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Preparation and characterisation of novel organoindium derivatives; X-ray crystal structure of dimethyl(salicylaldehyde)indium

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Abstract

The reaction of trimethylindium with one equivalent of salicylaldehyde, 2-carboxybenzaldehyde, 2-pyrrolidinone or *N,N'*-diphenylacetamidine results in the formation of the corresponding dimethylindium derivatives in good yields. The product obtained from the reaction with salicylaldehyde has been shown by a single crystal X-ray diffraction study to consist of centrosymmetric dimers with five-coordinate indium atoms, In–C 2.122(5), In–O(CHO) 2.341(4), In–O(OH) 2.188(3) and 2.383(3) Å. The reactions between triethylindium and phenylacetylene and ethanolamine are described.

Introduction

Trialkylindium compounds are known to react, via alkane elimination, with a range of compounds containing an acidic hydrogen, such as secondary amines and phosphines, alcohols, thiols, and carboxylic acids [1–8]. Although some reactions of this type are known in which two or three equivalents of alkane are eliminated, the majority that have been reported involve the elimination of only one equivalent. The organoindium derivatives thus prepared have generally been found to be associated into dimers or higher oligomers. This has been well demonstrated in the case of sterically hindered secondary amines and phosphines, which lead to the formation of dimeric products [3,9]. For acids containing more than one possible co-ordination site relatively little is known about the structures of the products, either in the solid or in solution. We report here some reactions of trimethyl- and triethyl-indium with several such acids and discuss the possible structures of the products.

Results and discussion

The addition of one equivalent of salicylaldehyde, 2-carboxybenzaldehyde, 2-pyrrolidinone, or *N,N'*-diphenyl-acetamidine to a toluene or hexane solution of trimethylindium etherate at room temperature resulted in the evolution of CH_4 and

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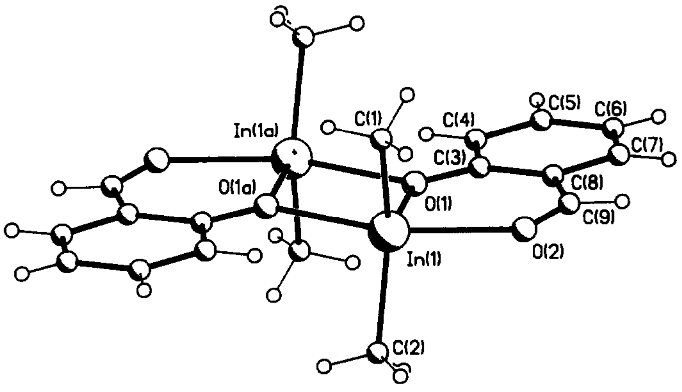

Fig. 1. View of the centrosymmetric dimer of $[Me_2In(OC_6H_4CHO)]$, **1**, showing the principal atomic numbering.

the formation of the corresponding dimethylindium derivatives in good yields.

$$In(CH_3)_3 + HL \rightarrow (CH_3)_2InL + CH_4$$

HL = salicylaldehyde (**1**); 2-carboxybenzaldehyde (**2**); 2-pyrrolidinone (**3**); *N,N'*-diphenylacetamidine (**4**)

Compound **1** was isolated as a yellow crystalline solid whereas **2**, **3** and **4** were isolated as colourless solids. All four compounds were found to be slightly air sensitive, and each compound was characterised by elemental analysis and NMR spectroscopy (see Experimental section). In each case the methyl protons were found to give rise to a singlet in the ^{1}H-NMR spectrum, the integral ratios of which confirmed the identities of the products as dimethyl-indium derivatives. The EI mass spectra of all of the products showed not only ions corresponding to the monomeric molecular ions but also ions deriving from dimeric species (see Experimental section).

A single crystal X-ray diffraction study of **1** confirmed the dimeric nature of this compound. The overall view of the molecule is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. The indium atoms in **1** are five coordinate and the dimerisation occurs through the oxygen atoms of the phenoxide groups giving rise to an essentially planar In_2O_2 ring. The dimeric unit consists of five fused rings that are all approximately in the same plane. The hydroxy-oxygen bridges both indium atoms, but the In–O bond lengths in the In_2O_2 ring are not identical with [In(1)–O(1)] being 2.188(3) Å while [In(1)–O(1a)] is 2.383(3) Å. The other bond from In(1) to the aldehyde oxygen [O(2)] has a length of 2.341(4) Å.

The structure of **1** shows similarities to the structure of the dimethyl gallium analogue [10]. Thus the internal angles in the M_2O_2 rings for the two compounds show the M–O–M angles as 104.5(1)°(Ga) and 105.2(1)°(In) and the O–M–O angle as 75.5(1)°(Ga) and 74.8(1)°(In). However, the major difference is in the bridging metal–oxygen bond distances. In the gallium analogue this bond has a length of 2.462(4) Å whereas in **1** the corresponding bond length is 2.383(3) Å [In(1)–O(1a)], despite the fact that gallium has a smaller covalent radius. Comparison of the Ga–C and In–C bond lengths in the two structures indicates that the gallium atom is smaller by approximately 0.18 Å. Taking this difference into account the gallium–oxygen bond is effectively 0.26 Å longer than in the indium

Table 1

Bond distances (Å) and angles (°) for $[Me_2In(OC_6H_4CHO)]_2$, **1**

(a) Bond distances			
In(1)–O(1)	2.188(3)	In(1)–O(2)	2.341(4)
In(1)–C(1)	2.121(5)	In(1)–C(2)	2.123(6)
In(1)–O(1A)	2.383(3)	O(1)–C(3)	1.325(5)
O(1)–In(1A)	2.383(3)	O(2)–C(9)	1.214(5)
C(3)–C(4)	1.394(5)	C(3)–C(8)	1.404(6)
C(4)–C(5)	1.381(6)	C(5)–C(6)	1.368(7)
C(6)–C(7)	1.374(6)	C(7)–C(8)	1.404(5)
C(8)–C(9)	1.425(6)		
(b) Bond angles			
O(1)–In(1)–O(2)	79.9(1)	O(1)–In(1)–C(1)	109.0(2)
O(2)–In(1)–C(1)	91.7(2)	O(1)–In(1)–C(2)	109.1(2)
O(2)–In(1)–C(2)	96.1(2)	C(1)–In(1)–C(2)	141.9(2)
O(1)–In(1)–O(1A)	74.8(1)	O(2)–In(1)–O(1A)	154.7(1)
C(1)–In(1)–O(1A)	94.5(2)	C(2)–In(1)–O(1A)	94.1(2)
In(1)–O(1)–C(3)	133.7(3)	In(1)–O(1)–In(1A)	105.2(1)
C(3)–O(1)–In(1A)	121.1(2)	In(1)–O(2)–C(9)	128.2(3)
O(1)–C(3)–C(4)	119.3(4)	O(1)–C(3)–C(8)	123.2(3)
C(4)–C(3)–C(8)	117.6(3)	C(3)–C(4)–C(5)	121.5(4)
C(4)–C(5)–C(6)	121.3(4)	C(5)–C(6)–C(7)	118.5(4)
C(6)–C(7)–C(8)	121.7(4)	C(3)–C(8)–C(7)	119.5(3)
C(3)–C(8)–C(9)	126.0(3)	C(7)–C(8)–C(9)	114.6(4)
O(2)–C(9)–C(8)	128.5(4)		

compound. The main reason for this is probably steric crowding around the smaller gallium atom, and the weakness of the association in the gallium compound has been noted [10]. Since such crowding is lessened in the indium compound, it is not unreasonable to suggest that the dimeric association is stronger in this case. The EI mass spectra of the compounds are compatible with this view, in that the gallium compound shows only ions attributable to the monomer [10], whereas we have found that the indium analogue exhibits ions derived from both the monomer and the dimer (see Experimental section).

The structure of **2** has not been determined unambiguously. 2-Carboxybenzaldehyde is known to exist in both cyclised form and ring opened form (Fig. 2A/B) [11]. The former exists in the solid state, while the latter is generally favoured in solution. The two forms are readily distinguishable in solution, by ^{1}H-NMR spectroscopy from the different chemical shifts of the aldehyde proton, the cyclised and ring opened forms showing chemical shifts of 6.75 ppm and 10.15 ppm, respectively. In the NMR spectrum of **2** (DMSO-d_6) the chemical shift for this proton is 10.39 ppm indicating that the ligand is in the ring-opened form in this compound in DMSO. The lack of solubility of **2** in other common solvents prevented further examination by NMR spectroscopy. The IR spectrum (Nujol mull) of the solid shows the carbonyl stretching band at 1667 cm^{-1}, which is close to that expected for a free aldehyde attached to an aromatic ring. It appears, therefore, that in **2** the aldehyde group is not strongly coordinated to the metal, and that coordination occurs through the carboxylate group. Consistent with this view, the $\nu_{as}(CO_2)$ and $\nu_s(CO_2)$ bands of the carboxylate group in **2** occur at 1541 and 1458 cm^{-1}, respectively,

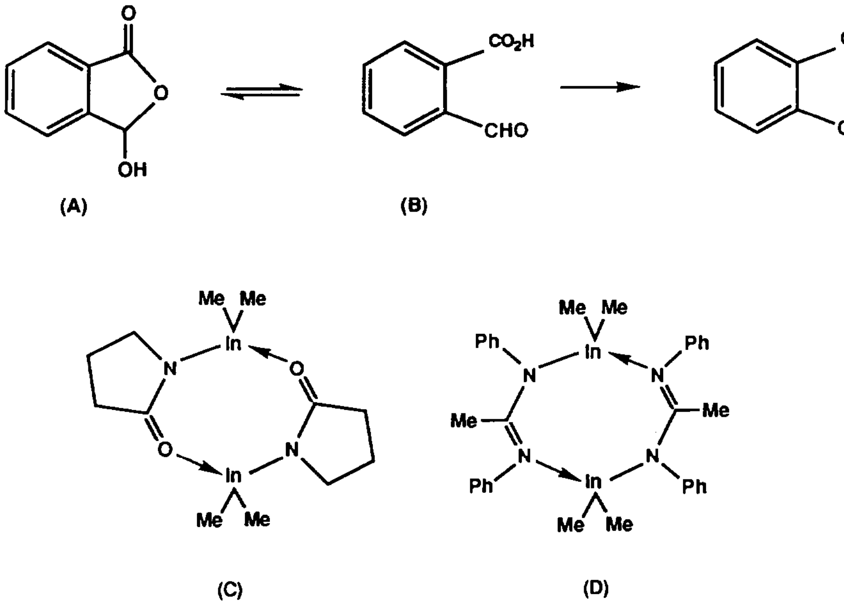

Fig. 2. Postulated models for $Me_2In(O_2CC_6H_4CHO)$ and $[Me_2In(N_2Ph_2CMe)]_2$.

while the ν_{sym} def(CO_2) band is observed at 701 cm^{-1}. These values are close to those observed for dimethylindium acetate at 1535, 1455 and 681 cm^{-1}, where X-ray crystallographic studies have shown that the acetate group acts as a bidentate ligand towards the metal [12]. It is interesting to compare this with the values in free carboxylate ions, where $\nu_{as}(CO_2)$ and $\nu_s(CO_2)$ are found at 1578 and 1414 cm^{-1}, respectively ($\Delta = \nu_{as} - \nu_s = 164$ cm^{-1}); the values for **2** give a Δ value of 83 cm^{-1}. Such relatively small values of Δ have been used by some authors to infer the presence of bidentate carboxylate groups, although it has been pointed that such inferences should be treated with caution [13].

Finally it is relevant that in dimethylindium acetate the indium centre is six-coordinate, the two further interactions arising from intermolecular contacts between oxygen atoms of carboxylate groups of neighbouring molecules [12]. Earlier studies have indicated that there also appears to be a correlation between the C–In–C bond angle in a number of Me_2InX compounds and $\Delta\nu_1$ [where $\Delta\nu_1 = \nu_{as}(InC_2) - \nu_s(InC_2)$] [14]. For Me_2InO_2CMe $\Delta\nu_1 = 49$ cm^{-1} with a C–In–C bond angle of 152°, and the salicylaldehyde derivative **1** shows $\Delta\nu_1 = 47$ cm^{-1} and a C–In–C angle of 142°, compared with the predicted angle (from $\Delta\nu_1$) of 144°. The value of $\Delta\nu_1$ for **2** is 45 cm^{-1} which gives a predicted angle of 142°. The similarity in the values of $\Delta\nu_1$ for the three compounds may be indicative that some intermolecular association in **2** occurs (as in the other two compounds) giving the indium atom a coordination number greater than four. However, it is probable that any such interaction in the solid state is rather weak, since the mass spectrum of **2** shows ions arising mainly from the monomeric species, with weak intensity peaks from the dimer accounting for only 0.4% of the total ion current (see Experimental section).

The EI mass spectrum of **3** shows ions corresponding to methyl loss from a dimeric species, although the dimeric molecular ion itself was not observed. The IR spectrum of **3** shows a band attributable to a carbonyl stretch at 1581 cm^{-1}. The corresponding band in the IR spectrum of 2-pyrrolidinone is found at 1685 cm^{-1}. The large difference between these two bands indicates that the association observed in the EI mass spectrum is most likely to involve the oxygen atom of the carbonyl group. Thus, a dimeric structure consisting of two $InMe_2$ units linked together by two pyrrolidinone residues bonded through both nitrogen and oxygen would be consistent with the IR and EIMS data. This structure is indicated in Fig. 2C.

The IR spectrum of **4** exhibits bands at 1475 and 1528 cm^{-1} which can be assigned as $\nu_s(CN_2)$ and $\nu_{as}(CN_2)$ respectively. These bands can be compared to those observed in the IR spectrum of dimethyl(N,N'-dimethylacetamidine)indium which have previously been reported at 1468 and 1540 cm^{-1} respectively [15]. A band at 392 cm^{-1} can be assigned to a symmetrical stretching mode of the InN_2 unit of **4**, which again compares well to the corresponding band in the IR spectrum of the dimethyl-acetamidine derivative observed at 384 cm^{-1}. This latter compound, and its gallium analogue, has previously been shown by vibrational spectroscopy to be dimeric in the solid state, and the presence of a puckered eight membered ring in the gallium compound has been confirmed by a single crystal X-ray study [16]. The similarity between the IR spectrum of **4** and dimethyl(N,N'-dimethylacetamidine)indium in conjunction with the EIMS evidence indicates that **4** also has a similar dimeric structure (see Fig. 2D).

Two reactions of similar type to those just discussed were carried out between triethylindium and phenylacetylene and ethanolamine.

$$InEt_3 + HL \rightarrow Et_2InL + C_2H_6$$

HL = phenylacetylene (**5**); ethanolamine (**6**)

The product **5** of the reaction between triethylindium and phenylacetylene is very soluble even in non-polar solvents such as hexane, and could not be isolated in a pure form. However, the addition of one equivalent of triphenylphosphine to a solution of **5** gave the 1 : 1 triphenylphosphine addict $Et_2InC{\equiv}CPh.PPh_3$, **7**, in high yield as a crystalline colourless solid which proved to be slightly air-sensitive.

The dimethylindium analogue of **5** has previously been reported [17]. The IR spectrum of this analogue in benzene solution shows a band at 2050 cm^{-1} attributable to the C≡C stretching mode. In THF solution this band was found to shift to 2100 cm^{-1}. This was interpreted as indicating that in benzene solution the phenylacetylene groups are bridging and that in THF these bridges are broken by the presence of the donor solvent. The C≡C stretch in **7** (Nujol mull) occurs at 2080 cm^{-1} which falls about midway between the values found for the dimethyl-indium analogue. It is unlikely that the phenylacetylene groups are bridging in this adduct, since such bridges would be expected to be extremely weak and would probably not survive the coordination of the bulky and relatively strong donor ligand triphenylphosphine.

The product of the reaction between triethylindium and ethanolamine (**6**) was isolated as a colourless crystalline solid that proved to be slightly air sensitive. The EI mass spectrum of **6** showed the presence of ions arising from both the monomeric species and a dimeric species as well as ions of higher mass which could not be assigned. The mass spectrum of the dimethyl gallium analogue of this compound has been reported and also showed the presence of ions attributable to monomeric,

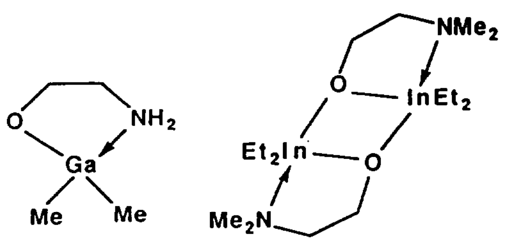

Fig. 3. Molecular arrangements in monomeric $Me_2Ga(OCH_2CH_2NH_2)$ [18] and dimeric $[Et_2In(OCH_2CH_2NMe_2)]_2$ [20].

dimeric and species of higher mass [18]. The structure of this gallium analogue was found by an X-ray crystallographic study to consist of monomeric units in which the ethanolamine unit is bonded in a chelating manner to the gallium atom giving rise to a five-membered ring [18]. These monomeric units are further associated through hydrogen bonds to give an overall polymeric structure. Unfortunately no IR data for this compound has been reported and so further spectroscopic comparisons are not possible. The dimethylgallium and diethylindium derivatives of dimethylethanolamine have also been prepared previously and shown to be dimeric [19,20]. However, again no IR data was reported for either compound and no comparisons with the spectrum of **6** are possible. The two known structures are indicated in Fig. 3.

The similarity in the mass spectrum of **6** with that of the dimethylgallium ethanolamine analogue indicates that the compounds may have similar structures. The presence of ions at higher masses than that of the dimeric species is also consistent with such a polymeric structure arising from intermolecular hydrogen bonds.

Experimental

General

All reactions were carried in an argon filled glove box and the products were subsequently isolated under dry nitrogen by standard Schlenk techniques. Trimethylindium was prepared from methyllithium and indium trichloride according to the method of Clark and Pickard [2]. The trimethylindium was obtained as an ether solution after distillation (b.p. 110 °C at 25 mmHg). Its concentration was determined by titration with EDTA and zinc acetate solutions on a hydrolysed sample, as described by Coates and Graham [21]. The solution was found to contain 53.8% (w/w) of trimethylindium. Triethylindium was prepared as described by Todt and Dotzer [22].

Salicylaldehyde, 2-carboxybenzaldehyde, 2-pyrrolidinone and *N*,*N*′-diphenylacetamidine were purchased from Aldrich and used as received. Toluene and hexane were dried by distillation from sodium benzophenone ketyl. Dichloromethane was dried over calcium hydride and distilled prior to use. NMR solvents were dried over molecular sieves.

^{1}H-NMR spectra were recorded with a Perkin Elmer R34 (220 MHz) instrument. Positive chemical shifts are given downfield from tetramethylsilane. IR spectra were recorded with a Perkin Elmer 580B spectrophotometer as Nujol mulls between CsI plates. Melting points were determined using open-end capillaries and are uncorrected. Mass spectra (EI) were obtained on a Kratos MS80 mass spectrometer at 70 eV.

(a) Dimethyl(salicylaldehyde)indium (1)

Salicylaldehyde (0.61 g, 5.00 mmol) was added slowly to a stirred solution of trimethylindium (0.80 g, 5.00 mmol) in hexane (20 cm^3) at room temperature. A yellow powder immediately precipitated, separated and this was filtered off and recrystallised from a mixture of dichloromethane and hexane to give a yellow crystalline product. Yield, 0.84 g (64%); m.p. 188–192 °C. Analysis. Found: C, 40.46; H, 4.03; In, 44.4. $C_9H_{11}O_2In$ calcd.: C, 40.64; H, 4.17; In, 43.2%; ^1H NMR (220 MHz, $CDCl_3$): δ −0.02 (6H, s, CH_3); 6.91 (2H, m, C_6H_4); 7.48 (2H, m, C_6H_4); 9.44 (1H, s, CHO). EI-MS, 70 eV, using ^{115}In, $(D-Me)^+$ 517, $(D-C_6H_3CHO)^+$ 428, $(D-C_6H_4CHO_2)^+$ 411, $(M)^+$ 266, $(M-Me)^+$ 251, $(M-Me_2)^+$ 236, $(M-C_6H_4CHO_2)^+$ 145. Crystals of this compound suitable for X-ray diffraction were grown from a chloroform solution of the compound layered with hexane (1 : 4).

(b) Dimethyl(2-carboxybenzaldehyde)indium (2)

2-Carboxybenzaldehyde (0.58 g, 3.86 mmol) was added slowly to a stirred solution of trimethylindium (0.62 g, 3.87 mmol) in hexane (20 cm^3) at room temperature. The mixture was heated at 50 °C for 30 min to ensure completion of the reaction and allowed to cool. Removal of the solvent under reduced pressure left a colourless solid which was recrystallised from a tetrahydrofuran/hexane mixture to give a white microcrystalline product. This was filtered off and dried *in vacuo*. Yield, 0.55 g (48%); m.p. 212–216 °C. Analysis Found: C, 40.67; H, 3.79; In, 38.3. $C_{10}H_{11}O_3In$ calcd.: C, 40.85; H, 3.77; In, 39.1%. ^1H NMR (220 MHz, $[(CD_3)_2SO]$): δ −0.23 (6H, s, CH_3); 7.43, 7.74, 7.97 (4H, m, C_6H_4); 10.39 (1H, s, CHO). EI-MS $(D-InMe_2)^+$ 443, $(M)^+$ 294, $(M-Me)^+$ 279, $(M-C_6H_4CHOCO_2)^+$ 145.

(c) Dimethyl(2-pyrrolidinone)indium (3)

2-Pyrrolidinone (0.44 g, 5.17 mmol) was added to a stirred solution of trimethylindium (0.83 g, 5.18 mmol) in toluene (20 cm^3) at room temperature. On addition, bubbles of gas were immediately evolved. The mixture was heated at 50 °C for 30 min, then concentrated *in vacuo* to a volume of ca 2 cm^3 and mixed with hexane (10 cm^3) and the solution kept at −20 °C for 3 h. The colourless microcrystalline powder formed was filtered off and dried *in vacuo*. Yield, 0.51 g (43%); m.p. 130–132 °C. Analysis. Found: C, 30.26; H, 5.21; N, 4.78; In, 49.8. $C_6H_{12}NOIn$ calcd.: C, 31.45; H, 5.24; N, 6.12; In, 50.2%. ^1H NMR (220 MHz, $[(CD_3)_2SO]$): δ −0.62 (6H, s, CH_3); 1.98 (4H, m, CH_2); 3.30 (2H, t, CH_2). EI-MS $(D-Me)^+$ 443, $(D-Me_2)^+$ 427, $(M-Me)^+$ 214, $(M-Me_2)^+$ 199, $(M-C_3H_6CONH)^+$ 145.

*(d) Dimethyl(*N,N′*-diphenylacetamidine)indium (4)*

N,N′-Diphenylacetamidine (0.82 g, 4.18 mmol) was added slowly to a stirred solution of trimethylindium (0.67 g, 4.19 mmol) in toluene (20 cm^3) at room temperature. A pale yellow solution was immediately formed, and this was concentrated under reduced pressure to a volume of ca 2 cm^3 and mixed with hexane (10 cm^3). The solution was kept at −20 °C for 3 h and the colourless crystalline product that separated was recrystallised from hexane and dried *in vacuo*. Yield, 0.82 g (58%); m.p. 82–85 °C. Analysis. Found: C, 54.03; H, 5.35; N, 7.90; In, 33.1. $C_{16}H_{19}N_2In$ calcd.: C, 54.26; H, 5.41; N, 7.91; In, 32.50%. ^1H NMR (220 MHz, $C_6D_5CD_3$): δ 0.03 (6H, s, CH_3); 1.58 (3H, s, CH_3); 6.90 and 7.13 (10H, m, C_6H_5). EI-MS $(D - Me + H)^+$ 694, $(D - Me - InMe_2)^+$ 548, $(D - Me_2InMe_2)^+$ 533, (M^+) 354, $(M - Me)^+$ 339, $(M - Me_2)^+$ 324, $(M - Ph_2N_2HCMe)^+$ 145.

(e) Diethyl(ethanolamino)indium (6)

Ethanolamine (0.34 g, 5.57 mmol) was added slowly to a stirred solution of triethylindium (1.13 g, 5.56 mmol) in tetrahydrofuran (40 cm^3) at room temperature. After 10 min the solvent was removed under reduced pressure to leave a colourless microcrystalline solid which was recrystallised from a tetrahydrofuran/hexane mixture and dried *in vacuo*. Yield, 0.96 g (74%). Analysis. Found: C, 31.02; H, 6.97; N, 6.01; In, 46.1. $C_6H_{16}NOIn$ calcd.: C, 30.93; H, 6.87; N, 6.01, In, 49.3%. ^1H NMR (220 MHz, $C_6D_5CD_3$): δ 0.31 (2H, t, NH_2); 0.65 (4H, q, CH_2); 1.50 (6H, t, CH_3); 2.22 (2H, m, CH_2); 3.54 (2H, t, CH_2). EI-MS $(D + H)^+$ 467, $(D - Et)^+$ 437, $(D - Et_2 + H_2)^+$ 406, $(M + H)^+$ 234, $(M - Et)^+$ 204, $(M - C_2H_4ONH_2)^+$ 173; also ions at m/z 565, 551 which could not be assigned.

(f) Diethyl(phenylacetylene)indium triphenylphosphine (7)

A solution of phenylacetylene (2.50 g, 24.51 mmol) in hexane (10 cm^3) was added slowly to a solution of triethylindium (5.17 g, 25.6 mmol) in hexane (20 cm^3) and the mixture was heated at 50 °C for 8 h. The colourless crystalline precipitate was filtered off, recrystallised from hexane, and dried *in vacuo*. Yield, 11.63 g (89%). Analysis. Found: C, 67.19; H, 5.60; In, 21.9. $C_{30}H_{30}OPIn$ calcd.: C, 67.20; H, 5.60; In 21.4%. ^1H NMR (220 MHz, $C_6D_5CD_3$): δ 1.08 (4H, q, CH_2); 1.64 (6H, t, CH_3), 7.0 and 7.3 (20H, m, C_6H_5).

X-ray crystallography

A single crystal (0.4 × 0.3 × 0.4 mm) of **1** was sealed in a thin-walled glass capillary under argon. The crystal data are as follows: $C_{18}H_{22}O_4In_2$, MW = 531.8, moniclinic space group $P2_1/n$, a 9.322(6), b 7.006(4), c 15.266(9) Å, β 100.03(4)°, V 982(1) Å^3, $Z = 2$, $D_c = 1.80$ g cm^{-3}, μ(Mo-K_α) 23.3 cm^{-1}, $F(000) = 520$.

The X-ray intensity data were collected on a Nicolet $R3M$ four circle diffractometer. A total of 1734 intensities within the range $3 < 2\theta < 50°$ were measured with $\omega - 2\theta$ scan mode. The intensities were corrected for Lorentz polarisation and for absorption by the Gaussian method (transmission factors 0.52–0.76) to give 1485 unique reflections with $I/\sigma(I) > 2.0$. The In atom of a centrosymmetric dimer was located by use of the Patterson interpretation section of SHELXTL [23] and the light atoms were located by successive Fourier syntheses. Anisotropic temperature factors were used for all non-H atoms, and H-atoms were placed at calculated positions and not refined. Final refinement was on F by the least-squares methods involving refinement of 100 parameters including an isotropic extinction parameter. Largest positive and negative peaks on a final difference Fourier synthesis were of height ± 0.3 e A^{-3}. A weighting scheme of the form $w = 1/(\sigma^2(F) + gF^2)$ with $g = 0.00054$ was used and shown to be satisfactory by a weight analysis. Final $R = 0.027$. $R_w = 0.038$. Maximum shift/error in final cycle 0.01. Computing was with SHELXTL PLUS [23] on a DEC Microvax-II. Scattering factors were used in the analytical form

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) of $[Me_2In(OC_6H_4CHO)]_2$, **1**

	x	y	z	U_{eq} [a]
In(1)	10077.6(3)	1224.8(4)	11056.2(2)	48.3(2)
O(1)	8580(3)	686(5)	9809(2)	52(1)
O(2)	7995(4)	2783(5)	11356(2)	70(1)
C(1)	9851(6)	−985(7)	11972(3)	74(2)
C(2)	11290(6)	3739(7)	10919(4)	76(2)
C(3)	7207(4)	1159(5)	9507(3)	44(1)
C(4)	6572(5)	635(7)	8646(3)	56(1)
C(5)	5135(5)	1055(7)	8307(3)	62(2)
C(6)	4285(5)	2038(7)	8798(3)	62(2)
C(7)	4879(4)	2557(6)	9652(3)	57(2)
C(8)	6338(4)	2160(5)	10016(2)	44(1)
C(9)	6788(5)	2829(7)	10903(3)	57(2)

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

and anomalous dispersion factors were taken from *International Tables of X-ray Crystallography* (1974). Final atomic coordinates are given in Table 2.

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