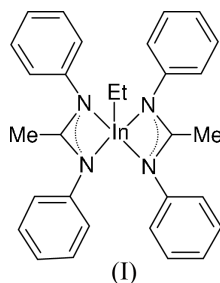


Damian R. Aris,<sup>a</sup> James Barker,<sup>b</sup>  
William Errington<sup>a\*</sup> and  
Malcolm G. H. Wallbridge<sup>a</sup><sup>a</sup>Department of Chemistry, University of  
Warwick, Coventry CV4 7AL, England, and  
<sup>b</sup>Associated Octel Co Ltd, Ellesmere Port,  
South Wirral L65 3HF, EnglandCorrespondence e-mail:  
w.errington@btopenworld.com

## Key indicators

Single-crystal X-ray study  
T = 240 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$   
R factor = 0.048  
wR factor = 0.124  
Data-to-parameter ratio = 13.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Bis(*N,N'*-diphenylacetamidinato)ethylindium(III)The title compound,  $[\text{In}(\text{C}_2\text{H}_5)(\text{C}_{14}\text{H}_{13}\text{N}_2)_2]$ , contains an  $\text{In}^{\text{III}}$  atom in a square-pyramidal geometry. The phenyl rings are almost coplanar in one of the amidinate ligands but significantly twisted with respect to each other in the other ligand.Received 20 January 2004  
Accepted 5 February 2004  
Online 14 February 2004

## Comment

The title compound, (I), was isolated as part of a wider study (Barker *et al.*, 1996) of the potential use of amidine compounds of group 13 elements as potential precursors in synthesis. The paucity of structural data, particularly for the amidinates of the heavier group 13 elements, is illustrated by the fact that the structures of only three indium complexes have been previously reported; these are the structures of chlorobis(*N,N'*-dicyclohexylneopentamidinato)indium(III) (Zhou & Richeson, 1996), bis( $\mu$ -2,2,6-diisopropylanilido)-bis(dimethylamido)(*N,N'*-diisopropyl-2,2-dimethylpropioamidinato)indiumtitanium and bis(2,6-diisopropylanilido)-(*N,N'*-diisopropyl-2,2-dimethylpropioamidinato)indium (Patton *et al.*, 2002).The title compound features a five-coordinate  $\text{In}^{\text{III}}$  atom in a slightly distorted square-pyramidal geometry. Of the previously reported structures, only the chloro derivative displays five-coordination, but in a stereochemistry better approximated by the trigonal-bipyramidal geometry. The indium–ligand bond lengths and angles are listed in Table 1. The  $\text{In}-\text{N}$  distances range from 2.292 (4) to 2.234 (4)  $\text{\AA}$  and the smallest bond angle is only  $58.22(15)^\circ$ , resulting from the small ‘bite’ associated with the bidentate amidinate ligand. The deviation of the stereochemistry from the square pyramidal ideal may be quantified by the method of Addison *et al.* (1984). In the present complex, the value of  $\tau$  is 0.01; this compares with  $\tau = 1$  for the trigonal-bipyramidal structure and  $\tau = 0$  for an ideal square-pyramidal structure.The orientation of the four aromatic rings is also of interest. In one of the amidinate ligands, the two phenyl groups are almost coplanar [actually inclined at  $8.1(2)^\circ$ ], whilst in the other case, there is significant twisting [ $32.9(2)^\circ$ ]. The asym-

metry in the latter case may be attributed to an interaction with the ethyl group (directly attached to In), which twists in the direction of this ligand.

## Experimental

*N,N'*-Diphenylacetamide, *A*, was purified by sublimation at 373 K under vacuum. Solid *A* (1.4 g, 6.6 mmol) was added slowly to a stirred suspension of  $\text{InEt}_3$  (0.67 g, 3.32 mmol) in hexane under an atmosphere of Ar at 195 K. On warming to room temperature, the solution became clear, and the mixture was then warmed at 353 K for 4 h to complete the reaction. On cooling the clear solution to room temperature, a crystalline solid was deposited as colourless blocks. Analytical data, calculated for  $\text{C}_{30}\text{H}_{31}\text{InN}_4$ : C 64.0, H 5.6, N 9.9%; found: C 65.0, H 5.7, N 9.7%.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  7.13 (t, 8H), 6.95 (tt, 4H), 6.76 (m, 8H), 2.06 (s, 6H), 1.41 (t, 3H), 1.13 (q, 2H).

### Crystal data

$[\text{In}(\text{C}_2\text{H}_5)(\text{C}_{14}\text{H}_{13}\text{N}_2)_2]$	$Z = 2$
$M_r = 562.41$	$D_x = 1.392 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.056$ (4) Å	Cell parameters from 18 reflections
$b = 12.014$ (7) Å	$\theta = 10\text{--}11^\circ$
$c = 12.842$ (8) Å	$\mu = 0.91 \text{ mm}^{-1}$
$\alpha = 107.11$ (5) $^\circ$	$T = 240$ (2) K
$\beta = 94.83$ (5) $^\circ$	Block, colourless
$\gamma = 111.92$ (4) $^\circ$	$0.37 \times 0.35 \times 0.27 \text{ mm}$
$V = 1342.3$ (15) Å <sup>3</sup>	

### Data collection

Siemens P3R3 diffractometer	$R_{\text{int}} = 0.076$
$\omega$ - $2\theta$ scans	$\theta_{\text{max}} = 24.1^\circ$
Absorption correction: Gaussian (Alcock, 1970)	$h = 0 \rightarrow 11$
$T_{\text{min}} = 0.731$ , $T_{\text{max}} = 0.792$	$k = -13 \rightarrow 12$
4504 measured reflections	$l = -14 \rightarrow 14$
4232 independent reflections	3 standard reflections
3640 reflections with $I > 2\sigma(I)$	every 200 reflections
	intensity decay: 6%

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2 + 0.2286P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 1.15 \text{ e \AA}^{-3}$
4232 reflections	$\Delta\rho_{\text{min}} = -1.23 \text{ e \AA}^{-3}$
319 parameters	
H-atom parameters constrained	

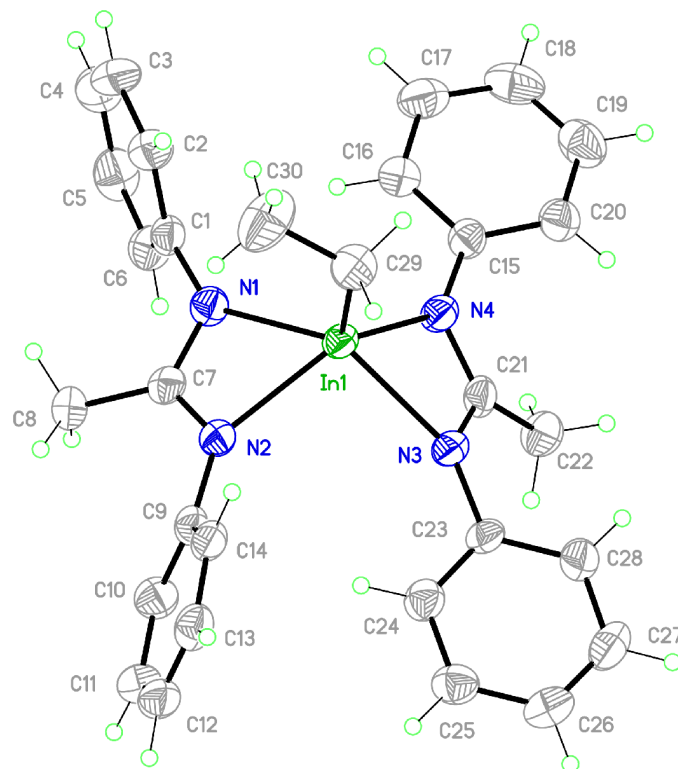
**Table 1**

Selected geometric parameters (Å,  $^\circ$ ).

In1—C29	2.157 (5)	N1—C7	1.333 (6)
In1—N4	2.234 (4)	N2—C7	1.315 (6)
In1—N2	2.255 (4)	N3—C21	1.341 (7)
In1—N1	2.274 (4)	N4—C21	1.331 (6)
In1—N3	2.292 (4)		
C29—In1—N4	120.3 (2)	C29—In1—N3	119.96 (19)
C29—In1—N2	113.2 (2)	N4—In1—N3	58.75 (15)
N4—In1—N2	126.51 (16)	N2—In1—N3	96.83 (15)
C29—In1—N1	114.18 (19)	N1—In1—N3	125.80 (15)
N4—In1—N1	96.12 (16)	N2—C7—N1	112.6 (4)
N2—In1—N1	58.22 (15)	N4—C21—N3	112.4 (4)

H atoms were added at calculated positions ( $\text{C—H} = 0.94\text{--}0.98$  Å) and refined using a riding model and were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of their parent atoms.

Data collection: Siemens P3R3 software; cell refinement: Siemens P3R3 software; data reduction: SHELXTL-Plus (Siemens, 1994);



**Figure 1**

View of the title molecule, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radii.

program(s) used to solve structure: SHELXS90 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL/PC (Sheldrick, 1994); software used to prepare material for publication: SHELXL97.

The authors acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen, 2002). We also acknowledge the use of the WinGX system of programs (Farrugia, 1999).

## References

- Addison, A. W., Rao, T. N., van Reedijk, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.
- Alcock, N. W. (1970). *Acta Cryst.* **A26**, 437–439.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Barker, J., Blacker, N. C., Phillips, P. R., Alcock, N. W., Errington, W. & Wallbridge, M. G. H. (1996). *J. Chem. Soc. Dalton Trans.* pp. 431–437.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
- Patton, J. T., Bokota, M. M. & Abboud, K. A. (2002). *Organometallics*, **21**, 2145–2148.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1994). SHELXTL/PC. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Siemens (1994). SHELXTL-Plus Reference Manual. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Zhou, Y. & Richeson, D. S. (1996). *Inorg. Chem.* **35**, 2448–2451.