GALLIUM AND INDIUM

ANNUAL SURVEY COVERING THE YEAR 1980

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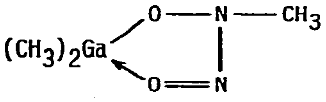
Introduction

This survey describes topics on organogallium and -indium chemistry reported during 1980 in the order of 'Synthesis, Analysis, and Reaction Chemistry' including one patent, and 'Structure and Bonding'.

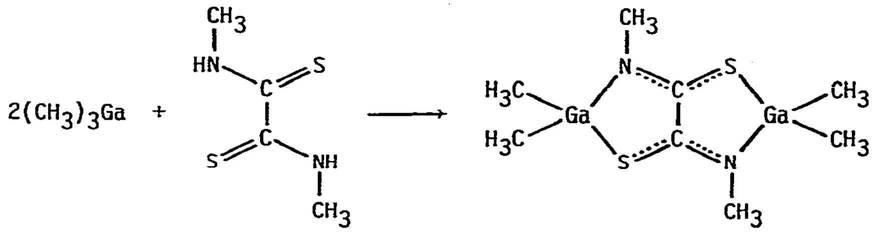
Synthesis, Analysis, and Reaction Chemistry

Nitric oxide was sealed with a pentane solution of $(CH_3)_3Ga$ (2:1 mole ratio) at -196°C, followed by the reaction at 20°C for 24 h, to give white crystals of the adduct, (N-methyl-N-nitrosohydroxylamido-O,O')-dimethylgallium (1) [1]. This reaction is analogous to that of NO with $(CH_3)_3Al$ [S. Amirkhalili, A. J. Conway, and J. D. Smith, *J. Organometal. Chem.*, 149 (1978) 407]. In contrast to the Al derivative, however, 1 does not rearrange to give species with hexa-coordinate gallium. The reaction of $(CH_3)_3Ga$ with N,N'-dimethyldithiooxamic acid at 2:1 mole ratio yielded bis(dimethylgallium)-N,N'-dimethyldithiooxamide (2 in Eq. 1). The vibrational and ^1H-NMR spectra have indicated that 2 exists as two configurational isomers with respect to the positions of N and S, both consisting of two fused five-membered rings with an almost planar structure. The preliminary result of the X-ray structure analysis was reported [2].



1

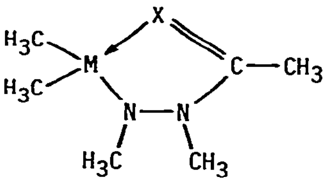
Gallium and Indium; Annual Survey Covering the Year 1979 see *J. Organometal. Chem.*, 207 (1980) 111-120.



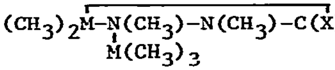
(1)

2

Trimethylgallium and -indium reacted with N,N'-dimethylacethydrazine, $HN(CH_3)-N(CH_3)-C(O)CH_3$, or N,N',N"-trimethylacetimidohydrazine, $HN(CH_3)-N(CH_3)-C(NCH_3)CH_3$, to give monomeric dimethylmetal derivatives with five-membered ring skeletons (3). The heterocyclic compounds thus formed immediately add an

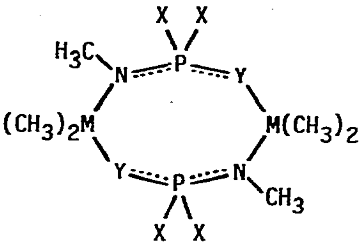


3: M = Ga, In; X = O, NCH_3

additional molecule of $(CH_3)_3M$ (M = Ga or In) to afford the adduct  $(CH_3)_2M-\underset{M(CH_3)_3}{N}(CH_3)-N(CH_3)-C(X)CH_3$, whose ^1H-NMR, IR, and Raman spectra were discussed [3]. X-Ray molecular structures of the two $(CH_3)_3Ga$ adducts will be described in the next section.

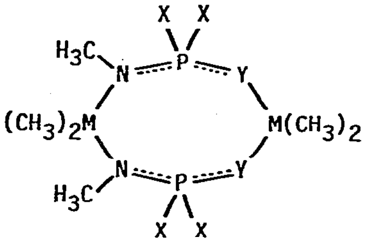
An 1:1 reaction of $(CH_3)_3Ga$ or $(CH_3)_3In$ with phosphonic or phosphinic acid amides, $HN(CH_3)-P(X_2)Y$ (X = F, Cl, CH_3; Y = O, S, but not all combinations), gave dimethylmetal derivatives of these acid amides, $(CH_3)_2MYP(X_2)NCH_3$ (M = Ga, In). Molecular weight determinations in benzene indicated that $(CH_3)_2MOP(X_2)NCH_3$ (M = Ga, X = F, Cl, CH_3; M = In, X = CH_3) is essentially dimeric, while $(CH_3)_2MSP(CH_3)_2NCH_3$ (M = Ga, In) is monomeric. The dimers exist as the structure with a centre of symmetry (4a), as evidenced from the appearance of only one N-CH_3 and one P-CH_3 signal in the ^1H-NMR spectra. This is in contrast to the Al analogs, $(CH_3)_2AlYP(CH_3)_2NCH_3$ (Y = O, S), whose ^1H-NMR spectra showed the presence of two structural isomers, 4a and 4b [4].

Two independent research groups reported the synthesis of tris(trimethylsilylmethyl)gallium, $[(CH_3)_3SiCH_2]_3Ga$, by the reaction of $GaCl_3$ with $(CH_3)_3SiCH_2MgCl$ in diethyl ether in good yields [5, 6]. The halide derivatives also were prepared from $[(CH_3)_3SiCH_2]_3Ga$ by elimination reactions with HCl or HBr



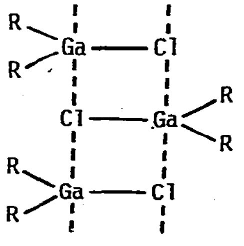
4a

X = F, Cl, CH_3
Y = O, S
M = Al, Ga, In

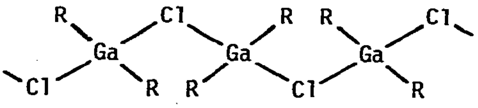


4b

and exchange reactions with $GaCl_3$ or $GaBr_3$, and fully characterized by elemental analyses, IR, and ^1H-NMR spectra [6]. Cryoscopic molecular weight determinations in benzene have indicated that $[(CH_3)_3SiCH_2]_3Ga$ is monomeric, whereas $[(CH_3)_3SiCH_2]_2GaBr$ and $(CH_3)_3SiCH_2GaX_2$ (X = Cl, Br) are dimers. The compound $[(CH_3)_3SiCH_2]_2GaCl$ had insufficient solubility in benzene for a molecular weight determination. In addition, this compound was obtained in surprisingly low yields only by the elimination reaction. On the basis of these unusual properties, $[(CH_3)_3SiCH_2]_2GaCl$ has been suggested to exist either as a 'ladder' polymer (5a) or a linear polymer (5b) in the solid state [6]. Diethyliodomethylgallium, $(C_2H_5)_2GaCH_2I$, reacted with $(CH_3)_3SiCH_2MgCl$ in diethyl ether to give $(CH_3)_3SiCH_2Ga(C_2H_5)_2$, the formation of which was confirmed in solution.



5a



5b

This compound was isolated as a more stable adduct with $(C_6H_5)_3P$ [5]. A report on the preparation of trialkylindium also appeared [7].

The reaction of MCl_3 (M = Ga, In) with sterically hindered $Li\{CH[Si(CH_3)_3]_2\}$ in diethyl ether at 0°C afforded white crystalline tris[bis(trimethylsilyl)-methyl]metals, $\{[(CH_3)_3Si]_2CH\}_3M$. Attempts to prepare $\{[(CH_3)_3Si]_2CH\}_xInCl_{3-x}$ (x = 1, 2) have been unsuccessful even at 2:1 mole ratio of $InCl_3$ and $Li\{CH[Si(CH_3)_3]_2\}$, where only the compound with x = 3 was obtained [8]. The first organosilylgallium compound $[(CH_3)_3Si]_3Ga$ was synthesized by the two reactions shown in Eqs. 2 and 3 [9]. The reaction 2 was accompanied by the formation

$$5Li + 4(CH_3)_3SiCl + Ga \xrightarrow[Hg/(C_2H_5)_2O]{14\ days,\ -4LiCl} [(CH_3)_3Si]_3Ga \qquad (2)$$

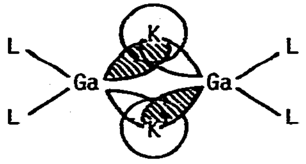
$$GaCl_3 + 6Li + 3(CH_3)_3SiCl \xrightarrow[THF]{-10°C,\ -6LiCl} [(CH_3)_3Si]_3Ga{\cdot}THF \qquad (3)$$

of $Li\{[(CH_3)_3Si]_4Ga\}{\cdot}3(C_2H_5)_2O$. Solvent-free $[(CH_3)_3Si]_3Ga$ exhibited IR absorption bands at 349 and 312 cm^{-1}, which have been assigned to $\nu_{asym}(GaSi_3)$ and $\nu_{sym}(GaSi_3)$, respectively.

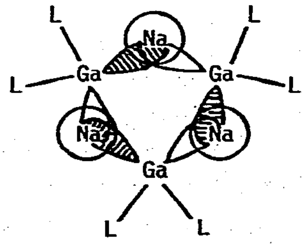
The chemistry of main group elements so far has focused on compounds with the metals in their highest oxidation states. The compounds C_5H_5In and C_5H_5Tl have been the only examples of low oxidation state Group III organometallic derivatives. At this time, a series of novel Ga(I) compounds, $MGa[CH_2Si(CH_3)_3]_2$ (M = Na, K) and the 1,2-dimethoxyethane solvate (M = Li, Na), were obtained in good yields by the apparent reductive elimination reaction shown in Eq. 4,

$$[(CH_3)_3SiCH_2]_3Ga + MH \xrightarrow[Solvent]{} MGa[CH_2Si(CH_3)_3]_2 + (CH_3)_4Si \qquad (4)$$

where the solvent is benzene for M = Na and K, and 1,2-dimethoxyethane for Li and Na. All the compounds other than the Li salt show high solubility in aliphatic and aromatic solvents and low melting points. The molecular weights obtained cryoscopically in benzene were consistent with the covalent formulas $\{KGa[CH_2Si(CH_3)_3]_2\}_2$ and $\{NaGa[CH_2Si(CH_3)_3]_2\}_3$, whereas the apparent molecular weight of $NaGa[CH_2Si(CH_3)_3]_2$ 1,2-dimethoxyethane solvate was dependent upon the concentration of the solution. In addition, all the $(CH_3)_3SiCH_2$ groups of $\{KGa[CH_2Si(CH_3)_3]_2\}_2$ and $\{NaGa[CH_2Si(CH_3)_3]_2\}_3$ occupy magnetically equivalent positions, as confirmed from the appearance of only two sharp signals in the both ^1H- and ^{13}C-NMR spectra for each compound. Based on these results, the structures involving Ga-Ga bonds shown in 6a and 6b have been proposed for the dimer and trimer [10]. Other attempts to prepare low oxidation state Group III organometallic derivatives by the reaction of InBr with $Li\{CH[Si(CH_3)_3]_2\}$



6a

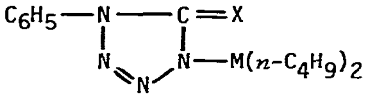


$L = CH_2Si(CH_3)_3$

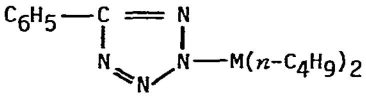
6b

or $Li\{N[Si(CH_3)_3]_2\}$ have been unsuccessful, liberating In metal almost quantitatively [8].

There have been two reports on the synthesis of new dibutylgallium and -indium (including -thallium) compounds; one is concerned with the pseudohalides $(n\text{-}C_4H_9)_2MX$ (M = Ga, In, Tl; X = N_3, NCO, NCS, NCSe) which were obtained by the metathetical reactions between $(n\text{-}C_4H_9)_2MCl$ and alkali metal pseudohalides [11]. The other dealt with the insertion of C_6H_5NCS, C_6H_5NCO, C_6H_5CN, and CS_2 into the metal-nitrogen bond of $(n\text{-}C_4H_9)_2MN_3$ (M = Ga, In, Tl), yielding 1-phenyl-4-(dibutylmetal)-tetrazole-5-thione (7a-7c), 1-phenyl-4-(dibutylmetal)-5-tetrazolinone (7d-7f), 1-phenyl-4-(dibutylmetal)-5-tetrazole (8a-8c), and $(n\text{-}C_4H_9)_2M$-NCS, respectively. These products were characterized by elemental analyses, electronic, IR, and ^1H-NMR spectra [12].



	M	X
7a	Ga	S
7b	In	S
7c	Tl	S
7d	Ga	O
7e	In	O
7f	Tl	O



	M
8a	Ga
8b	In
8c	Tl

Electrochemical oxidation of the metal at an electrode in the presence of appropriate alkyl halides and neutral ligands has been used recently with success in the synthesis of organotin, -cadmium, and -zinc compounds [J. J. Habeeb, A. Osman, and D. G. Tuck, *J. Organometal. Chem.*, 185 (1980) 117, and ref. cited therein]. This technique was applied to prepare a variety of organoindium compounds using cells of the type $Pt_{-}|RX + CH_3CN|In_{+}$. The initial voltage and current were 4-30 V and 7-35 mA, respectively. If 2,2'-bipyridine (bpy) was also present with RX in acetonitrile, the products were $RInX_2(bpy)$ (R = CH_3, C_2H_5, $C_6H_5CH_2$, C_6H_5, C_6F_5; X = Cl, Br, I, but not all combinations). When R'_4NX (R' = C_2H_5, $n\text{-}C_4H_9$) was present in the place of bpy, $[R'_4N][RInX_3]$ were obtained as products. Anomalous results were found in RX = CH_3I and C_2H_5I, where $R_2InI(bpy)$ or $[(n\text{-}C_4H_9)_4N][(CH_3)_2InI_2]$ also was deposited on the both electrodes [13].

Salts of tetra-coordinate Group III organometallic anions were prepared also by the following reactions (Eqs. 5-7). The reaction 5 was effectively

$$\mathrm{NaGaH_4} + \mathrm{RCH{=}CH_2} \xrightarrow[\mathrm{THF}]{50\text{–}60\,^\circ\mathrm{C}} \mathrm{Na[Ga(CH_2CH_2R)_4]} \tag{5}$$

$R = n\text{-}C_4H_9$, $n\text{-}C_5H_{11}$, $n\text{-}C_6H_{13}$ [14]

$$\mathrm{MGaH_4} + \mathrm{RC{\equiv}CH} \xrightarrow[\mathrm{(CH_2)_5NH}]{50\text{–}100\,^\circ\mathrm{C}} \mathrm{M[Ga(C{\equiv}CR)_4]} \tag{6}$$

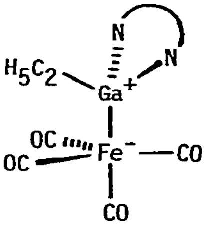
M = alkali metal; R = alkyl group [15]

$$\mathrm{R_2MX} + \mathrm{R'_4NX'} \longrightarrow \mathrm{[R'_4N][R_2MXX']} \tag{7}$$

M = Ga, In, Tl; $R = n\text{-}C_4H_9$, C_6H_5; $R' = CH_3$, C_2H_5, $n\text{-}C_4H_9$; X,X' = Cl, Br, I, N_3, NCSe [16]

catalyzed by the $(\eta^5\text{-}C_5H_5)_2TiCl_2\text{-}NaAl(C_2H_5)_4$ system [14]. Bis(η^5-cyclopentadienyl)acetylacetonatotitanium(IV) salts of Group III metal tetrahalides, $[(\eta^5\text{-}C_5H_5)_2Ti(acac)][MX_4]$ (M = Ga, In, Tl; X = Cl, Br, I), also were isolated and characterized by elemental analyses and IR spectra [17]. The ionic nature of these salts has been confirmed by conductance measurements in nitrobenzene.

The reaction of $C_2H_5GaI_2$ with $Na_2Fe(CO)_4$ in tetrahydrofuran (THF) gave a THF adduct of ethylgallium tetracarbonyliron, $(THF)C_2H_5GaFe(CO)_4$. The THF molecule was replaced by tetramethylethylenediamine (TMEDA), bpy, or pyridine (py) by reaction of the nitrogenous base in minimum amounts of THF to afford $(B)C_2H_5GaFe(CO)_4$ (B = TMEDA, bpy, 2py). Although the great air-sensitivity of these compounds has not allowed solution molecular weight determinations, the IR band position and pattern in the $\nu(C{\equiv}O)$ region have indicated the THF adduct to be associated. On the other hand, the nitrogenous base adducts have been suggested to exist as monomers (9) on the basis of a notable shift of $\nu(C{\equiv}O)$ to low frequency as compared with the THF adduct [18].



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There have been two reports on analysis this year; one is the mass spectrometric analysis of $(C_2H_5)_3In$ [19], and the other is quantitative gaschromatographic analysis of $(CH_3)_2MOCH_3$ (M = Ga, In), in which the procedure has been developed and the use of the atomic absorption detector has been recommended [20].

Structure and Bonding

Among the addition products between the heterocyclic compounds 3 and $(CH_3)_3M$ (M = Ga, In), two Ga compounds $(CH_3)_2\overline{\mathrm{Ga{-}}\underset{\mathrm{Ga(CH_3)_3}}{\mathrm{N}}\mathrm{(CH_3){-}N(CH_3){-}C(X)}}CH_3$ (X = O, NCH_3) were studied by the X-ray diffraction method [3]. The molecular structure of the compound with X = O is depicted in Fig. 1, which demonstrates that both Ga atoms are tetrahedrally coordinated. The distortion around the Ga(1) atom is great, ranging from ∠C(11)-Ga(1)-C(12) = 126.5(3)° to ∠O-Ga(1)-N(2) = 82.6(2)°. However, that around the Ga(2) atom is less serious. The compound with X = NCH_3 was isostructural to that with X = O.

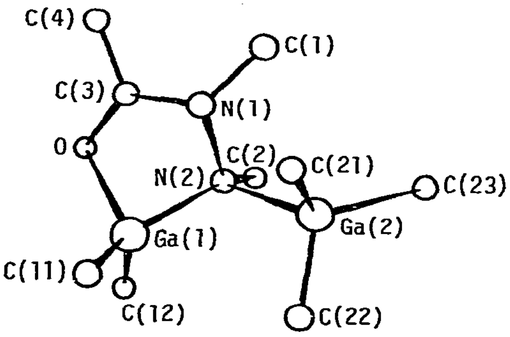
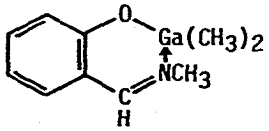


Fig. 1. Molecular structure of $(CH_3)_2\overline{\mathrm{Ga{-}}\underset{\mathrm{Ga(CH_3)_3}}{\mathrm{N}}\mathrm{(CH_3){-}N(CH_3){-}C(O)}}CH_3$

The electronic absorption and emission spectra in benzene, acetonitrile, or dimethyl sulfoxide and the dipole moments were measured for a series of dimethylgallium derivatives of aromatic and heterocyclic hydroxy- or mercapto-azomethines (10-13) as well as the diphenylboron derivatives. The results



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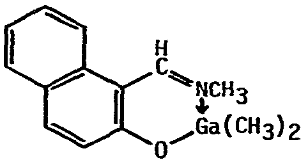

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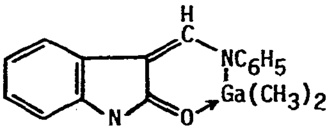

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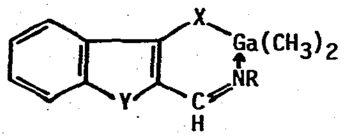
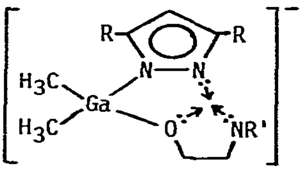


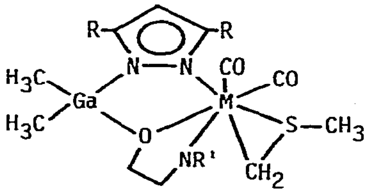
	X	Y	R
13a	O	CO	C_6H_5
13b	S	O	C_6H_5
13c	S	S	$CH_2C_6H_5$

have indicated that these Ga (and B) compounds have a mesomeric intra-complex structure with a tetra-coordinate Ga (or B) atom. Moreover, the structures of 10 and 13b were examined by X-ray analyses, which have revealed that both compounds are monomeric, and that the ligands on the Ga atoms have highly distorted tetrahedral configuration. The two Ga-C bonds in 10 were equal length, 1.98 Å, from which the normal covalent radius of Ga has been derived as 1.21 Å [21].

Two papers on the tridentate gallate anion 14 have been published [22, 23]. The equimolar reaction of the Na salt of 14 with $(CH_3CN)_3M(CO)_3$ (M = Mo, W) in THF gave the $LM(CO)_3^-$ anion (L = 14), which further reacted with $ClCH_2SCH_3$, affording bright yellow crystals of {dimethyl(N,N-dimethylaminoethoxy)(1-pyrazolyl)gallato[N(2),O,N(3)]}(η^2-thiomethoxymethyl)dicarbonylmetal (15) in 15–25% yields [22]. The ^1H-NMR (in benzene), IR (in dichloromethane or cyclohexane), and mass spectra indicated a monomeric pseudo-octahedral structure for 15 both in solution and in the gaseous state and the presence of two isomers in solution. X-Ray analyses for the two complexes with R = H and CH_3, R' = CH_3, and M = Mo, however, have shown that there exists only one isomer in the solid state; the Mo atom in the both complexes are best described as hepta-coordinate with the η^2-CH_2SCH_3 ligand and the Ga atom has a distorted tetrahedral geometry with the bond angles ranging from 92.24(7)° to 125.0(2)°. The tridentate gallate ligand 14 with R = CH_3 and R' = H or CH_3 reacted also with various transition metal salts at 2:1 mole ratio to give octahedral or trigonal bipyramidal complexes depending on the ligand, $[(CH_3)_2Ga(N_2C_5H_7)(OCH_2CH_2NH_2)]_2M$ (M = Fe, Co, $Ni{\cdot}C_6H_6$, $Ni{\cdot}(CH_3)_2CO$) or $\{(CH_3)_2Ga(N_2C_5H_7)[OCH_2CH_2N(CH_3)_2]\}M[(CH_3)_2Ga(N_2C_5H_7)_2]$ (M = Fe, Co, Ni, Cu, Zn) [23]. The electronic absorption spectra of these complexes in benzene or acetone ranging from 300 to 1400 nm were assigned. An X-ray structure analysis of the trigonal bipyramidal complex with M = Ni has revealed that the two N atoms of the bidentate $(CH_3)_2Ga(N_2C_5H_7)_2$ ligand occupy the equatorial positions and the tridentate ligand is meridionally coordinated with one oxygen in the equatorial and two nitrogens in the axial positions, as shown in Fig. 2.



14: R,R' = H or CH_3



15: M = Mo, W; R,R' = H or CH_3

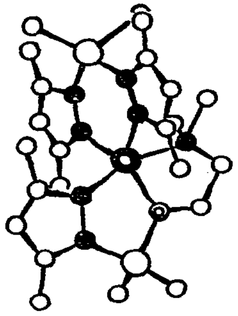
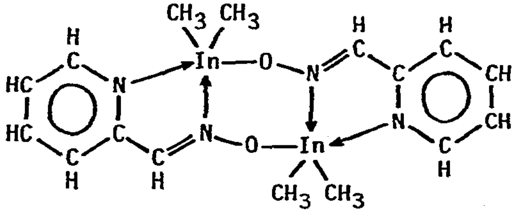


Fig. 2. Molecular structure of $\{(CH_3)_2Ga(N_2C_5H_7)[OCH_2CH_2N(CH_3)_2]\}$-$Ni[(CH_3)_2Ga(N_2C_5H_7)_2]$, ○: Ga, ◉: Ni, ○: C, ●: N, ◎: O.

The compound $In\{CH[Si(CH_3)_3]_2\}_3$ described in the preceding section was studied also by the X-ray diffraction method [8], which demonstrated that the In atom is three-coordinate, but that the geometry is not planar. The In atom lies 0.191 Å from the plane defined by the three C atoms; $\angle C\text{-}In\text{-}C = 119.3(2)°$. This compound is the first crystallographically authenticated monomeric, heavy Group III metal alkyl.

It had been found more than ten years ago that trimethyl derivatives of Group III metals react with pyridine-2-carbaldehyde oxime, $2\text{-}C_5H_4NCH{=}NOH$, to give dimeric $[(CH_3)_2M(ON{=}CHC_5H_4N)]_2$ ($M = Al$, Ga, In, Tl) with liberating CH_4 [I. Pattison and K Wade, *J. Chem. Soc., A*, (1968) 2618]. An X-ray structure analysis of the In compound was carried out [24]. The result has shown that each oximate group is coordinated through both N atoms to one In atom, and through the O atom to the other In atom of the dimer. Thus, the dimer consists of an assembly of five fused rings as depicted in 16. The In atoms adopt a distorted trigonal bipyramidal geometry, in which the N-In-N angle is only 69°.



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Although the distorted trigonal bipyramidal configuration has been found in a number of organoindium compounds, the penta-coordination of those compounds results from *inter*molecular association, such as in 16. As the first organoindium compound which has such a configuration as a result of *intra*molecular coordination, bis{2-[(dimethylamino)methyl]phenyl}chloroindium, $[2-(CH_3)_2NCH_2-C_6H_4]_2InCl$, was isolated by the reaction between $InCl_3$ and $Li[2-(CH_3)_2NCH_2C_6H_4]$ in diethyl ether in 1:2 mole ratio [25]. An X-ray analysis of this compound

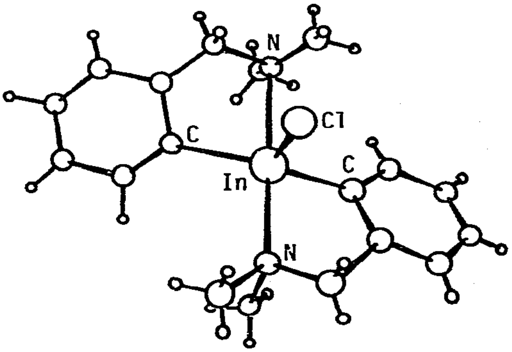

Fig. 3. Stereochemistry of $[(CH_3)_2NCH_2C_6H_4]_2InCl$

has shown that the crystal consists of discrete monomeric units and the molecule has a distorted trigonal bipyramidal geometry with an InC_2Cl equatorial plane and apical In–N bonds as illustrated in Fig. 3.

The crystal structure of methyl(tetraphenylporphinato)indium(III), $CH_3In-(TPP)$, also was reported [26]. The coordination polyhedron of the In atom is a square pyramid; In–N = 2.06(1) and In–C = 2.13(1) Å. The In atom lies 0.78(2) and 0.92(2) Å, respectively, from the plane defined by the four N atoms and from the porphinato plane. These are the largest values observed for such a coordination polyhedron in metalloporphyrin chemistry.

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