a 1.1 M TBAF·3H_2O solution (2.0 mmol) in THF was added. The clear red solution was warmed to −30 °C over 3 h and then kept at this temperature for 14 h. A brown precipitate was filtered off and the resulting red solution stripped in vacuo to yield 810 mg (70%) of **23** as a red oil. Anal. Calcd for $C_{29}H_{48}NO_4V$ (525.7): C, 66.26; H, 9.20; N, 2.66. Found: C, 67.87; H, 9.41; N, 2.72. Mixture of trans and cis isomers (70:30). *trans*-**23**: ^1H NMR (C_6D_6) δ 6.00, 5.50 (m, 1 H each, 3-H, 4-H), 5.29 (s, 5 H, CpV), 4.07, 3.97 (d, 1 H each, 2-H), 1.72 (br d, 3 H, CH_3), coupling constants (Hz) ^3J = 6.3 (2-H, 3-H), 6.5 (4-H, 5-H), tetrabutylammonium δ 2.90 (8 H, CH_2N), 1.26 (16 H, CH_2), 0.89 (12 H, CH_3); ^{13}C NMR (THF-d_8) δ 305 (br, C(1)), 265 (br, V–CO), 131.0 (158, C(4)), 123.1 (157, C(3)), 93.3 (171, CpV), 64.9 (C(2)), 18.4 (125, C(5)), tetrabutylammonium δ 59.3 (137, NCH_2), 24.7 (CH_2), 20.6 (121, CH_2), 14.1 (CH_3). *cis*-**23** (=minor isomer) δ 129.9 (158, C(4)), 121.2 (157, C(3)), 14.5 (C95)), remainder hidden under trans-**23** and tetrabutylammonium signals.

Tricarbonyl[ethoxy(but-2-en-1-yl)carbene]cyclopentadienylvanadium (24). The tetrabutylammonium vanadium salt **23** (550 mg, 1.05 mmol) was dissolved in 40 mL of dichloromethane and then cooled to −78 °C. During 2 h 1.05 mL of a 1.0 M triethyloxonium tetrafluoroborate solution in dichloromethane was added with stirring. The mixture was warmed up to −30 °C over 3 h. The resulting dark red solution was concentrated in vacuo to a volume of 10 mL. A 30-mL amount of cold pentane was added and the mixture kept at −30 °C for 14 h. The solution was then decanted from a brown precipitate at −30 °C. Removal of the solvent from the clear red solution at −30 °C gave 170 mg (52%) of **24**, which contained some CpV$(CO)_4$ and ammonium salts. The product was very sensitive toward further decomposition and was, therefore, stored at −78 °C. Mixture of two isomers (A:B ≈ 70:30). Major isomer: ^1H NMR (C_6D_6) δ 5.32, 5.29 (m, 2 H, 2-H, 3-H), 4.87 (s, 5 H, CpV), 4.41 (q, 2 H, OCH_2), 3.58 (m, 2 H, 1-H, 1-H′), 1.50 (d, 3 H, CH_3; the methyl resonance of the minor isomer was observed at δ 1.43), 1.11 (t, 3 H, OCH_2CH_3); ^{13}C NMR (CD_2Cl_2): δ 128.3, 126.4 (C(2), C(3); Minor isomer δ 125.0, 124.0), 94.2 (CpV), 73.6 (OCH_2), 62.4 (broad, C(1)), 18.0 (C(4), trans isomer; minor (cis) isomer δ 13.3), 14.8 (OCH_2CH_3), carbonyl and carbene carbon atoms at vanadium not localized. IR (CH_2Cl_2): ν(CO) 1964, 1867 cm^{-1}.

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Supplementary Material Available: Description of the X-ray crystal structure analysis of **13a** and listings of final positional and thermal parameters, distances, and angles (6 pages); a listing of final observed and calculated structure factors (18 pages). Ordering information is given on any current masthead page.

Indium Compounds That Contain Two Different Organic Substituents. Crystal Structure of $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$. An Interesting Case of Partial Ligand Disorder in the Solid State

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Three new indium compounds containing two different organic substituents, $In(CH_2CMe_3)(CH_2SiMe_3)Cl$, $In(CH_2CMe_3)(C_6H_5)Cl$, and $(Me_3CCH_2)(Me_3SiCH_2)InPEt_2$, have been prepared, purified, and characterized. All compounds were characterized by partial elemental analysis (C, H, and P as appropriate), cryoscopic molecular weight studies in benzene solution, and NMR and IR spectroscopic studies. All compounds exist as dimeric molecules in solution. NMR spectral data were consistent with the presence of mixtures of cis and trans isomers in solution. $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ crystallizes in the centrosymmetric triclinic space group $P\bar{1}$ (C_i^1, No. 2) with a = 6.1000 (10) Å, b = 10.337 (3) Å, c = 12.190 (3) Å, α = 77.28 (2)°, β = 84.41 (2)°, γ = 84.50 (2)°, V = 744.0 (3) Å^3, and Z = 1. Diffraction data (Mo Kα, 2θ = 5–50°) were collected on a Siemens R3m/V automated four-circle diffractometer, and the structure was solved and refined to R = 5.39% and R_w = 7.65% for those 1752 unique data with $|F_o| > 4\sigma(|F_o|)$ (R = 8.30% for all 2629 data). Distances within the centrosymmetric dimeric molecule in the trans conformation include In–CH_2CMe_3 = 2.140 (13) Å, In–CH_2SiMe_3 = 2.125 (12) Å, and In–Cl (bridging) = 2.572 (3) and 2.659 (3) Å. There is some slight disorder of CH_2CMe_3 and CH_2SiMe_3 ligands (~73%:27%), and there are weak intermolecular In···Cl interactions at 3.528 (3) Å along the a-direction. Attempts to prepare the related organogallium compounds, $Ga(Me)(CH_2CMe_3)Cl$ and $Ga(CH_2CMe_3)(CH_2CMe_2Ph)Cl$, were unsuccessful. Ligand redistribution reactions led to the formation and subsequent isolation of mixtures of the appropriate symmetrized products.

Introduction

The chemistry of organoindium compounds is important for gaining a more complete understanding of the reactions involved in the organometallic chemical vapor deposition process for making InP, an important electronic material. The most common organoindium compounds[1] employed in producing InP are $InMe_3$[2] and $InEt_3$[3,4], although a variety of other simple homoleptic trialkylindium compounds including In(n-Pr)$_3$,[4] In(i-Pr)$_3$,[4] In(n-Bu)$_3$,[4] In(sec-Bu)$_3$,[4] In(t-Bu)$_3$,[5] $In(CH_2CMe_3)_3$,[6] and $In(CH_2SiMe_3)_3$[7] are known.

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In contrast, only a few heteroleptic organoindium compounds including $InMe_2Et$,[8] $InEt_2Me$,[8] $In(CH_2CMe_3)_2Me$,[6] and $InMe_2(C_5H_5)$[9] have been reported. It is regretable that none of these compounds have been proven by appropriate structural studies to exist as pure compounds. Variable-temperature ^1H NMR studies[11] have shown that rapid ligand redistribution reactions occur in solutions of $InMe_2Et$. Consequently, the existence of $InMe_2Et$ as a single compound has been questioned.[11] A distillation[11] of $InMe_2Et$ in vacuo provided initial fractions of crystalline $InMe_3$ contaminated with traces of ethyl-containing material. Subsequent less volatile fractions contained increased Et:Me ratios according to ^1H NMR spectral studies. In contrast, a sample of $In(CH_2CMe_3)_2Me$ was distilled to give an analytically pure sample.[6] However, no experimental data proved that $In(CH_2CMe_3)_2Me$, a liquid at room temperature, existed as a single species. Solid $InMe_2(C_5H_5)$ might be expected to exist as a pure compound with a structure analogous to that observed for $GaMe_2(C_5H_5)$[12] and $AlMe_2(C_5H_5)$.[13] The X-ray structural studies of these compounds identify a polymer with four-coordinate metal atoms. The cyclopentadienyl group through its 1- and 3-positions bridges two metal atoms.

In this paper, we report the synthesis of the first fully characterized organoindium compound containing two different simple alkyl substituents, $In(CH_2CMe_3)(CH_2SiMe_3)Cl$. The compound was characterized by physical properties, partial elemental analyses (C, H), cryoscopic molecular weight data, infrared as well as ^1H and ^{13}C spectroscopic data, and an X-ray structural study. The related compounds $In(CH_2CMe_3)(C_6H_5)Cl$ and $(Me_3SiCH_2)(Me_3CCH_2)InPEt_2$ have also been prepared and partially characterized (no X-ray structural studies).

Experimental Section

All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or under a purified argon atmosphere. The compounds $In(CH_2SiMe_3)Cl_2$,[7] $In(CH_2SiMe_3)_2Cl$,[7] $In(CH_2CMe_3)_2Cl$,[6] $LiCH_2CMe_3$,[14] $LiPEt_2$,[15] $GaMe_2Cl$,[16] $Ga(CH_2CMe_3)_2Cl$,[17] $Ga(CH_2CMe_2Ph)Cl_2$,[18] and $Ga(CH_2CMe_2Ph)_2Cl$[18] were prepared and purified by literature methods. Solvents were dried by conventional procedures. Elemental analyses were performed by E+R Microanalytical Laboratory, Inc., Corona, NY. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. Absorption intensities are reported with the abbreviations vs (very strong), s (strong), m (medium), w (weak), vw (very weak), and sh (shoulder). The ^1H spectra were recorded at 300 MHz on a Varian Gemini-300 spectrometer. Proton chemical shifts are reported in δ units (ppm) and are referenced to $SiMe_4$ at δ 0.00 and C_6D_6 at δ 7.15. The proton-decoupled ^{13}C NMR spectra was recorded at 161.9 MHz on a Varian VXR-400 spectrometer. Proton-decoupled ^{31}P NMR spectra are referenced to 85% H_3PO_4 at δ 0.00. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries. Molecular weights were measured cryoscopically in benzene with use of an instrument similar to that described by Shriver and Drezdzon.[19]

Synthesis of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$. Metathesis Reaction. A side-arm dumper charged with 0.754 g (9.65 mmol) of $LiCH_2CMe_3$ was attached to a 100-mL two-neck flask charged with 2.634 g (9.650 mmol) of $In(CH_2SiMe_3)Cl_2$. After the reactants were dissolved in approximately 25 mL of Et_2O, the system was cooled to −78 °C. The $LiCH_2CMe_3$ was then slowly added with stirring to $In(CH_2SiMe_3)Cl_2$. After the reaction mixture was warmed to room temperature and stirred for 8 h, the Et_2O was removed by vacuum distillation. The product $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ was finally separated from the LiCl by extraction with 30 mL of pentane. Cooling the pentane solution to −20 °C produced crystals of the desired product, $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ (2.721 g, 8.819 mmol, 91.4%). **$In(CH_2CMe_3)(CH_2SiMe_3)Cl$** (sample recrystallized from pentane): colorless solid; mp 89–91 °C; sublimes at 70 °C (0.01 mm). ^1H NMR (C_6D_6, δ): 0.18 (s, 9.0 H, $SiMe_3$), 0.20 (s, 10.7 H, $SiMe_3$), 0.25 (s, 2.0 H, $InCH_2Si$), 0.27 (s, 2.3 H, $InCH_2Si$), 1.03 (s, 10.3 H, CMe_3), 1.08 (s, 9.3 H, CMe_3), 1.53 (s, 2.3 H, $InCH_2C$), 1.56 (s, 2.0 H, $InCH_2C$). $^{13}C\{^1H\}$ NMR (C_6D_6, δ): 1.75 (s, $SiMe_3$), 1.86 (s, $SiMe_3$) 9.71 (s, CH_2Si), 10.36 (s, CH_2Si), 31.93 (s, CH_2C), 32.11 (s, CH_2C), 34.18 (s, CMe_3), 34.28 (s, CMe_3), 46.07 (s, C), 46.68 (s, C). Anal. Calcd: C, 35.02; H, 7.19. Found: C, 35.23; H, 7.45. Cryoscopic molecular weight, benzene solution, formula weight 308.55 (observed molality, observed M_r, association): 0.0673, 634, 2.05; 0.0481, 624, 2.02; 0.0332, 617, 2.00. IR (Nujol mull, cm^{-1}): 2710 (vw), 1298 (w), 1256 (sh), 1243 (s), 1122 (w), 1112 (w), 1087 (w), 1011 (w), 995 (sh), 979 (m), 965 (sh), 847 (s), 825 (vs), 751 (s), 747 (sh), 716 (s), 682 (m), 609 (vw), 588 (w), 573 (w), 515 (vw), 494 (w). The ^1H NMR spectrum of a sample sublimed after recrystallization and dissolved in C_6D_6 was identical with that observed for a sample purified by recrystallization.

Synthesis of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$. Ligand Redistribution Reaction in Benzene. A 100-mL Schlenk flask was charged with 0.468 g (1.44 mmol) of $In(CH_2SiMe_3)_2Cl$ and 0.422 g (1.44 mmol) of $In(CH_2CMe_3)_2Cl$, and the components were dissolved in 30 mL of benzene. After the reaction mixture was stirred for 92 h at ambient temperature, the benzene was removed to leave 0.833 g of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ (2.70 mmol, 93.7%) as a colorless solid. **$In(CH_2CMe_3)(CH_2SiMe_3)Cl$**: mp 93–94 °C. The ^1H NMR spectrum (C_6D_6) was identical with the spectrum previously described.

Synthesis of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$. Ligand Redistribution Reaction without Solvent. A tube equipped with a 20-mm Solv-Seal joint was charged with stoichiometric quantities of $In(CH_2CMe_3)_2Cl$ (0.182 g, 0.622 mmol) and $In(CH_2SiMe_3)_2Cl$ (0.202 g, 0.622 mmol), and the contents were heated to 110 °C. After the two solids had melted (approximately 10 min), heating was stopped. The liquid solidified to a colorless crystalline solid. **$In(CH_2CMe_3)(CH_2SiMe_3)Cl$**: mp 94–96 °C. The ^1H NMR spectrum (C_6D_6) was identical with the spectrum previously described.

Synthesis of $In(CH_2CMe_3)(C_6H_5)Cl$. A solution of $Li(C_6H_5)$ (4.1 mL, 1.8 M, 7.4 mmol) in cyclohexane/Et_2O was added slowly at 0 °C to 30 mL of a solution of $In(CH_2CMe_3)Cl_2$ (1.862 g, 7.492 mmol) in Et_2O. The reaction mixture was stirred for 18 h at ambient temperature, and then the solvent was removed by vacuum distillation. The product $In(CH_2CMe_3)(C_6H_5)Cl$ was separated from the LiCl by extraction using 30 mL of pentane and then washed with 10 mL of pentane at −15 °C to obtain 1.658 g (5.553 mmol, 76.60% based on $In(CH_2CMe_3)Cl_2$) of $In(CH_2CMe_3)(C_6H_5)Cl$ as a colorless solid. The product then was recrystallized from methylcyclohexane at −15 °C. **$In(CH_2CMe_3)(C_6H_5)Cl$**: mp 126–131 °C; sublimes at 90 °C (0.01

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Synthesis of (Me_3CCH_2)(Me_3SiCH_2)InPEt$_2$. A side-arm dumper was charged with 0.378 g (3.94 mmol) of LiPEt$_2$ and attached to a 100-mL two-neck flask that was charged with 1.215 g (3.937 mmol) of In(CH_2CMe_3)(CH_2SiMe_3)Cl. After the indium reagent was dissolved in 25 mL of EtO$_2$, the solution was cooled to −78 °C and the LiPEt$_2$ was slowly added with stirring. After the reaction mixture was stirred for 8 h at room temperature, the Et$_2$O was removed by vacuum distillation. The product (CH_2CMe_3)(CH_2SiMe_3)InPEt$_2$ was separated from the LiCl by extraction using 30 mL of pentane. The resulting thick, colorless oil solidified upon standing to a tacky, colorless, semicrystalline material. The product was purified by sublimation to yield 1.157 g (3.193 mmol, 81.10%) of (CH_2CMe_3)(CH_2SiMe_3)InPEt$_2$. **(CH_2CMe_3)(CH_2SiMe_3)InPEt$_2$:** mp 45–50 °C; sublimes at 100 °C (0.01 mm). ^{1}H NMR (C_6D_6, δ) (see Results and Discussion): −0.38 (br, 2.0 H, CH_2Si), 0.04, 0.05, 0.06, 0.062, 0.07 (s, 9.2 H combined, $SiMe_3$), 0.87 (p, J = 7.2 Hz, 6.8 H, CH_3), 0.98, 0.99, 1.00, 1.01, 1.02 (s, 12.2 H combined, CH_2 and CMe_3), 1.62 (br, 4.0 H, PCH_2–). $^{31}P\{^1H\}$ NMR (C_6D_6, δ): −47.28 (s, 1.0), −48.74 (s, 3.0), −49.85 (s, 2.3), −50.10 (s, 1.4), −50.20 (s, 1.3), −51.26 (s, 3.5), −52.37 (s, 1.2). Anal. Calcd: C, 43.09: H, 8.90; P, 8.55. Found: C, 42.83; H, 8.97; P, 8.15. Cryoscopic molecular weight, benzene solution, formula weight 362.27 (observed molality, observed M_r, association): 0.0776, 780, 2.15; 0.0593, 771, 2.13; 0.0489, 773, 2.13. IR (Nujol mull, cm^{-1}): 2730 (vw), 2710 (vw), 1415 (m), 1357 (m), 1285 (w), 1252 (m), 1240 (vs), 1234 (sh), 1107 (w), 1093 (w), 1083 (m), 1022 (m), 1010 (m), 995 (m), 948 (s), 850 (vs), 820 (vs), 745 (vs), 719 (s), 680 (s), 605 (vw), 572 (m), 555 (m), 475 (w), 447 (w), 380 (vw).

Collection of X-ray Diffraction Data for [In(CH_2CMe_3)(CH_2SiMe_3)Cl]$_2$. The crystal selected for the diffraction study (dimensions 0.6 × 0.2 × 0.25) was sealed into a thin-walled capillary under anaerobic conditions and was aligned on a Siemens R3m/V diffractometer with its extended direction (a) close to a coincident with the ϕ-axis. Details of data collection appear in Table I.

The crystal belongs to the triclinic system, possible space groups being the noncentrosymmetric $P1$ and the centrosymmetric $P\bar{1}$. Intensity statistics favored the choice of $P\bar{1}$; this was confirmed by the successful solution of the structure. All data were corrected for Lorentz and polarization effects and for absorption. A total of 5258 reflections were collected (a complete shell for 2θ = 5–50° for Mo Kα radiation) and merged (R_{int} = 1.36%) to a unique set of 2629 reflections.

Solution and Refinement of the Structure of [In(CH_2CMe_3)(CH_2SiMe_3)Cl]$_2$. All crystallographic calculations were carried out with use of the Siemens SHELXTL PLUS program set.[20] The analytical scattering factors for neutral atoms were corrected for both components ($\Delta f'$ and $i\Delta f''$) of anomalous dispersion.[21]

The structure was solved by a combination of automatic Patterson search and direct methods. Positional and anisotropic thermal parameters of all non-hydrogen were refined. Hydrogen atoms were not located directly but were input in calculated positions with d(C–H) = 0.96 Å[22] and the appropriate staggered tetrahedral geometry. Refinement of the ordered model converged with R = 5.15% and R_w = 7.86% for 110 parameters refined against those 1503 reflections with $|F_o| > 6\sigma(|F_o|)$, and R = 8.69% for all 2629 reflections.

Table I. Structure Determination Summary

	Crystal Data
empirical formula	$C_{18}H_{44}Cl_2In_2Si_2$
color; habit	colorless
crystal size (mm)	0.60 × 0.25 × 0.20
crystal system	triclinic
space group	$P\bar{1}$
unit cell dimensions	a = 6.1000 (10) Å
	b = 10.337 (3) Å
	c = 12.190 (3) Å
	α = 77.28 (2)°
	β = 84.41 (2)°
	γ = 84.50 (2)°
volume	744.0 (3) Å^3
Z	1
formula weight	617.3
calcd density	1.378 Mg/m^3
absorption coefficient	1.789 mm^{-1}
F(000)	312
	Data Collection
diffractometer used	Siemens R3m/V
radiation	Mo Kα (λ = 0.710 73 Å)
temperature (K)	298
monochromator	highly oriented graphite crystal
2θ range	5.0–50.0°
scan type	2θ–θ
scan speed	constant; 2.00°/min in ω
scan range (ω)	0.47° plus Kα separation
background measurement	stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
standard reflections	3 measured every 97 reflections
index ranges	$-7 \le h \le 7$, $-12 \le k \le 12$, $-14 \le l \le 14$
reflections collected	5258
independent reflections	2629 (R_{int} = 1.36%)
observed reflections	1503 ($F > 6.0\sigma(F)$)
absorption correction	semiempirical
min/max transmission	0.5650/0.6258

A careful survey of our structure led to the following observations.

(1) The R factors seemd high when compared to the internal consistency of the data collected (i.e., R_{int} = 1.36% for averaging 2629 equivalent pairs of reflections).

(2) The thermal parameters for all atoms were high. Those for the external methyl groups were higher than any that we had even encountered in an ordered structure, with U_{eq} values of 0.150–0.445 Å^2 (B_{eq} = 11.8–35.1 Å^2).

(3) The thermal ellipsoids of C(Me) atoms appeared to represent a "wagging" motion of the CMe_3 system, in a direction perpendicular to the In–C(α)–[C(β) or Si] plane, in addition to the more normal libration about the C(α)–[C(β) or Si] axis.

(4) The thermal parameters for Si(1) were much *larger* than those for the bonded inner atom C(11) (U_{eq} = 0.148 Å^2 vs 0.107 Å^2), while those for C(22) were much *smaller* than those for the bonded inner atom C(21) (U_{eq} = 0.090 Å^2 vs 0.127 Å^2).

(5) One C–Me bond was alarmingly short ((C(22)–C(25) = 1.18 (5) Å) even though it clearly could be increased by a "riding" correction for librational motion.

Since the CH_2CMe_3 and CH_2SiMe_3 ligands are very similar in size and shape, we decided to check on possible disorder of these ligands in the crystal lattice, even though this had not been indicated by difference Fourier maps. We therefore refined an atom at the site of Si(1) as a composite silicon and carbon atom (respective occupancies x and $1 - x$) and an atom at the site of C(22) as a composite carbon and silicon atom (occupancies y and $1 - y$). Refinement led to the independent values of x = 0.724 (26) and y = 0.729 (37), indicating that while the net *composition* of the material is secure, there is a minor scrambling (~73%:27%) of CH_2CMe_3 and CH_2SiMe_3 ligands at the two crystallographically unique ligand sites.

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Table II. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$)[a]

	x	y	z	U(eq)[a]
In(1)	-2206 (1)	269 (1)	1199 (1)	76 (1)
Cl(1)	2162 (4)	182 (3)	796 (2)	94 (1)
Si(1)	-1320 (10)	-2340 (5)	3400 (4)	125 (3)
C(11)	-2738 (23)	-1622 (12)	2255 (11)	109 (5)
C(12)	-233 (53)	-1311 (20)	4051 (19)	289 (23)
C(13)	1133 (48)	-3337 (24)	2753 (33)	288 (24)
C(14)	-2684 (34)	-3626 (19)	4318 (14)	161 (9)
C(21)	-2948 (31)	2379 (13)	879 (14)	136 (8)
C(22)	-2846 (18)	3218 (10)	1781 (10)	130 (6)
C(23)	-3636 (53)	4680 (19)	1453 (25)	260 (19)
C(24)	-1048 (52)	2851 (25)	2563 (30)	269 (21)
C(25)	-4553 (76)	2576 (41)	2631 (41)	344 (35)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

This disordered model yielded $R = 4.88\%$ and $R_w = 5.28\%$ for the 1503 reflections with $|F_o| > 6\sigma(|F_o|)$ and $R = 8.30\%$ for all 2629 reflections. The previously anomalous features of the structure were all improved. U_{eq} values for methyl carbons are now 0.161–0.344 Å^2 (B_{eq} = 12.7–27.2 Å^2), the orientations of the ellipsoids are now closer to those expected for librational motion, U_{eq} values for α– and β–atoms are more internally consistent, and the C(22)–C(25) bond length is now acceptable at 1.490 (25) Å.

The largest features on a final difference Fourier map were in the range −0.51 to +0.81 e/Å^3. (In the ordered structure, these were little different at −0.71 to +0.81 e/Å^3.)

Final atomic coordinates are listed in Table II.

Results and Discussion

Three new indium compounds containing two different organic substituents, $In(CH_2CMe_3)(CH_2SiMe_3)Cl$, $In(CH_2CMe_3)(C_6H_5)Cl$, and $(Me_3CCH_2)(Me_3SiCH_2)InPEt_2$, have been prepared, purified, and characterized. The identity of the first compound in the above series as a dimer, $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$, was confirmed by an X-ray structural study. The elemental analyses (C, H, and P, as appropriate) of purified samples of all three compounds were consistent with the above formulae. Cryoscopic molecular weight measurements in benzene solutions of all three compounds identified the existence of dimeric molecules in solution. Spectroscopic NMR spectral data were consistent with the presence of cis and trans isomers. Attempts to prepare the related organogallium compounds, $Ga(Me)(CH_2CMe_3)Cl$ and $Ga(CH_2CMe_3)(CH_2CMe_2Ph)Cl$, were unsuccessful. Ligand redistribution reactions led to the formation and subsequent isolation of mixtures of the appropriate symmetrized products.

The compounds $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ and $In(CH_2CMe_3)(C_6H_5)Cl$ were synthesized by metathetical reactions between $In(CH_2SiMe_3)Cl_2$ or $In(CH_2CMe_3)Cl_2$ and the appropriate organolithium compounds in Et_2O solutions. In addition, $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ was prepared by ligand redistribution reactions between $[In(CH_2SiMe_3)_2Cl]_2$ and $[In(CH_2CMe_3)_2Cl]_2$, either in neat form at 110 °C or in benzene at room temperature. The products were purified by recrystallization and/or sublimation. The partial elemental analyses, melting points, and the X-ray structural study of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ confirm the purified products to be single compounds in the solid phase. Furthermore, samples of $In(CH_2CMe_3)(CH_2SiMe_3)Cl$ prepared by different routes and/or purified by different methods gave samples with essentially identical NMR spectra.

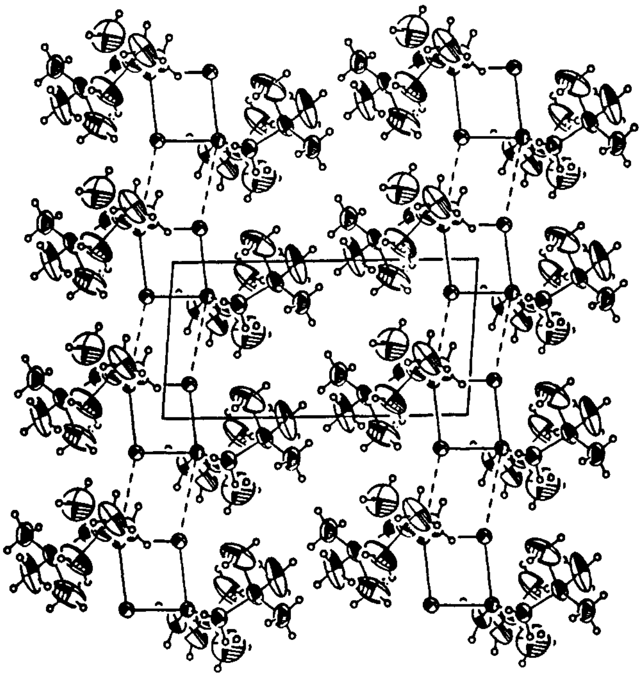

Figure 1. Packing diagram viewed down the *b*-axis with *a* vertical and *c* horizontal. Note the intermolecular contacts running down *a*.

Table III. Bond Lengths and Bond Angles

Bond Lengths (Å)			
In(1)–Cl(1)	2.659 (3)	In(1)–C(11)	2.125 (12)
In(1)–C(21)	2.140 (13)	In(1)–Cl(1A)	2.572 (3)
Cl(1)–In(1A)	2.572 (3)	Si(1)–C(11)	1.704 (14)
Si(1)–C(12)	1.676 (28)	Si(1)–C(13)	1.938 (31)
Si(1)–C(14)	1.757 (19)	C(21)–C(22)	1.522 (21)
C(22)–C(23)	1.520 (22)	C(22)–C(24)	1.490 (36)
C(22)–C(25)	1.490 (45)		
Bond Angles (deg)			
Cl(1)–In(1)–C(11)	103.1 (4)	Cl(1)–In(1)–C(21)	99.7 (5)
C(11)–In(1)–C(21)	148.1 (6)	Cl(1)–In(1)–Cl(1A)	84.5 (1)
C(11)–In(1)–Cl(1A)	102.9 (4)	C(21)–In(1)–Cl(1A)	101.3 (5)
In(1)–Cl(1)–In(1A)	95.5 (1)	C(11)–Si(1)–C(12)	116.8 (9)
C(11)–Si(1)–C(13)	102.5 (12)	C(12)–Si(1)–C(13)	106.9 (15)
C(11)–Si(1)–C(14)	113.0 (9)	C(12)–Si(1)–C(14)	114.2 (10)
C(13)–Si(1)–C(14)	101.2 (10)	In(1)–C(11)–Si(1)	125.1 (8)
In(1)–C(21)–C(22)	123.0 (9)	C(21)–C(22)–C(23)	116.3 (15)
C(21)–C(22)–C(24)	118.1 (15)	C(23)–C(22)–C(24)	117.4 (18)
C(21)–C(22)–C(25)	98.5 (22)	C(23)–C(22)–C(25)	106.4 (20)
C(24)–C(22)–C(25)	93.8 (23)		

The structure of a crystal of neopentyl[(trimethylsilyl)methyl]indium(III) chloride grown by slow sublimation consists of dimeric units of $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ (having crystallographically imposed $\bar{1}$ (C_i) symmetry), which are associated with intermolecular In···Cl contacts of 3.528 (3) Å and a Cl–In···Cl($-1 + x, y, z$) angle of 160.4 (1)° along the a–axis (see Figure 1). Interatomic distances and angles are collected in Table III, while an atomic labeling diagram is provided as Figure 2. It should be noted that all atoms are showing substantial "amplitudes of thermal motion", probably an artifical effect caused by disorder.

The In_2Cl_2 core is required to be strictly planar and has inequivalent In–Cl distances with In(1)–Cl(1) = In(1A)–Cl(1A) = 2.659 (3) Å and Cl(1)–In(1A) = Cl(1A)–In(1) = 2.572 (3) Å; internal angles are In(1)–Cl(1)–In(1A) = 95.5 (1)° and Cl(1)–In(1)–Cl(1A) = 84.5 (1)°. The CH_2SiMe_3 and CH_2CMe_3 ligands are associated with indium–carbon lengths of In(1)–C(11) = 2.125 (12) Å and In(1)–C(21) = 2.140 (13) Å, respectively, with an interligand angle of C(11)–In(1)–C(21) = 148.1 (6)°. These indium–carbon and indium–chlorine distances are similar to those observed for $[In(C_6Me_3H_2)_2Cl]_2$, the only other structurally characterized simple, four-coordinate indium chloride bridged dimer. The In–C distances in $[In(C_6Me_3H_2)_2Cl]_2$[23] are

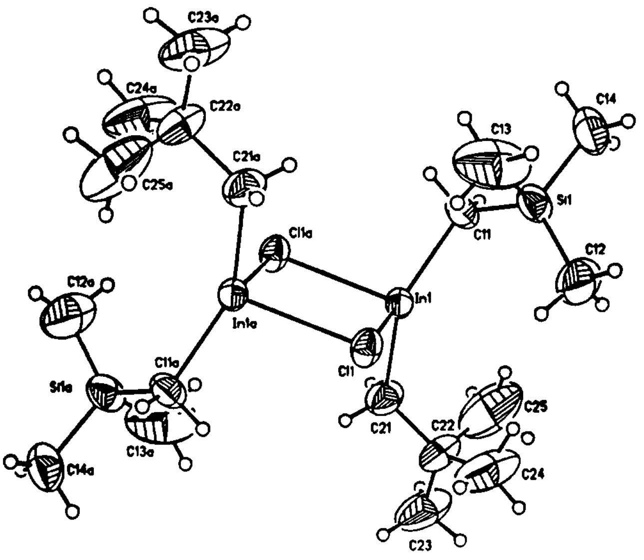

Figure 2. Labeling of atoms in the $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ molecule. ORTEPII diagram with 20% probability ellipsoids and with hydrogen atoms artifically reduced.

2.146 (9) and 2.174 (10) Å whereas the In–Cl distance is 2.574 (3) Å. The other structurally characterized organoindium chloride derivatives, $InMe_2Cl$,[24] $InMeCl_2$,[25] and $In(CH_2CMe_3)Cl_2$,[6] involve infinite chains in the solid state with trigonal-bipyramidal geometry about indium. The geometry of the α-carbon atom of each ligand in $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ is perturbed from the regular sp^3 angle of 109.5° with In(1)–C(11)–Si(1) = 125.1 (8)° and In(1)–C(21)–C(22) = 123.0 (9)°.

The structure of $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ involves some slight disorder. The "CH_2SiMe_3 site" is occupied ~73% by CH_2SiMe_3 and ~27% by CH_2CMe_3; similar structures apply, mutatis mutandis, to the "CH_2CMe_3" site. The interior "bonds" to the β-atoms, C(11)–Si(1) = 1.704 (14) Å and C(21)–C(22) = 1.552 (21) Å, should be compared to the weighted average of their components, rather than to true C–Si and C–C bond lengths. C–Si = 1.865 ± 0.008 Å for alkylsilanes[26] and C(α)–Si = 1.848 (9)–1.860 (8) Å for the CH_2SiMe_3 ligands in $[(Me_3SiCH_2)_2InPPh_2]_2$;[27] C–C = 1.541 ± 0.003 Å in alkanes[26] and C(α)–C(β) = 1.478 (13)–1.549 (9) Å for the CH_2CMe_3 ligand in $[(Me_3CCH_2)_2InPPh_2]_3$.[28] A 73%:27% average of the bond lengths from ref 26 would yield theoretical C(11)–"Si(1)" and C(21)–"C(22)" distances of 1.78 and 1.62 Å, respectively. Other "composite distances" observed are "Si(1)-"–Me = 1.676 (28)–1.938 (38) Å and "C(12)"–Me = 1.490 (36)–1.520 (22) Å.

The ^{13}C and ^1H NMR spectra of $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ are consistent with the existence in solution of an approximately equimolar mixture of cis and trans isomers. The ^{13}C NMR spectrum consists of ten lines, two sets of five lines each. One set of lines must be related to the cis isomer and the other set to the trans isomer. The (trimethylsilyl)methyl substituent exhibits two lines, whereas the neopentyl group has three lines for each isomer. None of the ten lines correspond with the ^{13}C NMR chemical shifts (δ) of lines observed for pure samples[29] of $[In(CH_2CMe_3)_2Cl]_2$ (32.43 (C), 34.69 (Me), 42.63 (CH_2)) and $[In(CH_2SiMe_3)_2Cl]_2$ (2.21 (Me), 10.44 (CH_2)). Thus, the ^{13}C NMR spectrum is inconsistent with an equilibrium between $[In(CH_2CMe_3)_2Cl]_2$, $[In(CH_2SiMe_3)_2Cl]_2$, and $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$. It is regrettable that it is not possible to assign a given set of lines to a specific isomer. The ^1H NMR spectrum has eight lines consisting of four pairs of lines. The two lines of a given pair are of similar intensities. These lines can be alternatively described as two sets of four lines. One set of four lines originates from the methylene and methyl protons of the neopentyl and (trimethylsilyl)methyl groups for the cis isomer; the other set is for the trans isomer. The lines due to the trans isomer have unique chemical shifts whereas the lines assigned to the cis isomer are essentially identical with those observed for $[In(CH_2CMe_3)_2Cl]_2$ and $[In(CH_2SiMe_3)_2Cl]_2$, as would be expected. The cis isomer by virtue of its structure has a "neopentyl group side" and a "(trimethylsilyl)methyl group side." Consequently, the interaction between substituent and solvent would be expected to be very similar to that in the corresponding diorganoindium chloride. Thus, the chemical shifts of the respective protons should be the same as that observed for the pure diorganoindium chloride dimer.

An alternative explanation for the ^1H NMR spectrum of benzene solutions of $[In(CH_2CMe_3)(CH_2SiMe_3)Cl_2]$, which involves an equilibrium mixture of $[In(CH_2CMe_3)_2Cl]_2$, $[In(CH_2SiMe_3)_2Cl]_2$, and $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$, is inconsistent with a variety of experimental observations, with the literature, and with the ^{13}C NMR spectrum, as previously discussed. If an equilibrium did exist, then of the observed eight ^1H NMR lines two lines would be assigned to $[In(CH_2CMe_3)_2Cl]_2$, two to $[In(CH_2SiMe_3)_2Cl]_2$, and four to $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$. The molecule $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ would either have to be undergoing fast exchange on the NMR time scale in order to average the chemical shifts of the lines for the different substituents for the cis and trans isomers or have to exist as only one isomer. When $[In(CH_2CMe_3)_2Cl]_2$ and $[In(CH_2SiMe_3)_2Cl]_2$ were mixed in benzene solution, the four lines expected for the two reactants were observed. Fast exchange between the reactants did not occur. Similarly, fast exchange was not observed for mixtures[6] of $[In(CH_2CMe_3)_2Cl]_2$ and $[In(CH_2CMe_3)Cl_2]_2$. The ^1H NMR study of the equimolar mixture of $[In(CH_2CMe_3)_2Cl]_2$ and $[In(CH_2SiMe_3)_2Cl]_2$ further revealed with time that four new lines appeared and increased in intensity and the four original lines decreased in intensity until the new and the old lines in each of the four pairs of lines were of approximately equal intensity (~72 h). A low-temperature ^1H NMR spectral study is inconsistent also with the proposal of a fast exchange between cis and trans isomers; the four "new" lines did not broaden and split as the sample was cooled. Instead, the lines for the CH_2SiMe_3 protons changed (moved and broadened) before a change was observed in the lines for the neopentyl group protons. Thus, complex rotational and/or dissociative processes (which will be discussed in a future paper) are occurring. In conclusion, the ^1H NMR spectrum is best explained by an approximately equimolar mixture of cis and trans isomers. A mixture of $[In(CH_2CMe_3)_2Cl]_2$, $[In(CH_2SiMe_3)_2Cl]_2$, and $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ in only one isomer conformation to give four pairs of lines of equal intensity is incon-

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sistent with the ^{13}C NMR spectrum and seems unlikely. Such a mixture in solution would be expected to give an impure product when the solvent was removed. Instead, a pure compound was obtained.

The hypothesis, that $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ exists as a single compound in solution rather than as an equilibrium mixture with $[In(CH_2CMe_3)_2Cl]_2$ and $[In(CH_2SiMe_3)_2Cl]_2$, is also supported by the observation that the compound reacts with $LiPEt_2$ in Et_2O solution at −78 °C to form $(Me_3CCH_2)(Me_3SiCH_2)InPEt_2$ and LiCl. The indium–phosphorus product, initially isolated as a thick, colorless oil, which crystallized upon standing, was purified by sublimation at 100 °C. The cryoscopic molecular weight study of the product in benzene solution identified the existence of dimeric molecules in solution. The most likely structure for the dimer would involve bridging PEt_2 groups as observed for $[(Me_3SiCH_2)_2InPPh_2]_2$,[30] $[(Me_3CCH_2)_2InPPh_2]_3$,[28] and other related groups 13–15 compounds.[31] The $^{31}P\{^1H\}$ NMR spectrum of seven lines suggests that the species in solution must have a variety of conformations including cis and trans isomers of planar and/or puckered indium–phosphorus rings. No line had a chemical shift that was identical with that observed for $[(Me_3CCH_2)_2InPEt]_2$[32] (δ −52.6). The observation that all lines for $[(Me_3CCH_2)(Me_3SiCH_2)InPEt_2]_2$ occurred in the narrow range of 5 ppm is consistent with the presence of species of only one degree of association, i.e., dimer. If planar and/or puckered rings are present, the rate of fluxionality between the conformations must be slow on the NMR time scale. The rather complex ^{1}H NMR spectrum is also consistent with multiple conformations and isomers.

The compound $In(CH_2CMe_3)(C_6H_5)Cl$ was prepared by reacting $In(CH_2CMe_3)Cl_2$ with $Li(C_6H_5)$ in Et_2O at 0 °C. The colorless, crystalline product was purified by recrystallization from methylcyclohexane or by sublimation at 90 °C. Molecular weight studies indicate that $In(CH_2CMe_3)(C_6H_5)Cl$ exists in benzene solution as dimeric molecules. The most likely structure for a dimer would involve chlorine bridges as observed for $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$. The ^{1}H and ^{13}C NMR spectra of the compound in benzene solution are consistent with the presence of cis and trans isomers and the absence of an equilibrium mixture of $[In(CH_2CMe_3)_2Cl]_2$, $[In(C_6H_5)_2Cl]_2$, and $[In(CH_2CMe_3)(C_6H_5)Cl]_2$. Two singlets were observed for the methylene protons, two singlets for methyl group protons, and two sets of lines for phenyl protons. The low-temperature (−85 °C) ^{1}H NMR spectrum did not show any splitting of the original lines for methylene and methyl protons as might be expected if two of the lines were due to neopentyl group protons in $[In(CH_2CMe_3)(C_6H_5)Cl]_2$, as previously described. Further confirmation of the absence of an equilibrium comes from the ^{13}C NMR spectrum. The neopentyl group CH_2 and CMe_3 carbon atoms exhibit two sets of lines with none corresponding to $[In(CH_2CMe_3)_2Cl]_2$. It is regrettable that the ^{13}C NMR spectrum of $[In(C_6H_5)_2Cl]_2$ is not available for comparison.

The successful synthesis and characterization of organoindium compounds with two different organic substituents prompted us to attempt the preparation of related organogallium chlorine compounds. However, the syntheses of $Ga(Me)(CH_2CMe_3)Cl$ and $Ga(CH_2CMe_3)(CH_2CMe_2Ph)Cl$ by metathesis and/or ligand redistribution reactions were unsuccessful. Our characterization data of the materials isolated after the utilization of separation and purification techniques confirmed that ligand redistribution reactions, which gave symmetrical products of the types GaR_2Cl and $GaR_2'Cl$, had occurred. Thus, the more symmetrical products must be thermodynamically more stable than the unsymmetrically substituted derivatives in gallium chemistry whereas the opposite appears to be true in indium chemistry. The reasons behind these observations are currently under investigation.

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Supplementary Material Available: Listings of anisotropic thermal parameters and calculated positions of hydrogen atoms (1 page); a listing of observed and calculated structure factors for $[In(CH_2CMe_3)(CH_2SiMe_3)Cl]_2$ (10 pages). Ordering information is given on any current masthead page.

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