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Synthesis and molecular structure of $CH_3\{2,6\text{-}[(C_2H_5)_2NCH_2]_2C_6H_3\}InCl$, an intramolecular stabilized monomeric diorganoindium chloride *

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Abstract

$InCl_3$ reacts with $2,6\text{-}[(CH_3)_2NCH_2]_2C_6H_3Li$ in hexane to yield $2,6\text{-}[(CH_3)_2NCH_2]_2C_6H_3InCl_2$ (**1**). The treatment of CH_3InCl_2 with $2,6\text{-}[(C_2H_5)_2NCH_2]_2C_6H_3Li$ affords $CH_3\{2,6\text{-}[(C_2H_5)_2NCH_2]_2C_6H_3\}InCl$ (**2**). Both compounds have been characterized by elemental analyses, ^1H NMR, ^{13}C NMR and mass spectra. **2** crystallizes monoclinic in the space group $P2_1/n$. The unit cell parameters are a 1090.2(1), b 1354.0(2), c 1300.0(2) pm, α 99.99(1)° and V 1.8899×10^6 pm^3 with a final R value of 0.049 for all 4055 independent reflections.

Introduction

Trimethylindium, the commonly used In source for the production of III–V semiconductor layers by organometallic vapor phase epitaxy [1], is air sensitive because of its coordinative unsaturation. The results presented here were obtained in connection with our investigations to increase the air stability of organoindium compounds via intramolecular base stabilization. Several organoindium (III) derivatives containing $2\text{-}(CH_3)_2NCH_2C_6H_4$ or $CH_3)_2NCH(CH_3)C_6H_4$ as ligands have been prepared recently and have been studied by NMR spectroscopy [2,3]. The crystal structure of $[2\text{-}(CH_3)_2NCH_2C_6H_4]_2InCl$ reveals that both nitrogen atoms coordinate the metal center and occupy the axial positions in the bipyramidal molecule [4].

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* In memoriam Professor Dr. Jerold J. Zuckerman.

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We now report on the synthesis of two organoindium compounds containing the tridentate 2,6-$(R_2NCH_2)_2C_6H_3$ group ($R = CH_3$, C_2H_5) as a ligand. Both compounds have been characterized by elemental analyses, ^1H NMR, ^{13}C NMR and mass spectra.

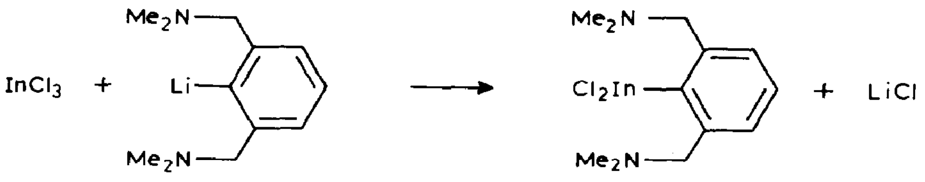
Results and discussion

Indium trichloride reacts in hexane with 2,6-bis(dimethylaminomethylene)phenyllithium to form 2,6-bis(dimethylaminomethylene)phenylindium dichloride (**1**). The reaction between methylindium dichloride and 2,6-bis(diethylaminomethylene)phenyllithium, conducted under the same conditions, affords 2,6-bis(diethylaminomethylene)phenyl(methyl)indium chloride (**2**).

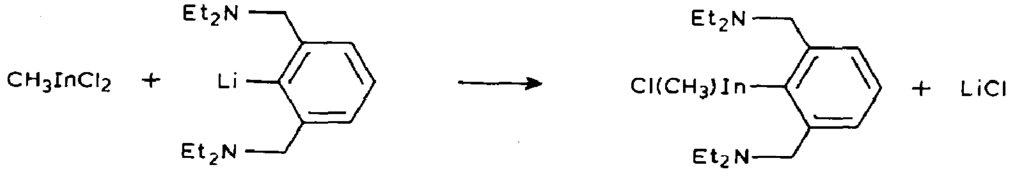
The compounds are colorless solids, melting at 108–110 °C or 52–54 °C, respectively. The ^1H NMR as well as the ^{13}C NMR spectra of the compounds, recorded in C_6D_6, show the expected results. The assigned values are listed in Table 1.

The indium nucleus has a spin of 9/2. The NMR resonances of carbon atoms bound to indium are thus expected to be broadened and of low intensity. The CH_3-In carbon atom in **2** in fact shows a broad signal at -9.3 ppm with a half-width of 8 Hz, as compared to normally about 1.5 Hz, and an intensity of only about 10% of that of a CH_3 group bound to another carbon atom. Since the ring carbon atom C1 of the ligand system, which is directly bound to indium, does not show an intensity enhancement in the broadband decoupled ^{13}C NMR due to the Nuclear Overhauser Effect, whereas H bearing carbons do, this signal for C1 is further diminished and could therefore not be detected.

The molecular structure of **2** has been determined by X-ray diffraction (Table 2, Fig. 1). The crystal packing consists of discrete monomeric units with the short distance of 296 pm between chlorine and a hydrogen atom of the CH_3-In group being the only unusual intermolecular contact (max. sum of Van der Waals radii



(1)



(2)

Table 1

^{1}H NMR and ^{13}C NMR data of **1** and **2**

	1	**2**
^{1}H NMR in C_6D_6, in ppm relative to $Si(CH_3)_4$		
$\delta(CH_3CH_2N)$	–	0.85(t)
$\delta(CH_3CH_2N)$	–	2.59(q)
$\delta(CH_3N)$	2.20(s)	–
$\delta(NCH_2C_6H_3)$	3.05(s)	3.51(s)
$\delta(C_6H_3)$	6.77–7.16(m)	7.00–7.42(m)
$\delta(CH_3In)$	–	0.06(s)
^{13}C-NMR in C_6D_6, in ppm relative to $Si(CH_3)_4$		
$\delta(CH_3CH_2N)$	–	8.7
$\delta(CH_3CH_2N)$	–	44.1
$\delta(CH_3N)$	45.6	–
$\delta(NCH_2C_6H_3)$	63.6	60.2
$\delta(C1)$	not observed	not observed
$\delta(C2,6)$	143.6	145.0
$\delta(C3,5)$	125.6	124.4
$\delta(C4)$	129.9	126.7
$\delta(CH_3In)$	–	-9.3

310 pm) [5]. The atom numbering is shown in Fig. 1, bond distances and bond angles are given in Tables 3 and 4, respectively.

The molecule exhibits distorted trigonal-bipyramidal geometry with the chlorine and the two carbon atoms bound to indium in equatorial and the nitrogen atoms in

Table 2

Final atomic coordinates for **2** with estimated standard deviations in parentheses and equivalent isotropic thermal parameters (Å^2)

Atom	x/a	y/b	z/c	B_{eq}
In	0.36989(3)	0.46814(2)	0.22136(2)	1.34
Cl	0.2938(1)	0.3552(1)	0.0852(1)	3.45
N1	0.5936(3)	0.3999(3)	0.2839(3)	1.22
N2	0.1505(3)	0.4928(2)	0.2714(3)	1.32
C1	0.4153(5)	0.6171(4)	0.1817(4)	1.84
C2	0.4849(4)	0.3744(3)	0.4344(3)	1.25
C3	0.3725(4)	0.4007(3)	0.3720(3)	1.15
C4	0.2606(4)	0.3762(3)	0.4030(3)	1.49
C5	0.2611(4)	0.3244(3)	0.4972(3)	1.71
C6	0.3741(4)	0.2987(3)	0.5582(4)	1.72
C7	0.4854(4)	0.3227(3)	0.5270(3)	1.57
C8	0.6034(4)	0.4068(4)	0.3993(3)	1.43
C9	0.6932(4)	0.4573(4)	0.2493(3)	1.77
C10	0.6871(5)	0.4562(4)	0.1315(4)	2.23
C11	0.5922(4)	0.2951(3)	0.2503(3)	1.54
C12	0.7066(5)	0.2339(4)	0.2933(4)	2.23
C13	0.1400(4)	0.3996(3)	0.3292(3)	1.46
C14	0.1666(4)	0.5793(3)	0.3413(3)	1.42
C15	0.0736(4)	0.5914(4)	0.4143(4)	2.16
C16	0.0429(4)	0.5037(3)	0.1849(3)	1.81
C17	0.0616(4)	0.5812(4)	0.1057(3)	1.96

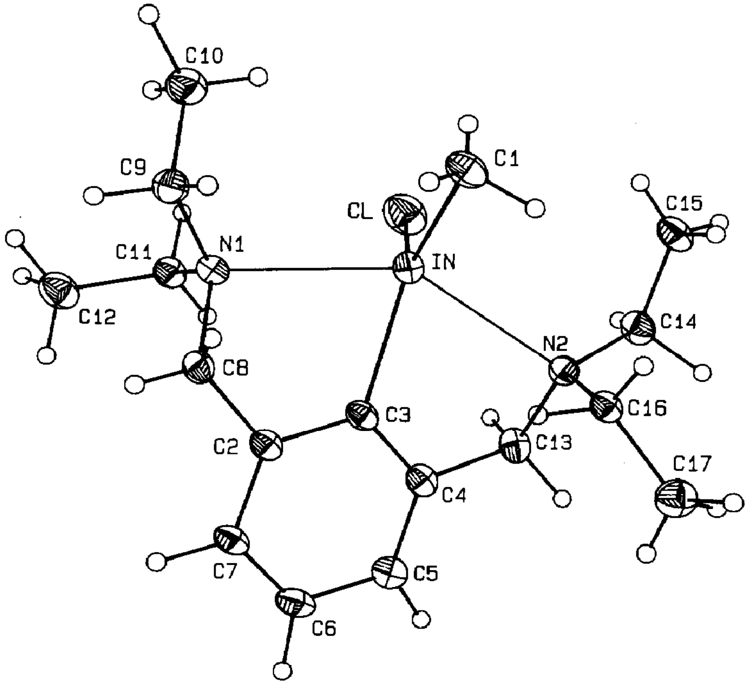


Fig. 1. ORTEP drawing [27] of **2** with 50 % probability ellipsoid for all atoms other than hydrogen. Hydrogens drawn for clarity with arbitrary radius.

axial position. The maximum deviation from the equatorial least-squares plane of In, C1, C3 and Cl is 2.7(3) pm for In. The considerable distortion of the metal coordination from the ideal trigonal-bipyramidal symmetry is caused by the geometry of the ligand. The bond angle N1–In–N2 (144.9(1)°) is significantly smaller than the respective angle in $[2\text{-}(CH_3)_2NCH_2C_6H_4]_2InCl$ (178.4(1)°) [4].

It is known that InL_3 (L = monodentate organic or inorganic ligand) forms stable adducts with nucleophiles like Et_2O, NR_3, HNR_2 and H_2NR [6]. The observed intramolecular N–In–N coordination in **2** emphasizes the acceptor beha-

Table 3

Final non-hydrogen bond distances (pm) in **2** with estimated standard deviations in parentheses

Atoms	Distance	Atoms	Distance
In–Cl	237.7(1)	C2–C7	139.2(6)
In–N1	260.3(3)	C2–C8	150.9(6)
In–N2	260.7(3)	C3–C4	139.0(6)
In–C1	216.0(5)	C4–C5	141.0(6)
In–C3	215.6(4)	C4–C13	152.1(6)
N1–C8	148.9(5)	C5–C6	138.9(6)
N1–C9	146.8(6)	C6–C7	138.3(7)
N1–C11	148.4(5)	C9–C10	152.1(7)
N2–C13	148.4(5)	C11–C12	152.0(6)
N2–C14	147.3(5)	C14–C15	151.4(7)
N2–C16	148.6(5)	C16–C17	150.9(7)
C2–C3	139.3(5)		

Table 4

Selected non-hydrogen bond angles (°) in **2** with estimated standard deviations in parentheses

Atoms	Angle	Atoms	Angle
C1–In–Cl	119.2(1)	C7–C6–C5	120.7(4)
C3–In–Cl	110.6(1)	C6–C7–C2	120.0(4)
C3–In–C1	130.1(2)	N1–C8–C2	111.8(3)
N1–In–Cl	101.0(1)	C8–N1–In	101.1(2)
N1–In–C1	99.5(2)	C9–N1–In	114.8(3)
N1–In–C3	72.7(1)	C9–N1–C8	110.3(3)
N2–In–Cl	93.0(1)	C11–N1–In	106.7(2)
N2–In–C1	101.4(2)	C11–N1–C8	110.6(3)
N2–In–C3	72.3(1)	C11–N1–C9	112.8(4)
N2–In–N1	144.9(1)	C10–C9–N1	113.5(4)
C7–C2–C3	120.2(4)	C12–C11–N1	116.6(3)
C8–C2–C3	117.5(4)	N2–C13–C4	111.2(3)
C8–C2–C7	122.2(4)	C13–N2–In	99.9(2)
C2–C3–In	120.6(3)	C14–N2–In	103.8(2)
C4–C3–In	119.4(3)	C14–N2–C13	112.1(3)
C4–C3–C2	119.8(4)	C16–N2–In	117.5(3)
C5–C4–C3	120.0(4)	C16–N2–C13	110.3(3)
C13–C4–C3	118.3(4)	C16–N2–C14	112.5(3)
C13–C4–C5	121.6(4)	C15–C14–N2	116.8(4)
C6–C5–C4	119.3(4)	C17–C16–N2	113.8(3)

vior of indium. The In–N distances are 260.3(3) and 260.7(3) pm, thus being longer than the sum of their covalent radii (219 pm) but significantly shorter than the sum of their Van der Waals radii (345 pm). Comparetively long In–N distances are reported for $CH_3In[CH_3NC(CH)_4N]_2$ (213.8(5) pm) [7], $[(CH_3)_2In]_2C_2(NCH_3)_4$ (218.3(5) pm) [8], $CH_3In\{[CH_3(CH_2)_2NCH_3]In(CH_3)_2\}_2$ (218.7(13) pm) [7], methyl(tetraphenylporphinato)indium(III) (220(1) pm) [9], $CH_3InCl_2 \cdot t\text{-}C_4H_9NH_2$ (221(1) pm) [10], $(CH_3)_2InN(CH_3)_2$ (223(1) pm) [11], $C_2H_5InBr_2 \cdot [(CH_3)_2NCH_2CH_2N(CH_3)_2]$ (230(1) and 249(1) pm) [12] and $C_2H_5InI_2 \cdot [(CH_3)_2NCH_2CH_2N(CH_3)_2]$ (233(1) and 244(1) pm) [12]. In the case of **2**, the ligand constraint prevents the nitrogens from moving closer to the metal center.

The dihedral angles C3–C2–C8–N1 (38.4(5)°) and C3–C4–C13–N2 (35.3(5)°) show out of plane bending of both nitrogens. N1 resides 75.6(3) pm below, N2 60.6(3) pm above the phenyl plane. The twist, most probably due to the coordination forces, affects carbon atoms C8 and C13, which bend away from the phenyl plane. The deviation is reflected in the dihedral angles C6–C7–C2–C8 (177.0(4)°) and C6–C5–C4–C13 (175.4(4)°).

The geometry of Cl, C1 and C3 around the indium center is not ideally trigonal. The bond angles C1–In–Cl, C3–In–Cl and C1–In–C3 are 119.2(1), 110.6(1) and 130.1(2)°. A similar lack of symmetry is reported for $[2\text{-}(CH_3)_2NCH_2C_6H_4]_2InCl$ (the respective angles are 102.9(1), 103.7(1) and 153.3(1)°) [4] and for $CH_3InCl_2 \cdot t\text{-}C_4H_9NH_2$ (Cl1–In–Cl 2 100.5(1)°, Cl1–In–C 115.9(3)° and Cl 2–In–C 119.8(3)°) [10]. A comparison of the C–In–C bond angle in **2** with values reported for $In(CH_3)_3$ (almost 120°) [13], $In(C_5H_5)_3$ (110.2(4), 117.7(4) and 118.7(3)°) [14], $In(C_6H_5)_3$ (121.6(3) and 116.8(6)°) [15] and for $Na[In(C_6H_5)_4]$ (110.2(3) and 108.1(3)°) [16] shows it to be significantly increased. We assume that the nitrogen coordination forces the methyl group to bend away from the phenyl ring leading to shortest non-bonding distances N2–H(C1) of 340(1) and 370(1) pm. A similar effect will cause a bending of the chlorine away from the phenyl ring resulting in a non-bonding distance Cl–N1 of 384.6(3) pm. The smaller C–In–Cl angles in $[2\text{-}(CH_3)_2NCH_2C_6H_4]_2InCl$ (102.9(1)° and 103.7(1)°) support this theory.

The bite angle C3–In–N1 (72.7(1)°) and C3–In–N2 (72.3(1)°) belong to the smallest cited in the literature [4]. The In–Cl distance of 237.7(1) pm is in agreement with reported literature values for indium(III) chlorides. The longest distances are found in inorganic compounds like $InCl_5^{2-}$ (241.5(1.2), 245.6(7) pm) [17], $[InCl_4(H_2O)_2]^-$ (248.5(2), 243.3(3), 241.7(3) pm) [18]. Neutral adducts like $InCl_3 \cdot PPh_3$ show similar In–Cl distances (237.7(5), 238.2(5) and 239.1(5) pm) [19]. In–Cl distances reported for organoindium compounds are 238.4(1) and 240.0(1) pm for $(CH_3InCl_2)_2$ [20], 240.5(2) and 239.3(2) pm for $CH_3InCl_2 \cdot t\text{-}C_4H_9NH_2$ [10].

The In–C_{alkyl} and In–C_{aryl} bond lengths (216.0(5) pm and 215.6(4) pm) are within the sum of the covalent radii (221 pm). The observed In–C_{alkyl} distance compares well with the respective values of $In(CH_3)_3$ (215(4), 212(4), 206(4) pm) [13], $(CH_3)_2InN(CH_3)_2$ (217(2) pm) [11], $(CH_3)_2InCl$ (218(1) pm), methyl(tetraphenylporphinato)indium(III) (213(1) pm) [9], $CH_3InCl_2 \cdot t\text{-}C_4H_9NH_2$ (214(1) pm) [10], bis(μ-4-methylpiperazino-*N*)tetramethyldiindium(III) (216.1(6), 216.3(6) pm), methylbis[methyl(2-pyridyl)amido-*N*,*N'*]indium(III) (213.7(7) pm), $CH_3In\{[CH_3N(CH_2)_2NCH_3]In(CH_3)_2\}_2$ (218.7(1.3), 219.8(1.5), 218.2(1.3) pm) [7], and $[(CH_3)_2In]_2C_2(NCH_3)_4$ (218.2(6), 217.3(6), 216.5(5), 218.1(6) pm) [8]. In^{III}–C_{aryl} distances in $[2\text{-}(CH_3)_2NCH_2C_6H_4]_2InCl$ (214.4(3), 215.4(3) pm) [4], in $In(C_6H_5)_3$ (211.1(1.4), 215.5(1.4) pm) [15], in $Na[In(C_6H_5)_4]$ (223.0(3) pm) [16] and in $In(C_5H_5)_3$ (223.7(9), 224.3(9), 237.4(7) and 246.6(8) pm) [14] are in good agreement with the respective values of **2**.

Experimental

All reactions were carried out under an atmosphere of dry oxygen-free argon, in solvents which were dried and distilled under argon before use. $(CH_3)InCl_2$ [21] and $2,6\text{-}[(CH_3)_2NCH_2]_2C_6H_3Li$ [22] were prepared according to literature procedures. $2,6\text{-}[(C_2H_5)_2NCH_2]_2C_6H_3Br$ was prepared from $2,6\text{-}(BrCH_2)_2C_6H_3Br$ [23] and $(C_2H_5)_2NH$ in C_6H_6. ^1H and ^{13}C NMR spectra were recorded on a Bruker WP 80-FT spectrometer at 80 MHz and 20.15 MHz, respectively, and the mass spectra on a Varian MAT 311 mass spectrometer. For the CHN analyses a Perkin–Elmer elemental analyzer 240C was used.

2,6-Bis(dimethylaminomethylene)phenylindium dichloride (1)

The respective lithium reagent, prepared from 1.72 g (6.3 mmol) $2,6\text{-}[(CH_3)_2NCH_2]_2C_6H_3Br$ and 3.96 ml n-butyllithium (1.6 mol/l in hexane) in 30 ml hexane, was added dropwise to a stirred suspension of 1.4 g (6.3 mmol) $InCl_3$ in 30 ml hexane. After stirring for 12 h, the solvent was removed and the product sublimed at 100 °C in vacuo onto a cold finger (15 °C). Yield: 1.51 g (63%) colorless solid, m.p. 108–110 °C. Found: C, 38.86; H, 5.08; N, 7.61. $C_{12}H_{19}Cl_2InN_2$ calcd.: C, 38.23; H, 5.08; N, 7.43%.

{2,6-Bis(diethylaminomethylene)phenyl}methylindium chloride (2)

The reaction mixture obtained after the addition of 9.4 mmol of the lithium reagent in hexane to 1.89 g (9.4 mmol) CH_3InCl_2 was refluxed for 4 h, filtered and the solvent removed in vacuo. Distillation yielded an orange oil (b.p. 160 °C/0.1 mbar), which was crystallized from hexane. Yield: 2.15 g (55%) colorless crystals, m.p. 52–54 °C. Found: C, 49.67; H, 7.28; N, 7.01. $C_{17}H_{30}ClInN_2$ calcd.: C, 49.47; H, 7.33; N, 6.79%. MS: 412 (molecular ion, 3.2%), 397($M - CH_3$, 2.8%), 377(M − Cl, 92.6%), 115 (In, 100%).

X-ray structure analysis

2 crystallizes as white parallelepipeds, monoclinic, crystal dimensions 0.43 × 0.65 × 0.40 mm^3; $P2_1/n$ from intensity statistics and structure refinement, cell parameters from 52 reflections (2θ range 40–53°), Mo-K_α radiation (graphite monochromator), 4472 unique reflections, intensities of all reflections with $1° \leq 2\theta \leq 54°$ measured at 140(5) K, $-14 \leq h \leq 14$, $-17 \leq k \leq 0$, $0 \leq l \leq 16$, θ–2θ scan technique, variable scan width $(0.95 + 0.34 \tan \theta)°$, variable horizontal receiving aperture 2.60 mm; three standard reflections measured every 1.5 h of X-ray exposure time, maximal fluctuation 1.3%, three orientation control reflections measured every 125 data, computation of a new orientation matrix from a list of 25 reflections if angular change in control reflections was more than 0.1°; 125 *h* of total exposure time; decay correction applied, Lorentz and polarization correction, empirical absorption correction (μ 12.72 cm^{-1}, psi scan method, max. transition 99.7% min. transition 86.5%), 4055 observed data (all F_o); structure determined by Patterson method and succesive difference Fourier syntheses; refinement by block-diagonal least squares in which the quantity $\Sigma w(|F_o| - |F_c|)^2$ was minimized; non-hydrogen atoms refined anisotropically, hydrogen atoms located and refined isotropically, final $R = 0.049$, $R_w = 0.040$ for 310 parameters and 4055 reflections (all F_o), maximum ratio of shift to error (Δ/σ) in final difference Fourier map 0.029, maximum electron density 1.64 e $Å^{-3}$ around In position.

All X-ray measurements on an Enraf–Nonius CAD-4 automatic diffractometer fitted with a liquid–nitrogen low temperature set-up; scattering factors for H from Stewart, Davidson and Simpson [24], scattering factors and anomalous dispersion terms for C, N, Cl and In from International Tables for X-Ray Crystallography [25]; all calculations with SHELX76 [26]. The final atomic and thermal parameters are given in Table 2.

Details of the crystal structure determination of **2** have been deposited with the Fachinformationszentrum Energie, Physik, Mathematik, D-5714 Eggenstein, Leopoldshafen 2 and can be obtained by quoting the journal, authors and file number CSD 53651.

Acknowledgements

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