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A primary monomeric alane: [*N*,*N*′-bis(2,6-diisopropylphenyl)pentane-2,4-diiminato-*N*,*N*′]dihydridoaluminium

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The title compound, $[AlH_2(C_{29}H_{41}N_2)]$, is an example of a sterically encumbered monomeric primary alane. The bond lengths are within normal ranges. The N—Al distance is 1.8989 (12) Å. The H atoms bonded to the aluminium center were located in a difference map and refined to Al—H distances of 1.51 (2) and 1.518 (19) Å. There are no significant intermolecular interactions.

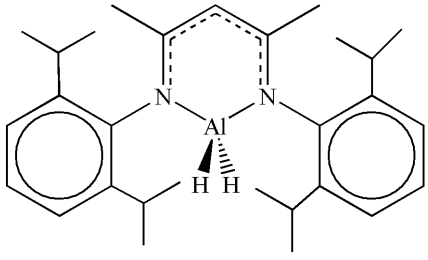
Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.135
Data-to-parameter ratio = 27.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, [*N*,*N*′-bis(2,6-diisopropylphenyl)pentane-2,4-diiminato-*N*,*N*′]dihydridoaluminium, (I), was obtained by the *in situ* reaction of $LiAlH_4$ with [*N*,*N*′-bis(2,6-diisopropylphenyl)pentane-2,4-diiminato]dichlorobismuth(III). Spectroscopic evidence indicates the presence of the primary alane and closely matches that recently reported by Cui, Roesky, Schmidt & Noltemeyer (2000).



(I)

The molecule crystallizes as colorless cube-like crystals in the monoclinic system. The compound is monomeric in the solid state with symmetric bonding of the diimine to the aluminium center with N—Al bond distances of 1.8989 (12) Å (Fig. 1). The H atoms were located in a difference map and refined, giving Al—H distances of 1.51 (2) and 1.518 (19) Å. The H—Al—H angle is 113.2 (11)° and the H—Al—H plane is orthogonal to the diketiminate plane (N1—C1—C2—C3—N2). The Al atom is essentially coplanar with the diketiminate moiety, and the planes of the pendant diisopropylphenyl groups are orthogonal to the plane of the central diimine [C6—C11 90.7 (3)° and C18—C23 87.4 (3)°]. The closely related three-coordinate dimethyl species $[\{HC(CMeNAr)_2\}Al(CH_3)_2]$ (Ar = 2,6-^{i}Pr$_2$C$_6$H$_3$; Qian *et al.*, 1998; Radzewich *et al.*, 1998) has similar N—Al distances [1.922 (2) and 1.935 (2) Å; 1.942 (3) and 1.919 (3) Å]. However, due to steric interactions of the methyl groups and the ^{i}Pr$_2$C$_6$H$_3$ rings, the diketiminate aluminium moiety is buckled and the planes of the diisopropylphenyl rings are no longer orthogonal to the diketiminate group. The unusual Al^{I} compound [{HC(CMe-

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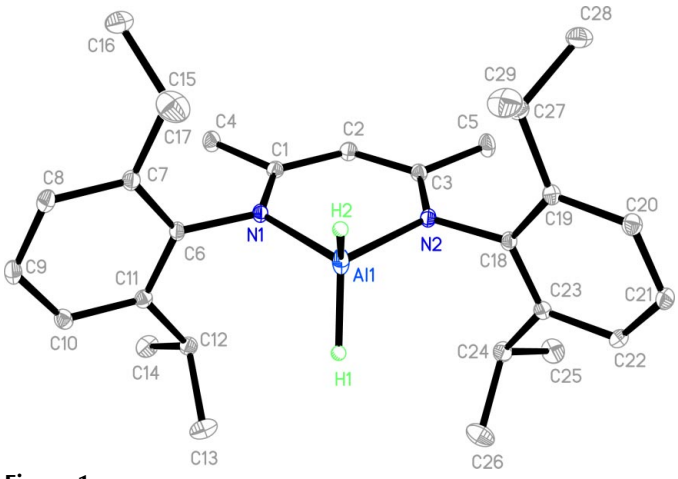

Figure 1
The molecular structure of (I) with 30% probability ellipsoids. H atoms have been omitted for clarity.

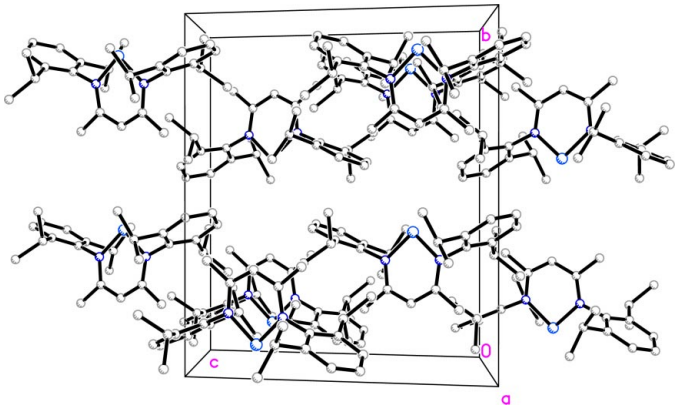

Figure 2
Packing diagram of (I) viewed down the [100] direction. H atoms have been omitted for clarity.

NAr)$_2$Al] (Cui, Roesky, Schmidt, Noltemeyer *et al.*, 2000) bears a closer resemblence to (I). In this complex, the Al—N1—C1—C2—C3—N2 moiety is also planar and the ^{i}Pr$_2$C$_6$H$_3$ rings are once more orthogonal to the diketiminate plane, as seen in (I). However, the Al—N bonding in this complex is slightly longer [1.957 (2) Å] as it is now a two-coordinate Al^{I} center. There are no significant intermolecular interactions in (I), with discrete separated molecules forming alternating stacks along the [100] direction, as shown in Fig. 2.

Experimental

Compound (I) can be synthesized according to the method of Cui, Roesky, Schmidt & Noltemeyer (2000). It was synthesized in this instance by the addition of LiAlH$_4$ (1.52 g, 0.04 mmol) in cold diethyl ether (100 ml) to an *in situ* solution of NacNacBiCl$_2$ (2.30 g, 0.033 mmol) in diethyl ether (50 ml). Immediate precipitation of a metal and salt was observed. After warming to room temperature, the mixture was filtered and concentrated, resulting in immediate precipitation of (I) as a colorless crystalline product (yield 70%; m.p. 467 K).

Crystal data

[AlH$_2$(C$_{29}$H$_{41}$N$_2$)]
M_r = 446.63
Monoclinic, $P2_1/n$
a = 12.4720 (9) Å
b = 16.0700 (12) Å
c = 14.0635 (11) Å
β = 104.66 (2)°
V = 2726.9 (4) Å^3
Z = 4
D_x = 1.088 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4245 reflections
θ = 2.3–31.1°
μ = 0.09 mm^{-1}
T = 90 (2) K
Cube, colorless
0.13 × 0.12 × 0.10 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1999)
T_{min} = 0.988, T_{max} = 0.991
26 394 measured reflections
8512 independent reflections
5366 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 31.5°
$h = -18 \rightarrow 17$
$k = -23 \rightarrow 23$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.135
S = 1.02
8512 reflections
307 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.3174P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

The H1 and H2 atoms were located and refined. All other H atoms were refined as riding (C—H 0.95–1.00 Å).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

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