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Notes

Synthesis and Characterization of Substituted (Thiomethyl)lithium Compounds. Structures of $[\{Li(CH_2SMe)(THF)\}_\infty]$ and $[Li_2(CH_2SPh)_2(THF)_4]$

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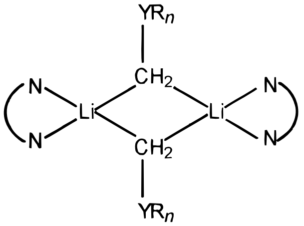
*Summary: Bu_3SnCH_2SR reacts with BuLi in n-hexane (R = Me, ^tBu) and in n-hexane/THF (R = Ph) to give the solvate-free compounds $LiCH_2SR$ (R = Me (**1a**), ^tBu (**1b**), Ph (**1c**)). The constitution of **1** was determined by microanalysis and by NMR (^1H, ^{13}C, ^7Li) spectroscopy. **1b** reacts with benzaldehyde and benzophenone to form $PhCH(OH)CH_2S^tBu$ (**2**) and $Ph_2C(OH)CH_2S^tBu$ (**3**), respectively. Recrystallization of **1a** and **1c** from n-hexane/THF solutions gives $[\{Li(CH_2SMe)(THF)\}_\infty]$ (**1a′**) and $[Li_2(CH_2SPh)_2(THF)_4]$ (**1c′**), respectively. The structures of **1a′** and **1c′** were determined by single-crystal X-ray analysis. **1a′** exhibits a polymeric ladder-like structure in which four-membered planar Li_2C_2 rings and six-membered $Li_2C_2S_2$ rings are alternately arranged. **1c′** crystallizes in the form of dimers with planar four-membered Li_2C_2 rings, but without sulfur coordination at lithium.*

Introduction

Stability, structure, and reactivity of organolithium compounds with functionalized methyl ligands of the type $LiCH_2YR_n$ (Y = heteroatom; R = alkyl, aryl, H) depend strongly on the nature of the heteroatom Y which can be (i) a neutral, coordinatively saturated heteroatom (e.g., $YR_n = SiR_3$, ...), (ii) a neutral, Lewis-basic heteroatom (e.g., $YR_n = NR_2, PR_2, OR, SR, F, Cl$, ...), or (iii) a cationic (ylidic) heteroatomic center (e.g., $YR_n = {}^+NR_3, {}^+PR_3, {}^+SR_2$, ...). Compounds of type ii are of special interest due to the possible involvement of Lewis-basic heteroatoms in the coordination at lithium, thus offering entirely new structures and reactions (e.g., the reactivity of Köbrich's carbenoids[1]). So far, all compounds of type ii whose solid state structures are known are either TMEDA adducts (TMEDA = *N,N,N′,N′*-tetramethylethylenediamine) or contain other nitrogen chelating ligands. All of them form dimeric molecules that can be classified according to their main structural feature. Type **I** is characterized by a four-membered Li_2C_2 ring (e.g., $[Li_2(CH_2SMe)_2(tmeda)_2]$[2] (**4**)) and type **II** by a six-membered $Li_2C_2Y_2$ ring (e.g., $[Li_2(CH_2SPh)_2(tmeda)_2]$[2] (**5**), $[Li_2(CH_2PPh_2)_2(tmeda)_2]$,[3] $[Li_2(CH_2PMe_2)_2(tmeda)_2]$,[4] $[Li_2(CH_2PPhMe)_2(tmeda)_2]$,[4] and $[Li_2(CH_2PPhMe)_2(sparteine)_2]$[4]).



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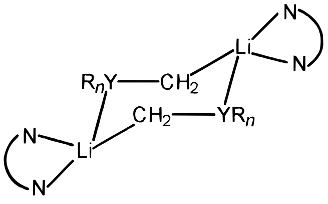

II

Sulfur-substituted methyllithium compounds $LiCH_2SR$ were prepared in solution for the first time by metalating MeSR with BuLi/DABCO[5] (DABCO = 1,4-diazabicyclo[2.2.2]octane), BuLi/TMEDA,[6,7] or PhLi.[5] From these solutions (R = Me, Ph), $[Li_2(CH_2SPh)_2(dabco)_{1.5}(THF)_{0.5}]$,[8] $LiCH_2SPh{\cdot}2THF$,[8] $[Li_2(CH_2SMe)_2(tmeda)_2]$[2,6] (**4**), and $[Li_2(CH_2SPh)_2(tmeda)_2]$[2] (**5**) were obtained as colorless crystals.

The transmetalation of Bu_3SnCH_2SMe with BuLi to form TMEDA-free $LiCH_2SMe$ was first mentioned by Peterson.[9] Here, we report on the analogous transmetalation reaction to give $LiCH_2SR$ (R = Me (**1a**), ^tBu (**1b**), Ph (**1c**)) and, for the first time, their isolation in the solid state as solvate-free compounds, as well as the solid state structures of $[\{Li(CH_2SMe)(THF)\}_\infty]$ (**1a′**) and $[Li_2(CH_2SPh)_2(THF)_4]$ (**1c′**).

Results and Discussion

(Thiomethyl)lithium complexes (**1**) were prepared by the reaction of BuLi (Bu = *n*-Bu) with the appropriate

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[⊗] Abstract published in *Advance ACS Abstracts,* May 15, 1997.

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tributyltin derivative in *n*-hexane, cf. eq 1. The formation of compound **1c** required a small amount of THF as a dipolar aprotic solvent. The products were isolated as colorless microcrystals in good yields (65−87%).

$$Bu_3SnCH_2SR + BuLi \xrightarrow[0\ °C,\ -Bu_4Sn]{n\text{-hexane (THF)}} \underset{\mathbf{1a\text{-}c}}{LiCH_2SR} \quad (1)$$

	1a	1b	1c
R	Me	^{t}Bu	Ph

The complexes **1** are very sensitive toward oxygen and moisture. **1a** explodes immediately in the presence of traces of oxygen. The compounds **1** are readily soluble in THF but nearly insoluble in diethyl ether and *n*-hexane.

Microanalyses (Li) and NMR spectra confirm the presence of solvate-free lithium organyls **1**. The ^{13}C NMR data of **1c** in THF-d_8 are in good agreement with the values observed for $LiCH_2SPh$ from the reaction of MeLi with MeSPh in THF/benzene-d_6.[10] The different aggregation of the lithium organyls in solution and the influence of TMEDA may be responsible for the upfield shift of the methylene and methyl protons of compound **1a** in THF-d_8 (0.36 and 1.78 ppm) compared with the values observed for $LiCH_2SMe$/tmeda in benzene-d_6 (0.74 and 2.08 ppm).[6]

In the ^{13}C NMR spectra of **1** in THF-d_8, the coupling constants $^1J(C,H)$ found for the CH_2 groups (116.0 (**1a**), 119.4 (**1b**), and 121.0 Hz (**1c**)) lie in between the values observed for methyllithium (98 Hz)[11] and $LiCH_2Ph$ (133 Hz).[12] The methylene proton shifts $\delta(CH_2)$ of **1** are only weakly dependent on the nature of the substituent R at sulfur (0.36 ppm (**1a**), 0.24 ppm (**1b**), 0.31 ppm (**1c**)). Generally, these signals are observed at higher field in comparison with those found in the corresponding (aminomethyl)lithium derivatives $LiCH_2NR_2$ (NR_2 = NMe_2 (0.94 ppm), NC_5H_{10} (0.98 ppm), NPh_2 (2.59 ppm)).[13] As expected, the ^7Li NMR spectra of **1** exhibit a singlet resonance (0.65 (**1a**), 0.86 ppm (**1b**), 0.67 ppm (**1c**)).

Additionally, compound **1b** was characterized by reaction with D_2O to give DCH_2S^tBu (degree of deuteration: 95% (^1H NMR)) and by reaction with benzaldehyde and benzophenone to form PhCH(OH)CH_2S^tBu (**2**) and $Ph_2C(OH)CH_2S^tBu$ (**3**), respectively.

Structures of [{Li(CH$_2$SMe)(THF)}$_\infty$] (1a′) and [Li$_2$(CH$_2$SPh)$_2$(THF)$_4$] (1c′). By recrystallization of **1a** and **1c** from *n*-hexane/THF, the complexes are obtained as THF adducts $[\{Li(CH_2SMe)(THF)\}_\infty]$ (**1a′**) and $[Li_2(CH_2SPh)_2(THF)_4]$ (**1c′**), respectively. **1a′** and **1c′** form well-shaped crystals whose air sensitivity is lower in comparison with that of **1a** and **1c**. Selected bond lengths and bond angles are listed in Table 1 for **1a′** and in Table 2 for **1c′**. ORTEP[14] drawings giving the atom-numbering schemes are shown in Figures 1 and 2.

Table 1. Selected Distances (Å) and Angles (deg) for [{Li(CH$_2$SMe)(THF)}$_\infty$] (1a′)[a]

C1−S1	1.769(3)	S1−C2	1.814(3)
C1−Li1′	2.225(5)	C1−Li1#	2.256(5)
Li1−O1	1.982(5)	Li1−S1	2.531(5)
Li1′−Li1#	2.499(9)		
C1−S1−C2	106.7(2)	C1′−Li1−C1*	112.2(2)
Li1′−C1−Li1#	67.8(2)	Li1′−C1−S1	128.0(2)
Li1#−C1−S1	103.2(2)	C1′−Li1−O1	100.3(2)
C1*−Li1−O1	114.8(2)	C1′−Li1−S1	116.6(2)
S1−Li1−O1	101.0(2)	Li1−S1−C1	105.2(2)

[a] Symmetry transformations used to generate equivalent atoms: (′)$-x + 1, -y + 1, -z + 2$; (#) $x - 1, y, z$; (*) $x + 1, y, z$.

Table 2. Selected Distances (Å) and Angles (deg) for [Li$_2$(CH$_2$SPh)$_2$(THF)$_4$] (1c′)[a]

C1−S	1.780(7)	S−C2	1.762(6)
C1−Li	2.22(1)	C1−Li′	2.27(1)
Li−O1	1.96(1)	Li−O2	1.96(1)
Li−Li′	2.55(2)		
C1−S−C2	109.3(3)	C1−Li−C1′	111.1(6)
Li−C1−Li′	68.9(6)	Li−C1−S	106.4(5)
C1−Li−O1	114.4(6)	C1−Li−O2	113.6(5)
C1′−Li−O1	110.0(5)	C1′−Li−O2	109.0(5)
O1−Li−O2	98.0(6)		

[a] Symmetry transformation used to generate equivalent atoms: (′) $-x + 1, -y, -z + 2$.

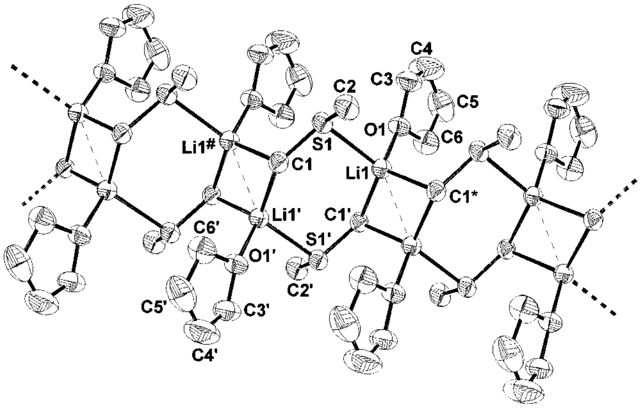

Figure 1. ORTEP drawing of a section of the polymeric structure of **1a′** (thermal ellipsoids at 50% probability). Hydrogen atoms are omitted for clarity.

Compound **1a′** is a polymeric species with a crystallographically imposed C_i symmetry. Owing to the μ_3-thiomethyl-1:2κ^2C:3κS ligands, planar four-membered Li_2C_2 rings and six-membered $Li_2C_2S_2$ rings in a chair conformation are arranged alternately. Thus, a ladder-like structure is obtained. This is indicated by an interplanar angle of 114.7° between the planes of the Li_2C_2 ring and the planar Li_2C_2 unit that is part of the neighboring six-membered ring. In contrast to **1a′**, the TMEDA adduct $[Li_2(CH_2SMe)_2(tmeda)_2]$[2] (**4**) is dimeric with a four-membered Li_2C_2 ring that is not exactly planar.

The Li−C bonds of **1a′** (d(Li1′−C1) = 2.225(5) Å and d(Li1#−C1) = 2.256(5) Å) are of the same lengths as those found in **4** (d(Li−C) = 2.227(7)−2.256(6) Å). In **1a′**, each Li is coordinated in a distorted tetrahedron manner by two methylene carbon atoms, one oxygen atom (THF), and one sulfur atom (d(Li1−S1) = 2.531(5) Å). The Li−S bond length is significantly shorter than the corresponding one in $[Li_2(CH_2SPh)_2(tmeda)_2]$[2] (**5**, 2.555(3) Å), which exhibits a structure of type **II**. The C−S bond lengths of **1a′** (d(C1−S1) = 1.769(3) Å, d(S1−

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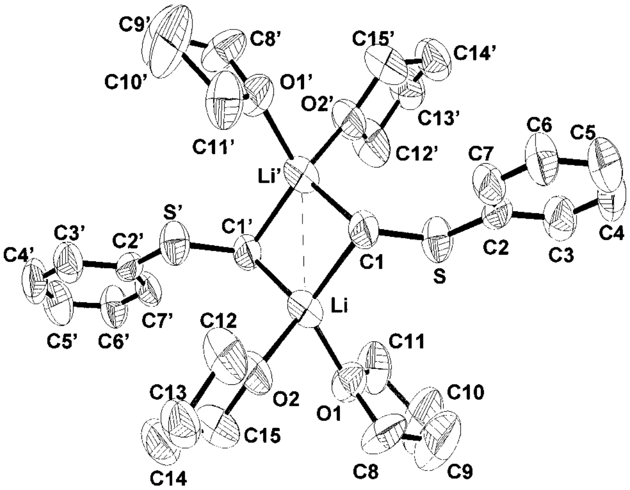

Figure 2. ORTEP drawing of the molecular structure of **1c′** (thermal ellipsoids at 50% probability). Hydrogen atoms are omitted for clarity.

C2) = 1.814(3) Å) do not differ significantly from those observed in **4** ($d(CH_2-S)$ = 1.777(4), 1.778(3) Å; $d(S-CH_3)$ = 1.808(5), 1.819(3) Å). The C−S−C bond angles are also indentical within the 3σ-criterion (**1a′**, 106.7(2)°; **4**, 107.2(3)°).

Compound **1c′** crystallizes as a centrosymmetric dimer. Thus, an exactly planar type **I** Li_2C_2 ring with μ-phenylthiomethyl-1:2κ^2*C* ligands is formed. The Li−C bond lengths in **1c′** (2.22(1) and 2.27(1) Å) are significantly longer than those in the TMEDA adduct **5**[2] (2.131(4) Å), which exhibits a structure of type **II**. Two oxygen atoms of THF molecules complete the distorted tetrahedron around lithium ($d(Li-O)$ = 1.96(1) Å). The CH_2-S bond lengths in **1c′** (1.780(7) Å) and **5** (1.759(2) Å) are equivalent within the 3σ-limit, whereas the $S-C_{Ph}$ distance in **1c′** is significantly shorter than the corresponding one in **5** (1.762(6) vs 1.792(2) Å).

Compounds **1a′** and **1c′** are the first structurally characterized functionalized methyllithium compounds $LiCH_2YR_n$ with a Lewis-basic heteroatomic center Y without chelating N donor ligands. **1a′** is the only example of a polymeric complex $LiCH_2YR_n$ in which both characteristic structural features, a six-membered ring $Li_2C_2Y_2$ and a four-membered ring Li_2C_2, occur together. Investigations on the structures of compounds $LiCH_2YR_n$ with other Lewis-basic heteroatoms (N, P) are in progress.

Experimental Section

General Comments. All reactions and manipulations were carried out under purified argon using standard Schlenk techniques. **(Warning: compound 1a explodes immediately in the presence of traces of oxygen. Appropriate precautions should be taken for its safe manipulation.)** *n*-Hexane, THF-d_8, and benzene-d_6 were dried with $LiAlH_4$. Diethyl ether and THF were distilled from sodium benzophenone ketyl. NMR spectra were recorded on Varian Gemini 300 or Varian Unity 500 NMR spectrometers using the protio impurities and the ^{13}C resonances of the deuterated solvents as references for the ^1H and ^{13}C NMR spectroscopy, respectively. The chemical shifts $\delta(^7Li)$ are reported relative to a solution of LiCl in THF-d_8 (external). The lithium contents were determined by atomic emission spectroscopy (AES). Elemental analyses (C,H,S) were obtained from the microanalytical laboratory of Martin-Luther-Universität. A 15% solution of BuLi in hexane was obtained from Merck. The tin compounds Bu_3SnCH_2SR (R = Me, Ph, ^{t}Bu) were prepared according to published procedures.[6,7,15]

Table 3. Crystal Data and Structure Refinement for 1a′ and 1c′

	1a′	1c′
empirical formula	$C_6H_{13}OSLi$	$C_{30}H_{46}O_4S_2Li_2$
fw	140.16	548.67
T (K)	200(2)	220(2)
cryst syst	triclinic	monoclinic
space group	$P\bar{1}$ (No. 2)	$P2_1/n$ (No. 14)
a (Å)	4.787(1)	9.285(9)
b (Å)	9.284(2)	19.794(12)
c (Å)	10.383(2)	9.792(9)
α (deg)	113.52(3)	90.0
β (deg)	96.90(3)	113.83(7)
γ (deg)	96.51(3)	90.0
V (Å^3)	413.4(2)	1646(2)
Z	2	2
D_{calcd} (g cm^{-3})	1.126	1.107
μ(Mo Kα) (mm^{-1})	0.312	0.191
F(000)	152	592
scan range (deg)	2.18 < θ < 22.98	2.50 < θ < 22.50
no. of reflns colld	2208	7299
no. of indep reflns	1127	2079
no. of params refined	121	185
goodness-of-fit on F^2	1.089	1.227
R_1 [$I > 2\sigma(I)$]	0.0377	0.0949
wR_2, all data	0.1030	0.2547
largest diff peak (e Å^{-3})	0.268	0.601
largest diff hole (e Å^{-3})	−0.250	−0.254

Synthesis of $LiCH_2SMe$ (1a) and $LiCH_2S^tBu$ (1b). To a stirred solution of Bu_3SnCH_2SR (R = Me, ^{t}Bu) (15 mmol) in *n*-hexane (5 mL) was added an equimolar amount of a solution of BuLi in *n*-hexane (0 °C). After the reaction mixture was stirred for 2 h at room temperature, colorless microcrystals were formed, which were filtered off, washed with cold *n*-hexane, and dried *in vacuo*. Yields: 87% (**1a**); 65% (**1b**).

Anal. Calcd for **1a**: Li, 10.19. Found: Li, 9.89. ^1H NMR (300.07 MHz, THF-d_8): δ 0.36 (s, 2H, CH_2), 1.78 (s, 3H, CH_3). ^{13}C NMR (coupled spectrum, 125.70 MHz, THF-d_8): δ 14.8 (t, CH_2, ^1J(C,H) = 116.0 Hz), 30.1 (q, CH_3, ^1J(C,H) = 133.2 Hz). ^7Li NMR (194.27 MHz, THF-d_8): δ 0.65 (s).

Anal. Calcd for **1b**: Li, 6.30. Found: Li, 6.43. ^1H NMR (300.07 MHz, THF-d_8): δ 0.24 (s, 2H, CH_2), 1.01 (s, 9H, CH_3). ^{13}C NMR (coupled spectrum, 125.70 MHz, THF-d_8): δ 2.7 (t, CH_2, ^1J(C,H) = 119.4 Hz), 46.0 (s, S*C*), 29.4 (q, CH_3, ^1J(C,H) = 129.1 Hz). ^7Li NMR (194.27 MHz, THF-d_8): δ 0.86 (s).

Synthesis of $LiCH_2SPh$ (1c). To a stirred solution of Bu_3SnCH_2SPh (10.8 g, 26.1 mmol) in *n*-hexane (20 mL) was added rapidly an equimolar amount of a solution of BuLi in *n*-hexane (25 °C). Then 3 mL of THF was added (0 °C). The reaction mixture continued to stir for 1 h at room temperature. The colorless precipitate was filtered off, washed with cold *n*-hexane, and vacuum dried. Yield: 2.6 g (77%). Anal. Calcd for **1c**: Li, 5.33. Found: Li, 5.19. ^1H NMR (300.07 MHz, THF-d_8): δ 0.31 (s, 2H, CH_2), 6.62 (t, 1H, *p*), 6.92 (t, 2H, *m*), 7.19 (d, 2H, *o*). ^{13}C NMR (coupled spectrum, 125.70 MHz, THF-d_8): δ 3.8 (t, CH_2, ^1J(C,H) = 121.0 Hz), 120.6 (d, C_p, ^1J(C,H) = 157.5 Hz), 123.9 (d, C_o, ^1J(C,H) = 158.7 Hz), 127.4 (d, C_m, ^1J(C,H) = 158.5 Hz), 158.5 (s, C_i). ^7Li NMR (194.27 MHz, THF-d_8): δ 0.67 (s).

Reaction of 1b with Benzaldehyde. To a stirred suspension of **1b** (1.1 g, 10.0 mmol) in diethyl ether (10 mL) an equimolar amount of PhCHO (1.1 g) was added rapidly at −78 °C. The mixture was stirred for 1 h at room temperature. After hydrolysis with water, the organic layer was separated, dried (Na_2SO_4), and concentrated. The residue was distilled *in vacuo* to give $PhCH(OH)CH_2S^tBu$ (**2**) as a colorless liquid (bp 120−140 °C/0.3 Torr). Yield: 1.2 g (56%). Anal. Calcd for **2**: C, 68.52; H, 8.62; S, 15.25. Found: C, 68.55; H, 8.66; S, 14.58. ^1H NMR ($PhCH^X(OH)CH^AH^MS^tBu$, 199.97 MHz, $CDCl_3$): δ

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1.35 (s, 9H, CH_3), 2.78 (dd, 1H, H^A, $^1J(H^A,H^M) = 12.9$ Hz, $^1J(H^A,H^X) = 9.1$ Hz), 2.92 (dd, 1H, H^M, $^1J(H^M,H^X) = 4.0$ Hz), 4.70 (dd, 1H, H^X), 3.19 (s, 1H, OH), 7.21−7.40 (m, 10H, *Ph*). ^{13}C NMR (50.29 MHz, CDCl$_3$): δ 30.9 (CH$_3$), 42.4 (SC), 38.3 (SCH$_2$), 72.4 (COH), 128.2 (C_p), 127.6 (C_o), 128.3 (C_m), 142.8 (C_i).

Reaction of 1b with Benzophenone. Analogous to the reaction with PhCHO, the reaction of **1b** (1.1 g, 10.0 mmol) with an equimolar amount of Ph_2CO (1.8 g) affords $Ph_2C(OH)CH_2S^tBu$ (**3**) as colorless crystals (mp 61−63 °C). Yield: 2.0 g (69%). Anal. Calcd for **3**: C, 75.48; H, 7.74; S, 11.19. Found: C, 75.36; H, 8.06; S, 10.99. ^{1}H NMR (199.97 MHz, CDCl$_3$): δ 1.34 (s, 9H, CH_3), 3.38 (s, 2H, CH_2), 3.84 (s, 1H, OH), 7.22−7.48 (m, 10H, *Ph*). ^{13}C NMR (50.29 MHz, CDCl$_3$): δ 30.9 (CH$_3$), 43.1 (SC), 41.9 (SCH$_2$), 76.5 (COH), 127.2 (C_p), 126.1 (C_o), 128.2 (C_m), 145.8 (C_i).

X-ray Structure Determination of 1a′ and 1c′. Suitable colorless single crystals were obtained from *n*-hexane/THF solutions at −40 °C (**1a′**) and −78 °C (**1c′**). X-ray measurements were performed on a Stoe STADI 4 (**1a′**) and a Stoe IPDS diffractometer (**1c′**) with Mo Kα radiation (0.710 73 Å, graphite monochromator). Crystal data, details of data collection, structure solution, and refinement are summarized in Table 3. The structures were solved by direct methods with SHELXS-86[16] and refined using full-matrix least-squares procedures on F^2 (SHELXL-93[17]). Non-hydrogen atoms were refined with anisotropic thermal parameters. For compound **1a′**, hydrogen atoms were located in a difference Fourier map and refined isotropically. For compound **1c′**, with the exception of the methylene group whose hydrogen atoms were located in a difference Fourier map and isotropically refined, all other hydrogen atoms were geometrically positioned.

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Supporting Information Available: Complete tables of atomic coordinates, H atom parameters, bond distances, bond angles, and anisotropic thermal parameters (11 pages). Ordering information is given on any current masthead page.

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