not reliable for such small energy differences, but the results do illustrate an alternative mode by which triplet ground states can, in principle, be obtained.

Introduction of the C2–C3 bond as a through-bond coupling unit in **9** once again causes each GVB orbital to incorporate that element in an antibonding way (Figure 5). This induces a small contribution from the far centers, which are included in such a way as to minimize one-electron energies. Since there is no significant through-space overlap, the net result can only be an increase in $|S_{lr}|$. It is worth noting that the sign of the overlap between the orbitals, as drawn in Figure 5, is positive, even though the overlap at the C2–C3 bond is negative. Evidently, the most important overlap occurs at the "ends" of the biradical, just as in the case of trimethylene (**4**). Because of the enhanced overlap from through-bond coupling in **9**, the system prefers the singlet state by 0.53 kcal/mol at the present level of theory. A recent study at the same level of theory but with an STO-3G basis set found that **9** has the largest singlet–triplet energy gap of any of the idealized forms of *trans*-tetramethylene.[19] The (0,90) form, in which the two p orbitals are orthogonal, has a very small preference for the triplet state, for reasons analogous to Hund's rule.[19] The (0,0) form, on the other hand, shows a very small preference for the singlet[19] because the ethano bridge is a very weak through-bond coupler.

Conclusion

It is now clear which factors are necessary for a simple biradical to have a triplet ground state. From eq 7, the most general requirement is that $|S_{lr}|$ must be significantly smaller than K_{lr}. This can only be accomplished when S_{lr} is diminished by a cancellation of positive and negative regions of overlap,[29] rather than by a general reduction in overlap throughout all regions of space (as in **3** for long distances, R). The cancellation may come about in a variety of ways. It may be forced by symmetry as in *trans*-(0,90)-tetramethylene. Alternatively, it may result from "accidental" nodal properties that have nothing to do with symmetry, as in the through-space interactions in **9**. Finally, the cancellation may be caused by a precise balancing of through-bond and through-space interactions (**1**, **4**, and **6**).

Closs' biradical (**1**) falls into the third category and is thus a triplet due to a fortuitous balance of through-bond and through-space effects. The results of the present work would seem to significantly restrict the class of biradicals that will be observable under the conditions of the Closs experiment. Prime candidates still include **6** and related trimethylene derivatives.

In another connection, Doubleday has recently speculated that for localized (1,n) biradicals derived from Norrish type I cleavage of cycloalkanones, the number of intervening σ bonds may influence ΔE_{S-T}.[12] While we have not studied any structures that are directly relevant to Doubleday's work, our results do support the general notion since the effectiveness of through-bond coupling should depend upon the number of intervening bonds. Note that for extended conformations of such structures, which would have very small through-space effects, through-bond interactions can only act to favor a singlet ground state.

Acknowledgment. We gratefully acknowledge the National Science Foundation (CHE-8024664) for support of this work. This work made use of the Dreyfus–NSF Theoretical Chemistry Computer, which was funded through grants from the Camille and Henry Dreyfus Foundation, the National Science Foundation (CHE-7820235), and the Sloan Fund of the California Institute of Technology. We especially thank Professor W. A. Goddard for helpful discussions and for access to the MQM library of programs. We are also indebted to John J. Low and Arthur F. Voter for their technical assistance. We also thank a referee for helpful comments.

Registry No. 1, 56564-28-4; **3**, 2229-07-4; **4**, 32458-33-6; **9**, 30967-48-7.

Communications to the Editor

Substituent Effects on Tin-119 Chemical Shifts in 4-Substituted Bicyclo[2.2.2]octyl- and Bicyclo[2.2.1]heptyl-1-trimethylstannanes

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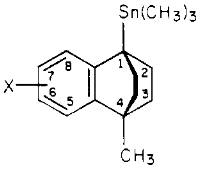
Tin-119 NMR chemical shifts are very reponsive to structural changes and hence can provide valuable insights into organotin structures, reactions, etc.[1-3] Despite this sensitivity, the direction of any chemical shift response to a tactical structural change is a priori difficult to predict, and in general the understanding of heavy-metal chemical shifts is poor.[4] In a previous study,[5] we observed that remote 6- and 7-substituents in 1-(trimethylstannyl)-4-methyl-1,4-ethano-1,2,3,4-tetrahydronaphthalenes (system **1**), in which direct π-type transmission mechanisms are prohibited, had a surprisingly large influence on the ^{119}Sn chemical shift, and we undertook to examine simpler rigid systems, so that field, through-bond, and other mechanisms for substituent–probe interactions could be assessed for the ^{119}Sn nucleus. A range of 4-substituted bicyclo[2.2.2]octyl- and bicyclo[2.2.1]heptylstannanes of types **2** and **3** have now been characterized,[6] and we



1

2, Y = $Sn(CH_3)_3$
4, Y = F

3, Y = $Sn(CH_3)_3$
5, Y = F

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(6) New compounds have been characterized by ^{1}H, ^{13}C and ^{119}Sn NMR and mass spectra and elemental analyses (C, H). All compounds have been prepared by treating the appropriate 4-substituted bicyclo[2.2.2]oct-1-yl iodides with (trimethyltin)lithium in tetrahydrofuran in the standard way. A full description of these syntheses will be presented later in a main writeup.

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Table I. ^{119}Sn Substituent Chemical Shifts[a,b] (SCS) of System 2: Polar-Field ($\rho_I\sigma_I$) and Residual Contributions

	SCS			$\rho_I\sigma_I$[d]		residual[e]	
substituent (X)	$CDCl_3$	c-C_6H_{12}	ΔSCS[c]	$CDCl_3$	c-C_6H_{12}	$CDCl_3$	c-C_6H_{12}
CN	10.00	9.19	0.81	3.92	3.16	6.08	6.03
F	11.67	10.83	0.84	2.94	2.09	8.73	8.74
Cl	10.35	9.66	0.69	3.01	2.30	7.34	7.36
Br	11.67	10.95	0.72	3.08	2.36	8.59	8.59
I	14.08	13.37	0.71	2.94	2.25	11.14	11.12
$N(CH_3)_2$	6.17	5.11	1.06	1.26	0.54	4.91	4.57
OCH_3	8.76	7.65	1.11	1.82	1.02	6.94	6.63
C_6H_5	4.90	4.46	0.44	1.19	0.80	3.71	3.66
p-FC_6H_4	5.32	5.05	0.27	1.61	1.39	3.71	3.66
CH_3	3.40	3.41	−0.01	0	0	3.40	3.41
$C(CH_3)_3$	2.04	1.89	0.15	0	0	2.04	1.89
$Sn(CH_3)_3$	−5.53	−5.90	0.37	0	0	−5.53	−5.90

[a] Chemical shifts (ppm) relative to parent system (2, X = H). Accurate to ±0.02 ppm. [b] ^{119}Sn spectra were obtained on a JEOL FX-100 spectrometer operating at 37.70 MHz for ca. 0.2 M solutions in deuteriochloroform and cyclohexane, with $(CH_3)_4Sn$ (TMT) as internal standard. The probe temperature was 295 ± 2 K. Relative to TMT, 2, X = H, has a shift of +0.46 ppm ($CDCl_3$) and +0.16 ppm (c-C_6H_{12}). A positive sign implies deshielding. [c] SCS ($CDCl_3$) − SCS (c-C_6H_{12}). [d] σ_I values taken from ref 7. ρ_I values for $CDCl_3$ and c-C_6H_{12} are 7.00 and 5.36, respectively (see text). [e] (Observed ^{119}Sn SCS) − $\rho_I\sigma_I$ in ppm.

draw attention to their ^{119}Sn substituent chemical shifts (SCS) and likely modes of substituent action. Some remarkable comparisons with related systems are also presented.

The ^{119}Sn SCS for system **2** along with a dissection into polar-field and "residual" contributions are presented in Table I and confirm our suggestion[5] that ^{119}Sn shifts are surprisingly sensitive to polar substituent influences. Note the range of some 17 ppm between the most electropositive ($Sn(CH_3)_3$) and electronegative (F) substituents, such range being greater than the corresponding one for ^{19}F SCS (system **4**, 14.15 ppm for $CDCl_3$ solvent).[7] In broad terms, the directions of the shifts accord with the primitive idea that electron-withdrawing polar substituents induce downfield shifts. Correlative analyses of SCS values with electric field parameters (σ_I)[7] were unsatisfactory (r = 0.86 ($CDCl_3$), 0.82 c-C_6H_{12}), as observed with the 4-substituted bicyclo[2.2.2]oct-1-yl fluorides (**4**).[7] Polar susceptibility parameters (ρ_I); 7.00 for $CDCl_3$ and 5.36 for c-C_6H_{12}) may be determined independently by dividing the chemical shift difference between **2**, X = *p*-FC_6H_4, and **2**, X = C_6H_5 (0.42 ppm ($CDCl_3$), 0.59 ppm (c-C_6H_{12})) by $\Delta\sigma_I$ for *p*-FC_6H_4 and C_6H_5 (0.06 for $CDCl_3$, 0.11 for c-C_6H_{12}).[8] Accepting the idea[9] that the electric field effect on NMR screening constants can be ascribed to differential polarization of the bonds about the magnetic nucleus, the significant polar susceptibility parameters imply greater polarization of the Sn–C_1 bond vs. Sn–CH_3 bonds. Factorization of the SCS (Table I) demonstrates that although the polar-field term ($\rho_I\sigma_I$) is significant, it is *not* the dominant factor regulating the ^{119}Sn shifts in **2**. Furthermore, the close agreement in "residual" values for both solvents requires that the solvent effect (ΔSCS, Table I) is embodied in the polar field term.

Our suspicion that "through-bond" and/or "through-space" electron delocalization[10] may be responsible for the large "residual" (Table I) encouraged examination of system **3**, for which a change in the blend of possible through-bond and through-space effects on physicochemical parameters has been demonstrated.[10-12] This suspicion was supported by the relative magnitudes of the "residuals" for ^{119}Sn SCS (**2**) vs. ^{19}F SCS (**4**)[7] as substantial differences in the "mix" of through-bond and through-space effects for these probes would be reasonable[10-12] (see below). The available SCS results for system **3** ($CDCl_3$) (X = OCH_3, −3.10; F, −1.83; I, −3.33; $Sn(CH_3)_3$, +2.52)[13] are dramatically different. Note the change in sign and magnitude when these are compared with data for **2**. Given that the polar-field terms for **2** and **3** (rough constancy of angle and distance) will be similar in magnitude and sign, the conclusion follows that the residual contributions in **3** are *opposite* in sign to those (Table I) for **2**. The evidence is that "through-three-bond" delocalization is antagonistic[11,12] to both "through-two-bond" (as in **3**) and "through-space" interactions. Accepting that the former is more important in **2**,[10] the results for ^{119}Sn shifts are sensible only if the through-space effect is *shielding* and the through-bond effect is *deshielding*. In contrast, a recent comparison[14] for these two bicycloalkane systems (with ^{19}F probe, systems **4** and **5**) demonstrated *shielding through-three-bond* and *deshielding through-space* contributions. A further manifestation of a change in the "mix" of effects concerns the striking variations in long-range coupling constants (Hz, $CDCl_3$)[15] for **2** and **4** vs. **3** and **5**, respectively ($^5J_{^{119}Sn-^{19}F}$ = 74.5 (**4**), 6.6 (**5**); $^5J_{^{119}Sn-^{119}Sn}$ = 120 (**2**), and 20.7 (**3**)).

Some qualitative understanding of these apparently puzzling shift variations can be achieved by considering the dominant orbital interactions governing the electron-delocalization mechanisms. A scrutiny of pertinent molecular orbital parameters (orbital coefficients, resonance integrals, and energy differentials)[16] suggests that for an *electronegative* substituent (X) the dominant through-three-bond orbital interactions must be σ^*_{CF}–σ_{CC}–σ^*_{CX} (for ^{19}F) and σ_{CSn}–σ^*_{CC}–σ_{CX} (for ^{119}Sn). Thus, utilizing the information that a *decrease* in σ^*_{CF}–π(benzene) (e.g. electron-withdrawing para substituent in benzyl fluoride) results in an *upfield shift*[17] and an *increase* in σ_{CSn}–π^*(benzene) (e.g., electron-withdrawing para substituent in benzyltrimethylstannane) leads to a *downfield* shift,[5] we conclude that the observed *upfield* (for ^{19}F) and *downfield* (for ^{119}Sn) through-three-bond contributions for electronegative substituents (see above) is understandable in terms of a *decrease* and *increase* in σ^*_{CF}–σ_{CC} and σ_{CSn}–σ^*_{CC}, respectively, as a result of an *increase* and *decrease* in σ_{CC}–σ^*_{CX} and σ^*_{CC}–σ_{CX}, respectively (i.e., hyperconjugative charge-transfer effects). The likely dominant through-space in-

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(13) Relative to $(CH_3)_4Sn$ (TMT), **3**, X = H, has a shift of +4.50 ppm ($CDCl_3$) and +4.20 ppm (c-C_6H_{12}).

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teractions are σ^*_{CF}–σ^*_{CX} (for ^{19}F) and σ_{CSn}–σ^*_{CX} (for ^{119}Sn); however, we do not have a simple interpretation of their mode of action on the shifts since there are no model systems from which parallels can be drawn. It is possible that these orbital interactions may significantly govern the average excitation energy term (ΔE) in the expression for the dominant paramagnetic contribution to the shifts of both probe nuclei ($\Delta E = \sigma_{CF} \rightarrow \sigma^*_{CF}$ (for ^{19}F) and $\sigma_{CSn} \rightarrow \sigma^*_{CSn}$ (for ^{119}Sn)).[18] The different relative magnitude of the "residual" contributions for both probes (^{19}F[7] and ^{119}Sn) in the bicyclo[2.2.2]octane ring system probably reflects the different nature of the orbital interactions underlying the electron-delocalization mechanisms for both probes as well as their mix.

These and additional features of this work will be reported in full at a later date.

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Registry No. 2 (X = CN), 84010-79-7; **2** (X = F), 78385-88-3; **2** (X = Cl), 84010-80-0; **2** (X = Br), 84010-81-1; **2** (X = I), 84010-82-2; **2** (X = N(CH$_3$)$_2$), 84010-83-3; **2** (X = OMe), 84010-84-4; **2** (X = C$_6$H$_5$), 68756-27-4; **2** (X = *p*-FC$_6$H$_4$), 68756-25-2; **2** (X = CH$_3$), 84010-85-5; **2** (X = C(CH$_3$)$_3$), 84010-86-6; **2** (X = Sn(CH$_3$)$_3$), 84010-87-7; **3** (X = OCH$_3$), 84010-88-8; **3** (X = F), 84010-89-9; **3** (X = I), 84010-90-2; **3** (X = Sn(CH$_3$)$_3$), 84010-91-3.

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[18] As X becomes more electronegative, both the σ^*_{CF}–σ^*_{CX} and σ_{CSn}–σ^*_{CX} orbital interactions will *increase* and, hence, lead to a decrease and increase, respectively, in ΔE for ^{19}F and ^{119}Sn chemical shifts, i.e., downfield (for ^{19}F) and upfield (for ^{119}Sn) contributions for the through-space effect, as observed.

Oxidative Additions to (W≡W)$^{6+}$ Centers: The Influence of Lewis Base Association Reactions

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Oxidative-addition reactions have been extensively studied in mononuclear organometallic chemistry since they represent one of the key steps in many catalytically important reactions.[1] Oxidative addition occurs to coordinatively unsaturated metal centers, and thus Lewis base association or dissociation steps may influence the overall rate of reaction. This is well exemplified by the studies of molecular hydrogen addition to Wilkinson's compound, RhCl(PPh$_3$)$_3$[2] and is generally true for oxidative-addition reactions involving phosphine complexes of the group 8 transition elements.[3] We here show that a parallel situation occurs in dinuclear transition-metal chemistry.

(1) W$_2$(NMe$_2$)$_6$ (M≡M) has been previously shown[4] to react with *i*-PrOH to give the tetranuclear complex W$_4$(μ-H)$_2$(O-*i*-Pr)$_{14}$. The reaction was proposed to proceed in three stages: (i) alcoholysis, W$_2$(NMe$_2$)$_6$ + *i*-PrOH → W$_2$(O-*i*-Pr)$_6$ + HNMe$_2$; (ii) oxidative addition, W$_2$(O-*i*-Pr)$_6$ + *i*-PrOH → W$_2$(H)(O-*i*-Pr)$_7$ (M═M); (iii) dimerization by RO bridge formation, 2W$_2$(H)(O-*i*-Pr)$_7$ → W$_4$(H)$_2$(O-*i*-Pr)$_{14}$. We have now found that W$_2$(O-*i*-Pr)$_6$(HNMe$_2$)$_2$ can be isolated from the reaction between W$_2$(NMe$_2$)$_6$ and *i*-PrOH in hexane at 0 °C as a yellow crystalline compound.[5] The molecular structure deduced from an X-ray study[6] shows that the HNMe$_2$ ligands complete roughly square-planar coordination sites at each tungsten. The overall W$_2$O$_6$N$_2$ moiety has virtual C$_2$ symmetry, and the two WO$_3$N units are nearly eclipsed as a result of hydrogen bonding, N–H···OR, across the W≡W bond. ^{1}H NMR studies show that below 0 °C in toluene-*d*$_8$ the HNMe$_2$ ligands are tightly bound, and the ^{1}H NMR spectra are consistent with expectations based on the solid-state molecular structure. At ambient temperatures, the HNMe$_2$ ligands are labile according to the generalized equation (1) and may readily be exchanged for ligands that coordinate more strongly such as pyridine, PMe$_3$, and Me$_2$PCH$_2$CH$_2$PMe$_2$.[7] However, at 0 °C the HNMe$_2$ ligands are sufficiently tightly bound to prevent reaction with excess *i*-PrOH.

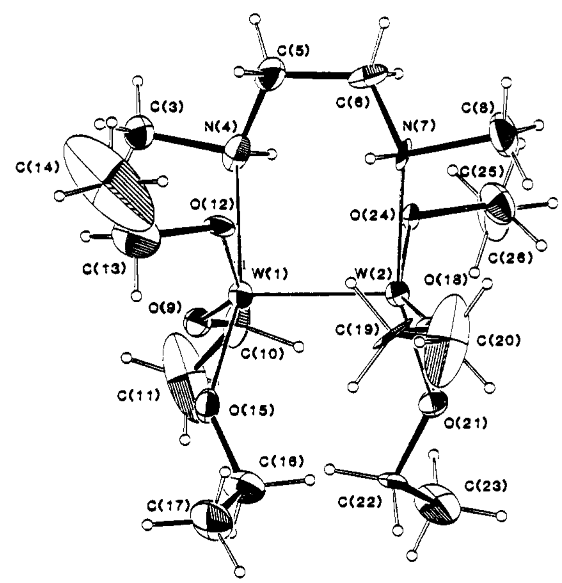

Figure 1. ORTEP view of the W$_2$(OEt)$_6$(Me(H)NCH$_2$CH$_2$N(H)Me) molecule. Atoms are represented as ellipsoids drawn to include 20% probability of thermal displacement. Some pertinent bond distances (Å) and angles (deg) are as follows: W(1)–W(2) = 2.296 (2), W(1)–O(9), –O(12), –O(15), –N(4) = 1.97 (2), 1.96 (2), 1.88 (2), 2.26 (2); W(2)–O(18), –O(21), –O(24), –N(7) = 1.89 (2), 1.90 (2), 1.95 (2), 2.31 (2); W(2)–W(1)–O(9), –O(12), –O(15), –N(4) = 106.0 (5), 101.2 (5), 106.8 (5), 90.0 (5); W(1)–W(2)–O(18), –O(21), –O(24), –N(7) = 104.1 (5), 106.9 (5), 98.7 (4), 91.3 (4).

$$W_2(OR)_6L_2 \rightleftharpoons W_2(OR)_6 + 2L \tag{1}$$

(2) W$_2$(NMe$_2$)$_6$ (M≡M) has been shown[8] to react with EtOH and MeOH according to eq 2. The tetranuclear complex may

$$2W_2(NMe_2)_6 + 16ROH \rightarrow W_4(OR)_{16} + 2H_2 + 12HNMe_2 \tag{2}$$

be viewed as the dimerized product of W$_2$(OR)$_8$ (M═M).[9] Irrespective of the detailed pathway of the formation of W$_4$(OEt)$_{16}$, tungsten has been oxidized from oxidation state 3+ to 4+ in reaction 2.

In marked contrast, when W$_2$(NMe$_2$)$_6$ is allowed to react with EtOH in hexane in the presence of Me(H)NCH$_2$CH$_2$N(H)Me at room temperature, oxidative addition does not occur and the

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