

The structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares methods, with anisotropic displacement parameters for all non-H atoms.

Data collection: Enraf-Nonius (1982) CAD-4 software. Structure solution and refinement: *TEXSAN* (Molecular Structure Corporation, 1988). Molecular graphics: *ORTEPII* (Johnson, 1976) and *PLUTO* (Motherwell, 1974).

Financial support for this work was provided by the National Science Foundation of China and by a grant for a Major Project from the State Science and Technology Commission of China.

Lists of structure factors, anisotropic displacement parameters, least-squares-planes data, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1132). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Arend, H. & Huber, W. (1978). *J. Cryst. Growth*, **43**, 213–223.  
 Enraf-Nonius (1982). *CAD-4 Operations Manual*. Enraf-Nonius, Delft, The Netherlands.  
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 Molecular Structure Corporation (1988). *TEXSAN. TEXRAY Structure Analysis Package*. Version 2.1. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.  
 Motherwell, W. D. S. (1974). *PLUTO. Program for Plotting Molecular and Crystal Structures*. University of Cambridge, England.  
 Needham, G. F. & Willett, R. D. (1984). *J. Phys. Chem.* **88**, 674–682.  
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.  
 Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

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## Adduct of AlMe<sub>3</sub> with Benzo[*f*]quinoline

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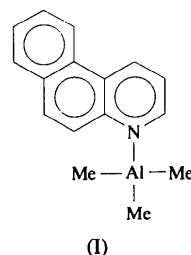
### Abstract

The reaction of AlMe<sub>3</sub> with benzo[*f*]quinoline gave the Lewis acid–base adduct Me<sub>3</sub>Al–BQ, (BQ = benzo[*f*]quinoline) {(benzo[*f*]quinoline-*N*)trimethylaluminium,

[Al(CH<sub>3</sub>)<sub>3</sub>(C<sub>13</sub>H<sub>9</sub>N)]}, with an Al–N bond length of 2.057(2) Å. This adduct is much less air and water sensitive than the trialkyl metal. The complex was found to be a discrete 1:1 molecular adduct of trimethylaluminium bonded to BQ. The compound contains a tetracoordinate Al atom with distorted tetrahedral geometry.

### Comment

The growth of epitaxial layers of Group III–V and Group II–VI compound semiconductors by metal-organic chemical vapour deposition (MOCVD) is being studied extensively and is progressing towards the production phase for *e.g.* gallium arsenide. The electrical properties of these layers depend critically upon the purity of the precursors (metal alkyls and hydrides) employed in the semiconductor growth. The development of purification techniques for the removal of metal-containing impurities in these precursors to levels ≤ 1 p.p.m. has provided an exciting challenge for the organometallic chemist (Beachley & Coates, 1965; Bradley, Dawes, Frigo, Hursthouse & Smith, 1990). In this paper we report the synthesis, characterization and X-ray structure determination of a novel intermolecular-stabilized organoaluminium complex, (I), as a MOCVD precursor.



The structure consists of a discrete 1:1 molecular adduct of trimethylaluminium bonded to BQ. The compound contains tetracoordinate Al atoms within distorted tetrahedra, with the distortion being towards trigonal pyramidal geometry. The base of the pyramid is defined by the three methyl C atoms. The Al atom is almost coplanar with the plane of the coordinated benzo[*f*]quinoline (Bradford, Bradley, Hursthouse & Motevalli, 1992). The C–Al–N bond angles [103.9(1)–105.5(1)°] are consistently *ca* 4–5° smaller than the tetrahedral angle. The Al–C bond lengths [1.965(4)–1.987(3) Å] are within the expected range for metal–carbon single bonds. Thus, it is noteworthy that whereas the Al–C and Ga–C [1.972(5)–1.984(6) Å] (Sun, Wang, Huang & You, 1995) bond distances are very similar to each other, the Al–N bond [2.057(2) Å] is significantly shorter than the Ga–N bond [2.152(3) Å], implying stronger bonding of BQ to AlMe<sub>3</sub> than to GaMe<sub>3</sub>.

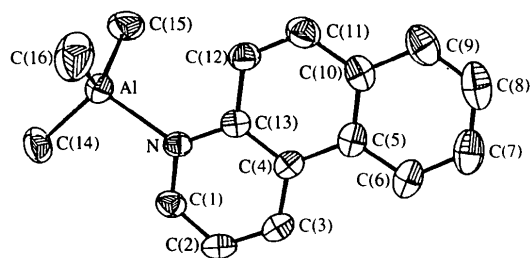


Fig. 1. Molecular structure of Me<sub>3</sub>Al-BQ showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

## Experimental

The synthesis was carried out under an atmosphere of specially dried oxygen-free nitrogen using Schlenk technology and a dry glove box. Solvents were carefully dried by distillation from sodium diphenyl ketone under nitrogen before use. The product was prepared by reaction of benzo[*f*]quinoline and AlMe<sub>3</sub> (ratio 1:1.2) in Et<sub>2</sub>O for 5 h at room temperature. It was recrystallized from Et<sub>2</sub>O. The crystal was sealed in a Lindemann capillary under nitrogen.

### Crystal data

[Al(CH<sub>3</sub>)<sub>3</sub>(C<sub>13</sub>H<sub>9</sub>N)]

*M<sub>r</sub>* = 251.31

Monoclinic

*P*2<sub>1</sub>/*c*

*a* = 7.242 (7) Å

*b* = 14.634 (4) Å

*c* = 13.772 (5) Å

β = 97.89 (7)°

*V* = 1446 (2) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.15 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 9.98–14.78°

μ = 0.118 mm<sup>-1</sup>

*T* = 296 K

Columnar

1.0 × 0.45 × 0.30 mm

Colourless

### Data collection

Enraf-Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction: none

2885 measured reflections

2669 independent reflections

1794 observed reflections

[*I* > 3σ(*I*)]

*R<sub>int</sub>* = 0.018

θ<sub>max</sub> = 25°

*h* = 0 → 8

*k* = 0 → 17

*l* = -16 → 16

3 standard reflections

monitored every 300 reflections

intensity decay: 9.6%

### Refinement

Refinement on *F*

*R* = 0.064

*wR* = 0.073

*S* = 2.11

1794 reflections

163 parameters

H-atom parameters not refined

*w* = 1/σ<sup>2</sup>(*F*)

(Δ/σ)<sub>max</sub> = 0.0002

Δρ<sub>max</sub> = 0.25 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.27 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B<sub>eq</sub></i>
Al	0.4691 (1)	0.17042 (4)	0.03475 (5)	3.27 (4)
N	0.3986 (3)	0.3037 (1)	0.0595 (1)	2.59 (8)
C(1)	0.4433 (4)	0.3353 (1)	0.1504 (2)	3.2 (1)
C(2)	0.4090 (4)	0.4234 (2)	0.1775 (2)	3.5 (1)
C(3)	0.3277 (4)	0.4826 (1)	0.1081 (2)	3.0 (1)
C(4)	0.2777 (3)	0.4536 (1)	0.0110 (2)	2.4 (1)
C(5)	0.1940 (3)	0.5134 (1)	-0.0671 (2)	2.9 (1)
C(6)	0.1556 (4)	0.6064 (1)	-0.0500 (2)	3.7 (1)
C(7)	0.0825 (4)	0.6612 (2)	-0.1277 (3)	4.7 (1)
C(8)	0.0446 (5)	0.6272 (2)	-0.2218 (3)	4.9 (2)
C(9)	0.0765 (5)	0.5368 (2)	-0.2400 (2)	4.5 (1)
C(10)	0.1504 (4)	0.4791 (1)	-0.1624 (2)	3.1 (1)
C(11)	0.1862 (4)	0.3846 (2)	-0.1800 (2)	4.0 (1)
C(12)	0.2619 (4)	0.3285 (1)	-0.1085 (2)	3.3 (1)
C(13)	0.3154 (3)	0.3617 (1)	-0.0105 (2)	2.5 (1)
C(14)	0.6075 (5)	0.1295 (2)	0.1620 (2)	4.6 (1)
C(15)	0.6325 (5)	0.1759 (2)	-0.0686 (2)	5.0 (1)
C(16)	0.2275 (6)	0.1091 (2)	0.0012 (3)	6.6 (2)

Table 2. Selected geometric parameters (Å, °)

Al—C(16)	1.965 (4)	C(4)—C(5)	1.453 (4)
Al—C(15)	1.974 (3)	C(5)—C(10)	1.401 (4)
Al—C(14)	1.987 (3)	C(5)—C(6)	1.415 (3)
Al—N	2.057 (2)	C(6)—C(7)	1.383 (4)
N—C(1)	1.332 (3)	C(7)—C(8)	1.380 (5)
N—C(13)	1.361 (3)	C(8)—C(9)	1.372 (4)
C(1)—C(2)	1.374 (3)	C(9)—C(10)	1.408 (4)
C(2)—C(3)	1.362 (4)	C(10)—C(11)	1.434 (3)
C(3)—C(4)	1.402 (4)	C(11)—C(12)	1.339 (4)
C(4)—C(13)	1.412 (3)	C(12)—C(13)	1.437 (4)
C(16)—Al—C(15)	116.9 (2)	C(10)—C(5)—C(6)	118.4 (2)
C(16)—Al—C(14)	113.3 (2)	C(10)—C(5)—C(4)	119.7 (2)
C(16)—Al—N	103.9 (1)	C(6)—C(5)—C(4)	121.8 (2)
C(15)—Al—C(14)	111.6 (1)	C(7)—C(6)—C(5)	119.5 (3)
C(15)—Al—N	105.5 (1)	C(8)—C(7)—C(6)	121.5 (2)
C(14)—Al—N	104.1 (1)	C(9)—C(8)—C(7)	120.2 (3)
C(1)—N—C(13)	118.4 (2)	C(8)—C(9)—C(10)	119.7 (3)
C(1)—N—Al	116.8 (2)	C(5)—C(10)—C(9)	120.6 (2)
C(13)—N—Al	124.7 (1)	C(5)—C(10)—C(11)	118.7 (2)
N—C(1)—C(2)	123.5 (2)	C(9)—C(10)—C(11)	120.6 (2)
C(3)—C(2)—C(1)	118.9 (2)	C(12)—C(11)—C(10)	122.2 (2)
C(2)—C(3)—C(4)	120.5 (2)	C(11)—C(12)—C(13)	120.9 (2)
C(3)—C(4)—C(13)	117.2 (2)	N—C(13)—C(4)	121.6 (2)
C(3)—C(4)—C(5)	123.4 (2)	N—C(13)—C(12)	119.4 (2)
C(13)—C(4)—C(5)	119.4 (2)	C(4)—C(13)—C(12)	119.0 (2)

The anomalous-dispersion terms were included in the structure-factor calculations. All calculations were carried out on a MicroVAX 3100 computer.

Cell refinement: *CAD-4 Software* (Enraf-Nonius, 1989). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1987). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

Financial support for this work was provided by the National Science Foundation of China and by a grant for a Major Project from the State Science and Technology Commission of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Beachley, O. T. & Coates, G. E. (1965). *J. Chem. Soc.* pp. 3324–3326.  
 Bradford, A. M., Bradley, D. C., Hursthouse, M. G. & Motevalli, M. (1992). *Organometallics*, **11**, 113–115.  
 Bradley, D. C., Dawes, H. M., Frigo, D. M., Hursthouse, M. G. & Smith, L. M. (1990). *Polyhedron*, **9**, 343–346.  
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.  
 Gilmore, C. J. (1983). *MITHRIL. Computer Program for the Automatic Solution of Crystal Structure from X-ray Data*. Department of Chemistry, University of Glasgow, Scotland.  
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 Molecular Structure Corporation (1987). *TEXSAN. TEXRAY Structure Analysis Package*. Revised. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.  
 Sun, H.-S., Wang, X.-M., Huang, X.-Y. & You, X.-Z. (1995). *Polyhedron*. In the press.

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## *mer*-Bis[2,6-bis(1-phenyliminoethyl)pyridine-*N,N',N''*]copper(II) Diperchlorate

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### Abstract

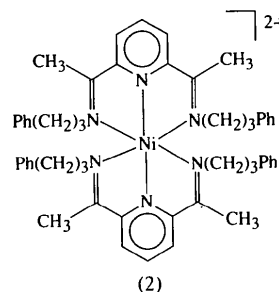
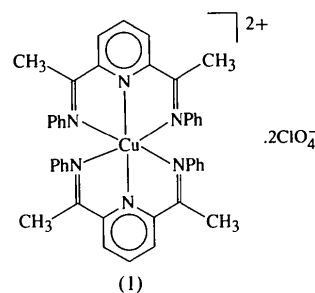
In  $[\text{Cu}(\text{C}_{21}\text{H}_{19}\text{N}_3)_2](\text{ClO}_4)_2$ , two tridentate N-donor ligands coordinate to a copper(II) centre to give a distorted octahedral environment. The two ligands are approximately orthogonal, with an angle of  $89.7(2)^\circ$  between the two  $\text{CuN}_3$  planes. One of the ligands lies further from the Cu atom, its  $\text{Cu}-\text{N}_{\text{imino}}$  distances (mean  $2.31 \text{ \AA}$ ) being  $0.21 \text{ \AA}$  longer than the corresponding distances for the second ligand, an effect which is due to Jahn–Teller distortion.

### Comment

In the title compound, *mer*-bis[2,6-bis(1-phenyliminoethyl)pyridine-*N,N',N''*]copper(II) diperchlorate, (1), the copper(II) centre lies at the centre of a distorted octahedron of N-donor atoms (Fig. 1). This distortion arises in part from the dimensions, shape and rigidity of the 2,6-bis(1-phenyliminoethyl)pyridine ligand, which also

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imposes a *mer* configuration on the complex. The two  $\text{N}_3$ -donor sets are approximately orthogonal, with an angle of  $89.7(2)^\circ$  between the plane of Cu, N7, N10 and N13 and that of Cu, N7', N10' and N13'.



The  $\text{Cu}-\text{N}$  distances in compound (1) fall into three groups: approximately  $1.95 \text{ \AA}$  for the pyridyl N atoms, and  $2.10$  and  $2.31 \text{ \AA}$  for the imino N atoms. A closer

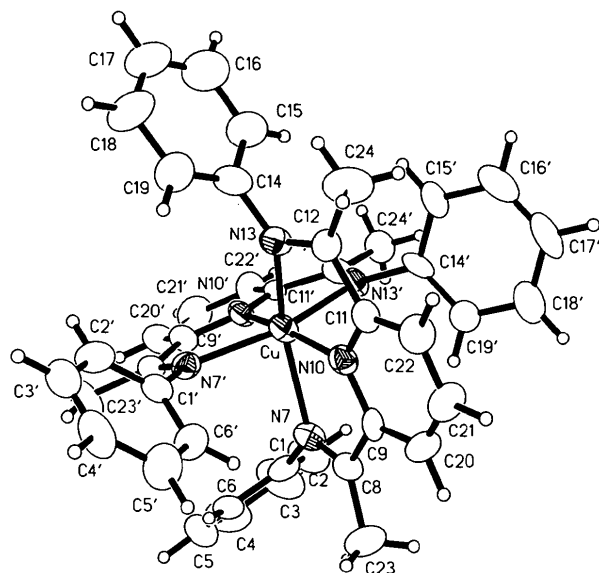


Fig. 1. A view of the title molecule with the atom-numbering scheme. Displacement ellipsoids enclose 30% electron-probability surfaces and H atoms are drawn as small spheres of arbitrary radii. The ligand whose donor set comprises atoms N7, N10 and N13 lies further from the metal centre as a result of a Jahn–Teller elongation of the  $\text{Cu}-\text{N7}$  and  $\text{Cu}-\text{N13}$  distances.