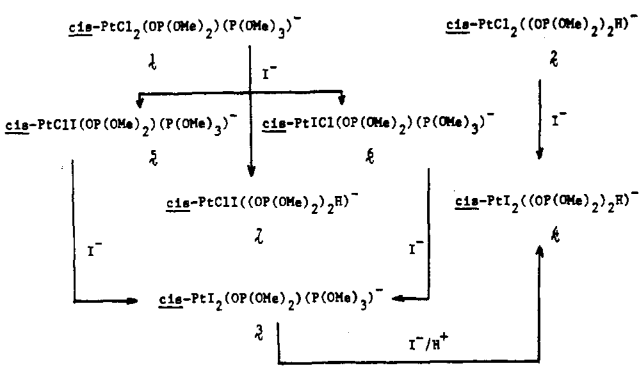
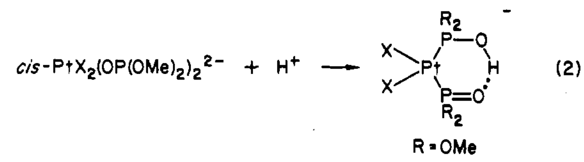
Scheme I



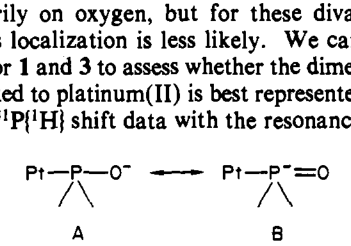
in the reaction mixture to give the observed monoanionic product complexes **2** and **4** (eq 2).[8] We do not observe a resonance due



to the single acidic proton at the expected large downfield shift position, but such resonances are frequently unobserved in these complexes due to exchange.[7]

If halide nucleophiles other than those complexed to platinum are used, mixed-halo complexes are obtained (Scheme I). These complexes have been characterized by ^{31}P{^{1}H} NMR spectroscopy. We cannot, however, assign the stereochemistries of complexes **5** and **6** with any degree of certainty.

In practice, therefore, the route to prepare bifunctional ligands through the dianionic complex is not a viable one for the assemblage of heterobimetallic complexes. For the P-bonded phosphinito ligand of molybdenum(0) it is apparent that the negative charge resides primarily on oxygen, but for these divalent-platinum complexes this localization is less likely. We can now use the spectral data for **1** and **3** to assess whether the dimethyl phosphito ligand complexed to platinum(II) is best represented by tautomer A or B. The ^{31}P{^{1}H} shift data with the resonance for the com-



plexed dimethyl phosphito ligand at δ 30.4 (**1**) and δ 36.8 (**3**) show unambiguously that the phosphonato tautomer B is the canonical form that corresponds with this large upfield shift.[1] Thus in these platinum(II) complexes the negative charge is not localized on oxygen. The changes in δ(^{195}Pt) are small, as are changes in δ(^{1}H) and δ(^{13}C). This suggests that no significant changes occur in the shielding density at the Pt nucleus.[9] In support of this premise, we find that treating complex **3** with Ph$_2$PCl gives impure products that show, by ^{31}P{^{1}H} NMR spectroscopy, substitution at platinum by the phosphine. It is apparent therefore that Wong's procedure cannot be used with platinum(II) complexes to effect template syntheses of bridging *P,P*-ligands.

Registry No. **1**, 101997-95-9; **2**, 101997-97-1; **3**, 101997-99-3; **4**, 101998-01-0; **5**, 101998-02-1; **6**, 102129-57-7; **7**, 101998-03-2; *cis*-PtCl$_2$(P(OMe)$_3$)$_2$, 28374-51-8; *cis*-PtI$_2$(P(OMe)$_3$)$_2$, 28374-52-9; P(OMe)$_3$, 121-45-9; Ph$_4$AsCl, 507-28-8; Ph$_4$AsI, 7422-32-4.

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(8) The hydroxylated complex is formed even when dried acetonitrile solvent is used in the reaction. Under these experimental conditions the hydrogen source is water in Ph$_4$AsX, since we find by ^{1}H NMR spectroscopy that vacuum-dried samples of purified salt contain small quantities of water.

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Synthesis and Properties of (Ethylenebis(dithiocarbamato))diorganotin(IV) Complexes. Structure of [(*t*-Bu)$_2$Sn(ebdtc)]$_2$·4THF

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Interest in dithiocarbamate complexes of diorganotin(IV) species[1-5] arises in part because of their varied structures[6-14] and because of their biological activity.[15,16] Crystallographic studies of these complexes[6-9] have revealed a variety of coordination geometries about the Sn atom, ranging from tetrahedral to distorted octahedral, as well as dithiocarbamate ligands that are either anisobidentate[8,9] or monodentate.[6,7] In an effort to extend this chemistry we have prepared the first organotin(IV) complexes of the ethylenebis(dithiocarbamate) ligand $^-$S$_2$CNHCH$_2$CH$_2$NHCS$_2$$^-$, (ebdtc), a ligand that is potentially tetradentate. The sodium salt of ebdtc displays biological activity a few times greater than that of the sodium salt of dimethyldithiocarbamic acid.[16] Here we report the syntheses and properties of diphenyl- and di-*tert*-butyltin(IV) complexes of ebdtc together with a crystal structure of the di-*tert*-butyltin(IV) complex.

Experimental Section

All preparations were performed under a dinitrogen atmosphere with the use of standard Schlenkware techniques. Reagent grade chemicals were used without further purification. Chemical analyses were carried out by the Chemical Analysis Laboratory at KAIST. Mass spectra were recorded on a Varian Model 212 GC-MASS system (THF solution). ^{1}H-NMR spectra were recorded on fresh samples in Me$_2$SO-d_6 on a JEOL JNM-PMX 60 or a Varian FT-80A spectrometer with Me$_4$Si as the reference. The infrared spectra in the 300–4000-cm^{-1} region were measured on KBr pellets with a Perkin-Elmer 283B spectrophotometer.

Preparation of the ebdtc Ligand. A solution of Na$_2$(ebdtc) was prepared in the following manner:[17] to 3.0 g of 1,2-diaminoethane dissolved in 30 g of H$_2$O was added 8.0 g of CS$_2$ slowly with stirring. The solution was maintained at 35 °C and was slowly neutralized by addition of concentrated NaOH solution. To this reaction mixture was added ethanol until crystals started to form, and the mixture was left overnight. The white crystalline solid thus obtained was recrystallized from a H$_2$O–EtOH solvent pair (1:2), to produce the hexahydrate in 79% yield. Mp: 82 °C. Anal. Calcd for C$_4$H$_6$N$_2$Na$_2$S$_4$·6H$_2$O: C, 13.16; H, 4.94; N, 7.69; CS$_2$, 41.7. Found: C, 13.50; H, 4.90; N, 7.72; CS$_2$, 41.7.

Preparation of (*t*-Bu)$_2$Sn(ebdtc). Na$_2$(ebdtc) (3 mmol) in 100 mL of ethanol was added dropwise to an equimolar solution of di-*tert*-butyltin dichloride in 100 mL of ethanol. After the reaction mixture was cooled to about 0 °C, the NaCl formed was removed by filtration. The filtrate was condensed to approximately 100 mL with a rotary evaporator, and then *n*-hexane was added slowly to the filtrate to precipitate the product. The resultant off-white solid was recrystallized from a benzene–petroleum ether (1:1) solvent pair. The monobenzene-solvate crystalline solid (mp 189 °C) was obtained in 72% yield. ^{1}H NMR (δ): 1.5 (s, *t*-Bu), 3.5 (s, CH$_2$), 7.3 (s, Ph), 10.1 (br, NH). IR (cm^{-1}): ν(C–N), 1525 (s), 1505 (s); ν(C–S), 975 (s), 950 (sh); ν(Sn–S), 390 (s). Anal. Calcd for C$_{12}$H$_{24}$N$_2$S$_4$Sn·C$_6$H$_6$: C, 41.43; H, 5.75; N, 5.37; Sn, 22.8. Found: C, 41.20; H, 5.83; N, 5.13; Sn, 22.5.

When recrystallization is performed in a THF–*n*-hexane solvent pair, the THF-solvated product (*t*-Bu)$_2$Sn(ebdtc)·2THF is obtained. ^{1}H NMR (δ): 1.5 (s, *t*-Bu), 1.8 (qui, THF), 3.5 (s, CH$_2$), 3.7 (t, THF), 10.1 (br, NH). IR (cm^{-1}): ν(C–N), 1520 (s), 1495 (s); ν(C–S), 975 (s), 950 (sh); ν(Sn–S) 390 (s). The composition of [(*t*-Bu)$_2$Sn(ebdtc)]$_2$·4THF was established in the X-ray study.

Preparation of Ph$_2$Sn(ebdtc). To a solution of diphenyltin(IV) dichloride (3 mmol) in 100 mL of ethanol was added an equimolar ethanol solution of Na$_2$(ebdtc). After removal of NaCl and concentration of the filtrate, *n*-hexane was added. The resultant solid was recrystalized from a THF–petroleum ether pair (1:3) to yield the pale yellow product (mp 113 °C) in 78% yield. ^{1}H NMR (δ): 3.5 (s, CH$_2$), 7.3 (m, Ph), 7.8 (br,

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[‡] Northwestern University.

0020-1669/86/1325-2273$01.50/0

Table I. Crystal Data and Experimental Details for $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$

formula	$C_{40}H_{80}O_4S_8Sn_2$
fw	1175.0
a, Å	15.892 (7)
b, Å	11.228 (5)
c, Å	16.295 (8)
β, deg	111.44 (2)
V, Å^3	2706
Z	2
d_{calcd}, g/cm^3	1.441 (112 K)[a]
space group	C_{2h}^5-$P2_1/n$
cryst shape	crystal bounded by faces {10$\bar{1}$}, {011}, and (00$\bar{1}$); max dimens 0.13 × 0.2 × 0.2 mm
cryst vol, mm^3	0.0058
radiation	graphite-monochromated Mo Kα ($\lambda(K\alpha_1) = 0.7093$ Å)
μ, cm^{-1}	12.61
transmissn factors	0.803–0.887 (analytical method)
take-off angle, deg	2.0
receiving aperture, mm	6.0 × 6.0, 33 cm from crystal
scan speed, deg in 2θ/min	2.0
scan width, deg	0.85 below Kα_1 to 0.85 above Kα_2
bkgd counts	10 s with rescan option[b]
data collcd	$\pm h,+k,+l$; $3.4 \leq 2\theta \leq 55.0°$
no. of unique data	6090
p for calculation of $\sigma(F_o^2)$	0.03
no. unique data with $F_o^2 \geq 3\sigma(F_o^2)$	3467
no. of variables	262
R on F_o^2	0.131
R_w on F_o^2	0.147
R on F_o ($F_o^2 > 3\sigma(F_o^2)$)	0.069
R_w on F_o ($F_o^2 > 3\sigma(F_o^2)$)	0.069
error in observn unit of weight, e^2	1.38

[a] The low-temperature system is based on a design by: Huffman, J. C. Ph.D. Thesis, Indiana University, 1974. [b] The diffractometer was run under the disk-oriented Vanderbilt system: Lenhert, P. G. *J. Appl. Crystallogr.* **1975**, *8*, 568–570.

Ph), 7.9 (br, NH). IR (cm^{-1}): ν(C–N), 1520–1500 (br); ν(C–S), 980 (s), 960 (s); ν(Sn–S), 370 (s). Anal. Calcd for $C_{16}H_{16}N_2S_4Sn$: C, 39.76; H, 3.33; N, 5.79; Sn, 24.5. Found: C, 38.77; H, 3.28; N, 6.01; Sn, 23.5.

Attempted Preparations of $R_2Sn(ebdtc)$, R = Me or Et. When Me_2SnCl_2 or Et_2SnCl_2 was used in place of Ph_2SnCl_2, similar products were obtained but the materials could not be obtained in pure form through recrystallization apparently because they are unstable in solution.

Crystallographic Study of $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$. A suitable crystal was removed from the mother liquor and inserted immediately into the cold stream (112 K) of a Picker FACS-1 X-ray diffractometer, as the crystals decompose rapidly owing to loss of solvent. No significant change in the intensities of six standard reflections, monitored every 100 reflections during data collection, was observed. Hence the crystal was stable in the nitrogen cold stream. Crystal data and other details are given in Table I.

In the solution and refinement of this structure, procedures standard in this laboratory were employed.[18] The structure was solved with the use of direct methods and a combination of refinement and difference electron density calculations. The structure was refined by full-matrix least-squares methods. The final cycles were carried out on F_o^2. Prior to these final cycles the positions of the H atoms of the THF and methylene C atoms were calculated (C–H = 0.95 Å); those of the methyl groups were located in difference electron density maps and then idealized. These H positions were not varied in the final cycle of refinement. Details on the refinement, including agreement indices, are given in Table I. Final positional parameters are given in Table II. Table III[19] presents

Table II. Positional Parameters for the Non-Hydrogen Atoms of $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$

atom	*x*	*y*	*z*
Sn	0.667 598 (39)	0.163 766 (48)	0.381 442 (38)
S(1)	0.610 51 (15)	0.028 50 (17)	0.467 83 (14)
S(2)	0.635 97 (15)	−0.073 70 (19)	0.310 17 (15)
S(3)	0.328 79 (13)	−0.330 52 (19)	0.509 94 (13)
S(4)	0.529 51 (14)	−0.280 60 (18)	0.586 34 (14)
O(1)	0.211 37 (38)	0.033 49 (57)	0.208 11 (42)
O(2)	0.636 06 (46)	0.269 59 (67)	0.863 57 (44)
N(1)	0.562 26 (43)	−0.186 09 (49)	0.411 72 (44)
N(2)	0.437 98 (43)	−0.373 25 (53)	0.428 55 (44)
C(1)	0.600 50 (55)	−0.089 71 (66)	0.397 22 (56)
C(2)	0.540 27 (61)	−0.295 14 (70)	0.356 30 (55)
C(3)	0.521 09 (58)	−0.394 97 (69)	0.408 42 (60)
C(4)	0.437 28 (51)	−0.328 85 (70)	0.502 92 (52)
C(5)	0.592 73 (63)	0.232 53 (81)	0.249 24 (54)
C(6)	0.500 91 (57)	0.179 97 (76)	0.209 90 (57)
C(7)	0.583 01 (62)	0.370 83 (73)	0.257 68 (60)
C(8)	0.648 49 (61)	0.206 88 (78)	0.192 68 (55)
C(9)	0.814 87 (54)	0.155 51 (77)	0.429 17 (56)
C(10)	0.846 30 (61)	0.080 59 (90)	0.365 42 (68)
C(11)	0.850 18 (59)	0.102 06 (83)	0.520 63 (67)
C(12)	0.851 80 (60)	0.284 12 (79)	0.432 70 (66)
C(13)	0.207 94 (64)	−0.045 58 (84)	0.274 52 (62)
C(14)	0.294 20 (59)	−0.116 65 (75)	0.298 59 (57)
C(15)	0.361 32 (60)	−0.026 63 (81)	0.288 49 (58)
C(16)	0.301 36 (57)	0.071 83 (80)	0.232 39 (63)
C(17)	0.724 36 (69)	0.246 9 (12)	0.926 71 (67)
C(18)	0.716 74 (65)	0.152 6 (10)	0.990 39 (73)
C(19)	0.617 82 (76)	0.120 00 (97)	0.952 81 (71)
C(20)	0.574 21 (68)	0.223 6 (10)	0.898 63 (65)

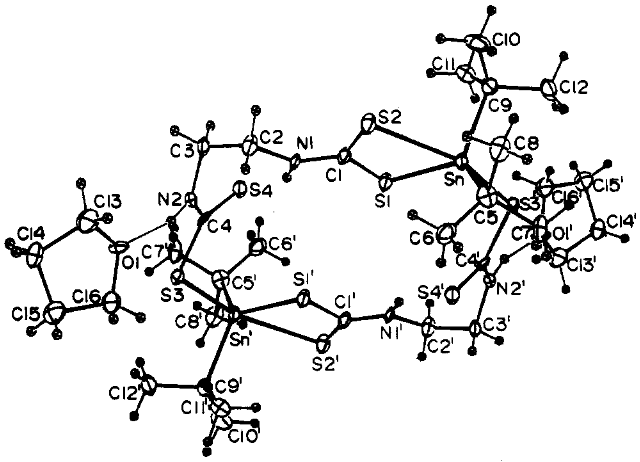

Figure 1. The molecular structure of $[(t\text{-Bu})_2Sn(ebdtc)]_2$, showing the THF molecule that is hydrogen-bonded to the 18-membered ring system. The thermal ellipsoids are drawn at their 50% probability values, except for hydrogen atoms, which are drawn arbitrarily small. Primed atoms are related to unprimed atoms by a crystallographically imposed center of symmetry.

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Table V. Selected Bond Distances (Å) and Angles (deg) for $[(t\text{-Bu})_2Sn(ebdtc)]_2 \cdot 4THF$

Sn–S(1)	2.459 (2)	S(1)–Sn–C(9)	110.0 (2)
Sn–S(2)	2.878 (2)	S(2)–Sn–S(3′)	155.79 (7)
Sn–S(3′)	2.563 (2)	S(2)–Sn–C(5)	88.7 (2)
Sn–C(5)	2.186 (8)	S(2)–Sn–C(9)	96.4 (2)
Sn–C(9)	2.182 (8)	S(3′)–Sn–C(5)	106.9 (2)
S(1)–C(1)	1.725 (8)	S(3′)–Sn–C(9)	92.0 (2)
S(2)–C(1)	1.717 (8)	C(5)–Sn–C(9)	119.5 (3)
C(1)–N(1)	1.304 (9)	Sn–S(1)–C(1)	93.3 (3)
S(1)–Sn–S(2)	67.18 (7)	Sn–S(2)–C(1)	79.9 (3)
S(1)–Sn–S(3′)	88.61 (8)	S(1)–C(1)–N(1)	116.9 (6)
S(1)–Sn–C(5)	126.9 (2)	S(2)–C(1)–N(1)	123.5 (6)

the anisotropic thermal parameters and the positions of the H atoms while Table IV[19] lists structure amplitudes (×10). A negative entry in Table IV indicates that $F_o^2 < 0$.

Results and Discussion

Syntheses. Reaction of R_2SnCl_2 with the ebdtc ligand in ethanol, followed by recrystallization, affords the products $(t\text{-Bu})_2Sn(ebdtc)\cdot C_6H_6$, $(t\text{-Bu})_2Sn(ebdtc)\cdot 2THF$, and $Ph_2Sn(ebdtc)$, as established by chemical analysis, spectroscopy, and, for the THF derivative, a crystal structure. Most bivalent transition-metal ions react with the ebdtc ligand to form insoluble polymeric products of the type $(-M-S_2CNHCH_2CH_2NHCS_2-)_n$, where M = Mn, Zn, Fe, etc.[20-23] Unlike such transition-metal complexes, the diorganotin(IV) complexes with the ebdtc ligand are dimeric both in solution and in the solid state, as will be shown below. To prevent possible formation of polymeric species, we originally carried out the syntheses of $R_2Sn(ebdtc)$ in dilute solution. However, when more concentrated solutions were used the same products were obtained. Thus the absence of polymeric materials in the R_2Sn system is intrinsic and is not the result of a particular synthetic strategy.

Structure of $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$. The molecular geometry and labeling scheme for this complex are shown in Figure 1. $[(t\text{-Bu})_2Sn(ebdtc)]_2$ is a centrosymmetric dimer that consists of an 18-membered ring in which the ebdtc ligands are tridentate, with atoms S(1) and S(2) bound to the Sn atom and atom S(3) to its centrosymmetric equivalent, Sn′. Atom S(4) does not coordinate. One of the independent THF molecules is hydrogen-bonded to the ring (N(2)···O(1) = 2.797 (9) Å, ∠N(2)–H···O(1) = 164°, and H1N(2)–O(1) = 1.86 Å). The other THF molecule is not bonded, but rather makes van der Waals contacts with the ring. It is presumably the loose association of these two species that accounts for the rapid loss of solvent from the material.

In Table V selected bond distances and angles are tabulated. Table VI[19] contains a more complete tabulation. There are no unexpected values. However, of particular interest is the geometry about the Sn atom, which is that of a distorted trigonal bipyramid. Atoms C(5) and C(9) of the *t*-Bu groups and atom S(1) occupy equatorial positions, while atoms S(2) and S(3) are axial. The small bite angle (67.2 (1)°) of the ebdtc ligand is in part responsible for the distortion of the trigonal bipyramid. The geometry about the Sn center in $Me_2SnCl(dtc)$[8] is similar. The bite angle of the dtc ligand is 68.2 (3)°, and the corresponding angles between axial groups are nearly equal (S–Sn–Cl = 154.5 (4)° in the dtc complex versus S–Sn–S = 155.8 (1)° in the ebdtc complex). The bidentate portion of the ebdtc ligand is bonded to the Sn atom in the anisobidentate manner typical of the dtc ligand. Thus the Sn–S(1) and Sn–S(2) distances of 2.459 (2) and 2.878 (2) Å are similar to those of 2.48 (1) and 2.79 (1) Å in $Me_2SnCl(dtc)$.

Although this is the first Sn(ebdtc) complex to be studied, the coexistence in one structure of an anisobidentate and a monodentate CS_2^- group is not without precedent. Thus in the structure of $Sn(Et_2dtc)_4$,[24,25] two CS_2^- groups are bidentate while the other two are monodentate, resulting in six-coordination about the central Sn(IV) center.

Why the Sn(IV) center in the present complex is five-coordinate, rather than six-coordinate, is not clear. There is a temptation to invoke the steric influence of the bulky *t*-Bu groups. But Mössbauer studies[10-13] have been interpreted in favor of six-coordination of the Sn(IV) centers in a series of bulky $Ph_2Sn(S_2CNR_2)_2$ complexes.

Physical and Spectroscopic Properties of $R_2Sn(ebdtc)$ Complexes. These complexes are soluble in polar solvents such as THF, Me_2SO, and DMF, less soluble in benzene, toluene, acetone, and ethanol, but insoluble in water and aliphatic hydrocarbons. Cryoscopic molecular weight measurements of these complexes have been attempted in Me_2SO, but the results have not been reproducible, probably because of partial decomposition of the materials. Instead, mass spectra were obtained for $(t\text{-Bu})_2Sn(ebdtc)\cdot 2THF$ and $Ph_2Sn(ebdtc)$; the region of *m/e* 500–1200 is shown in Figure 2.[19] The mass spectrum was scanned up to *m/e* 2400, but no molecular ion peaks for $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$ or $[Ph_2Sn(ebdtc)]_2$ (1174.96 and 966.49, respectively) were found. However, the highest peaks appear around *m/e* 739 and 839 for these two compounds; these must be fragments from the parent dimers.

There are many IR and NMR studies on Sn–dtc complexes.[1-5,26-28] The characteristic IR bands that are sensitive to molecular structure are the stretching modes for the C–N, C–S, and Sn–S bonds. In particular, the ν(C–N) band in the region 1450–1550 cm^{-1} and the ν(C–S) band in the region 850–1050 cm^{-1} are known to depend on the nature of coordination of the dtc ligand to the metal. Splittings of the ν(C–N) and ν(C–S) bands are characteristic of a monodentate dtc ligand whereas the appearance of a single band in each region is characteristic of an anisobidentate chelation of the dtc ligand. The ebdtc ligand in $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$ acts both as a monodentate and as a anisobidentate ligand; the patterns for ν(C–N) and ν(C–S) are consistent with this (see Experimental Section). The ν(C–S) mode as well as the ν(Sn–S) mode in the THF and C_6H_6 solvates of $(t\text{-Bu})_2Sn(ebdtc)$ have almost exactly the same band shape and intensity, but the ν(C–N) modes appear at lower frequencies in the THF solvate, probably owing to the hydrogen bonding of the amine proton to the oxygen atom of the THF molecule. The ν(C–N) mode in $Ph_2Sn(ebdtc)$ appears as a broad band without clear splitting, but the ν(C–S) mode has a split pattern similar to that shown in the *t*-Bu complexes.

In the ^1H NMR spectra the chemical shifts of the *t*-Bu, $N-CH_2$, and NH protons in the two different solvated forms of $(t\text{-Bu})_2Sn(ebdtc)$ are the same (see Experimental Section). The appearance as singlets of the resonances from the *t*-Bu and $N-CH_2$ protons indicates that the molecules are stereochemically nonrigid in solution with the *t*-Bu groups rotating around the Sn–C bonds.

These molecular weight and spectroscopic data are consistent with the same dimeric molecular structure in the solid state and in solution for these $R_2Sn(ebdtc)$ complexes.

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Registry No. $(t\text{-Bu})_2Sn(ebdtc)$, 101347-98-2; $Ph_2Sn(ebdtc)$, 101347-99-3; $[(t\text{-Bu})_2Sn(ebdtc)]_2\cdot 4THF$, 101401-92-7; $Na_2(ebdtc)$, 13036-89-0; $H_2NCH_2CH_2NH_2$, 107-15-3; CS_2, 75-15-0; $(t\text{-Bu})_2SnCl_2$, 19429-30-2; Ph_2SnCl_2, 1135-99-5.

Supplementary Material Available: Table III (anisotropic thermal parameters and hydrogen atom positions), Table IV (structure amplitudes), Table VI (bond distances and angles), and Figure 2 (GC-mass spectra) (29 pages). Ordering information is given on any current masthead page.

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