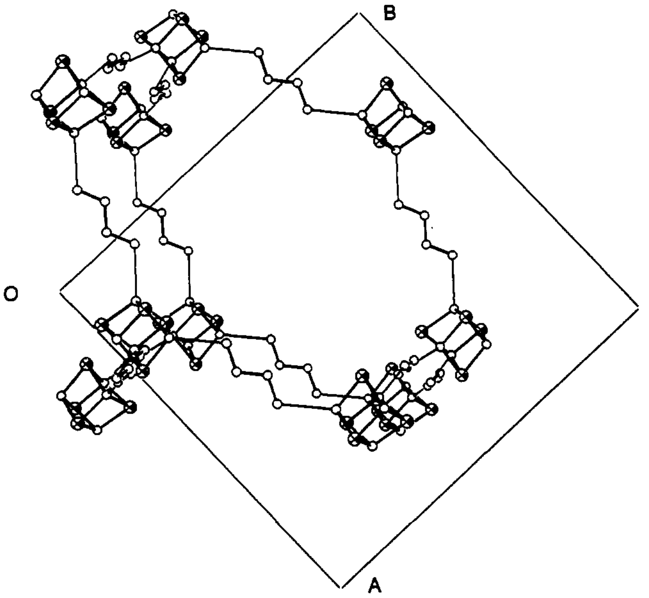

Figure 2. Perspective view of the tertiary structure of **3** viewed down the *c*-axis. For the sake of clarity, an adamantane-like unit of only one of the three networks is illustrated and all hydrogen atoms and carbonyl ligands are omitted.

that **3** exists in the solid state as an undistorted triple diamondoid network of **2** linked by en bridges. O···N and O–H···N distances of 2.774 (8) and 1.94 (6) Å, respectively, O–H···N angles of 165 (5)°, and IR spectroscopy[8] confirm the presence of hydrogen bonds. The O–H···en···H–O linkages therefore serve the same geometric function as C–C bonds in diamond but can be much more readily disassembled and reassembled. The rigid tetrahedral geometry of **2**, which is illustrated in Figure 1, is reinforced crystallographically since **2** lies around a $\bar{4}$ position. The adamantane-like architecture of one of the independent diamondoid networks of **3** is illustrated in Figure 2.

The relative size and shape of **2**, the length of en, and the interpenetration of the networks do not facilitate enclathration of guest molecules; however, the concept of assembly of complementary molecules to generate diamondoid networks is confirmed. A significant feature of this concept (as opposed to self-assembly of identical molecules) is that judicious choice of the bridging H-bond-acceptor moiety should permit rational design of cavity size without extensive synthetic procedures (i.e., breakage or formation of covalent bonds). As noted previously,[3,4] there are numerous potential applications of diamondoid networks. In addition to those already discussed, we point out that molecules such as **2** may be photoreactive[10] and that **2** is not the only cubane cluster with such geometric features.[11] The generality of the approach to construction of diamondoid and other hydrogen-bonded networks outlined herein is currently under further investigation in our laboratory.[12]

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Supplementary Material Available: Crystallographic report, atomic parameters, *U* values, and interatomic distances and angles for **3** (3 pages); listing of observed and calculated structure factors for **3** (4 pages). Ordering information is given on any current masthead page.

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(9) Crystals of **3** belong to the noncentrosymmetric cubic space group $I\bar{4}3d$ (T_d, No. 220) with a = 20.733 (3) Å, V = 8912.2 (13) Å^3, D_{calcd} = 1.66 g cm^{-3}, and Z = 12. Molecules of **2** lie around a $\bar{4}$ position whereas the bridging en molecules lie around 2-fold axes. Full matrix least-squares refinement of all non-hydrogen atoms with anisotropic thermal parameters, the μ_3-OH hydrogen atom with an isotropic thermal parameter, and with all other hydrogen atoms fixed in calculated positions, afforded R = 0.027 and R_w = 0.026. The tertiary structure consists of three independent diamondoid networks based upon molecules of **2** that lie 11.59 Å from each other around corresponding sets of four $\bar{4}$ positions (Set 1: $^7/_8$, 0, $^1/_4$; $^5/_8$, 0, $^3/_4$; $^3/_8$, $^1/_2$, $^3/_4$, $^1/_8$, $^1/_2$, $^1/_4$. Set 2: $^1/_4$, $^7/_8$, 0; $^3/_4$, $^5/_8$, 0; $^3/_4$; $^3/_8$, $^1/_2$; $^1/_4$; $^1/_8$; $^1/_2$. Set 3: 0, $^1/_4$, $^7/_8$; 0, $^3/_4$, $^5/_8$; $^1/_2$, $^3/_4$, $^3/_8$; $^1/_2$, $^1/_4$, $^5/_8$). The network illustrated in Figure 2 is based upon set 2.

(10) Mn_4O_4 cubes are considered a model for one of the "S-states" of PS-II, a water oxidation enzyme: Proserpio, D. M.; Hoffmann, R.; Dismukes, G. C. *J. Am. Chem. Soc.* **1992**, *114*, 4364 and references therein.

(11) There are numerous examples of rigid M_4X_4 cubanes spanning a wide range of metals (including main group) and X moieties. **2** has close analogues in $[Mo(CO)_2(NO)(\mu_3\text{-}OH)]_4$, which also forms a 1:4 adduct with triphenylphosphine oxide (Albano, V.; Bellon, P.; Ciani, G.; Manassero, M. *J. Chem. Soc., Chem. Commun.* **1969**, 1242) and $[Re(CO)_3(\mu_3\text{-}OH)]_4$ (Herberhold, M.; Suss, G.; Ellermann, J.; Gabelein, H. *Chem. Ber.* **1978**, *111*, 2931). X could also be an H-bond acceptor, thereby facilitating assembly of diamondoid networks with rigid tetrafunctional H-bond acceptors and simple difunctional H-bond-donor molecules.

(12) A plethora of potential bridging molecules exists. A double diamondoid network is obtained when **2** is cocrystallized with the bulkier but longer hydrogen-bond acceptor 1,2-bis(diphenylphosphinyl)ethane). The two interweaved diamondoid networks are tetragonally distorted, and the phenyl rings are disordered, resulting in an R value of 0.11. Space group $P4_2/n$, a = 14.290 (5) Å, c = 18.028 (8) Å, V = 3681.4 Å^3, Z = 2. Full details will be published at a later date.

Trisilaallyl Anion Structures. Is Conjugation Effective?

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To what extent can analogies between carbon and silicon species be applied to the trisilaallyl anion $SiH_2SiHSiH_2^-$, **1**? Is the potential resonance energy in **1** sufficient to overcome the inherent nonplanar preferences of the related silicon species? While the methyl anion inversion barrier is only 2.3 kcal/mol at MP2/6-31+G*,[1] the SiH_3^- barrier is an order of magnitude higher (25.9 kcal/mol).[2] Unsaturated carbon species are planar, while the silicon analogs are not. The bent structures of disilaethylene[3] and hexasilabenzene[4] are examples.[4e]

The structure and stabilization energy of the allyl anion (**2**) have been determined both experimentally and theoretically,[5] but

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(1) See: Salzner, U.; Schleyer, P. v. R. *Chem. Phys. Lett.* **1992**, in press and references cited for other values.

(2) Reliable earlier estimations of the SiH_3^- inversion barrier are in the 25.4–26.5 kcal/mol range: (a) Keil, F.; Ahlrichs, R. *Chem. Phys.* **1975**, *8*, 384. (b) Eades, R. A.; Dixon, D. A. *J. Chem. Phys.* **1980**, *72*, 3309. (c) Reed, A. E.; Schleyer, P. v. R. *Chem. Phys. Lett.* **1987**, *133*, 553. (d) Shen, M.; Xie, Ya.; Schaefer, H. F., III. *J. Chem. Phys.* **1990**, *93*, 8098–8104. (e) Hopkinson, A. C.; Rodriquez, C. F. *Can. J. Chem.* **1990**, *68*, 1309–1316.

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(4) (a) Nagase, S.; Kudo, T.; Aoki, M. *J. Chem. Soc., Chem. Commun.* **1985**, 1121. (b) Clabo, D. A.; Schaefer, H. F., III. *J. Chem. Phys.* **1986**, *84*, 1664. (c) Nagase, S.; Teramae, H.; Kudo, T. *J. Chem. Phys.* **1987**, *86*, 4513. (d) Sax, A. F.; Kalcher, J.; Janoschek, R. *J. Comput. Chem.* **1988**, *9*, 564. (e) Apeloig, Y. In *The Chemistry of Organic Silicon Compounds Part 1*; Patai, S., Rappoport, Z., Eds.; Wiley Interscience: New York, 1989; pp 57–225.

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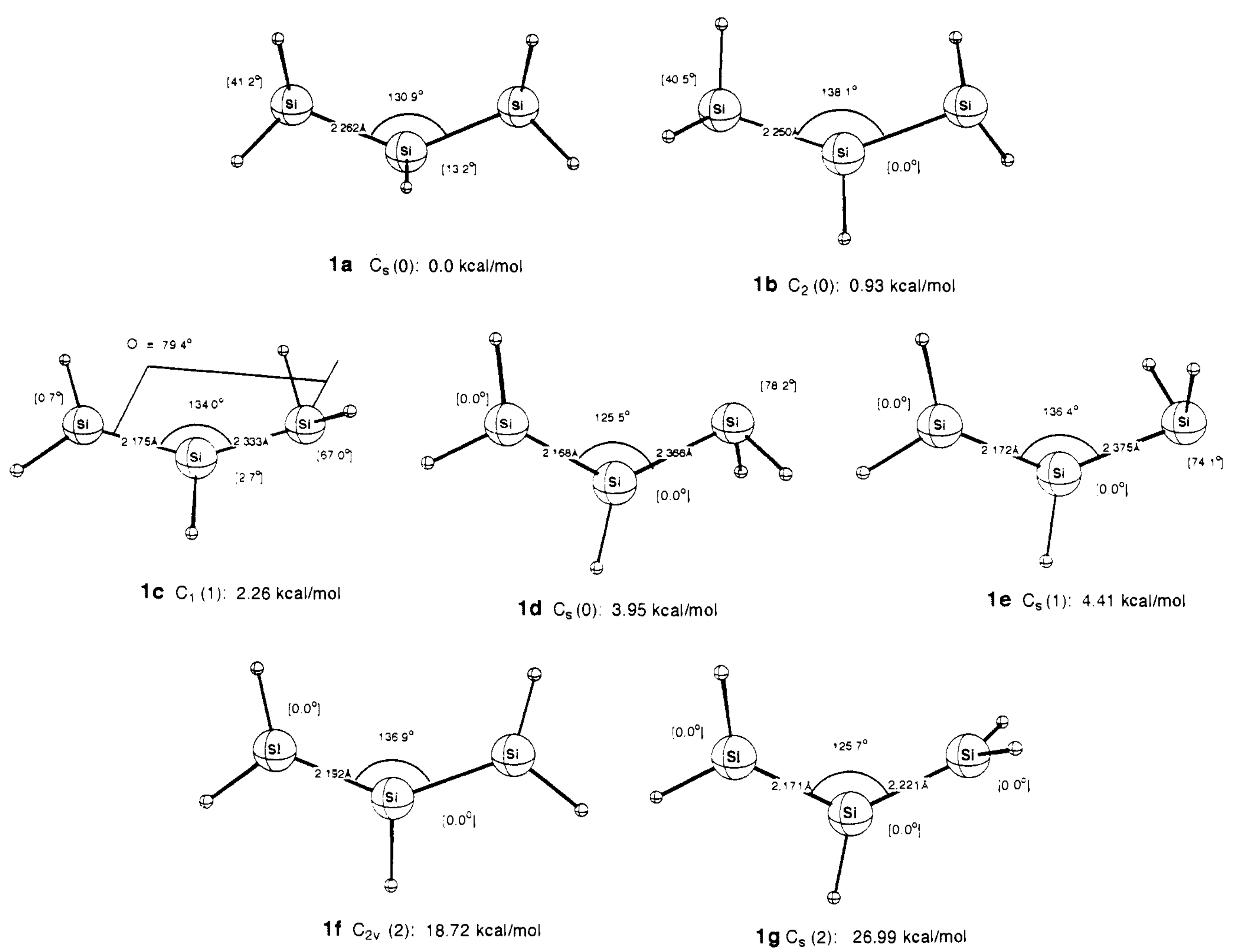

1a C_s(0): 0.0 kcal/mol

1b C_2 (0): 0.93 kcal/mol

1c C_1 (1): 2.26 kcal/mol

1d C_s(0): 3.95 kcal/mol

1e C_s(1): 4.41 kcal/mol

1f C_{2v} (2): 18.72 kcal/mol

1g C_s (2): 26.99 kcal/mol

Figure 1. Trisilaallyl anion stationary point structures and relative energies in kilocalories/mole (MP2(full)/6-31+G*). The degrees of nonplanarity (DNP, see text) are given in square brackets. The numbers of imaginary frequencies (NIMAG) are shown in parentheses.

data for the silicon analogues are still unknown. The recent ab initio CISD/6-31G*//6-31G* study of the trisilaallyl radical[6] prompts us to present an ab initio study of **1** together with data for the reference molecules, SiH_4, Si_2H_6, Si_3H_6, SiH_3^-, and $Si_2H_5^-$, as well as the corresponding carbon analogs. The final geometries of all species were optimized at MP2(full)/6-31+G*,[7a] and the isodesmic[7b] stabilization energies (eqs 1–4) are estimated at this level. Unless specified otherwise, the discussion is based on this data. The frequencies were calculated analytically at HF/6-31G* for all conformers of **1** and at MP2(full)/6-31+G*[8] for the two most stable structures, **1a** and **1b**.

The allyl anion is planar[5] and possesses a substantial rotational barrier (21 kcal/mol at MP2/6-31+G**, via the C_s anti-rotated transition structure). The behavior of **1** is quite different. Seven stationary points (**1a–g**) have been located at MP2/6-31+G*. These are depicted in Figure 1 together with the relative energies (in kilocalories/mole vs **1a**). The degrees of nonplanarity (DNP) at the silicon centers are given by the differences between the sums of valence bond angles and 360°.[9]

The most stable $Si_3H_5^-$ structure, **1a**, has C_s symmetry. All three silicon groups are nonplanar, and the SiSi bond length (2.262 Å) is intermediate between double-bond (2.165 Å in Si_2H_4) and single-bond (2.337 Å in Si_2H_6 and 2.362 Å in $Si_2H_5^-$) values. The SiSiSi bond angle in **1a** (130.9°) is very close to the CCC angle in the C_{2v} allyl anion, **2** (131.5°); both angles are substantially larger than 120°. The second lowest energy minimum, **1b** (C_2), is only 1 kcal/mol less stable than **1a** (0.93 kcal/mol at our standard MP2(full)/6-31+G* level and 1.01 kcal/mol at MP4SDTQ/6-311+G**); the zero-point energy correction reduces this difference to 0.4 kcal/mol. The central silicon in **1b** is planar, and the SiSiSi angle (138.1°) is even 7° greater than that in **1a**.

The **1a** $\rightarrow$ **1b** barrier is only 2.3 kcal/mol; the transition structure, **1c**, lacks symmetry, but retains some conjugation between the lone pair and the double bond. A fully nonconjugated transition structure, **1e** with C_s symmetry, is higher in energy. Rotation of a SiH_2^- group leads to another minimum (**1d**), which also is fully nonconjugated. Note that **1c–e** all possess Lewis structures, $H_2Si{=}SiH{-}SiH_2^-$, with distinctly different SiSi bond lengths.

The planar C_{2v} structure **1f** is of special interest, since it corresponds to the allyl anion minimum, **2**. Remarkably, **1f** has *two* imaginary frequencies and is 18.7 kcal/mol higher in energy than **1a**. As in **1a** and **1b**, the SiSi bonds in **1f** prefer to be equal in length; the vectors of the imaginary frequencies both correspond

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(9) For example, the degree of nonplanarity (DNP) for XH_3 species with 109.47° bond angles is 31.6°; the DNP for SiH_3^- here is 71.0°.

to out-of-plane rotation modes. While the cost in bending energy is considerable, planarization to **1f** results in enhanced π conjugation. However, the extent of this conjugation is underestimated by the rigid rotation barrier via **1g** (8.3 kcal/mol). This value is only a little higher than the relaxed barrier (**1a** → **1e**, 4.4 kcal/mol) and is much lower than the rotational barrier in the allyl anion (ca. 21 kcal/mol).

An entirely different conclusion regarding the magnitude of the conjugation energy in planar $Si_3H_5^-$ is reached by comparison of the stabilization energies of $C_3H_5^-$ (**2**) vs $Si_3H_5^-$ (**1f**) based on the appropriate *planar* XH_3^- (D_{3h}) species (eq 1).

$$X_3H_5^-(C_{2v}) + XH_4 \rightarrow X_3H_6 + XH_3^-(D_{3h}) \qquad (1)$$

34.1 kcal/mol for $Si_3H_5^-$ (**1f**)

30.1 kcal/mol for $C_3H_5^-$ (**2**)

The *larger* stabilization energy of **1f** than **2** (eq 1) shows that charge delocalization in the second row can be as effective as that in the first. However, p–π conjugation in the second row must compete with the strong preference of lone pairs or single electrons (including those comprising double bonds) to occupy orbitals having a high degree of s-character. This preference results, for example, in the nonplanar $Si_3H_5^-$ structures (e.g., **1a**, **1b**) found here (Figure 1).

When eq 1 is reevaluated by employing data for the most stable *nonplanar* structures of SiH_3^- (C_{3v}) and $Si_3H_5^-$ (**1a**) (as well as C_{3v} CH_3^-), the energy comparison with **2** (eq 2) is remarkable: the stabilization energy for $Si_3H_5^-$ (**1a**) is almost as large as that for allyl anion (**2**)! Even if the fully nonconjugated minimum **1d** were substituted for **1a**, the X = C vs Si comparison in eq 2 would give nearly the same energies!

However, when the lowest energy $X_2H_5^-$ (and X_2H_6) species are used as the references (instead of XH_3^- and XH_4), **2** is clearly favored over **1a** (eq 3). The disilaethyl anion, $Si_2H_5^-$, is *stabilized* by its SiH_3 substituent, whereas $C_2H_5^-$ is slightly *destabilized* by the methyl group.[10] This difference is brought out in eq 4. The Si_2H_3 grouping, even in the nonconjugated **1d**, functions similarly to a SiH_3 substituent in stabilizing a SiH_2^- anion. The $Si_3H_5^-$ rotational potential energy surface is relatively flat, because all of the conformations (**1a–e**) benefit to nearly equal extents from different combinations of conjugation, hyperconjugation, and electron delocalization.

$$X_3H_5^- + XH_4 \rightarrow X_3H_6 + XH_3^-(C_{3v}) \qquad (2)$$

26.9 kcal/mol for X = Si (**1a**)

27.8 kcal/mol for X = C (**2**)

$$X_3H_5^- + X_2H_6 \rightarrow X_3H_6 + X_2H_5^- \qquad (3)$$

13.6 kcal/mol for X = Si (**1a**)

31.0 kcal/mol for X = C (**2**)

$$X_2H_5^- + XH_4 \rightarrow X_2H_6 + XH_3^- \qquad (4)$$

13.3 kcal/mol for X = Si

−3.2 kcal/mol for X = C

The trisilaallyl radical (**3**) was found[6] to have a minimum similar to **1a**: all silicons are nonplanar. The stabilization energy of **3** with respect to $Si_2H_5^{\bullet}$ (6.0 kcal/mol; cf. eq 3) is about half the value for **1a** (13.6 kcal/mol). The rotation barrier of **3** (with imposed planarity of the rotating SiH_2 group) also is lower (5.0 kcal/mol) than the corresponding value (8.3 kcal/mol), **1f** → **1g**.

We conclude that the lowest energy structure of the trisilaallyl anion (**1a**) is governed by the inherent nonplanarity of tricoordinated silyl anions rather than by conjugation. When planarity is imposed (eq 1), the stabilization energy of **1f** (relative to planar SiH_3^-) is even larger than that of allyl anion. Hyperconjugation provides substantial stabilization of the nonplanar $Si_3H_5^-$ conformers, as well as $SiH_3SiH_2^-$.

Acknowledgment. This work was supported by the Deutsche Forschungsgemeinschaft, the Fonds der Chemischen Industrie, the Stiftung Volkswagenwerk, the Convex Computer Corporation, and the awards of fellowships by the Alexander von Humboldt Foundation to A.A.K.

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Aminobenzannulation via Metathesis of Isonitriles Using Chromium Carbene Complexes

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The Dötz benzannulation reaction[1] based on alkyne cycloaddition to chromium carbene complexes is the most important reaction of Fischer carbene complexes.[2] Numerous advances have been achieved in application of the Dötz reaction to total synthesis projects[3] and in further development of benzannulation reactions based on Fischer carbene complexes.[4] However, a long-standing problem has been development of general annulation reactions which incorporate triple bonds other than carbon–carbon triple bonds for the synthesis of heterosubstituted benzene derivatives from chromium carbene complexes. Although reactions utilizing phosphaalkynes successfully generate phosphaarenes,[5] use of nitriles or isonitriles fails in a general sense to produce pyridine or aminobenzene derivatives.[6,7] Typical reactions of carbene complexes with nitriles led to imino carbene complexes,[8] while isonitriles provide, initially, metal complexed ketenimines,[9] then

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