons is not borne out by their published spectra. The NH proton has only two possible sites which are shown in Ia and Ie, and thus its behavior is different from that of the  $\beta$ -protons.

(3) Higgins, R. H.; Cromwell, N. H.; Paudler, W. W. *J. Heterocycl. Chem.* 1971, 8, 961-966. The chemical shifts of the  $\alpha$ - and  $\beta$ -protons in I in CCl<sub>4</sub> solution, with tetramethylsilane as the internal reference, are  $\delta$  3.53 and 2.33, respectively, whereas the chemical shifts reported by FCT for these protons are between  $\delta$  1 and 2. These latter high-field shifts point to unusual shielding, such as occurs in a three- but not in a four-membered ring.

(4) Lopez, A.; Gauthier, M. M.; Martino, R.; Lattes, A. *Org. Magn. Reson.* 1979, 12, 418-428. In the major conformer (II<sub>t</sub>), the assignments are as follows: CH<sub>3</sub>,  $\delta$  1.04,  $J = 5.5$  Hz; CH<sub>2</sub>,  $\delta$  0.96 and 1.66; CH,  $\delta$  1.89; NH,  $\delta$  0.61. In the minor conformer (II<sub>c</sub>), the CH<sub>3</sub> and NH protons are at  $\delta$  1.23 and 0.08, respectively, and the other protons lie between  $\delta$  1.2 and 1.4.

chemical shifts are expected to be influenced by hydrogen bonding and thus to depend on both concentration and solvent. The conformational ratio (II<sub>t</sub>:II<sub>c</sub>) is 2:1 in the liquid phase and the small difference from the gas-phase value (3:1) is again not unexpected.

The free energy barrier to nitrogen inversion found by FCT is very close to that in aziridine itself ( $\Delta G^\ddagger = 17.2 \pm 0.1$  kcal/mol in either the gas<sup>5</sup> or the liquid phase<sup>6</sup>) and much higher than that in 1-methylazetidine (III) ( $\Delta G^\ddagger = 10.0$  kcal/mol in the liquid phase<sup>7</sup>). At present, the barrier to nitrogen inversion in I remains unknown,<sup>8</sup> but its value should be similar to that in its *N*-methyl derivative, III.<sup>9</sup>

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(9) Professor, N. S. True (personal communication) has informed me that the compound studied by them was indeed 2-methylaziridine (see Additions and Corrections; True, N. S. *J. Am. Chem. Soc.*, in press.)

## A Diacridine Derivative That Binds by Bisintercalation at Two Contiguous Sites on DNA

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When two DNA-intercalating chromophores are joined by a linker chain, the nature of this chain becomes a major constraint upon bisintercalative binding.<sup>1-3</sup> Results for derivatives of 9-aminoacridine indicate that compounds with polymethylene or amide-containing linker chains as short as 8.8 Å undergo bisintercalative binding.<sup>1-6</sup> Such compounds (e.g., 1 and 2) must intercalate at contiguous sites, with one chromophore on either side of the same base pair, in violation of the "excluded-site" principle proposed as a thermodynamic limitation in some theoretical models of the binding of monointercalators and observed in practice for such compounds.<sup>5,7</sup>

However, monointercalative binding of a related bis(acridine) hydrazine 3 was indicated in a recent study.<sup>2</sup> Also, NMR studies show that 1 and 2 bind to the oligodeoxyribonucleotide d(A-T)<sub>5</sub>d(A-T)<sub>5</sub> by monointercalation,<sup>6</sup> suggesting that bisintercalation of chromophores joined by flexible chains is condition dependent.

Molecules where the chromophores are held by a rigid framework in a suitable orientation can show an increased propensity for intercalative binding. The quinoxaline chromophores of triostin A are known to both intercalate DNA<sup>8</sup> (although not

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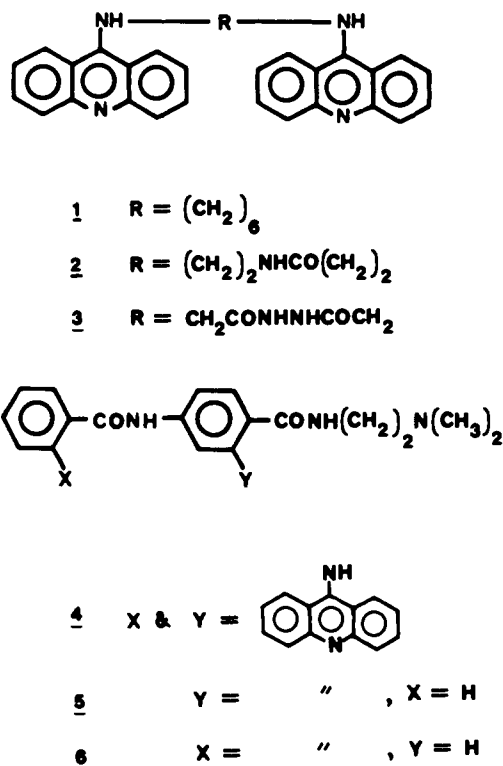
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at contiguous sites), whereas the free chromophores do not bind in this manner.

We now report the synthesis of the bis(acridine) derivative **4**, specifically designed as a relatively rigid molecule in which the two chromophores are essentially coplanar and 7 Å apart in the only reasonably strain-free conformation (as determined using Courtald molecular models), forcing them to bind simultaneously to contiguous DNA sites. The structure of the linker chain was based on the known conformation of 9-anilinoacridines determined by X-ray crystallography.<sup>9,10</sup> The side chain incorporated a cationic charge to enhance water solubility and DNA-binding affinity and was linked to the second aniline ring via a carboxamide function to keep the electronic environment of both acridines as similar as possible, with aqueous  $pK_a$  values of about 7<sup>11</sup> (ignoring proximity effects).

The linker chain of **4** was constructed from 4-amino-2-nitrobenzoic acid. Schotten-Baumann reaction with 2-nitrobenzoyl chloride followed by 1,1'-carbonyldiimidazole-induced coupling with *N,N*-dimethylethylenediamine gave the desired dinitro precursor. Catalytic hydrogenation (Pd/C/MeOH/20 °C), followed by reaction of the resulting diamine with 2 mol of 9-chloroacridine gave **4** as the yellow, crystalline, water-soluble trihydrochloride salt. Obvious modifications of the above scheme provided the model compounds **5** and **6**, bearing only one chromophore.<sup>12</sup>

Figure 1 shows that all three compounds (**4**–**6**) unwind and rewind the supercoils of closed circular supercoiled DNA (*E. coli* plasmid pNZ 116), a necessary criterion for intercalative binding.<sup>1,13</sup> Equilibrium dialysis against calf thymus DNA in 0.01 M acetate buffer at pH 5 showed free drug levels of 0%, 3% and

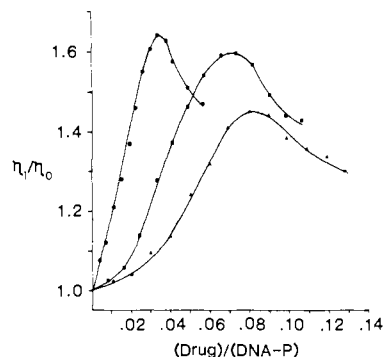


Figure 1. Viscometric titrations of closed circular superhelical DNA with **4** (●), **5** (▲), and **6** (■) at 25 °C in 0.01 M SHE buffer. Results for **5** and **6** are corrected for unbound drug (see text).

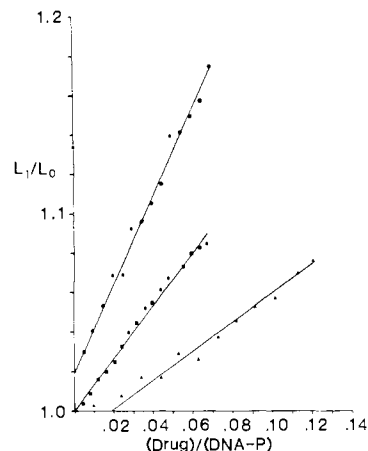


Figure 2. Length increase of sonicated calf thymus DNA by **4** (●), **5** (▲), and **6** (■) at 25.3 °C in 0.01 M SHE buffer. Results for **5** and **6** are corrected for unbound drug (see text).

20% for compounds **4**–**6**, respectively (corresponding to approximate association constants of  $\gg 10^6$ ,  $1.37 \times 10^6$ , and  $1.73 \times 10^5 \text{ M}^{-1}$ ). Using the above free drug levels to correct the drug/DNA phosphate values for the two mono(acridines) **5** and **6**, unwinding angles of 34°, 16°, and 14° were calculated for **4**–**6** respectively (assuming an unwinding angle of 26° for ethidium<sup>1</sup>). These values are consistent with monintercalation for **5** and **6** and bisintercalation for **4**.

A further demonstration of this binding mode for **4** comes from helix extension measurements using short rodlike DNA molecules obtained by sonication (Figure 2).<sup>1</sup> While a theoretical slope of **4** is expected for bisintercalation, plots of  $L_r/L_0$  vs.  $r$  with slopes of greater than 2 are usually acceptable for a putative bisintercalator, provided that this value is also approximately double that of related monointercalators.<sup>2,14</sup> This is the case for compounds **4**–**6**, where a slope of 2.2 for the bis(acridine) **4** should be compared with slopes of 1.3 and 1.1 for the monomers **5** and **6**, respectively.

The above data strongly suggest bisintercalative binding of the bis(acridine) **4**, which must be at contiguous sites on the DNA. Bis(acridine) **4** represents the first molecule of rigid geometry successfully designed to exhibit such binding, which must result in a unique distortion of the DNA substrate.

Further proof of this binding mode was sought by high-field NMR, but complexes of **4** (and of related more water-soluble molecules containing azaacridine chromophores) with defined-sequence oligonucleotides and short, random-sequence DNA were too insoluble to provide useful data.

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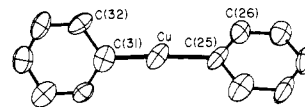
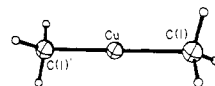
### Isolation and X-ray Crystal Structures of the Mononuclear Cuprates $[\text{CuMe}_2]^-$ , $[\text{CuPh}_2]^-$ , and $[\text{Cu}(\text{Br})\text{CH}(\text{SiMe}_3)_2]^-$

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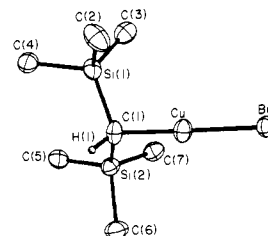
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Lithium diorganocuprates (Gilman reagents,  $\text{LiCuR}_2$ )<sup>1</sup> have been widely used in organic synthesis.<sup>2</sup> Nevertheless, details for their reaction pathways remain unclear, partly because the structures of the solution species have not been completely defined. Earlier <sup>1</sup>H NMR and molecular weight investigations have suggested the presence of species such as  $\text{LiCuR}_2$ ,  $\text{LiCu}_2\text{R}_3$ ,  $\text{Li}_2\text{Cu}_2\text{R}_3$ , or  $\text{Li}_2\text{Cu}_3\text{R}_5$  in ether solution.<sup>3</sup> Recent work by van Koten, Noltes, and co-workers, using the chelating ligand  $2\text{-Me}_2\text{NCH}_2\text{C}_6\text{H}_4^-$ , has resulted in the characterization of the complex  $[\text{Li}_2\text{Cu}_2(2\text{-Me}_2\text{NCH}_2\text{C}_6\text{H}_4)_4]$  in solution and solid phases.<sup>4</sup> Work in this laboratory has shown that aggregates involving lithium and copper atom frameworks with simple aryl substituents, e.g.,  $[\text{Li}_2\text{Cu}_3\text{Ph}_6]^-$ , can be isolated and structurally characterized.<sup>5</sup> Parallel X-ray structural work by Bau et al. has shown that the closely related complexes  $[\text{Cu}_3\text{Ph}_6]^-$  and  $[\text{LiCu}_4\text{Ph}_6]^-$  can also be present in these solutions.<sup>6</sup> Further information has come from the use of very large groups such as  $-\text{C}_6\text{H}_2\text{Me}_3\text{-2,4,6}$  or  $-\text{C}(\text{SiMe}_3)_3$  which has allowed the isolation of the first monomeric organocuprates of formula  $[\text{Cu}\{\text{C}(\text{SiMe}_3)_2\}]^-$ <sup>7</sup> and  $[\text{Cu}\{\text{C}_6\text{H}_2\text{Me}_3\text{-2,4,6}\}]^-$ <sup>8</sup> In these cases it is thought<sup>7</sup> that, because of the very large substituent size, their structures are not representative of the more frequently encountered lithium cuprates such as "LiCuMe<sub>2</sub>" which form polynuclear aggregates in solution. We now report a facile route to simple mononuclear cuprates crystallized as their lithium crown ether salts. These complexes, which have been characterized by



**Figure 1.** Computer-generated drawings of the anions in **1** and **2**. Selected bond distances (Å) and angles (deg) for  $[\text{CuPh}_2]^-$  not given in text: C(25)–C(26) = 1.387 (16); C(26)–C(27) = 1.379 (14); C(27)–C(28) = 1.385 (22); C(28)–C(29) = 1.371 (18); C(29)–C(30) = 1.368 (14); C(25)CuC(31) = 178.5 (4); CuC(25)C(26) = 125.0 (11); CuC(25)C(30) = 122.0 (8); C(25)C(26)C(27) = 123.9 (14); C(26)C(27)C(28) = 120.5 (12); C(27)C(28)C(29) = 118.2 (10); C(28)C(29)C(30) = 120.3 (14); C(29)C(30)C(25) = 124.0 (12). Dihedral angle between the phenyl rings = 47.3 (9)°.



**Figure 2.** Computer-generated drawing of **3**. Bond distances (Å) and angles (deg) not given in text: Cu–Br = 2.267 (2); C(1)–Si(1) = 1.842 (7); C(1)–Si(2) = 1.837 (8); other Si–C distances average ca. 1.88 Å; C(1)CuBr = 178.7 (2).

X-ray diffraction, are  $[\text{Li}(12\text{-crown-4})_2][\text{CuMe}_2]$  (**1**),  $[\text{Li}(12\text{-crown-4})_2][\text{CuPh}_2]\cdot\text{THF}$  (**2**), and the intermediate, monosubstituted species  $[\text{Li}(12\text{-crown-4})_2][\text{Cu}(\text{Br})\text{CH}(\text{SiMe}_3)_2]\cdot\text{PhMe}$  (**3**).

The compounds **1** and **2** were prepared by the addition of 1 equiv of the halide-free organolithium reagent to 1 equiv of CuI in ether at 0 °C. The slurry was stirred for 15 min and the solid isolated by filtration. Suspension in Et<sub>2</sub>O and addition of a second equivalent of MeLi or PhLi gave a clear solution (occasionally slightly colored). Two equivalents of the crown ether were added via syringe and the solid product redissolved in a minimum volume of warm THF. Cooling to –10 °C afforded the products **1** or **2** as colorless crystals in about 50% yield. In the case of the  $-\text{CH}(\text{SiMe}_3)_2$ -substituted compound the only product we were able to characterize was **3** which was isolated as colorless crystals by adding a 1:1 toluene/hexane mixture to the THF solution of **3**.

The structures of **1–3** were determined by X-ray crystallography.<sup>9</sup> Their structures consist of well-separated cations,  $[\text{Li}(12\text{-crown-4})_2]^+$ , and anions,  $[\text{CuMe}_2]^-$ ,  $[\text{CuPh}_2]^-$ , or  $[\text{Cu}(\text{Br})\text{CH}(\text{SiMe}_3)_2]^-$ . The anion structures are illustrated in Figures 1 and 2. In these the copper atom has the rare<sup>7,8,10</sup> mononuclear, two-coordinate configuration with essentially linear geometry at copper. The Cu–C distances in all three anions are fairly uniform, with values of 1.935 (8), 1.925 (10)<sub>av</sub>, and 1.920 (6) Å for **1**, **2**, and **3**, respectively. These compare well with bond lengths in other

(9) Crystal data at 140 K with Mo K $\alpha$  ( $\lambda = 0.71069$  Å, **1** and **3**) or Cu K $\alpha$  ( $\lambda = 1.54178$  Å, **2**) radiation: **1**, triclinic  $P\bar{1}$ ,  $Z = 1$ ,  $a = 7.951$  (4) Å,  $b = 8.237$  (7) Å,  $c = 9.517$  (6) Å,  $\alpha = 88.70$  (6)°,  $\beta = 86.59$  (4)°,  $\gamma = 61.85$  (6)°,  $R = 0.070$  at the present stage of refinement, 90 parameters, 1984 unique observed data. **2**, triclinic  $P\bar{1}$ ,  $Z = 2$ ,  $a = 11.859$  (5) Å,  $b = 12.483$  (12) Å,  $c = 13.634$  (11) Å,  $\alpha = 67.59$  (6)°,  $\beta = 66.54$  (5)°,  $\gamma = 64.61$  (6)°,  $R = 0.081$ , 388 parameters, 2056 unique observed data. **3**, monoclinic,  $P2_1/c$ ,  $Z = 4$ ,  $a = 11.810$  (9) Å,  $b = 23.362$  (19) Å,  $c = 13.881$  (7) Å,  $\beta = 98.21$  (5)°,  $R = 0.054$ , 395 parameters, 3445 unique observed data.

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