Electronic Supplementary Information

Synthesis of Al Complexes Containing Phenoxy-imine Ligands and their Use as the Catalyst Precursors for Efficient Living Ring-Opening Polymerisation of ε-Caprolactone

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Contents

Crystal structure determinations, reports for $Me_2Al[O\text{-}2\text{-}R^1\text{-}6\text{-}(R^2N{=}CH)C_6H_3]$ [R^1 = Me (**1**), ^{t}Bu (**2**), H (**3**); R^2 = 2,6-^{i}Pr$_2$C$_6$H$_3$ (**a**), ^{t}Bu (**b**), cyclohexyl (**c**), Ph (**e**), C_6F_5 (**g**)], (**1a**,**b**, **2c**,**e**,**g**), and $Me_2Al[\mu_2\text{-}O\text{-}2\text{-}(R^2N{=}CH)C_6H_4](AlMe_3)$ [R^2 = 2,6-^{i}Pr$_2$C$_6$H$_3$ (**3a**), ^{t}Bu (**3b**)].

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X-ray Structure Report for $Me_2Al[O\text{-}2\text{-}Me\text{-}6\text{-}\{(2,6\text{-}^{i}Pr_2C_6H_3)N{=}CH\}C_6H_3]$ (**1a**)

May 15, 2007

Experimental

Data Collection

A colorless block crystal of $C_{22}H_{30}NOAl$ having approximate dimensions of 0.32 x 0.28 x 0.14 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 360 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 34.0799(16) Å
b = 8.8107(3) Å β = 107.7934(18)^o
c = 15.1777(6) Å
V = 4339.3(3) Å^3

For Z = 8 and F.W. = 351.47, the calculated density is 1.076 g/cm^3. Based on the systematic absences of:

hkl: h+k ± 2n
h0l: l ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 55 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 4.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 250.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 4.0^o step, at χ=45.0^o and ϕ = 210.0^o. The exposure rate was 250.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 20662 reflections that were collected, 4947 were unique (R_{int} = 0.041); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.018 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.713 to 0.986. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2742 observed reflections and 256 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0430$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1333$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.16 and -0.16 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

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(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{22}H_{30}NOAl$
Formula Weight	351.47
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.32 X 0.28 X 0.14 mm
Crystal System	monoclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 360.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 34.0799(16) Å b = 8.8107(3) Å c = 15.1777(6) Å β = 107.7934(18) ^{o} V = 4339.3(3) $Å^3$
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.076 g/cm^3
F_{000}	1520.00
μ(MoKα)	1.018 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	55 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	250.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=210.0)	0.0 - 160.0^{o}
Exposure Rate	250.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 20662 Unique: 4947 (R_{int} = 0.041)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.713 - 0.986)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0027Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	2742
No. Variables	256
Reflection/Parameter Ratio	10.71
Residuals: R1 (I>2.00σ(I))	0.0430
Residuals: wR2 (I>2.00σ(I))	0.1333
Goodness of Fit Indicator	1.005
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.16 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.16 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.12132(2)	0.32905(7)	0.68553(4)	3.677(15)
O(1)	0.17114(5)	0.24788(18)	0.72364(10)	4.54(3)
N(1)	0.12386(5)	0.36746(17)	0.55871(11)	3.12(3)
C(1)	0.11857(11)	0.5201(3)	0.7489(2)	6.18(7)
C(2)	0.07904(9)	0.1787(3)	0.68313(19)	5.77(7)
C(3)	0.19405(7)	0.1873(2)	0.67660(15)	3.56(4)
C(4)	0.22764(7)	0.0943(2)	0.72277(18)	4.63(5)
C(5)	0.25158(8)	0.0331(2)	0.6727(2)	5.63(7)
C(6)	0.24375(8)	0.0608(3)	0.5797(2)	5.92(7)
C(7)	0.21110(8)	0.1512(2)	0.5333(2)	4.83(6)
C(8)	0.18576(6)	0.2151(2)	0.58064(15)	3.46(4)
C(9)	0.15232(6)	0.3096(2)	0.52876(15)	3.30(4)
C(10)	0.23507(10)	0.0631(3)	0.8233(2)	7.58(9)
C(11)	0.09309(6)	0.4621(2)	0.49464(14)	3.38(4)
C(12)	0.05718(7)	0.3955(2)	0.43854(15)	4.31(5)
C(13)	0.02773(8)	0.4902(3)	0.38040(19)	6.01(7)
C(14)	0.03449(10)	0.6452(4)	0.3788(2)	6.51(8)
C(15)	0.07003(10)	0.7071(3)	0.4341(2)	5.89(7)
C(16)	0.10071(8)	0.6188(2)	0.49389(17)	4.19(5)
C(17)	0.05081(9)	0.2244(3)	0.43367(17)	5.64(6)
C(18)	0.06299(12)	0.1590(3)	0.3531(2)	7.30(9)
C(19)	0.00700(11)	0.1787(4)	0.4270(2)	9.63(12)
C(20)	0.14114(9)	0.6903(2)	0.5489(2)	5.35(6)
C(21)	0.16813(10)	0.7186(3)	0.4867(2)	7.22(9)
C(22)	0.13479(12)	0.8366(3)	0.5984(2)	8.40(10)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.0992	0.5857	0.7082	7.69
H(2)	0.1101	0.5002	0.8019	7.69
H(3)	0.1449	0.5669	0.7674	7.68
H(4)	0.0772	0.1653	0.7438	7.06
H(5)	0.0533	0.2130	0.6433	7.10
H(6)	0.0860	0.0848	0.6609	7.08
H(7)	0.2776	-0.0361	0.7147	6.51
H(8)	0.2596	0.0076	0.5402	7.44
H(9)	0.2063	0.1836	0.4609	6.10
H(10)	0.1509	0.3348	0.4592	4.03
H(11)	0.2574	-0.0057	0.8450	8.30
H(12)	0.2414	0.1552	0.8573	8.29
H(13)	0.2109	0.0197	0.8315	8.30
H(14)	-0.0005	0.4510	0.3376	6.96
H(15)	0.0089	0.7058	0.3315	7.70
H(16)	0.0755	0.8221	0.4272	7.11
H(17)	0.0708	0.1730	0.4938	6.36
H(18)	0.0481	0.2106	0.2981	8.56
H(19)	0.0917	0.1729	0.3637	8.58
H(20)	0.0567	0.0538	0.3467	8.58
H(21)	0.0008	0.2147	0.4802	11.29
H(22)	-0.0116	0.2224	0.3731	11.28
H(23)	0.0044	0.0713	0.4239	11.29
H(24)	0.1556	0.6088	0.6019	6.19
H(25)	0.1938	0.7611	0.5214	8.60
H(26)	0.1727	0.6254	0.4599	8.61
H(27)	0.1542	0.7871	0.4390	8.62
H(28)	0.1240	0.9125	0.5529	9.88
H(29)	0.1159	0.8182	0.6319	9.87
H(30)	0.1603	0.8702	0.6397	9.88

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0507(4)	0.0493(3)	0.0384(3)	0.0062(3)	0.0117(3)	-0.0019(2)
O(1)	0.0539(10)	0.0715(10)	0.0411(9)	0.0140(8)	0.0057(7)	0.0004(7)
N(1)	0.0394(10)	0.0349(8)	0.0411(9)	0.0009(7)	0.0075(8)	-0.0001(6)
C(1)	0.109(2)	0.0679(16)	0.0657(17)	0.0114(16)	0.0384(17)	-0.0082(13)
C(2)	0.0738(19)	0.0914(19)	0.0576(16)	-0.0090(15)	0.0254(14)	0.0023(13)
C(3)	0.0386(12)	0.0390(10)	0.0526(13)	-0.0031(9)	0.0062(10)	-0.0019(9)
C(4)	0.0450(14)	0.0504(13)	0.0688(16)	0.0039(10)	-0.0003(12)	0.0049(11)
C(5)	0.0452(15)	0.0535(14)	0.107(2)	0.0101(11)	0.0105(15)	0.0062(14)
C(6)	0.0629(18)	0.0667(16)	0.106(2)	0.0188(14)	0.0413(17)	0.0057(16)
C(7)	0.0619(16)	0.0526(13)	0.0779(17)	0.0080(12)	0.0343(14)	0.0048(11)
C(8)	0.0394(12)	0.0351(10)	0.0567(13)	-0.0015(9)	0.0144(10)	0.0004(9)
C(9)	0.0450(13)	0.0350(10)	0.0471(12)	-0.0020(9)	0.0167(10)	0.0008(8)
C(10)	0.084(2)	0.100(2)	0.077(2)	0.0298(19)	-0.0140(17)	0.0134(17)
C(11)	0.0406(12)	0.0463(11)	0.0406(11)	0.0051(9)	0.0111(9)	0.0057(9)
C(12)	0.0457(14)	0.0751(15)	0.0396(12)	-0.0045(12)	0.0078(10)	0.0081(11)
C(13)	0.0445(15)	0.118(2)	0.0583(17)	0.0080(16)	0.0039(13)	0.0158(16)
C(14)	0.0609(19)	0.109(2)	0.074(2)	0.0379(18)	0.0148(16)	0.0265(17)
C(15)	0.083(2)	0.0647(16)	0.0773(19)	0.0312(15)	0.0262(17)	0.0195(13)
C(16)	0.0565(15)	0.0451(12)	0.0583(14)	0.0119(10)	0.0185(12)	0.0063(10)
C(17)	0.0780(19)	0.0786(16)	0.0454(14)	-0.0366(15)	0.0008(13)	0.0060(12)
C(18)	0.137(3)	0.0694(17)	0.0646(18)	-0.0240(18)	0.0216(19)	-0.0033(14)
C(19)	0.115(3)	0.162(3)	0.080(2)	-0.081(2)	0.016(2)	-0.002(2)
C(20)	0.0733(18)	0.0416(12)	0.0810(19)	-0.0059(12)	0.0128(15)	0.0022(11)
C(21)	0.074(2)	0.085(2)	0.114(2)	-0.0154(17)	0.0262(19)	0.0079(18)
C(22)	0.142(3)	0.0496(16)	0.122(3)	-0.0172(17)	0.031(2)	-0.0195(16)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.7688(17)	Al(1)	N(1)	1.9821(18)
Al(1)	C(1)	1.955(2)	Al(1)	C(2)	1.949(3)
O(1)	C(3)	1.321(3)	N(1)	C(9)	1.295(2)
N(1)	C(11)	1.455(2)	C(3)	C(4)	1.408(2)
C(3)	C(8)	1.418(3)	C(4)	C(5)	1.383(4)
C(4)	C(10)	1.494(4)	C(5)	C(6)	1.376(4)
C(6)	C(7)	1.374(3)	C(7)	C(8)	1.399(3)
C(8)	C(9)	1.437(2)	C(11)	C(12)	1.389(2)
C(11)	C(16)	1.405(2)	C(12)	C(13)	1.393(3)
C(12)	C(17)	1.522(3)	C(13)	C(14)	1.386(4)
C(14)	C(15)	1.359(4)	C(15)	C(16)	1.393(3)
C(16)	C(20)	1.513(3)	C(17)	C(18)	1.519(4)
C(17)	C(19)	1.519(4)	C(20)	C(21)	1.527(5)
C(20)	C(22)	1.541(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(5)	H(7)	1.104	C(6)	H(8)	1.033
C(7)	H(9)	1.099	C(9)	H(10)	1.067
C(10)	H(11)	0.950	C(10)	H(12)	0.950
C(10)	H(13)	0.950	C(13)	H(14)	1.042
C(14)	H(15)	1.087	C(15)	H(16)	1.042
C(17)	H(17)	1.059	C(18)	H(18)	0.950
C(18)	H(19)	0.950	C(18)	H(20)	0.950
C(19)	H(21)	0.950	C(19)	H(22)	0.950
C(19)	H(23)	0.950	C(20)	H(24)	1.079
C(21)	H(25)	0.950	C(21)	H(26)	0.950
C(21)	H(27)	0.950	C(22)	H(28)	0.950
C(22)	H(29)	0.950	C(22)	H(30)	0.950

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	93.68(7)	O(1)	Al(1)	C(1)	111.89(11)
O(1)	Al(1)	C(2)	111.20(10)	N(1)	Al(1)	C(1)	110.71(10)
N(1)	Al(1)	C(2)	110.44(9)	C(1)	Al(1)	C(2)	116.61(15)
Al(1)	O(1)	C(3)	130.81(12)	Al(1)	N(1)	C(9)	122.24(13)
Al(1)	N(1)	C(11)	120.69(14)	C(9)	N(1)	C(11)	117.07(17)
O(1)	C(3)	C(4)	119.4(2)	O(1)	C(3)	C(8)	121.34(17)
C(4)	C(3)	C(8)	119.2(2)	C(3)	C(4)	C(5)	118.7(2)
C(3)	C(4)	C(10)	118.3(2)	C(5)	C(4)	C(10)	123.1(2)
C(4)	C(5)	C(6)	122.2(2)	C(5)	C(6)	C(7)	120.1(3)
C(6)	C(7)	C(8)	120.0(2)	C(3)	C(8)	C(7)	119.82(19)
C(3)	C(8)	C(9)	122.6(2)	C(7)	C(8)	C(9)	117.6(2)
N(1)	C(9)	C(8)	126.1(2)	N(1)	C(11)	C(12)	119.17(18)
N(1)	C(11)	C(16)	118.11(16)	C(12)	C(11)	C(16)	122.70(18)
C(11)	C(12)	C(13)	117.7(2)	C(11)	C(12)	C(17)	122.28(19)
C(13)	C(12)	C(17)	119.9(2)	C(12)	C(13)	C(14)	120.6(2)
C(13)	C(14)	C(15)	120.4(2)	C(14)	C(15)	C(16)	121.9(2)
C(11)	C(16)	C(15)	116.7(2)	C(11)	C(16)	C(20)	122.94(19)
C(15)	C(16)	C(20)	120.2(2)	C(12)	C(17)	C(18)	110.2(2)
C(12)	C(17)	C(19)	112.8(2)	C(18)	C(17)	C(19)	110.5(2)
C(16)	C(20)	C(21)	110.2(2)	C(16)	C(20)	C(22)	112.2(2)
C(21)	C(20)	C(22)	111.5(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.4	Al(1)	C(1)	H(2)	109.3
Al(1)	C(1)	H(3)	109.7	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.5	Al(1)	C(2)	H(5)	109.3
Al(1)	C(2)	H(6)	109.6	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
C(4)	C(5)	H(7)	113.7	C(6)	C(5)	H(7)	124.1
C(5)	C(6)	H(8)	122.9	C(7)	C(6)	H(8)	116.8
C(6)	C(7)	H(9)	120.8	C(8)	C(7)	H(9)	118.9
N(1)	C(9)	H(10)	117.0	C(8)	C(9)	H(10)	116.8
C(4)	C(10)	H(11)	109.8	C(4)	C(10)	H(12)	109.7
C(4)	C(10)	H(13)	108.9	H(11)	C(10)	H(12)	109.5
H(11)	C(10)	H(13)	109.5	H(12)	C(10)	H(13)	109.5
C(12)	C(13)	H(14)	123.1	C(14)	C(13)	H(14)	116.3
C(13)	C(14)	H(15)	113.0	C(15)	C(14)	H(15)	126.6
C(14)	C(15)	H(16)	118.8	C(16)	C(15)	H(16)	119.1
C(12)	C(17)	H(17)	109.7	C(18)	C(17)	H(17)	105.7
C(19)	C(17)	H(17)	107.7	C(17)	C(18)	H(18)	108.8
C(17)	C(18)	H(19)	109.6	C(17)	C(18)	H(20)	110.1
H(18)	C(18)	H(19)	109.5	H(18)	C(18)	H(20)	109.5
H(19)	C(18)	H(20)	109.5	C(17)	C(19)	H(21)	108.9
C(17)	C(19)	H(22)	109.6	C(17)	C(19)	H(23)	110.0
H(21)	C(19)	H(22)	109.5	H(21)	C(19)	H(23)	109.5
H(22)	C(19)	H(23)	109.5	C(16)	C(20)	H(24)	105.5
C(21)	C(20)	H(24)	110.2	C(22)	C(20)	H(24)	107.1
C(20)	C(21)	H(25)	110.4	C(20)	C(21)	H(26)	109.4
C(20)	C(21)	H(27)	108.6	H(25)	C(21)	H(26)	109.5
H(25)	C(21)	H(27)	109.5	H(26)	C(21)	H(27)	109.5
C(20)	C(22)	H(28)	108.4	C(20)	C(22)	H(29)	109.7
C(20)	C(22)	H(30)	110.3	H(28)	C(22)	H(29)	109.5
H(28)	C(22)	H(30)	109.5	H(29)	C(22)	H(30)	109.5

Table 8. Torsion Angles(O)

atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(9)	11.98(15)
N(1)	Al(1)	O(1)	C(3)	-20.80(19)
C(2)	Al(1)	O(1)	C(3)	92.8(2)
C(1)	Al(1)	N(1)	C(11)	-53.51(17)
C(2)	Al(1)	N(1)	C(11)	77.17(16)
Al(1)	O(1)	C(3)	C(8)	17.7(3)
Al(1)	N(1)	C(11)	C(12)	-88.7(2)
C(9)	N(1)	C(11)	C(12)	90.8(2)
C(11)	N(1)	C(9)	C(8)	179.14(17)
O(1)	C(3)	C(4)	C(10)	2.3(3)
O(1)	C(3)	C(8)	C(9)	-0.1(2)
C(4)	C(3)	C(8)	C(9)	-179.45(19)
C(8)	C(3)	C(4)	C(10)	-178.3(2)
C(10)	C(4)	C(5)	C(6)	178.7(2)
C(5)	C(6)	C(7)	C(8)	-0.0(3)
C(6)	C(7)	C(8)	C(9)	179.5(2)
C(7)	C(8)	C(9)	N(1)	173.80(19)
N(1)	C(11)	C(12)	C(17)	-7.0(3)
N(1)	C(11)	C(16)	C(20)	6.7(3)
C(12)	C(11)	C(16)	C(20)	-174.8(2)
C(16)	C(11)	C(12)	C(17)	174.5(2)
C(11)	C(12)	C(17)	C(18)	-94.6(2)
C(13)	C(12)	C(17)	C(18)	80.6(3)
C(17)	C(12)	C(13)	C(14)	-175.0(2)
C(13)	C(14)	C(15)	C(16)	-0.0(5)
C(14)	C(15)	C(16)	C(20)	175.3(3)
C(11)	C(16)	C(20)	C(22)	-136.0(2)
C(15)	C(16)	C(20)	C(22)	48.7(4)
O(1)	Al(1)	N(1)	C(11)	-168.58(13)
C(1)	Al(1)	O(1)	C(3)	-134.9(2)
C(1)	Al(1)	N(1)	C(9)	127.04(17)
C(2)	Al(1)	N(1)	C(9)	-102.27(17)
Al(1)	O(1)	C(3)	C(4)	-162.92(16)
Al(1)	N(1)	C(9)	C(8)	-1.4(2)
Al(1)	N(1)	C(11)	C(16)	89.9(2)
C(9)	N(1)	C(11)	C(16)	-90.7(2)
O(1)	C(3)	C(4)	C(5)	-179.3(2)
O(1)	C(3)	C(8)	C(7)	178.87(19)
C(4)	C(3)	C(8)	C(7)	-0.5(3)
C(8)	C(3)	C(4)	C(5)	0.1(2)
C(3)	C(4)	C(5)	C(6)	0.4(3)
C(4)	C(5)	C(6)	C(7)	-0.4(3)
C(6)	C(7)	C(8)	C(3)	0.5(3)
C(3)	C(8)	C(9)	N(1)	-7.2(3)
N(1)	C(11)	C(12)	C(13)	177.8(2)
N(1)	C(11)	C(16)	C(15)	-177.8(2)
C(12)	C(11)	C(16)	C(15)	0.7(4)
C(16)	C(11)	C(12)	C(13)	-0.7(3)
C(11)	C(12)	C(13)	C(14)	0.4(4)
C(11)	C(12)	C(17)	C(19)	141.4(2)
C(13)	C(12)	C(17)	C(19)	-43.4(3)
C(12)	C(13)	C(14)	C(15)	-0.0(4)
C(14)	C(15)	C(16)	C(11)	-0.3(4)
C(11)	C(16)	C(20)	C(21)	99.1(3)
C(15)	C(16)	C(20)	C(21)	-76.2(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
C(7)	C(7)[1)]	3.567(4)			

Symmetry Operators:

(1) -X+1/2,-Y+1/2,-Z+1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Al(1)	H(7)[1)]	3.519	O(1)	H(7)[1)]	2.559
O(1)	H(27)[2)]	3.503	O(1)	H(30)[3)]	3.542
C(1)	H(7)[1)]	3.446	C(1)	H(10)[2)]	3.299
C(1)	H(19)[2)]	3.488	C(1)	H(26)[2)]	3.420
C(2)	H(15)[4)]	3.106	C(2)	H(20)[5)]	3.478
C(2)	H(23)[6)]	3.571	C(2)	H(29)[3)]	3.587
C(3)	H(7)[1)]	2.936	C(3)	H(11)[1)]	3.239
C(3)	H(28)[3)]	3.514	C(3)	H(30)[3)]	3.008
C(4)	H(7)[1)]	3.412	C(4)	H(30)[3)]	2.998
C(5)	H(3)[7)]	3.376	C(5)	H(12)[7)]	3.379
C(5)	H(25)[3)]	3.482	C(5)	H(30)[3)]	3.323
C(6)	H(9)[8)]	2.997	C(6)	H(25)[3)]	3.120
C(6)	H(26)[8)]	3.492	C(7)	H(8)[8)]	3.458
C(7)	H(9)[8)]	3.145	C(7)	H(11)[1)]	3.534
C(7)	H(13)[9)]	3.412	C(7)	H(25)[3)]	3.482
C(8)	H(11)[1)]	3.121	C(8)	H(28)[3)]	3.343
C(8)	H(30)[3)]	3.357	C(9)	H(11)[1)]	3.490
C(10)	H(7)[1)]	3.583	C(10)	H(8)[5)]	3.200
C(10)	H(9)[5)]	3.361	C(10)	H(24)[7)]	3.570
C(10)	H(25)[7)]	3.314	C(10)	H(30)[3)]	3.579
C(11)	H(2)[10)]	3.169	C(12)	H(2)[10)]	3.269
C(13)	H(2)[10)]	3.370	C(13)	H(14)[11)]	3.171
C(14)	H(2)[10)]	3.389	C(14)	H(4)[10)]	3.305
C(14)	H(5)[4)]	3.163	C(14)	H(14)[11)]	3.569
C(14)	H(15)[11)]	3.126	C(14)	H(21)[4)]	3.020
C(15)	H(2)[10)]	3.302	C(15)	H(4)[10)]	3.178
C(15)	H(20)[12)]	3.306	C(15)	H(21)[4)]	3.148
C(16)	H(2)[10)]	3.204	C(18)	H(4)[9)]	3.412
C(18)	H(16)[3)]	3.157	C(18)	H(22)[11)]	3.390
C(19)	H(18)[11)]	3.369	C(19)	H(23)[6)]	3.266
C(21)	H(2)[10)]	3.479	C(21)	H(8)[8)]	3.290
C(21)	H(11)[1)]	3.593	C(21)	H(12)[1)]	3.313
C(22)	H(6)[12)]	3.068	H(1)	H(14)[4)]	3.237
H(1)	H(18)[2)]	3.095	H(1)	H(19)[2)]	3.245
H(1)	H(22)[4)]	3.323	H(2)	C(11)[2)]	3.169
H(2)	C(12)[2)]	3.269	H(2)	C(13)[2)]	3.370
H(2)	C(14)[2)]	3.389	H(2)	C(15)[2)]	3.302

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(2)	C(16)[2)]	3.204	H(2)	C(21)[2)]	3.479
H(2)	H(10)[2)]	2.778	H(2)	H(18)[2)]	3.300
H(2)	H(19)[2)]	3.151	H(2)	H(26)[2)]	2.899
H(2)	H(27)[2)]	3.329	H(3)	C(5)[1)]	3.376
H(3)	H(7)[1)]	2.726	H(3)	H(10)[2)]	2.982
H(3)	H(19)[2)]	3.507	H(3)	H(26)[2)]	3.258
H(4)	C(14)[2)]	3.305	H(4)	C(15)[2)]	3.178
H(4)	C(18)[5)]	3.412	H(4)	H(15)[4)]	3.024
H(4)	H(15)[2)]	3.221	H(4)	H(16)[2)]	2.804
H(4)	H(19)[5)]	3.448	H(4)	H(20)[5)]	2.706
H(4)	H(27)[2)]	3.330	H(5)	C(14)[4)]	3.163
H(5)	H(14)[4)]	3.521	H(5)	H(15)[4)]	2.374
H(5)	H(23)[6)]	3.155	H(6)	C(22)[3)]	3.068
H(6)	H(20)[5)]	3.488	H(6)	H(23)[6)]	3.258
H(6)	H(28)[3)]	2.823	H(6)	H(29)[3)]	2.650
H(6)	H(30)[3)]	3.254	H(7)	Al(1)[7)]	3.519
H(7)	O(1)[7)]	2.559	H(7)	C(1)[7)]	3.446
H(7)	C(3)[7)]	2.936	H(7)	C(4)[7)]	3.412
H(7)	C(10)[7)]	3.583	H(7)	H(3)[7)]	2.726
H(7)	H(12)[7)]	2.929	H(7)	H(24)[7)]	3.269
H(8)	C(7)[8)]	3.458	H(8)	C(10)[9)]	3.200
H(8)	C(21)[8)]	3.290	H(8)	H(9)[8)]	2.960
H(8)	H(10)[8)]	3.348	H(8)	H(11)[9)]	2.941
H(8)	H(12)[9)]	3.016	H(8)	H(12)[7)]	3.478
H(8)	H(13)[9)]	3.104	H(8)	H(25)[3)]	3.071
H(8)	H(25)[8)]	3.148	H(8)	H(26)[8)]	2.588
H(9)	C(6)[8)]	2.997	H(9)	C(7)[8)]	3.145
H(9)	C(10)[9)]	3.361	H(9)	H(8)[8)]	2.960
H(9)	H(9)[8)]	3.078	H(9)	H(11)[9)]	3.233
H(9)	H(13)[9)]	2.698	H(10)	C(1)[10)]	3.299
H(10)	H(2)[10)]	2.778	H(10)	H(3)[10)]	2.982
H(10)	H(8)[8)]	3.348	H(11)	C(3)[7)]	3.239
H(11)	C(7)[7)]	3.534	H(11)	C(8)[7)]	3.121
H(11)	C(9)[7)]	3.490	H(11)	C(21)[7)]	3.593
H(11)	H(8)[5)]	2.941	H(11)	H(9)[5)]	3.233
H(11)	H(24)[7)]	2.998	H(11)	H(25)[7)]	3.216
H(11)	H(26)[7)]	3.385	H(12)	C(5)[1)]	3.379

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	C(21)[7)]	3.313	H(12)	H(7)[1)]	2.929
H(12)	H(8)[5)]	3.016	H(12)	H(8)[1)]	3.478
H(12)	H(24)[7)]	3.398	H(12)	H(25)[2)]	3.441
H(12)	H(25)[7)]	2.580	H(12)	H(26)[7)]	3.372
H(12)	H(27)[2)]	3.588	H(13)	C(7)[5)]	3.412
H(13)	H(8)[5)]	3.104	H(13)	H(9)[5)]	2.698
H(13)	H(27)[2)]	3.350	H(13)	H(30)[3)]	3.180
H(14)	C(13)[11)]	3.171	H(14)	C(14)[11)]	3.569
H(14)	H(1)[4)]	3.237	H(14)	H(5)[4)]	3.521
H(14)	H(14)[11)]	2.671	H(14)	H(15)[11)]	3.356
H(14)	H(18)[11)]	3.053	H(15)	C(2)[4)]	3.106
H(15)	C(14)[11)]	3.126	H(15)	H(4)[4)]	3.024
H(15)	H(4)[10)]	3.221	H(15)	H(5)[4)]	2.374
H(15)	H(14)[11)]	3.356	H(15)	H(15)[11)]	2.361
H(15)	H(20)[12)]	3.446	H(15)	H(21)[4)]	3.056
H(15)	H(23)[12)]	3.534	H(16)	C(18)[12)]	3.157
H(16)	H(4)[10)]	2.804	H(16)	H(17)[12)]	3.273
H(16)	H(19)[12)]	3.334	H(16)	H(20)[12)]	2.365
H(16)	H(21)[4)]	3.328	H(16)	H(23)[12)]	3.259
H(17)	H(16)[3)]	3.273	H(17)	H(28)[3)]	2.893
H(18)	C(19)[11)]	3.369	H(18)	H(1)[10)]	3.095
H(18)	H(2)[10)]	3.300	H(18)	H(14)[11)]	3.053
H(18)	H(18)[11)]	3.155	H(18)	H(22)[11)]	2.517
H(18)	H(23)[11)]	3.517	H(19)	C(1)[10)]	3.488
H(19)	H(1)[10)]	3.245	H(19)	H(2)[10)]	3.151
H(19)	H(3)[10)]	3.507	H(19)	H(4)[9)]	3.448
H(19)	H(16)[3)]	3.334	H(19)	H(28)[3)]	3.577
H(20)	C(2)[9)]	3.478	H(20)	C(15)[3)]	3.306
H(20)	H(4)[9)]	2.706	H(20)	H(6)[9)]	3.488
H(20)	H(15)[3)]	3.446	H(20)	H(16)[3)]	2.365
H(20)	H(22)[11)]	3.545	H(20)	H(28)[3)]	3.496
H(21)	C(14)[4)]	3.020	H(21)	C(15)[4)]	3.148
H(21)	H(15)[4)]	3.056	H(21)	H(16)[4)]	3.328
H(21)	H(23)[6)]	2.942	H(22)	C(18)[11)]	3.390
H(22)	H(1)[4)]	3.323	H(22)	H(18)[11)]	2.517
H(22)	H(20)[11)]	3.545	H(22)	H(29)[4)]	3.548
H(23)	C(2)[6)]	3.571	H(23)	C(19)[6)]	3.266

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(23)	H(5)[6)]	3.155	H(23)	H(6)[6)]	3.258
H(23)	H(15)[3)]	3.534	H(23)	H(16)[3)]	3.259
H(23)	H(18)[11)]	3.517	H(23)	H(21)[6)]	2.942
H(23)	H(23)[6)]	2.727	H(24)	C(10)[1)]	3.570
H(24)	H(7)[1)]	3.269	H(24)	H(11)[1)]	2.998
H(24)	H(12)[1)]	3.398	H(25)	C(5)[12)]	3.482
H(25)	C(6)[12)]	3.120	H(25)	C(7)[12)]	3.482
H(25)	C(10)[1)]	3.314	H(25)	H(8)[12)]	3.071
H(25)	H(8)[8)]	3.148	H(25)	H(11)[1)]	3.216
H(25)	H(12)[10)]	3.441	H(25)	H(12)[1)]	2.580
H(26)	C(1)[10)]	3.420	H(26)	C(6)[8)]	3.492
H(26)	H(2)[10)]	2.899	H(26)	H(3)[10)]	3.258
H(26)	H(8)[8)]	2.588	H(26)	H(11)[1)]	3.385
H(26)	H(12)[1)]	3.372	H(27)	O(1)[10)]	3.503
H(27)	H(2)[10)]	3.329	H(27)	H(4)[10)]	3.330
H(27)	H(12)[10)]	3.588	H(27)	H(13)[10)]	3.350
H(28)	C(3)[12)]	3.514	H(28)	C(8)[12)]	3.343
H(28)	H(6)[12)]	2.823	H(28)	H(17)[12)]	2.893
H(28)	H(19)[12)]	3.577	H(28)	H(20)[12)]	3.496
H(29)	C(2)[12)]	3.587	H(29)	H(6)[12)]	2.650
H(29)	H(22)[4)]	3.548	H(30)	O(1)[12)]	3.542
H(30)	C(3)[12)]	3.008	H(30)	C(4)[12)]	2.998
H(30)	C(5)[12)]	3.323	H(30)	C(8)[12)]	3.357
H(30)	C(10)[12)]	3.579	H(30)	H(6)[12)]	3.254
H(30)	H(13)[12)]	3.180			

Symmetry Operators:

(1) -X+1/2,Y+1/2,-Z+1/2+1
(2) X,-Y+1,Z+1/2
(3) X,Y-1,Z
(4) -X,-Y+1,-Z+1
(5) X,-Y,Z+1/2
(6) -X,-Y,-Z+1
(7) -X+1/2,Y+1/2-1,-Z+1/2+1
(8) -X+1/2,-Y+1/2,-Z+1
(9) X,-Y,Z+1/2-1
(10) X,-Y+1,Z+1/2-1
(11) -X,Y,-Z+1/2
(12) X,Y+1,Z

X-ray Structure Report for $Me_2A[O\text{-}2\text{-}Me\text{-}6\text{-}(^tBuN{=}CH)C_6H_3]$ (**1b**)

February 20, 2007

Experimental

Data Collection

A colorless block crystal of $C_{14}H_{22}NOAl$ having approximate dimensions of 0.46 x 0.44 x 0.34 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 9.7023(5) Å
b = 9.7378(5) Å β = 92.9412(18)^o
c = 15.6108(8) Å
V = 1472.97(13) Å^3

For Z = 4 and F.W. = 247.32, the calculated density is 1.115 g/cm^3. The systematic absences of:

h0l: l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 5.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 80.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 5.0^o step, at χ=45.0^o and ϕ = 180.0^o. The exposure rate was 80.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 14044 reflections that were collected, 3354 were unique (R_{int} = 0.022); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.237 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.803 to 0.959. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2752 observed reflections and 176 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0449$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1466$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.25 and -0.28 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2006). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{14}H_{22}NOAl$
Formula Weight	247.32
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.46 X 0.44 X 0.34 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 9.7023(5) Å b = 9.7378(5) Å c = 15.6108(8) Å β = 92.9412(18) ^{o} V = 1472.97(13) $Å^3$
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.115 g/cm^3
F_{000}	536.00
μ(MoKα)	1.237 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	80.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0^{o}
Exposure Rate	80.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 14044 Unique: 3354 (R_{int} = 0.022)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.803 - 0.959)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF99 PATTY)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0042Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	2752
No. Variables	176
Reflection/Parameter Ratio	15.64
Residuals: R1 (I>2.00σ(I))	0.0449
Residuals: wR2 (I>2.00σ(I))	0.1466
Goodness of Fit Indicator	1.001
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.25 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.28 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.19308(4)	0.24440(4)	0.39644(3)	3.140(11)
O(1)	0.26868(12)	0.40991(11)	0.40758(7)	4.03(2)
N(1)	0.20434(12)	0.23435(12)	0.27033(8)	3.01(2)
C(1)	0.3129(2)	0.10977(19)	0.45678(12)	4.78(3)
C(2)	-0.00116(19)	0.2485(2)	0.42611(13)	4.57(4)
C(3)	0.31002(13)	0.49787(15)	0.34983(8)	3.00(2)
C(4)	0.35832(14)	0.62892(15)	0.37692(10)	3.40(2)
C(5)	0.39963(15)	0.72081(15)	0.31631(13)	3.94(3)
C(6)	0.39603(18)	0.68937(17)	0.22891(12)	4.34(3)
C(7)	0.34932(17)	0.56287(16)	0.20238(10)	3.87(3)
C(8)	0.30553(14)	0.46506(14)	0.26154(9)	3.10(2)
C(9)	0.25307(15)	0.33628(15)	0.22820(9)	3.19(2)
C(10)	0.15219(15)	0.11190(15)	0.21840(10)	3.53(2)
C(11)	0.0233(2)	0.1540(2)	0.16589(17)	6.13(5)
C(12)	0.2626(2)	0.0585(2)	0.16237(16)	6.28(5)
C(13)	0.1122(2)	-0.0015(2)	0.27989(14)	5.83(4)
C(14)	0.36057(19)	0.66316(18)	0.47170(11)	4.66(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.3477	0.1481	0.5095	5.75
H(2)	0.2618	0.0290	0.4678	5.78
H(3)	0.3876	0.0874	0.4223	5.75
H(4)	-0.0169	0.3280	0.4595	5.55
H(5)	-0.0587	0.2513	0.3750	5.53
H(6)	-0.0218	0.1688	0.4581	5.51
H(7)	0.4328	0.8138	0.3352	4.72
H(8)	0.4399	0.7566	0.1889	5.23
H(9)	0.3478	0.5388	0.1434	4.66
H(10)	0.2606	0.3242	0.1660	3.85
H(11)	-0.0115	0.0778	0.1335	7.23
H(12)	-0.0449	0.1853	0.2028	7.29
H(13)	0.0463	0.2259	0.1281	7.26
H(14)	0.3428	0.0415	0.1982	7.65
H(15)	0.2332	-0.0244	0.1351	7.65
H(16)	0.2831	0.1246	0.1201	7.62
H(17)	0.0676	-0.0740	0.2486	6.99
H(18)	0.1925	-0.0358	0.3100	7.02
H(19)	0.0510	0.0353	0.3196	6.99
H(20)	0.3844	0.7571	0.4795	5.68
H(21)	0.2724	0.6468	0.4934	5.62
H(22)	0.4274	0.6073	0.5016	5.62

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0406(2)	0.0381(2)	0.0406(2)	-0.00158(16)	0.0027(2)	0.00318(16)
O(1)	0.0670(7)	0.0433(5)	0.0430(5)	-0.0117(5)	0.0059(4)	-0.0019(4)
N(1)	0.0355(5)	0.0351(5)	0.0437(6)	-0.0021(4)	0.0026(4)	-0.0038(4)
C(1)	0.0610(9)	0.0559(9)	0.0638(10)	0.0037(8)	-0.0072(8)	0.0101(8)
C(2)	0.0486(8)	0.0697(11)	0.0563(10)	0.0016(7)	0.0120(7)	0.0031(7)
C(3)	0.0316(6)	0.0362(6)	0.0463(7)	0.0012(4)	0.0029(5)	-0.0018(5)
C(4)	0.0316(6)	0.0383(6)	0.0591(8)	0.0030(5)	0.0026(5)	-0.0076(6)
C(5)	0.0399(7)	0.0338(6)	0.0761(10)	-0.0017(5)	0.0057(7)	-0.0029(7)
C(6)	0.0532(8)	0.0430(8)	0.0694(10)	-0.0052(7)	0.0088(7)	0.0100(7)
C(7)	0.0505(8)	0.0456(8)	0.0514(8)	-0.0032(6)	0.0069(6)	0.0054(6)
C(8)	0.0352(6)	0.0360(6)	0.0468(7)	0.0006(5)	0.0034(5)	0.0010(5)
C(9)	0.0398(6)	0.0410(7)	0.0405(6)	0.0000(5)	0.0035(5)	-0.0017(5)
C(10)	0.0433(7)	0.0398(7)	0.0511(7)	-0.0071(6)	0.0022(6)	-0.0080(6)
C(11)	0.0744(13)	0.0597(11)	0.0953(14)	-0.0055(9)	-0.0309(11)	-0.0166(10)
C(12)	0.0721(12)	0.0606(11)	0.1087(17)	-0.0200(9)	0.0336(12)	-0.0403(11)
C(13)	0.0971(15)	0.0483(9)	0.0763(11)	-0.0246(10)	0.0073(10)	-0.0057(8)
C(14)	0.0615(10)	0.0499(9)	0.0657(10)	-0.0067(7)	0.0046(8)	-0.0188(7)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.7756(11)	Al(1)	N(1)	1.9797(13)
Al(1)	C(1)	1.9607(19)	Al(1)	C(2)	1.9640(19)
O(1)	C(3)	1.3208(17)	N(1)	C(9)	1.2935(18)
N(1)	C(10)	1.5142(18)	C(3)	C(4)	1.417(2)
C(3)	C(8)	1.4133(18)	C(4)	C(5)	1.377(2)
C(4)	C(14)	1.516(2)	C(5)	C(6)	1.397(2)
C(6)	C(7)	1.369(2)	C(7)	C(8)	1.407(2)
C(8)	C(9)	1.4406(19)	C(10)	C(11)	1.516(2)
C(10)	C(12)	1.509(2)	C(10)	C(13)	1.526(2)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(5)	H(7)	1.000	C(6)	H(8)	1.014
C(7)	H(9)	0.949	C(9)	H(10)	0.985
C(11)	H(11)	0.950	C(11)	H(12)	0.950
C(11)	H(13)	0.950	C(12)	H(14)	0.950
C(12)	H(15)	0.950	C(12)	H(16)	0.950
C(13)	H(17)	0.950	C(13)	H(18)	0.950
C(13)	H(19)	0.950	C(14)	H(20)	0.950
C(14)	H(21)	0.950	C(14)	H(22)	0.950

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	95.66(5)	O(1)	Al(1)	C(1)	109.16(6)
O(1)	Al(1)	C(2)	110.79(7)	N(1)	Al(1)	C(1)	112.53(6)
N(1)	Al(1)	C(2)	109.76(6)	C(1)	Al(1)	C(2)	116.91(8)
Al(1)	O(1)	C(3)	131.32(9)	Al(1)	N(1)	C(9)	120.53(9)
Al(1)	N(1)	C(10)	122.50(9)	C(9)	N(1)	C(10)	116.93(12)
O(1)	C(3)	C(4)	119.20(12)	O(1)	C(3)	C(8)	121.59(12)
C(4)	C(3)	C(8)	119.21(12)	C(3)	C(4)	C(5)	118.94(14)
C(3)	C(4)	C(14)	118.53(13)	C(5)	C(4)	C(14)	122.53(14)
C(4)	C(5)	C(6)	122.36(14)	C(5)	C(6)	C(7)	118.94(15)
C(6)	C(7)	C(8)	121.11(14)	C(3)	C(8)	C(7)	119.45(12)
C(3)	C(8)	C(9)	122.80(12)	C(7)	C(8)	C(9)	117.71(12)
N(1)	C(9)	C(8)	128.01(13)	N(1)	C(10)	C(11)	108.69(13)
N(1)	C(10)	C(12)	110.71(13)	N(1)	C(10)	C(13)	108.77(13)
C(11)	C(10)	C(12)	111.70(16)	C(11)	C(10)	C(13)	107.83(15)
C(12)	C(10)	C(13)	109.05(14)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.2	Al(1)	C(1)	H(2)	109.8
Al(1)	C(1)	H(3)	109.4	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.2	Al(1)	C(2)	H(5)	109.4
Al(1)	C(2)	H(6)	109.8	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
C(4)	C(5)	H(7)	119.1	C(6)	C(5)	H(7)	118.5
C(5)	C(6)	H(8)	118.0	C(7)	C(6)	H(8)	122.5
C(6)	C(7)	H(9)	120.3	C(8)	C(7)	H(9)	118.6
N(1)	C(9)	H(10)	117.2	C(8)	C(9)	H(10)	114.7
C(10)	C(11)	H(11)	109.8	C(10)	C(11)	H(12)	109.9
C(10)	C(11)	H(13)	108.8	H(11)	C(11)	H(12)	109.5
H(11)	C(11)	H(13)	109.5	H(12)	C(11)	H(13)	109.5
C(10)	C(12)	H(14)	107.7	C(10)	C(12)	H(15)	110.3
C(10)	C(12)	H(16)	110.5	H(14)	C(12)	H(15)	109.5
H(14)	C(12)	H(16)	109.5	H(15)	C(12)	H(16)	109.5
C(10)	C(13)	H(17)	109.8	C(10)	C(13)	H(18)	109.7
C(10)	C(13)	H(19)	109.0	H(17)	C(13)	H(18)	109.5
H(17)	C(13)	H(19)	109.5	H(18)	C(13)	H(19)	109.5
C(4)	C(14)	H(20)	109.2	C(4)	C(14)	H(21)	110.1
C(4)	C(14)	H(22)	109.2	H(20)	C(14)	H(21)	109.5
H(20)	C(14)	H(22)	109.5	H(21)	C(14)	H(22)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(9)	-1.81(11)	O(1)	Al(1)	N(1)	C(10)	-179.32(10)
N(1)	Al(1)	O(1)	C(3)	3.48(13)	C(1)	Al(1)	O(1)	C(3)	119.71(13)
C(2)	Al(1)	O(1)	C(3)	-110.17(13)	C(1)	Al(1)	N(1)	C(9)	-115.27(12)
C(1)	Al(1)	N(1)	C(10)	67.22(12)	C(2)	Al(1)	N(1)	C(9)	112.68(12)
C(2)	Al(1)	N(1)	C(10)	-64.83(12)	Al(1)	O(1)	C(3)	C(4)	175.41(10)
Al(1)	O(1)	C(3)	C(8)	-3.7(2)	Al(1)	N(1)	C(9)	C(8)	0.7(2)
Al(1)	N(1)	C(10)	C(11)	109.46(14)	Al(1)	N(1)	C(10)	C(12)	-127.50(13)
Al(1)	N(1)	C(10)	C(13)	-7.70(16)	C(9)	N(1)	C(10)	C(11)	-68.14(18)
C(9)	N(1)	C(10)	C(12)	54.91(17)	C(9)	N(1)	C(10)	C(13)	174.71(14)
C(10)	N(1)	C(9)	C(8)	178.35(13)	O(1)	C(3)	C(4)	C(5)	-179.18(12)
O(1)	C(3)	C(4)	C(14)	-0.03(15)	O(1)	C(3)	C(8)	C(7)	179.30(13)
O(1)	C(3)	C(8)	C(9)	1.6(2)	C(4)	C(3)	C(8)	C(7)	0.14(19)
C(4)	C(3)	C(8)	C(9)	-177.60(12)	C(8)	C(3)	C(4)	C(5)	-0.01(17)
C(8)	C(3)	C(4)	C(14)	179.15(13)	C(3)	C(4)	C(5)	C(6)	-0.2(2)
C(14)	C(4)	C(5)	C(6)	-179.31(14)	C(4)	C(5)	C(6)	C(7)	0.2(2)
C(5)	C(6)	C(7)	C(8)	-0.1(2)	C(6)	C(7)	C(8)	C(3)	-0.1(2)
C(6)	C(7)	C(8)	C(9)	177.77(14)	C(3)	C(8)	C(9)	N(1)	-0.2(2)
C(7)	C(8)	C(9)	N(1)	-177.99(14)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
C(3)	C(11)[1)]	3.571(2)	C(11)	C(3)[2)]	3.571(2)

Symmetry Operators:

(1) -X,Y+1/2,-Z+1/2 (2) -X,Y+1/2-1,-Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(8)[1)]	3.595	O(1)	H(11)[2)]	3.024
O(1)	H(16)[3)]	3.330	O(1)	H(22)[4)]	3.210
N(1)	H(8)[1)]	3.484	N(1)	H(17)[2)]	3.233
C(1)	H(9)[3)]	3.255	C(1)	H(10)[3)]	3.392
C(1)	H(20)[5)]	3.518	C(1)	H(20)[4)]	3.316
C(2)	H(11)[2)]	3.343	C(2)	H(13)[3)]	3.172
C(2)	H(15)[2)]	3.263	C(2)	H(17)[2)]	3.265
C(2)	H(21)[6)]	3.143	C(3)	H(8)[1)]	3.453
C(3)	H(11)[2)]	3.023	C(3)	H(12)[2)]	3.226
C(3)	H(14)[7)]	3.514	C(3)	H(22)[4)]	3.509
C(4)	H(11)[2)]	3.397	C(4)	H(12)[2)]	3.272
C(4)	H(14)[7)]	3.295	C(4)	H(16)[7)]	3.477
C(4)	H(22)[4)]	3.579	C(5)	H(10)[7)]	3.444
C(5)	H(12)[2)]	3.457	C(5)	H(14)[7)]	3.066
C(5)	H(16)[7)]	3.320	C(5)	H(18)[8)]	3.106
C(6)	H(3)[7)]	3.388	C(6)	H(14)[8)]	3.497
C(6)	H(14)[7)]	3.081	C(6)	H(15)[8)]	3.489
C(6)	H(18)[8)]	3.595	C(7)	H(3)[7)]	3.297
C(7)	H(5)[2)]	3.525	C(7)	H(7)[1)]	3.290
C(7)	H(12)[2)]	3.575	C(7)	H(14)[7)]	3.301
C(8)	H(7)[1)]	3.359	C(8)	H(8)[1)]	3.259
C(8)	H(11)[2)]	3.536	C(8)	H(12)[2)]	3.383
C(8)	H(14)[7)]	3.517	C(9)	H(1)[9)]	3.584
C(9)	H(7)[1)]	3.259	C(9)	H(8)[1)]	3.279
C(9)	H(17)[2)]	3.270	C(9)	H(19)[2)]	3.577
C(11)	H(4)[9)]	3.230	C(11)	H(17)[2)]	3.114
C(12)	H(8)[5)]	3.421	C(12)	H(21)[9)]	3.315
C(12)	H(22)[9)]	3.447	C(13)	H(5)[10)]	3.433
C(13)	H(12)[10)]	3.133	C(13)	H(13)[10)]	3.421
C(14)	H(1)[4)]	3.375	C(14)	H(4)[6)]	3.556
C(14)	H(8)[11)]	3.526	C(14)	H(15)[3)]	3.192
C(14)	H(22)[4)]	3.355	H(1)	C(9)[3)]	3.584
H(1)	C(14)[4)]	3.375	H(1)	H(3)[12)]	3.565
H(1)	H(7)[4)]	3.166	H(1)	H(9)[3)]	2.772
H(1)	H(10)[3)]	2.639	H(1)	H(16)[3)]	2.895
H(1)	H(20)[4)]	2.756	H(1)	H(22)[4)]	3.241
H(2)	H(6)[13)]	3.278	H(2)	H(7)[5)]	3.434

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(2)	H(9)[3)]	2.900	H(2)	H(10)[3)]	3.408
H(2)	H(20)[5)]	2.905	H(3)	C(6)[1)]	3.388
H(3)	C(7)[1)]	3.297	H(3)	H(1)[12)]	3.565
H(3)	H(7)[5)]	3.034	H(3)	H(8)[1)]	2.972
H(3)	H(9)[1)]	2.853	H(3)	H(20)[5)]	3.338
H(3)	H(20)[4)]	3.032	H(4)	C(11)[3)]	3.230
H(4)	C(14)[6)]	3.556	H(4)	H(4)[6)]	3.589
H(4)	H(11)[2)]	2.853	H(4)	H(11)[3)]	2.865
H(4)	H(13)[3)]	2.722	H(4)	H(15)[2)]	2.886
H(4)	H(17)[2)]	3.397	H(4)	H(21)[6)]	2.633
H(5)	C(7)[10)]	3.525	H(5)	C(13)[2)]	3.433
H(5)	H(9)[10)]	3.485	H(5)	H(11)[2)]	3.256
H(5)	H(15)[2)]	2.763	H(5)	H(17)[2)]	2.571
H(5)	H(21)[6)]	3.153	H(6)	H(2)[13)]	3.278
H(6)	H(6)[13)]	3.556	H(6)	H(13)[3)]	2.889
H(6)	H(21)[6)]	3.146	H(7)	C(7)[7)]	3.290
H(7)	C(8)[7)]	3.359	H(7)	C(9)[7)]	3.259
H(7)	H(1)[4)]	3.166	H(7)	H(2)[8)]	3.434
H(7)	H(3)[8)]	3.034	H(7)	H(9)[7)]	3.061
H(7)	H(10)[7)]	2.977	H(7)	H(14)[8)]	3.171
H(7)	H(14)[7)]	3.487	H(7)	H(16)[7)]	3.359
H(7)	H(18)[8)]	2.765	H(8)	O(1)[7)]	3.595
H(8)	N(1)[7)]	3.484	H(8)	C(3)[7)]	3.453
H(8)	C(8)[7)]	3.259	H(8)	C(9)[7)]	3.279
H(8)	C(12)[8)]	3.421	H(8)	C(14)[14)]	3.526
H(8)	H(3)[7)]	2.972	H(8)	H(14)[8)]	2.935
H(8)	H(14)[7)]	3.399	H(8)	H(15)[8)]	3.017
H(8)	H(20)[14)]	3.288	H(8)	H(21)[14)]	3.512
H(8)	H(22)[14)]	3.208	H(9)	C(1)[9)]	3.255
H(9)	H(1)[9)]	2.772	H(9)	H(2)[9)]	2.900
H(9)	H(3)[7)]	2.853	H(9)	H(5)[2)]	3.485
H(9)	H(7)[1)]	3.061	H(9)	H(20)[14)]	3.273
H(10)	C(1)[9)]	3.392	H(10)	C(5)[1)]	3.444
H(10)	H(1)[9)]	2.639	H(10)	H(2)[9)]	3.408
H(10)	H(7)[1)]	2.977	H(11)	O(1)[10)]	3.024
H(11)	C(2)[10)]	3.343	H(11)	C(3)[10)]	3.023
H(11)	C(4)[10)]	3.397	H(11)	C(8)[10)]	3.536

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(11)	H(4)$^{10)}$	2.853	H(11)	H(4)$^{9)}$	2.865
H(11)	H(5)$^{10)}$	3.256	H(11)	H(21)$^{10)}$	3.204
H(12)	C(3)$^{10)}$	3.226	H(12)	C(4)$^{10)}$	3.272
H(12)	C(5)$^{10)}$	3.457	H(12)	C(7)$^{10)}$	3.575
H(12)	C(8)$^{10)}$	3.383	H(12)	C(13)$^{2)}$	3.133
H(12)	H(17)$^{2)}$	2.477	H(12)	H(18)$^{2)}$	3.072
H(12)	H(19)$^{2)}$	3.426	H(13)	C(2)$^{9)}$	3.172
H(13)	C(13)$^{2)}$	3.421	H(13)	H(4)$^{9)}$	2.722
H(13)	H(6)$^{9)}$	2.889	H(13)	H(17)$^{2)}$	2.991
H(13)	H(18)$^{2)}$	3.453	H(13)	H(19)$^{2)}$	3.274
H(14)	C(3)$^{1)}$	3.514	H(14)	C(4)$^{1)}$	3.295
H(14)	C(5)$^{1)}$	3.066	H(14)	C(6)$^{5)}$	3.497
H(14)	C(6)$^{1)}$	3.081	H(14)	C(7)$^{1)}$	3.301
H(14)	C(8)$^{1)}$	3.517	H(14)	H(7)$^{5)}$	3.171
H(14)	H(7)$^{1)}$	3.487	H(14)	H(8)$^{5)}$	2.935
H(14)	H(8)$^{1)}$	3.399	H(14)	H(22)$^{9)}$	3.529
H(15)	C(2)$^{10)}$	3.263	H(15)	C(6)$^{5)}$	3.489
H(15)	C(14)$^{9)}$	3.192	H(15)	H(4)$^{10)}$	2.886
H(15)	H(5)$^{10)}$	2.763	H(15)	H(8)$^{5)}$	3.017
H(15)	H(21)$^{9)}$	2.558	H(15)	H(22)$^{9)}$	2.992
H(16)	O(1)$^{9)}$	3.330	H(16)	C(4)$^{1)}$	3.477
H(16)	C(5)$^{1)}$	3.320	H(16)	H(1)$^{9)}$	2.895
H(16)	H(7)$^{1)}$	3.359	H(16)	H(21)$^{9)}$	3.299
H(16)	H(22)$^{1)}$	3.474	H(16)	H(22)$^{9)}$	3.279
H(17)	N(1)$^{10)}$	3.233	H(17)	C(2)$^{10)}$	3.265
H(17)	C(9)$^{10)}$	3.270	H(17)	C(11)$^{10)}$	3.114
H(17)	H(4)$^{10)}$	3.397	H(17)	H(5)$^{10)}$	2.571
H(17)	H(12)$^{10)}$	2.477	H(17)	H(13)$^{10)}$	2.991
H(18)	C(5)$^{5)}$	3.106	H(18)	C(6)$^{5)}$	3.595
H(18)	H(7)$^{5)}$	2.765	H(18)	H(12)$^{10)}$	3.072
H(18)	H(13)$^{10)}$	3.453	H(19)	C(9)$^{10)}$	3.577
H(19)	H(12)$^{10)}$	3.426	H(19)	H(13)$^{10)}$	3.274
H(20)	C(1)$^{8)}$	3.518	H(20)	C(1)$^{4)}$	3.316
H(20)	H(1)$^{4)}$	2.756	H(20)	H(2)$^{8)}$	2.905
H(20)	H(3)$^{8)}$	3.338	H(20)	H(3)$^{4)}$	3.032
H(20)	H(8)$^{11)}$	3.288	H(20)	H(9)$^{11)}$	3.273
H(21)	C(2)$^{6)}$	3.143	H(21)	C(12)$^{3)}$	3.315

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(21)	H(4)[6)]	2.633	H(21)	H(5)[6)]	3.153
H(21)	H(6)[6)]	3.146	H(21)	H(8)[11)]	3.512
H(21)	H(11)[2)]	3.204	H(21)	H(15)[3)]	2.558
H(21)	H(16)[3)]	3.299	H(22)	O(1)[4)]	3.210
H(22)	C(3)[4)]	3.509	H(22)	C(4)[4)]	3.579
H(22)	C(12)[3)]	3.447	H(22)	C(14)[4)]	3.355
H(22)	H(1)[4)]	3.241	H(22)	H(8)[11)]	3.208
H(22)	H(14)[3)]	3.529	H(22)	H(15)[3)]	2.992
H(22)	H(16)[7)]	3.474	H(22)	H(16)[3)]	3.279
H(22)	H(22)[4)]	2.522			

Symmetry Operators:

(1) -X+1,Y+1/2-1,-Z+1/2
(2) -X,Y+1/2,-Z+1/2
(3) X,-Y+1/2,Z+1/2
(4) -X+1,-Y+1,-Z+1
(5) X,Y-1,Z
(6) -X,-Y+1,-Z+1
(7) -X+1,Y+1/2,-Z+1/2
(8) X,Y+1,Z
(9) X,-Y+1/2,Z+1/2-1
(10) -X,Y+1/2-1,-Z+1/2
(11) X,-Y+1/2+1,Z+1/2
(12) -X+1,-Y,-Z+1
(13) -X,-Y,-Z+1
(14) X,-Y+1/2+1,Z+1/2-1

X-ray Structure Report for $Me_2Al[O\text{-}2\text{-}^tBu\text{-}6\text{-}\{(cyclohexyl)N{=}CH\}C_6H_3]$ (**2c**)

May 11, 2007

Experimental

Data Collection

A colorless block crystal of $C_{19}H_{30}NOAl$ having approximate dimensions of 0.50 x 0.44 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 150 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 13.4480(3) Å
b = 12.7111(4) Å
c = 22.4540(5) Å
V = 3838.26(17) Å^3

For Z = 8 and F.W. = 315.43, the calculated density is 1.092 g/cm^3. The systematic absences of:

0kl: k ± 2n
h0l: l ± 2n
hk0: h ± 2n

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 5.0^o step, at χ=45.0^o and ϕ = 120.0^o. The exposure rate was 80.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 5.0^o step, at χ=45.0^o and ϕ = 270.0^o. The exposure rate was 80.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 34904 reflections that were collected, 4366 were unique (R_{int} = 0.034); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.080 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.685 to 0.979. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2620 observed reflections and 229 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0401$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1046$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.21 and -0.21 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

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(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{30}NOAl$
Formula Weight	315.43
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.50 X 0.44 X 0.20 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 150.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 13.4480(3) Å b = 12.7111(4) Å c = 22.4540(5) Å V = 3838.26(17) Å^3
Space Group	Pbca (#61)
Z value	8
D_{calc}	1.092 g/cm^3
F_{000}	1376.00
μ(MoKα)	1.080 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=120.0)	130.0 - 190.0^{o}
Exposure Rate	80.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=270.0)	0.0 - 160.0^{o}
Exposure Rate	80.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 34904 Unique: 4366 (R_{int} = 0.034)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.685 - 0.979)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0013Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	2620
No. Variables	229
Reflection/Parameter Ratio	11.44
Residuals: R1 (I>2.00σ(I))	0.0401
Residuals: wR2 (I>2.00σ(I))	0.1046
Goodness of Fit Indicator	1.000
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.21 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.21 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.51135(4)	0.06681(5)	0.68000(2)	3.013(12)
O(1)	0.42123(9)	0.09628(10)	0.62473(5)	3.21(2)
N(1)	0.43087(11)	0.12024(12)	0.74657(6)	2.86(3)
C(1)	0.62723(17)	0.1585(2)	0.67194(11)	5.52(6)
C(2)	0.5363(2)	-0.08452(18)	0.68526(10)	4.91(5)
C(3)	0.35899(13)	0.17786(14)	0.62373(8)	2.81(3)
C(4)	0.32809(12)	0.22423(15)	0.67793(8)	2.94(3)
C(5)	0.36193(13)	0.18827(15)	0.73568(8)	3.00(4)
C(6)	0.32187(13)	0.21705(15)	0.56883(8)	3.00(3)
C(7)	0.25419(15)	0.29983(17)	0.57184(9)	3.71(4)
C(8)	0.22300(15)	0.34574(18)	0.62495(10)	4.18(5)
C(9)	0.25992(15)	0.30826(17)	0.67788(9)	3.67(4)
C(10)	0.35724(15)	0.17263(16)	0.50874(8)	3.42(4)
C(11)	0.47018(16)	0.1868(2)	0.50272(9)	4.68(5)
C(12)	0.32965(18)	0.05572(18)	0.50381(9)	4.44(5)
C(13)	0.30774(19)	0.2294(2)	0.45621(9)	5.07(5)
C(14)	0.44822(15)	0.09490(16)	0.81057(8)	3.27(4)
C(15)	0.39898(16)	-0.00859(18)	0.82712(9)	4.23(5)
C(16)	0.41749(19)	-0.0340(2)	0.89284(10)	5.29(6)
C(17)	0.52682(19)	-0.0348(2)	0.90749(10)	5.30(6)
C(18)	0.57584(19)	0.0668(2)	0.89002(10)	5.35(6)
C(19)	0.55867(15)	0.09242(18)	0.82464(9)	4.12(5)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.6586	0.1458	0.6347	6.66
H(2)	0.6065	0.2298	0.6739	6.68
H(3)	0.6728	0.1445	0.7033	6.68
H(4)	0.5219	-0.1168	0.6481	5.96
H(5)	0.6040	-0.0967	0.6952	5.99
H(6)	0.4948	-0.1137	0.7152	5.97
H(7)	0.3268	0.2224	0.7708	3.61
H(8)	0.2289	0.3298	0.5315	4.44
H(9)	0.1726	0.4068	0.6257	5.01
H(10)	0.2379	0.3439	0.7191	4.42
H(11)	0.4902	0.1664	0.4638	5.60
H(12)	0.4876	0.2583	0.5093	5.62
H(13)	0.5027	0.1437	0.5313	5.62
H(14)	0.3482	0.0305	0.4656	5.35
H(15)	0.3640	0.0170	0.5336	5.32
H(16)	0.2600	0.0473	0.5091	5.34
H(17)	0.3296	0.3004	0.4552	6.09
H(18)	0.3255	0.1955	0.4200	6.10
H(19)	0.2376	0.2275	0.4609	6.08
H(20)	0.4152	0.1525	0.8347	3.91
H(21)	0.4279	-0.0628	0.8037	5.08
H(22)	0.3298	-0.0045	0.8189	5.08
H(23)	0.3860	0.0188	0.9161	6.34
H(24)	0.3894	-0.1006	0.9020	6.36
H(25)	0.5573	-0.0905	0.8860	6.34
H(26)	0.5354	-0.0462	0.9490	6.39
H(27)	0.6454	0.0617	0.8969	6.42
H(28)	0.5490	0.1219	0.9137	6.39
H(29)	0.5890	0.0397	0.8008	4.93
H(30)	0.5868	0.1590	0.8153	4.97

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0386(3)	0.0360(3)	0.0399(3)	0.0062(2)	-0.0016(2)	0.0010(2)
O(1)	0.0453(7)	0.0348(7)	0.0418(6)	0.0098(6)	-0.0040(5)	-0.0016(5)
N(1)	0.0372(8)	0.0310(8)	0.0403(7)	-0.0013(7)	-0.0009(6)	0.0009(7)
C(1)	0.0532(13)	0.088(2)	0.0685(15)	-0.0116(13)	0.0035(11)	0.0098(13)
C(2)	0.0811(16)	0.0519(15)	0.0534(12)	0.0258(12)	-0.0084(11)	-0.0020(10)
C(3)	0.0327(9)	0.0263(9)	0.0477(10)	-0.0013(7)	-0.0019(7)	0.0032(7)
C(4)	0.0337(8)	0.0318(10)	0.0463(10)	0.0002(7)	0.0016(8)	0.0025(8)
C(5)	0.0370(9)	0.0305(10)	0.0465(10)	-0.0033(8)	0.0049(7)	-0.0013(7)
C(6)	0.0374(9)	0.0293(10)	0.0473(10)	-0.0037(8)	-0.0041(7)	0.0054(8)
C(7)	0.0463(10)	0.0383(12)	0.0563(12)	0.0047(9)	-0.0046(9)	0.0111(9)
C(8)	0.0479(12)	0.0434(13)	0.0673(14)	0.0149(10)	0.0017(9)	0.0079(11)
C(9)	0.0434(10)	0.0392(12)	0.0570(12)	0.0073(9)	0.0051(9)	0.0014(9)
C(10)	0.0513(11)	0.0364(11)	0.0422(10)	0.0015(9)	-0.0028(8)	0.0070(8)
C(11)	0.0612(14)	0.0632(16)	0.0532(12)	-0.0003(12)	0.0083(10)	0.0044(10)
C(12)	0.0777(15)	0.0416(13)	0.0492(12)	-0.0027(11)	-0.0061(10)	-0.0019(9)
C(13)	0.0846(16)	0.0591(15)	0.0491(12)	0.0100(14)	-0.0059(11)	0.0120(11)
C(14)	0.0512(11)	0.0359(11)	0.0372(9)	0.0051(8)	0.0034(8)	0.0010(7)
C(15)	0.0527(12)	0.0503(14)	0.0579(13)	-0.0038(10)	0.0083(9)	0.0102(10)
C(16)	0.0882(17)	0.0545(15)	0.0584(13)	0.0053(13)	0.0226(12)	0.0137(11)
C(17)	0.0933(18)	0.0641(17)	0.0442(12)	0.0234(14)	0.0003(11)	0.0093(10)
C(18)	0.0808(17)	0.0698(17)	0.0525(12)	0.0037(14)	-0.0182(11)	-0.0006(12)
C(19)	0.0547(12)	0.0533(15)	0.0486(11)	-0.0096(10)	-0.0101(9)	0.0048(10)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.7746(12)	Al(1)	N(1)	1.9664(14)
Al(1)	C(1)	1.954(2)	Al(1)	C(2)	1.956(2)
O(1)	C(3)	1.333(2)	N(1)	C(5)	1.291(2)
N(1)	C(14)	1.491(2)	C(3)	C(4)	1.415(2)
C(3)	C(6)	1.420(2)	C(4)	C(5)	1.448(2)
C(4)	C(9)	1.408(2)	C(6)	C(7)	1.393(2)
C(6)	C(10)	1.538(2)	C(7)	C(8)	1.392(3)
C(8)	C(9)	1.373(3)	C(10)	C(11)	1.535(2)
C(10)	C(12)	1.536(3)	C(10)	C(13)	1.534(2)
C(14)	C(15)	1.519(3)	C(14)	C(19)	1.519(2)
C(15)	C(16)	1.531(3)	C(16)	C(17)	1.507(3)
C(17)	C(18)	1.502(3)	C(18)	C(19)	1.521(3)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(5)	H(7)	1.016	C(7)	H(8)	1.040
C(8)	H(9)	1.030	C(9)	H(10)	1.072
C(11)	H(11)	0.950	C(11)	H(12)	0.950
C(11)	H(13)	0.950	C(12)	H(14)	0.950
C(12)	H(15)	0.950	C(12)	H(16)	0.950
C(13)	H(17)	0.950	C(13)	H(18)	0.950
C(13)	H(19)	0.950	C(14)	H(20)	1.014
C(15)	H(21)	0.950	C(15)	H(22)	0.950
C(16)	H(23)	0.950	C(16)	H(24)	0.950
C(17)	H(25)	0.950	C(17)	H(26)	0.950
C(18)	H(27)	0.950	C(18)	H(28)	0.950
C(19)	H(29)	0.950	C(19)	H(30)	0.950

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	94.75(6)	O(1)	Al(1)	C(1)	110.73(8)
O(1)	Al(1)	C(2)	111.54(8)	N(1)	Al(1)	C(1)	107.66(9)
N(1)	Al(1)	C(2)	112.85(8)	C(1)	Al(1)	C(2)	117.05(11)
Al(1)	O(1)	C(3)	127.22(11)	Al(1)	N(1)	C(5)	118.85(11)
Al(1)	N(1)	C(14)	124.88(11)	C(5)	N(1)	C(14)	116.08(14)
O(1)	C(3)	C(4)	119.61(15)	O(1)	C(3)	C(6)	120.55(15)
C(4)	C(3)	C(6)	119.82(16)	C(3)	C(4)	C(5)	123.12(16)
C(3)	C(4)	C(9)	120.45(16)	C(5)	C(4)	C(9)	116.41(16)
N(1)	C(5)	C(4)	127.34(16)	C(3)	C(6)	C(7)	116.91(16)
C(3)	C(6)	C(10)	121.60(16)	C(7)	C(6)	C(10)	121.46(16)
C(6)	C(7)	C(8)	123.71(18)	C(7)	C(8)	C(9)	119.14(19)
C(4)	C(9)	C(8)	119.96(18)	C(6)	C(10)	C(11)	109.89(15)
C(6)	C(10)	C(12)	110.11(15)	C(6)	C(10)	C(13)	111.57(16)
C(11)	C(10)	C(12)	110.24(18)	C(11)	C(10)	C(13)	107.84(17)
C(12)	C(10)	C(13)	107.13(17)	N(1)	C(14)	C(15)	110.77(15)
N(1)	C(14)	C(19)	110.98(15)	C(15)	C(14)	C(19)	110.94(17)
C(14)	C(15)	C(16)	110.34(17)	C(15)	C(16)	C(17)	111.75(19)
C(16)	C(17)	C(18)	111.4(2)	C(17)	C(18)	C(19)	111.67(19)
C(14)	C(19)	C(18)	110.71(17)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.6	Al(1)	C(1)	H(2)	109.3
Al(1)	C(1)	H(3)	109.5	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.6	Al(1)	C(2)	H(5)	109.8
Al(1)	C(2)	H(6)	109.0	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
N(1)	C(5)	H(7)	118.2	C(4)	C(5)	H(7)	114.4
C(6)	C(7)	H(8)	116.6	C(8)	C(7)	H(8)	119.6
C(7)	C(8)	H(9)	121.8	C(9)	C(8)	H(9)	119.0
C(4)	C(9)	H(10)	120.0	C(8)	C(9)	H(10)	120.1
C(10)	C(11)	H(11)	109.2	C(10)	C(11)	H(12)	110.0
C(10)	C(11)	H(13)	109.2	H(11)	C(11)	H(12)	109.5
H(11)	C(11)	H(13)	109.5	H(12)	C(11)	H(13)	109.5
C(10)	C(12)	H(14)	109.1	C(10)	C(12)	H(15)	109.5
C(10)	C(12)	H(16)	109.8	H(14)	C(12)	H(15)	109.5
H(14)	C(12)	H(16)	109.5	H(15)	C(12)	H(16)	109.5
C(10)	C(13)	H(17)	109.3	C(10)	C(13)	H(18)	109.6
C(10)	C(13)	H(19)	109.5	H(17)	C(13)	H(18)	109.5
H(17)	C(13)	H(19)	109.5	H(18)	C(13)	H(19)	109.5
N(1)	C(14)	H(20)	106.9	C(15)	C(14)	H(20)	107.7
C(19)	C(14)	H(20)	109.4	C(14)	C(15)	H(21)	108.3
C(14)	C(15)	H(22)	109.4	C(16)	C(15)	H(21)	108.3
C(16)	C(15)	H(22)	110.9	H(21)	C(15)	H(22)	109.5
C(15)	C(16)	H(23)	108.0	C(15)	C(16)	H(24)	109.4
C(17)	C(16)	H(23)	108.6	C(17)	C(16)	H(24)	109.6
H(23)	C(16)	H(24)	109.5	C(16)	C(17)	H(25)	108.3
C(16)	C(17)	H(26)	109.5	C(18)	C(17)	H(25)	108.6
C(18)	C(17)	H(26)	109.5	H(25)	C(17)	H(26)	109.5
C(17)	C(18)	H(27)	109.3	C(17)	C(18)	H(28)	108.7
C(19)	C(18)	H(27)	108.8	C(19)	C(18)	H(28)	109.0
H(27)	C(18)	H(28)	109.5	C(14)	C(19)	H(29)	108.5
C(14)	C(19)	H(30)	109.0	C(18)	C(19)	H(29)	109.1
C(18)	C(19)	H(30)	110.0	H(29)	C(19)	H(30)	109.5

Table 8. Torsion Angles(O)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(5)	-22.89(14)	O(1)	Al(1)	N(1)	C(14)	162.36(14)
N(1)	Al(1)	O(1)	C(3)	34.80(14)	C(1)	Al(1)	O(1)	C(3)	-76.11(16)
C(2)	Al(1)	O(1)	C(3)	151.65(14)	C(1)	Al(1)	N(1)	C(5)	90.64(15)
C(1)	Al(1)	N(1)	C(14)	-84.11(16)	C(2)	Al(1)	N(1)	C(5)	-138.66(15)
C(2)	Al(1)	N(1)	C(14)	46.59(17)	Al(1)	O(1)	C(3)	C(4)	-27.9(2)
Al(1)	O(1)	C(3)	C(6)	153.49(13)	Al(1)	N(1)	C(5)	C(4)	6.9(2)
Al(1)	N(1)	C(14)	C(15)	-84.31(18)	Al(1)	N(1)	C(14)	C(19)	39.4(2)
C(5)	N(1)	C(14)	C(15)	100.81(19)	C(5)	N(1)	C(14)	C(19)	-135.49(17)
C(14)	N(1)	C(5)	C(4)	-177.92(17)	O(1)	C(3)	C(4)	C(5)	0.9(2)
O(1)	C(3)	C(4)	C(9)	-177.95(16)	O(1)	C(3)	C(6)	C(7)	177.56(16)
O(1)	C(3)	C(6)	C(10)	-4.4(2)	C(4)	C(3)	C(6)	C(7)	-1.1(2)
C(4)	C(3)	C(6)	C(10)	177.00(16)	C(6)	C(3)	C(4)	C(5)	179.50(16)
C(6)	C(3)	C(4)	C(9)	0.7(2)	C(3)	C(4)	C(5)	N(1)	8.5(2)
C(3)	C(4)	C(9)	C(8)	-0.0(2)	C(5)	C(4)	C(9)	C(8)	-178.90(18)
C(9)	C(4)	C(5)	N(1)	-172.65(18)	C(3)	C(6)	C(7)	C(8)	0.9(2)
C(3)	C(6)	C(10)	C(11)	-59.3(2)	C(3)	C(6)	C(10)	C(12)	62.3(2)
C(3)	C(6)	C(10)	C(13)	-178.91(17)	C(7)	C(6)	C(10)	C(11)	118.7(2)
C(7)	C(6)	C(10)	C(12)	-119.7(2)	C(7)	C(6)	C(10)	C(13)	-0.9(2)
C(10)	C(6)	C(7)	C(8)	-177.23(19)	C(6)	C(7)	C(8)	C(9)	-0.2(3)
C(7)	C(8)	C(9)	C(4)	-0.2(3)	N(1)	C(14)	C(15)	C(16)	179.84(16)
N(1)	C(14)	C(19)	C(18)	179.84(17)	C(15)	C(14)	C(19)	C(18)	-56.6(2)
C(19)	C(14)	C(15)	C(16)	56.1(2)	C(14)	C(15)	C(16)	C(17)	-55.4(2)
C(15)	C(16)	C(17)	C(18)	54.9(2)	C(16)	C(17)	C(18)	C(19)	-55.1(2)
C(17)	C(18)	C(19)	C(14)	55.9(2)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Al(1)	C(3)	2.7905(18)	Al(1)	C(4)	3.1749(18)
Al(1)	C(5)	2.8257(18)	Al(1)	C(14)	3.0731(18)
Al(1)	C(19)	3.325(2)	O(1)	N(1)	2.7557(17)
O(1)	C(1)	3.070(2)	O(1)	C(2)	3.086(2)
O(1)	C(4)	2.375(2)	O(1)	C(5)	2.865(2)
O(1)	C(6)	2.391(2)	O(1)	C(10)	2.910(2)
O(1)	C(11)	3.043(2)	O(1)	C(12)	3.026(2)
N(1)	O(1)	2.7557(17)	N(1)	C(1)	3.165(2)
N(1)	C(2)	3.268(2)	N(1)	C(3)	3.013(2)
N(1)	C(4)	2.456(2)	N(1)	C(15)	2.477(2)
N(1)	C(19)	2.480(2)	C(1)	O(1)	3.070(2)
C(1)	N(1)	3.165(2)	C(1)	C(2)	3.335(3)
C(2)	O(1)	3.086(2)	C(2)	N(1)	3.268(2)
C(2)	C(1)	3.335(3)	C(3)	Al(1)	2.7905(18)
C(3)	N(1)	3.013(2)	C(3)	C(5)	2.518(2)
C(3)	C(7)	2.397(2)	C(3)	C(8)	2.811(2)
C(3)	C(9)	2.450(2)	C(3)	C(10)	2.583(2)
C(3)	C(11)	3.103(2)	C(3)	C(12)	3.133(2)
C(4)	Al(1)	3.1749(18)	C(4)	O(1)	2.375(2)
C(4)	N(1)	2.456(2)	C(4)	C(6)	2.453(2)
C(4)	C(7)	2.754(2)	C(4)	C(8)	2.408(2)
C(5)	Al(1)	2.8257(18)	C(5)	O(1)	2.865(2)
C(5)	C(3)	2.518(2)	C(5)	C(9)	2.427(2)
C(5)	C(14)	2.363(2)	C(5)	C(15)	3.275(2)
C(5)	C(19)	3.532(2)	C(6)	O(1)	2.391(2)
C(6)	C(4)	2.453(2)	C(6)	C(8)	2.456(2)
C(6)	C(9)	2.834(2)	C(6)	C(11)	2.516(2)
C(6)	C(12)	2.519(2)	C(6)	C(13)	2.541(2)
C(7)	C(3)	2.397(2)	C(7)	C(4)	2.754(2)
C(7)	C(9)	2.385(2)	C(7)	C(10)	2.558(2)
C(7)	C(11)	3.593(3)	C(7)	C(13)	2.839(2)
C(8)	C(3)	2.811(2)	C(8)	C(4)	2.408(2)
C(8)	C(6)	2.456(2)	C(9)	C(3)	2.450(2)
C(9)	C(5)	2.427(2)	C(9)	C(6)	2.834(2)
C(9)	C(7)	2.385(2)	C(10)	O(1)	2.910(2)
C(10)	C(3)	2.583(2)	C(10)	C(7)	2.558(2)
C(11)	O(1)	3.043(2)	C(11)	C(3)	3.103(2)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(11)	C(6)	2.516(2)	C(11)	C(7)	3.593(3)
C(11)	C(12)	2.519(3)	C(11)	C(13)	2.481(3)
C(12)	O(1)	3.026(2)	C(12)	C(3)	3.133(2)
C(12)	C(6)	2.519(2)	C(12)	C(11)	2.519(3)
C(12)	C(13)	2.470(3)	C(13)	C(6)	2.541(2)
C(13)	C(7)	2.839(2)	C(13)	C(11)	2.481(3)
C(13)	C(12)	2.470(3)	C(14)	Al(1)	3.0731(18)
C(14)	C(5)	2.363(2)	C(14)	C(16)	2.503(3)
C(14)	C(17)	2.928(3)	C(14)	C(18)	2.501(3)
C(15)	N(1)	2.477(2)	C(15)	C(5)	3.275(2)
C(15)	C(17)	2.515(3)	C(15)	C(18)	2.928(3)
C(15)	C(19)	2.503(3)	C(16)	C(14)	2.503(3)
C(16)	C(18)	2.486(3)	C(16)	C(19)	2.921(3)
C(17)	C(14)	2.928(3)	C(17)	C(15)	2.515(3)
C(17)	C(19)	2.502(3)	C(18)	C(14)	2.501(3)
C(18)	C(15)	2.928(3)	C(18)	C(16)	2.486(3)
C(19)	Al(1)	3.325(2)	C(19)	N(1)	2.480(2)
C(19)	C(5)	3.532(2)	C(19)	C(15)	2.503(3)
C(19)	C(16)	2.921(3)	C(19)	C(17)	2.502(3)

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Al(1)	H(1)	2.442	Al(1)	H(2)	2.439
Al(1)	H(3)	2.441	Al(1)	H(4)	2.445
Al(1)	H(5)	2.447	Al(1)	H(6)	2.437
Al(1)	H(9)[1)]	3.428	Al(1)	H(13)	3.481
Al(1)	H(21)	3.419	Al(1)	H(29)	2.927
Al(1)	H(30)	3.411	O(1)	H(1)	3.262
O(1)	H(2)	3.211	O(1)	H(4)	3.072
O(1)	H(6)	3.497	O(1)	H(9)[1)]	2.720
O(1)	H(12)	3.429	O(1)	H(13)	2.442
O(1)	H(15)	2.408	O(1)	H(16)	3.440
N(1)	H(2)	3.191	N(1)	H(3)	3.409
N(1)	H(6)	3.174	N(1)	H(7)	1.985
N(1)	H(20)	2.033	N(1)	H(21)	2.657
N(1)	H(22)	2.646	N(1)	H(29)	2.656
N(1)	H(30)	2.650	C(1)	H(5)	3.300
C(1)	H(7)[2)]	3.085	C(1)	H(13)	3.580
C(1)	H(22)[2)]	3.428	C(1)	H(24)[3)]	3.490
C(1)	H(29)	3.304	C(1)	H(30)	3.265
C(2)	H(1)	3.544	C(2)	H(3)	3.464
C(2)	H(7)[4)]	3.222	C(2)	H(9)[1)]	3.115
C(2)	H(11)[5)]	3.524	C(2)	H(18)[5)]	3.321
C(2)	H(20)[4)]	3.435	C(2)	H(21)	3.046
C(2)	H(29)	3.118	C(3)	H(2)	3.576
C(3)	H(7)	3.378	C(3)	H(8)	3.329
C(3)	H(9)[1)]	3.472	C(3)	H(10)	3.419
C(3)	H(12)	3.262	C(3)	H(13)	2.869
C(3)	H(15)	2.879	C(3)	H(16)	3.340
C(3)	H(25)[3)]	3.160	C(3)	H(27)[6)]	3.263
C(4)	H(3)[6)]	3.536	C(4)	H(7)	2.085
C(4)	H(9)	3.336	C(4)	H(10)	2.154
C(4)	H(25)[3)]	3.159	C(4)	H(30)[6)]	3.352
C(5)	H(3)[6)]	2.943	C(5)	H(5)[3)]	3.176
C(5)	H(6)[3)]	3.356	C(5)	H(10)	2.614
C(5)	H(20)	2.381	C(5)	H(22)	3.112
C(5)	H(30)	3.533	C(6)	H(8)	2.079
C(6)	H(9)	3.387	C(6)	H(11)	3.332
C(6)	H(12)	2.651	C(6)	H(13)	2.737

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(6)	H(14)	3.335	C(6)	H(15)	2.724
C(6)	H(16)	2.674	C(6)	H(17)	2.764
C(6)	H(18)	3.353	C(6)	H(19)	2.678
C(6)	H(25)[3)]	3.108	C(6)	H(26)[3)]	3.592
C(6)	H(27)[6)]	3.182	C(7)	H(9)	2.124
C(7)	H(10)	3.361	C(7)	H(12)	3.479
C(7)	H(15)[7)]	3.299	C(7)	H(16)	3.507
C(7)	H(16)[7)]	3.452	C(7)	H(17)	2.808
C(7)	H(19)	2.664	C(7)	H(25)[3)]	3.045
C(7)	H(26)[3)]	3.472	C(7)	H(27)[6)]	3.435
C(7)	H(28)[6)]	3.582	C(8)	H(4)[7)]	3.368
C(8)	H(6)[7)]	3.599	C(8)	H(8)	2.110
C(8)	H(10)	2.124	C(8)	H(15)[7)]	3.212
C(8)	H(25)[3)]	3.074	C(8)	H(27)[3)]	3.303
C(8)	H(30)[6)]	3.284	C(9)	H(3)[6)]	3.582
C(9)	H(5)[3)]	3.595	C(9)	H(7)	2.520
C(9)	H(8)	3.325	C(9)	H(9)	2.078
C(9)	H(25)[3)]	3.123	C(9)	H(30)[6)]	3.007
C(10)	H(8)	2.689	C(10)	H(11)	2.055
C(10)	H(12)	2.064	C(10)	H(13)	2.054
C(10)	H(14)	2.054	C(10)	H(15)	2.058
C(10)	H(16)	2.061	C(10)	H(17)	2.055
C(10)	H(18)	2.058	C(10)	H(19)	2.057
C(11)	H(4)[5)]	3.503	C(11)	H(8)[8)]	3.569
C(11)	H(14)	2.708	C(11)	H(15)	2.679
C(11)	H(15)[5)]	3.513	C(11)	H(16)	3.340
C(11)	H(17)	2.607	C(11)	H(18)	2.692
C(11)	H(19)	3.307	C(11)	H(26)[3)]	3.564
C(11)	H(28)[9)]	3.322	C(12)	H(8)[1)]	3.042
C(12)	H(9)[1)]	3.328	C(12)	H(11)	2.729
C(12)	H(12)	3.340	C(12)	H(13)	2.654
C(12)	H(13)[5)]	3.483	C(12)	H(17)	3.296
C(12)	H(18)	2.588	C(12)	H(19)	2.689
C(12)	H(27)[6)]	3.334	C(13)	H(1)[10)]	3.273
C(13)	H(4)[5)]	3.575	C(13)	H(8)	2.369
C(13)	H(11)	2.587	C(13)	H(12)	2.721
C(13)	H(13)	3.302	C(13)	H(14)	2.594

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(13)	H(15)	3.298	C(13)	H(16)	2.679
C(13)	H(20)[9)]	3.432	C(13)	H(23)[9)]	3.488
C(13)	H(24)[11)]	3.345	C(14)	H(6)	3.465
C(14)	H(7)	2.468	C(14)	H(21)	2.029
C(14)	H(22)	2.041	C(14)	H(23)	2.693
C(14)	H(24)	3.319	C(14)	H(25)	3.251
C(14)	H(27)	3.312	C(14)	H(28)	2.705
C(14)	H(29)	2.031	C(14)	H(30)	2.037
C(15)	H(2)[4)]	3.326	C(15)	H(6)	3.124
C(15)	H(7)	3.341	C(15)	H(10)[1)]	3.576
C(15)	H(20)	2.067	C(15)	H(23)	2.036
C(15)	H(24)	2.052	C(15)	H(25)	2.713
C(15)	H(26)	3.329	C(15)	H(28)	3.256
C(15)	H(29)	2.694	C(15)	H(30)	3.315
C(16)	H(2)[4)]	3.371	C(16)	H(16)[12)]	3.541
C(16)	H(17)[13)]	3.489	C(16)	H(19)[12)]	3.569
C(16)	H(20)	2.706	C(16)	H(21)	2.040
C(16)	H(22)	2.070	C(16)	H(25)	2.018
C(16)	H(26)	2.032	C(16)	H(27)	3.298
C(16)	H(28)	2.697	C(16)	H(29)	3.236
C(17)	H(12)[4)]	3.233	C(17)	H(20)	3.254
C(17)	H(21)	2.708	C(17)	H(22)	3.335
C(17)	H(23)	2.022	C(17)	H(24)	2.033
C(17)	H(26)[14)]	3.485	C(17)	H(27)	2.025
C(17)	H(28)	2.019	C(17)	H(29)	2.708
C(17)	H(30)	3.316	C(18)	H(16)[2)]	3.366
C(18)	H(20)	2.719	C(18)	H(21)	3.230
C(18)	H(23)	2.690	C(18)	H(24)	3.300
C(18)	H(25)	2.017	C(18)	H(26)	2.028
C(18)	H(29)	2.041	C(18)	H(30)	2.051
C(19)	H(3)	3.196	C(19)	H(20)	2.087
C(19)	H(21)	2.685	C(19)	H(22)	3.318
C(19)	H(23)	3.238	C(19)	H(25)	2.702
C(19)	H(26)	3.316	C(19)	H(27)	2.036
C(19)	H(28)	2.039	H(1)	Al(1)	2.442
H(1)	O(1)	3.262	H(1)	C(2)	3.544
H(1)	C(13)[8)]	3.273	H(1)	H(2)	1.551

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(1)	H(3)	1.551	H(1)	H(5)	3.447
H(1)	H(7)[2)]	3.251	H(1)	H(13)	3.129
H(1)	H(14)[5)]	3.178	H(1)	H(17)[8)]	3.136
H(1)	H(18)[8)]	3.258	H(1)	H(19)[8)]	2.887
H(1)	H(20)[2)]	3.519	H(1)	H(22)[2)]	3.167
H(1)	H(24)[3)]	3.389	H(2)	Al(1)	2.439
H(2)	O(1)	3.211	H(2)	N(1)	3.191
H(2)	C(3)	3.576	H(2)	C(15)[3)]	3.326
H(2)	C(16)[3)]	3.371	H(2)	H(1)	1.551
H(2)	H(3)	1.551	H(2)	H(6)[3)]	3.465
H(2)	H(7)[2)]	3.214	H(2)	H(10)[2)]	3.315
H(2)	H(19)[8)]	3.545	H(2)	H(21)[3)]	2.723
H(2)	H(22)[3)]	3.488	H(2)	H(24)[3)]	2.748
H(2)	H(25)[3)]	3.447	H(2)	H(30)	3.311
H(3)	Al(1)	2.441	H(3)	N(1)	3.409
H(3)	C(2)	3.464	H(3)	C(4)[2)]	3.536
H(3)	C(5)[2)]	2.943	H(3)	C(9)[2)]	3.582
H(3)	C(19)	3.196	H(3)	H(1)	1.551
H(3)	H(2)	1.551	H(3)	H(5)	3.206
H(3)	H(7)[2)]	2.369	H(3)	H(10)[2)]	3.198
H(3)	H(20)[2)]	3.372	H(3)	H(22)[2)]	2.879
H(3)	H(29)	2.799	H(3)	H(30)	2.775
H(4)	Al(1)	2.445	H(4)	O(1)	3.072
H(4)	C(8)[1)]	3.368	H(4)	C(11)[5)]	3.503
H(4)	C(13)[5)]	3.575	H(4)	H(5)	1.551
H(4)	H(6)	1.551	H(4)	H(7)[4)]	3.412
H(4)	H(9)[1)]	2.680	H(4)	H(11)[5)]	2.596
H(4)	H(14)[5)]	3.281	H(4)	H(18)[5)]	2.749
H(4)	H(20)[4)]	3.076	H(4)	H(30)[4)]	3.307
H(5)	Al(1)	2.447	H(5)	C(1)	3.300
H(5)	C(5)[4)]	3.176	H(5)	C(9)[4)]	3.595
H(5)	H(1)	3.447	H(5)	H(3)	3.206
H(5)	H(4)	1.551	H(5)	H(6)	1.551
H(5)	H(7)[4)]	2.595	H(5)	H(10)[4)]	2.965
H(5)	H(18)[5)]	3.029	H(5)	H(20)[4)]	3.269
H(5)	H(21)	3.425	H(5)	H(22)[2)]	3.270
H(5)	H(29)	2.943	H(6)	Al(1)	2.437

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(6)	O(1)	3.497	H(6)	N(1)	3.174
H(6)	C(5)[4)]	3.356	H(6)	C(8)[1)]	3.599
H(6)	C(14)	3.465	H(6)	C(15)	3.124
H(6)	H(2)[4)]	3.465	H(6)	H(4)	1.551
H(6)	H(5)	1.551	H(6)	H(7)[4)]	3.193
H(6)	H(9)[1)]	3.030	H(6)	H(10)[1)]	3.177
H(6)	H(20)[4)]	3.399	H(6)	H(21)	2.275
H(6)	H(22)	3.503	H(6)	H(29)	3.016
H(6)	H(30)[4)]	3.166	H(7)	N(1)	1.985
H(7)	C(1)[6)]	3.085	H(7)	C(2)[3)]	3.222
H(7)	C(3)	3.378	H(7)	C(4)	2.085
H(7)	C(9)	2.520	H(7)	C(14)	2.468
H(7)	C(15)	3.341	H(7)	H(1)[6)]	3.251
H(7)	H(2)[6)]	3.214	H(7)	H(3)[6)]	2.369
H(7)	H(4)[3)]	3.412	H(7)	H(5)[3)]	2.595
H(7)	H(6)[3)]	3.193	H(7)	H(10)	2.271
H(7)	H(18)[13)]	3.510	H(7)	H(20)	2.066
H(7)	H(22)	3.080	H(8)	C(3)	3.329
H(8)	C(6)	2.079	H(8)	C(8)	2.110
H(8)	C(9)	3.325	H(8)	C(10)	2.689
H(8)	C(11)[10)]	3.569	H(8)	C(12)[7)]	3.042
H(8)	C(13)	2.369	H(8)	H(9)	2.450
H(8)	H(11)[10)]	3.211	H(8)	H(12)[10)]	3.552
H(8)	H(13)[10)]	3.369	H(8)	H(14)[7)]	3.127
H(8)	H(15)[7)]	2.687	H(8)	H(16)[7)]	2.814
H(8)	H(17)	2.216	H(8)	H(18)	3.297
H(8)	H(19)	2.053	H(8)	H(25)[3)]	3.568
H(8)	H(26)[3)]	3.567	H(9)	Al(1)[7)]	3.428
H(9)	O(1)[7)]	2.720	H(9)	C(2)[7)]	3.115
H(9)	C(3)[7)]	3.472	H(9)	C(4)	3.336
H(9)	C(6)	3.387	H(9)	C(7)	2.124
H(9)	C(9)	2.078	H(9)	C(12)[7)]	3.328
H(9)	H(4)[7)]	2.680	H(9)	H(6)[7)]	3.030
H(9)	H(8)	2.450	H(9)	H(10)	2.411
H(9)	H(11)[10)]	3.305	H(9)	H(15)[7)]	2.546
H(9)	H(16)[7)]	3.296	H(9)	H(27)[3)]	3.182
H(10)	C(3)	3.419	H(10)	C(4)	2.154

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(10)	C(5)	2.614	H(10)	C(7)	3.361
H(10)	C(8)	2.124	H(10)	C(15)[7)]	3.576
H(10)	H(2)[6)]	3.315	H(10)	H(3)[6)]	3.198
H(10)	H(5)[3)]	2.965	H(10)	H(6)[7)]	3.177
H(10)	H(7)	2.271	H(10)	H(9)	2.411
H(10)	H(21)[7)]	3.160	H(10)	H(22)[7)]	3.093
H(10)	H(29)[3)]	3.437	H(10)	H(30)[6)]	3.202
H(11)	C(2)[5)]	3.524	H(11)	C(6)	3.332
H(11)	C(10)	2.055	H(11)	C(12)	2.729
H(11)	C(13)	2.587	H(11)	H(4)[5)]	2.596
H(11)	H(8)[8)]	3.211	H(11)	H(9)[8)]	3.305
H(11)	H(12)	1.551	H(11)	H(13)	1.551
H(11)	H(14)	2.575	H(11)	H(15)	2.990
H(11)	H(15)[5)]	3.046	H(11)	H(16)	3.593
H(11)	H(17)	2.758	H(11)	H(18)	2.452
H(11)	H(19)	3.486	H(11)	H(28)[9)]	3.022
H(12)	O(1)	3.429	H(12)	C(3)	3.262
H(12)	C(6)	2.651	H(12)	C(7)	3.479
H(12)	C(10)	2.064	H(12)	C(12)	3.340
H(12)	C(13)	2.721	H(12)	C(17)[3)]	3.233
H(12)	H(8)[8)]	3.552	H(12)	H(11)	1.551
H(12)	H(13)	1.551	H(12)	H(14)	3.586
H(12)	H(15)	3.531	H(12)	H(17)	2.505
H(12)	H(18)	3.067	H(12)	H(19)	3.555
H(12)	H(19)[8)]	3.432	H(12)	H(24)[3)]	3.150
H(12)	H(25)[3)]	3.098	H(12)	H(26)[3)]	2.674
H(12)	H(28)[9)]	2.758	H(13)	Al(1)	3.481
H(13)	O(1)	2.442	H(13)	C(1)	3.580
H(13)	C(3)	2.869	H(13)	C(6)	2.737
H(13)	C(10)	2.054	H(13)	C(12)	2.654
H(13)	C(12)[5)]	3.483	H(13)	C(13)	3.302
H(13)	H(1)	3.129	H(13)	H(8)[8)]	3.369
H(13)	H(11)	1.551	H(13)	H(12)	1.551
H(13)	H(14)	2.926	H(13)	H(14)[5)]	2.989
H(13)	H(15)	2.465	H(13)	H(15)[5)]	3.083
H(13)	H(16)	3.521	H(13)	H(17)	3.508
H(13)	H(18)	3.515	H(13)	H(19)[8)]	3.562

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(14)	C(6)	3.335	H(14)	C(10)	2.054
H(14)	C(11)	2.708	H(14)	C(13)	2.594
H(14)	H(1)[5)]	3.178	H(14)	H(4)[5)]	3.281
H(14)	H(8)[1)]	3.127	H(14)	H(11)	2.575
H(14)	H(12)	3.586	H(14)	H(13)	2.926
H(14)	H(13)[5)]	2.989	H(14)	H(15)	1.551
H(14)	H(16)	1.551	H(14)	H(17)	3.447
H(14)	H(18)	2.352	H(14)	H(19)	2.914
H(14)	H(23)[11)]	3.398	H(15)	O(1)	2.408
H(15)	C(3)	2.879	H(15)	C(6)	2.724
H(15)	C(7)[1)]	3.299	H(15)	C(8)[1)]	3.212
H(15)	C(10)	2.058	H(15)	C(11)	2.679
H(15)	C(11)[5)]	3.513	H(15)	C(13)	3.298
H(15)	H(8)[1)]	2.687	H(15)	H(9)[1)]	2.546
H(15)	H(11)	2.990	H(15)	H(11)[5)]	3.046
H(15)	H(12)	3.531	H(15)	H(13)	2.465
H(15)	H(13)[5)]	3.083	H(15)	H(14)	1.551
H(15)	H(16)	1.551	H(15)	H(18)	3.452
H(15)	H(19)	3.566	H(15)	H(27)[6)]	3.377
H(16)	O(1)	3.440	H(16)	C(3)	3.340
H(16)	C(6)	2.674	H(16)	C(7)	3.507
H(16)	C(7)[1)]	3.452	H(16)	C(10)	2.061
H(16)	C(11)	3.340	H(16)	C(13)	2.679
H(16)	C(16)[11)]	3.541	H(16)	C(18)[6)]	3.366
H(16)	H(8)[1)]	2.814	H(16)	H(9)[1)]	3.296
H(16)	H(11)	3.593	H(16)	H(13)	3.521
H(16)	H(14)	1.551	H(16)	H(15)	1.551
H(16)	H(17)	3.562	H(16)	H(17)[1)]	3.573
H(16)	H(18)	2.884	H(16)	H(19)	2.551
H(16)	H(23)[11)]	2.986	H(16)	H(24)[11)]	3.206
H(16)	H(26)[6)]	3.379	H(16)	H(27)[6)]	2.620
H(16)	H(28)[6)]	3.458	H(17)	C(6)	2.764
H(17)	C(7)	2.808	H(17)	C(10)	2.055
H(17)	C(11)	2.607	H(17)	C(12)	3.296
H(17)	C(16)[9)]	3.489	H(17)	H(1)[10)]	3.136
H(17)	H(8)	2.216	H(17)	H(11)	2.758
H(17)	H(12)	2.505	H(17)	H(13)	3.508

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(17)	H(14)	3.447	H(17)	H(16)	3.562
H(17)	H(16)[7)]	3.573	H(17)	H(18)	1.551
H(17)	H(19)	1.551	H(17)	H(20)[9)]	3.000
H(17)	H(23)[9)]	2.574	H(17)	H(26)[3)]	3.424
H(17)	H(28)[9)]	3.248	H(18)	C(2)[5)]	3.321
H(18)	C(6)	3.353	H(18)	C(10)	2.058
H(18)	C(11)	2.692	H(18)	C(12)	2.588
H(18)	H(1)[10)]	3.258	H(18)	H(4)[5)]	2.749
H(18)	H(5)[5)]	3.029	H(18)	H(7)[9)]	3.510
H(18)	H(8)	3.297	H(18)	H(11)	2.452
H(18)	H(12)	3.067	H(18)	H(13)	3.515
H(18)	H(14)	2.352	H(18)	H(15)	3.452
H(18)	H(16)	2.884	H(18)	H(17)	1.551
H(18)	H(19)	1.551	H(18)	H(20)[9)]	2.976
H(18)	H(24)[11)]	3.157	H(19)	C(6)	2.678
H(19)	C(7)	2.664	H(19)	C(10)	2.057
H(19)	C(11)	3.307	H(19)	C(12)	2.689
H(19)	C(16)[11)]	3.569	H(19)	H(1)[10)]	2.887
H(19)	H(2)[10)]	3.545	H(19)	H(8)	2.053
H(19)	H(11)	3.486	H(19)	H(12)	3.555
H(19)	H(12)[10)]	3.432	H(19)	H(13)[10)]	3.562
H(19)	H(14)	2.914	H(19)	H(15)	3.566
H(19)	H(16)	2.551	H(19)	H(17)	1.551
H(19)	H(18)	1.551	H(19)	H(24)[11)]	2.696
H(20)	N(1)	2.033	H(20)	C(2)[3)]	3.435
H(20)	C(5)	2.381	H(20)	C(13)[13)]	3.432
H(20)	C(15)	2.067	H(20)	C(16)	2.706
H(20)	C(17)	3.254	H(20)	C(18)	2.719
H(20)	C(19)	2.087	H(20)	H(1)[6)]	3.519
H(20)	H(3)[6)]	3.372	H(20)	H(4)[3)]	3.076
H(20)	H(5)[3)]	3.269	H(20)	H(6)[3)]	3.399
H(20)	H(7)	2.066	H(20)	H(17)[13)]	3.000
H(20)	H(18)[13)]	2.976	H(20)	H(21)	2.830
H(20)	H(22)	2.330	H(20)	H(23)	2.527
H(20)	H(24)	3.572	H(20)	H(27)	3.586
H(20)	H(28)	2.556	H(20)	H(29)	2.846
H(20)	H(30)	2.350	H(21)	Al(1)	3.419

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(21)	N(1)	2.657	H(21)	C(2)	3.046
H(21)	C(14)	2.029	H(21)	C(16)	2.040
H(21)	C(17)	2.708	H(21)	C(18)	3.230
H(21)	C(19)	2.685	H(21)	H(2)[4)]	2.723
H(21)	H(5)	3.425	H(21)	H(6)	2.275
H(21)	H(10)[1)]	3.160	H(21)	H(20)	2.830
H(21)	H(22)	1.551	H(21)	H(23)	2.787
H(21)	H(24)	2.318	H(21)	H(25)	2.562
H(21)	H(26)	3.575	H(21)	H(29)	2.529
H(21)	H(30)	3.547	H(22)	N(1)	2.646
H(22)	C(1)[6)]	3.428	H(22)	C(5)	3.112
H(22)	C(14)	2.041	H(22)	C(16)	2.070
H(22)	C(17)	3.335	H(22)	C(19)	3.318
H(22)	H(1)[6)]	3.167	H(22)	H(2)[4)]	3.488
H(22)	H(3)[6)]	2.879	H(22)	H(5)[6)]	3.270
H(22)	H(6)	3.503	H(22)	H(7)	3.080
H(22)	H(10)[1)]	3.093	H(22)	H(20)	2.330
H(22)	H(21)	1.551	H(22)	H(23)	2.328
H(22)	H(24)	2.370	H(22)	H(25)	3.580
H(22)	H(29)	3.554	H(23)	C(13)[13)]	3.488
H(23)	C(14)	2.693	H(23)	C(15)	2.036
H(23)	C(17)	2.022	H(23)	C(18)	2.690
H(23)	C(19)	3.238	H(23)	H(14)[12)]	3.398
H(23)	H(16)[12)]	2.986	H(23)	H(17)[13)]	2.574
H(23)	H(20)	2.527	H(23)	H(21)	2.787
H(23)	H(22)	2.328	H(23)	H(24)	1.551
H(23)	H(25)	2.774	H(23)	H(26)	2.295
H(23)	H(26)[14)]	3.227	H(23)	H(27)	3.556
H(23)	H(28)	2.555	H(24)	C(1)[4)]	3.490
H(24)	C(13)[12)]	3.345	H(24)	C(14)	3.319
H(24)	C(15)	2.052	H(24)	C(17)	2.033
H(24)	C(18)	3.300	H(24)	H(1)[4)]	3.389
H(24)	H(2)[4)]	2.748	H(24)	H(12)[4)]	3.150
H(24)	H(16)[12)]	3.206	H(24)	H(18)[12)]	3.157
H(24)	H(19)[12)]	2.696	H(24)	H(20)	3.572
H(24)	H(21)	2.318	H(24)	H(22)	2.370
H(24)	H(23)	1.551	H(24)	H(25)	2.290

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(24)	H(26)	2.335	H(24)	H(28)	3.561
H(25)	C(3)[4)]	3.160	H(25)	C(4)[4)]	3.159
H(25)	C(6)[4)]	3.108	H(25)	C(7)[4)]	3.045
H(25)	C(8)[4)]	3.074	H(25)	C(9)[4)]	3.123
H(25)	C(14)	3.251	H(25)	C(15)	2.713
H(25)	C(16)	2.018	H(25)	C(18)	2.017
H(25)	C(19)	2.702	H(25)	H(2)[4)]	3.447
H(25)	H(8)[4)]	3.568	H(25)	H(12)[4)]	3.098
H(25)	H(21)	2.562	H(25)	H(22)	3.580
H(25)	H(23)	2.774	H(25)	H(24)	2.290
H(25)	H(26)	1.551	H(25)	H(27)	2.281
H(25)	H(28)	2.772	H(25)	H(29)	2.564
H(25)	H(30)	3.567	H(26)	C(6)[4)]	3.592
H(26)	C(7)[4)]	3.472	H(26)	C(11)[4)]	3.564
H(26)	C(15)	3.329	H(26)	C(16)	2.032
H(26)	C(17)[14)]	3.485	H(26)	C(18)	2.028
H(26)	C(19)	3.316	H(26)	H(8)[4)]	3.567
H(26)	H(12)[4)]	2.674	H(26)	H(16)[2)]	3.379
H(26)	H(17)[4)]	3.424	H(26)	H(21)	3.575
H(26)	H(23)	2.295	H(26)	H(23)[14)]	3.227
H(26)	H(24)	2.335	H(26)	H(25)	1.551
H(26)	H(26)[14)]	2.745	H(26)	H(27)	2.330
H(26)	H(28)	2.286	H(26)	H(28)[14)]	3.424
H(26)	H(29)	3.575	H(27)	C(3)[2)]	3.263
H(27)	C(6)[2)]	3.182	H(27)	C(7)[2)]	3.435
H(27)	C(8)[4)]	3.303	H(27)	C(12)[2)]	3.334
H(27)	C(14)	3.312	H(27)	C(16)	3.298
H(27)	C(17)	2.025	H(27)	C(19)	2.036
H(27)	H(9)[4)]	3.182	H(27)	H(15)[2)]	3.377
H(27)	H(16)[2)]	2.620	H(27)	H(20)	3.586
H(27)	H(23)	3.556	H(27)	H(25)	2.281
H(27)	H(26)	2.330	H(27)	H(28)	1.551
H(27)	H(29)	2.305	H(27)	H(30)	2.346
H(28)	C(7)[2)]	3.582	H(28)	C(11)[13)]	3.322
H(28)	C(14)	2.705	H(28)	C(15)	3.256
H(28)	C(16)	2.697	H(28)	C(17)	2.019
H(28)	C(19)	2.039	H(28)	H(11)[13)]	3.022

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(28)	H(12)[13)]	2.758	H(28)	H(16)[2)]	3.458
H(28)	H(17)[13)]	3.248	H(28)	H(20)	2.556
H(28)	H(23)	2.555	H(28)	H(24)	3.561
H(28)	H(25)	2.772	H(28)	H(26)	2.286
H(28)	H(26)[14)]	3.424	H(28)	H(27)	1.551
H(28)	H(29)	2.794	H(28)	H(30)	2.315
H(29)	Al(1)	2.927	H(29)	N(1)	2.656
H(29)	C(1)	3.304	H(29)	C(2)	3.118
H(29)	C(14)	2.031	H(29)	C(15)	2.694
H(29)	C(16)	3.236	H(29)	C(17)	2.708
H(29)	C(18)	2.041	H(29)	H(3)	2.799
H(29)	H(5)	2.943	H(29)	H(6)	3.016
H(29)	H(10)[4)]	3.437	H(29)	H(20)	2.846
H(29)	H(21)	2.529	H(29)	H(22)	3.554
H(29)	H(25)	2.564	H(29)	H(26)	3.575
H(29)	H(27)	2.305	H(29)	H(28)	2.794
H(29)	H(30)	1.551	H(30)	Al(1)	3.411
H(30)	N(1)	2.650	H(30)	C(1)	3.265
H(30)	C(4)[2)]	3.352	H(30)	C(5)	3.533
H(30)	C(8)[2)]	3.284	H(30)	C(9)[2)]	3.007
H(30)	C(14)	2.037	H(30)	C(15)	3.315
H(30)	C(17)	3.316	H(30)	C(18)	2.051
H(30)	H(2)	3.311	H(30)	H(3)	2.775
H(30)	H(4)[3)]	3.307	H(30)	H(6)[3)]	3.166
H(30)	H(10)[2)]	3.202	H(30)	H(20)	2.350
H(30)	H(21)	3.547	H(30)	H(25)	3.567
H(30)	H(27)	2.346	H(30)	H(28)	2.315
H(30)	H(29)	1.551			

Symmetry Operators:

(1) -X+1/2,Y+1/2-1,Z
(2) X+1/2,Y,-Z+1/2+1
(3) -X+1,Y+1/2,-Z+1/2+1
(4) -X+1,Y+1/2-1,-Z+1/2+1
(5) -X+1,-Y,-Z+1
(6) X+1/2-1,Y,-Z+1/2+1
(7) -X+1/2,Y+1/2,Z
(8) X+1/2,-Y+1/2,-Z+1
(9) X,-Y+1/2,Z+1/2-1
(10) X+1/2-1,-Y+1/2,-Z+1
(11) -X+1/2,-Y,Z+1/2-1
(12) -X+1/2,-Y,Z+1/2
(13) X,-Y+1/2,Z+1/2
(14) -X+1,-Y,-Z+2

X-ray Structure Report for $Me_2Al[O\text{-}2\text{-}^{t}Bu\text{-}6\text{-}(PhN{=}CH)C_6H_3]$ (**2e**)

May 10, 2007

Experimental

Data Collection

A yellow block crystal of $C_{19}H_{24}NOAl$ having approximate dimensions of 0.40 x 0.24 x 0.24 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 360 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 12.1416(5) Å
b = 7.2786(3) Å β = 100.5986(13)^o
c = 21.0147(9) Å
V = 1825.47(13) Å^3

For Z = 4 and F.W. = 309.39, the calculated density is 1.126 g/cm^3. The systematic absences of:

h0l: h+l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 5.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 180.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 5.0^o step, at χ=45.0^o and ϕ = 180.0^o. The exposure rate was 180.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 17113 reflections that were collected, 4157 were unique (R_{int} = 0.027); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.127 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.667 to 0.973. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3154 observed reflections and 223 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0394$$

$$wR2 = [\, \Sigma \, (\, w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.1195$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.18 and -0.24 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

$_{19}H_{24}NOAl$

Formula Weight	309.39
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.40 X 0.24 X 0.24 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 360.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 12.1416(5) Å b = 7.2786(3) Å c = 21.0147(9) Å β = 100.5986(13) ^{o} V = 1825.47(13) Å^{3}
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.126 g/cm^{3}
F_{000}	664.00
μ(MoKα)	1.127 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	180.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0^{o}
Exposure Rate	180.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 17113 Unique: 4157 (R_{int} = 0.027)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.667 - 0.973)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0022Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	3154
No. Variables	223
Reflection/Parameter Ratio	14.14
Residuals: R1 (I>2.00σ(I))	0.0394
Residuals: wR2 (I>2.00σ(I))	0.1195
Goodness of Fit Indicator	1.012
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.18 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.24 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.47981(4)	0.10284(6)	0.37697(2)	2.924(10)
O(1)	0.34219(8)	0.17874(17)	0.38155(5)	3.54(2)
N(1)	0.44849(10)	0.09340(15)	0.28071(6)	2.74(2)
C(1)	0.59166(15)	0.2922(2)	0.40498(9)	4.54(4)
C(2)	0.50491(18)	-0.1466(2)	0.41182(9)	4.72(4)
C(3)	0.24559(12)	0.15283(19)	0.34146(7)	2.70(2)
C(4)	0.24513(12)	0.10181(18)	0.27628(7)	2.67(2)
C(5)	0.34536(13)	0.08369(18)	0.24976(7)	2.76(2)
C(6)	0.14248(12)	0.1808(2)	0.36302(7)	2.93(2)
C(7)	0.04476(12)	0.1595(2)	0.31801(7)	3.25(3)
C(8)	0.04348(13)	0.1088(2)	0.25323(8)	3.44(3)
C(9)	0.14235(13)	0.07923(19)	0.23279(7)	3.06(3)
C(10)	0.14099(14)	0.2288(2)	0.43398(7)	3.75(3)
C(11)	0.20203(19)	0.4100(2)	0.45308(10)	5.20(4)
C(12)	0.19577(17)	0.0713(3)	0.47769(9)	5.10(4)
C(13)	0.01990(16)	0.2508(3)	0.44600(9)	5.13(4)
C(14)	0.53494(12)	0.09533(19)	0.24278(7)	2.77(2)
C(15)	0.52169(13)	0.1931(2)	0.18462(7)	3.16(3)
C(16)	0.60629(15)	0.1912(2)	0.14881(8)	3.87(3)
C(17)	0.70353(15)	0.0924(2)	0.16907(9)	3.93(3)
C(18)	0.71793(14)	-0.0019(2)	0.22714(9)	4.10(3)
C(19)	0.63448(13)	0.0010(2)	0.26438(8)	3.51(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.5978	0.3126	0.4501	5.51
H(2)	0.6622	0.2540	0.3962	5.47
H(3)	0.5693	0.4027	0.3822	5.51
H(4)	0.5226	-0.1423	0.4577	5.65
H(5)	0.4389	-0.2175	0.3987	5.68
H(6)	0.5652	-0.2013	0.3956	5.69
H(7)	0.3323	0.0607	0.2029	3.40
H(8)	-0.0282	0.1843	0.3304	3.95
H(9)	-0.0311	0.0969	0.2219	4.14
H(10)	0.1448	0.0410	0.1874	3.64
H(11)	0.2774	0.4011	0.4469	6.36
H(12)	0.1650	0.5051	0.4264	6.38
H(13)	0.2010	0.4372	0.4972	6.37
H(14)	0.1920	0.0969	0.5216	6.26
H(15)	0.1568	-0.0397	0.4648	6.28
H(16)	0.2719	0.0587	0.4734	6.26
H(17)	-0.0148	0.3515	0.4214	6.37
H(18)	-0.0206	0.1414	0.4328	6.41
H(19)	0.0203	0.2720	0.4906	6.39
H(20)	0.4521	0.2685	0.1709	3.81
H(21)	0.5965	0.2598	0.1048	4.79
H(22)	0.7697	0.0834	0.1458	4.95
H(23)	0.7839	-0.0866	0.2396	5.11
H(24)	0.6449	-0.0742	0.3070	4.28

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0326(2)	0.0447(2)	0.0339(2)	-0.00179(18)	0.00619(18)	0.00248(18)
O(1)	0.0326(5)	0.0696(7)	0.0326(5)	-0.0013(5)	0.0073(4)	-0.0072(5)
N(1)	0.0332(6)	0.0374(6)	0.0354(6)	0.0024(4)	0.0111(4)	0.0014(4)
C(1)	0.0496(10)	0.0643(11)	0.0574(10)	-0.0130(8)	0.0069(8)	-0.0001(8)
C(2)	0.0804(14)	0.0543(9)	0.0431(9)	0.0015(9)	0.0078(8)	0.0065(7)
C(3)	0.0331(7)	0.0363(6)	0.0341(7)	-0.0000(5)	0.0086(5)	0.0023(5)
C(4)	0.0334(7)	0.0340(6)	0.0351(7)	0.0007(5)	0.0087(5)	0.0010(5)
C(5)	0.0380(7)	0.0361(6)	0.0320(6)	0.0009(5)	0.0093(5)	-0.0011(5)
C(6)	0.0375(7)	0.0379(7)	0.0384(7)	0.0021(6)	0.0140(6)	0.0050(5)
C(7)	0.0320(7)	0.0445(7)	0.0495(8)	0.0014(6)	0.0141(6)	0.0063(6)
C(8)	0.0337(7)	0.0489(8)	0.0468(8)	-0.0018(6)	0.0036(6)	0.0018(6)
C(9)	0.0382(8)	0.0401(7)	0.0369(7)	-0.0011(6)	0.0046(6)	-0.0007(5)
C(10)	0.0439(8)	0.0639(10)	0.0380(8)	0.0069(7)	0.0163(6)	0.0029(7)
C(11)	0.0745(14)	0.0772(12)	0.0499(10)	-0.0011(10)	0.0218(9)	-0.0199(9)
C(12)	0.0619(12)	0.0925(14)	0.0434(9)	0.0186(10)	0.0203(8)	0.0197(9)
C(13)	0.0557(11)	0.0964(15)	0.0498(10)	0.0180(10)	0.0275(8)	0.0059(9)
C(14)	0.0348(7)	0.0361(6)	0.0366(7)	-0.0007(5)	0.0128(5)	-0.0033(5)
C(15)	0.0429(8)	0.0431(7)	0.0361(7)	0.0015(6)	0.0126(6)	-0.0015(6)
C(16)	0.0554(10)	0.0573(9)	0.0383(8)	-0.0063(7)	0.0191(7)	-0.0037(7)
C(17)	0.0452(9)	0.0540(9)	0.0565(9)	-0.0050(7)	0.0262(7)	-0.0111(7)
C(18)	0.0385(8)	0.0489(8)	0.0735(11)	0.0028(7)	0.0233(7)	-0.0006(8)
C(19)	0.0372(8)	0.0457(8)	0.0528(9)	0.0026(6)	0.0143(6)	0.0060(6)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.7792(11)	Al(1)	N(1)	1.9896(13)
Al(1)	C(1)	1.9482(18)	Al(1)	C(2)	1.9607(18)
O(1)	C(3)	1.3257(16)	N(1)	C(5)	1.3024(18)
N(1)	C(14)	1.430(2)	C(3)	C(4)	1.418(2)
C(3)	C(6)	1.422(2)	C(4)	C(5)	1.435(2)
C(4)	C(9)	1.4142(19)	C(6)	C(7)	1.3833(18)
C(6)	C(10)	1.535(2)	C(7)	C(8)	1.408(2)
C(8)	C(9)	1.364(2)	C(10)	C(11)	1.530(2)
C(10)	C(12)	1.542(2)	C(10)	C(13)	1.545(2)
C(14)	C(15)	1.398(2)	C(14)	C(19)	1.392(2)
C(15)	C(16)	1.381(2)	C(16)	C(17)	1.381(2)
C(17)	C(18)	1.383(2)	C(18)	C(19)	1.390(2)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(5)	H(7)	0.983	C(7)	H(8)	0.985
C(8)	H(9)	1.021	C(9)	H(10)	0.999
C(11)	H(11)	0.950	C(11)	H(12)	0.950
C(11)	H(13)	0.950	C(12)	H(14)	0.950
C(12)	H(15)	0.950	C(12)	H(16)	0.950
C(13)	H(17)	0.950	C(13)	H(18)	0.950
C(13)	H(19)	0.950	C(15)	H(20)	1.004
C(16)	H(21)	1.039	C(17)	H(22)	1.017
C(18)	H(23)	1.007	C(19)	H(24)	1.038

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	93.44(5)	O(1)	Al(1)	C(1)	111.93(7)
O(1)	Al(1)	C(2)	110.51(8)	N(1)	Al(1)	C(1)	108.90(6)
N(1)	Al(1)	C(2)	109.59(6)	C(1)	Al(1)	C(2)	119.36(8)
Al(1)	O(1)	C(3)	130.64(10)	Al(1)	N(1)	C(5)	119.74(11)
Al(1)	N(1)	C(14)	122.94(8)	C(5)	N(1)	C(14)	117.32(12)
O(1)	C(3)	C(4)	119.79(13)	O(1)	C(3)	C(6)	120.33(13)
C(4)	C(3)	C(6)	119.86(12)	C(3)	C(4)	C(5)	123.11(12)
C(3)	C(4)	C(9)	120.06(13)	C(5)	C(4)	C(9)	116.68(13)
N(1)	C(5)	C(4)	127.39(13)	C(3)	C(6)	C(7)	117.43(13)
C(3)	C(6)	C(10)	120.69(12)	C(7)	C(6)	C(10)	121.86(14)
C(6)	C(7)	C(8)	123.10(14)	C(7)	C(8)	C(9)	119.47(13)
C(4)	C(9)	C(8)	120.07(13)	C(6)	C(10)	C(11)	111.08(14)
C(6)	C(10)	C(12)	109.18(14)	C(6)	C(10)	C(13)	111.35(12)
C(11)	C(10)	C(12)	110.48(13)	C(11)	C(10)	C(13)	107.30(16)
C(12)	C(10)	C(13)	107.39(15)	N(1)	C(14)	C(15)	120.94(12)
N(1)	C(14)	C(19)	119.72(13)	C(15)	C(14)	C(19)	119.33(14)
C(14)	C(15)	C(16)	119.60(14)	C(15)	C(16)	C(17)	121.12(15)
C(16)	C(17)	C(18)	119.51(17)	C(17)	C(18)	C(19)	120.17(15)
C(14)	C(19)	C(18)	120.21(15)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.5	Al(1)	C(1)	H(2)	109.7
Al(1)	C(1)	H(3)	109.2	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.8	Al(1)	C(2)	H(5)	109.3
Al(1)	C(2)	H(6)	109.3	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
N(1)	C(5)	H(7)	118.2	C(4)	C(5)	H(7)	114.4
C(6)	C(7)	H(8)	119.9	C(8)	C(7)	H(8)	117.0
C(7)	C(8)	H(9)	119.8	C(9)	C(8)	H(9)	120.7
C(4)	C(9)	H(10)	118.1	C(8)	C(9)	H(10)	121.8
C(10)	C(11)	H(11)	109.8	C(10)	C(11)	H(12)	108.6
C(10)	C(11)	H(13)	110.0	H(11)	C(11)	H(12)	109.5
H(11)	C(11)	H(13)	109.5	H(12)	C(11)	H(13)	109.5
C(10)	C(12)	H(14)	109.9	C(10)	C(12)	H(15)	108.9
C(10)	C(12)	H(16)	109.6	H(14)	C(12)	H(15)	109.5
H(14)	C(12)	H(16)	109.5	H(15)	C(12)	H(16)	109.5
C(10)	C(13)	H(17)	109.4	C(10)	C(13)	H(18)	108.8
C(10)	C(13)	H(19)	110.3	H(17)	C(13)	H(18)	109.5
H(17)	C(13)	H(19)	109.5	H(18)	C(13)	H(19)	109.5
C(14)	C(15)	H(20)	119.0	C(16)	C(15)	H(20)	121.3
C(15)	C(16)	H(21)	120.4	C(17)	C(16)	H(21)	118.4
C(16)	C(17)	H(22)	126.3	C(18)	C(17)	H(22)	114.2
C(17)	C(18)	H(23)	119.9	C(19)	C(18)	H(23)	119.4
C(14)	C(19)	H(24)	120.2	C(18)	C(19)	H(24)	119.5

Table 8. Torsion Angles(O)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(5)	-20.46(10)	O(1)	Al(1)	N(1)	C(14)	160.04(10)
N(1)	Al(1)	O(1)	C(3)	27.57(13)	C(1)	Al(1)	O(1)	C(3)	139.53(13)
C(2)	Al(1)	O(1)	C(3)	-84.83(14)	C(1)	Al(1)	N(1)	C(5)	-135.04(11)
C(1)	Al(1)	N(1)	C(14)	45.45(12)	C(2)	Al(1)	N(1)	C(5)	92.74(12)
C(2)	Al(1)	N(1)	C(14)	-86.76(12)	Al(1)	O(1)	C(3)	C(4)	-19.6(2)
Al(1)	O(1)	C(3)	C(6)	162.10(11)	Al(1)	N(1)	C(5)	C(4)	8.55(18)
Al(1)	N(1)	C(14)	C(15)	-141.27(11)	Al(1)	N(1)	C(14)	C(19)	37.74(17)
C(5)	N(1)	C(14)	C(15)	39.21(18)	C(5)	N(1)	C(14)	C(19)	-141.77(13)
C(14)	N(1)	C(5)	C(4)	-171.92(12)	O(1)	C(3)	C(4)	C(5)	-2.8(2)
O(1)	C(3)	C(4)	C(9)	-178.14(12)	O(1)	C(3)	C(6)	C(7)	177.15(13)
O(1)	C(3)	C(6)	C(10)	-4.1(2)	C(4)	C(3)	C(6)	C(7)	-1.1(2)
C(4)	C(3)	C(6)	C(10)	177.65(13)	C(6)	C(3)	C(4)	C(5)	175.50(12)
C(6)	C(3)	C(4)	C(9)	0.2(2)	C(3)	C(4)	C(5)	N(1)	6.7(2)
C(3)	C(4)	C(9)	C(8)	0.9(2)	C(5)	C(4)	C(9)	C(8)	-174.78(13)
C(9)	C(4)	C(5)	N(1)	-177.80(13)	C(3)	C(6)	C(7)	C(8)	1.2(2)
C(3)	C(6)	C(10)	C(11)	61.00(19)	C(3)	C(6)	C(10)	C(12)	-61.07(19)
C(3)	C(6)	C(10)	C(13)	-179.47(15)	C(7)	C(6)	C(10)	C(11)	-120.26(17)
C(7)	C(6)	C(10)	C(12)	117.66(16)	C(7)	C(6)	C(10)	C(13)	-0.7(2)
C(10)	C(6)	C(7)	C(8)	-177.58(15)	C(6)	C(7)	C(8)	C(9)	-0.2(2)
C(7)	C(8)	C(9)	C(4)	-0.8(2)	N(1)	C(14)	C(15)	C(16)	-179.49(13)
N(1)	C(14)	C(19)	C(18)	178.39(13)	C(15)	C(14)	C(19)	C(18)	-2.6(2)
C(19)	C(14)	C(15)	C(16)	1.5(2)	C(14)	C(15)	C(16)	C(17)	0.8(2)
C(15)	C(16)	C(17)	C(18)	-1.9(2)	C(16)	C(17)	C(18)	C(19)	0.8(2)
C(17)	C(18)	C(19)	C(14)	1.4(2)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Al(1)	C(3)	2.8277(14)	Al(1)	C(4)	3.2216(13)
Al(1)	C(5)	2.8680(14)	Al(1)	C(14)	3.0162(16)
Al(1)	C(19)	3.3629(18)	O(1)	N(1)	2.7474(17)
O(1)	C(1)	3.090(2)	O(1)	C(2)	3.075(2)
O(1)	C(4)	2.3743(16)	O(1)	C(5)	2.8617(18)
O(1)	C(6)	2.3841(17)	O(1)	C(10)	2.882(2)
O(1)	C(11)	2.988(2)	O(1)	C(12)	3.029(2)
N(1)	O(1)	2.7474(17)	N(1)	C(1)	3.204(2)
N(1)	C(2)	3.228(2)	N(1)	C(3)	3.007(2)
N(1)	C(4)	2.4545(19)	N(1)	C(15)	2.461(2)
N(1)	C(19)	2.440(2)	C(1)	O(1)	3.090(2)
C(1)	N(1)	3.204(2)	C(1)	C(2)	3.374(2)
C(2)	O(1)	3.075(2)	C(2)	N(1)	3.228(2)
C(2)	C(1)	3.374(2)	C(3)	Al(1)	2.8277(14)
C(3)	N(1)	3.007(2)	C(3)	C(5)	2.509(2)
C(3)	C(7)	2.398(2)	C(3)	C(8)	2.8086(19)
C(3)	C(9)	2.4536(19)	C(3)	C(10)	2.571(2)
C(3)	C(11)	3.121(2)	C(3)	C(12)	3.090(2)
C(4)	Al(1)	3.2216(13)	C(4)	O(1)	2.3743(16)
C(4)	N(1)	2.4545(19)	C(4)	C(6)	2.458(2)
C(4)	C(7)	2.766(2)	C(4)	C(8)	2.407(2)
C(5)	Al(1)	2.8680(14)	C(5)	O(1)	2.8617(18)
C(5)	C(3)	2.509(2)	C(5)	C(9)	2.425(2)
C(5)	C(14)	2.335(2)	C(5)	C(15)	2.858(2)
C(5)	C(19)	3.519(2)	C(6)	O(1)	2.3841(17)
C(6)	C(4)	2.458(2)	C(6)	C(8)	2.454(2)
C(6)	C(9)	2.835(2)	C(6)	C(11)	2.527(2)
C(6)	C(12)	2.507(2)	C(6)	C(13)	2.544(2)
C(7)	C(3)	2.398(2)	C(7)	C(4)	2.766(2)
C(7)	C(9)	2.394(2)	C(7)	C(10)	2.5518(19)
C(7)	C(12)	3.574(2)	C(7)	C(13)	2.840(2)
C(7)	C(14)[1)]	3.490(2)	C(7)	C(15)[2)]	3.487(2)
C(7)	C(19)[1)]	3.539(2)	C(8)	C(3)	2.8086(19)
C(8)	C(4)	2.407(2)	C(8)	C(6)	2.454(2)
C(8)	C(15)[2)]	3.445(2)	C(8)	C(19)[1)]	3.558(2)
C(9)	C(3)	2.4536(19)	C(9)	C(5)	2.425(2)
C(9)	C(6)	2.835(2)	C(9)	C(7)	2.394(2)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(10)	O(1)	2.882(2)	C(10)	C(3)	2.571(2)
C(10)	C(7)	2.5518(19)	C(11)	O(1)	2.988(2)
C(11)	C(3)	3.121(2)	C(11)	C(6)	2.527(2)
C(11)	C(12)	2.523(3)	C(11)	C(13)	2.477(3)
C(12)	O(1)	3.029(2)	C(12)	C(3)	3.090(2)
C(12)	C(6)	2.507(2)	C(12)	C(7)	3.574(2)
C(12)	C(11)	2.523(3)	C(12)	C(13)	2.488(2)
C(13)	C(6)	2.544(2)	C(13)	C(7)	2.840(2)
C(13)	C(11)	2.477(3)	C(13)	C(12)	2.488(2)
C(14)	Al(1)	3.0162(16)	C(14)	C(5)	2.335(2)
C(14)	C(7)[2)]	3.490(2)	C(14)	C(16)	2.401(2)
C(14)	C(17)	2.785(2)	C(14)	C(18)	2.411(2)
C(15)	N(1)	2.461(2)	C(15)	C(5)	2.858(2)
C(15)	C(7)[1)]	3.487(2)	C(15)	C(8)[1)]	3.445(2)
C(15)	C(17)	2.405(2)	C(15)	C(18)	2.776(2)
C(15)	C(19)	2.408(2)	C(16)	C(14)	2.401(2)
C(16)	C(18)	2.388(2)	C(16)	C(19)	2.762(2)
C(17)	C(14)	2.785(2)	C(17)	C(15)	2.405(2)
C(17)	C(19)	2.403(2)	C(18)	C(14)	2.411(2)
C(18)	C(15)	2.776(2)	C(18)	C(16)	2.388(2)
C(19)	Al(1)	3.3629(18)	C(19)	N(1)	2.440(2)
C(19)	C(5)	3.519(2)	C(19)	C(7)[2)]	3.539(2)
C(19)	C(8)[2)]	3.558(2)	C(19)	C(15)	2.408(2)
C(19)	C(16)	2.762(2)	C(19)	C(17)	2.403(2)

Symmetry Operators:

(1) -X+1/2,Y+1/2,-Z+1/2

(2) -X+1/2,Y+1/2-1,-Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Al(1)	H(1)	2.436	Al(1)	H(2)	2.438
Al(1)	H(3)	2.432	Al(1)	H(4)	2.452
Al(1)	H(4)[1)]	3.491	Al(1)	H(5)	2.445
Al(1)	H(6)	2.445	Al(1)	H(16)	3.530
Al(1)	H(24)	2.987	O(1)	H(1)	3.318
O(1)	H(3)	3.201	O(1)	H(4)	3.396
O(1)	H(4)[1)]	3.483	O(1)	H(5)	3.110
O(1)	H(10)[2)]	3.027	O(1)	H(11)	2.350
O(1)	H(12)	3.451	O(1)	H(15)	3.479
O(1)	H(16)	2.412	N(1)	H(2)	3.416
N(1)	H(3)	3.259	N(1)	H(5)	3.376
N(1)	H(6)	3.343	N(1)	H(7)	1.968
N(1)	H(10)[2)]	3.553	N(1)	H(20)	2.644
N(1)	H(24)	2.645	C(1)	H(4)	3.503
C(1)	H(4)[1)]	3.593	C(1)	H(9)[2)]	3.442
C(1)	H(13)[3)]	3.542	C(1)	H(22)[4)]	3.021
C(1)	H(24)	3.501	C(2)	H(1)	3.572
C(2)	H(1)[1)]	3.572	C(2)	H(2)	3.534
C(2)	H(3)[5)]	3.455	C(2)	H(4)[1)]	3.519
C(2)	H(9)[6)]	3.436	C(2)	H(10)[6)]	3.380
C(2)	H(16)[1)]	3.342	C(2)	H(24)	3.065
C(3)	H(7)	3.342	C(3)	H(7)[2)]	3.201
C(3)	H(8)	3.298	C(3)	H(10)	3.344
C(3)	H(10)[2)]	3.228	C(3)	H(11)	2.831
C(3)	H(12)	3.371	C(3)	H(15)	3.297
C(3)	H(16)	2.816	C(4)	H(7)	2.047
C(4)	H(7)[2)]	3.519	C(4)	H(9)	3.338
C(4)	H(10)	2.081	C(4)	H(10)[2)]	3.495
C(5)	H(10)	2.563	C(5)	H(10)[2)]	3.575
C(5)	H(20)	2.651	C(6)	H(7)[2)]	3.132
C(6)	H(8)	2.060	C(6)	H(9)	3.363
C(6)	H(11)	2.702	C(6)	H(12)	2.700
C(6)	H(13)	3.347	C(6)	H(14)	3.331
C(6)	H(15)	2.654	C(6)	H(16)	2.701
C(6)	H(17)	2.750	C(6)	H(18)	2.686
C(6)	H(19)	3.361	C(6)	H(20)[6)]	3.245
C(7)	H(7)[2)]	3.345	C(7)	H(9)	2.110

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(7)	H(10)	3.313	C(7)	H(12)	3.525
C(7)	H(15)	3.453	C(7)	H(17)	2.789
C(7)	H(18)	2.677	C(7)	H(20)[6)]	2.855
C(8)	H(3)[6)]	3.285	C(8)	H(5)[2)]	3.478
C(8)	H(6)[2)]	3.451	C(8)	H(8)	2.052
C(8)	H(10)	2.073	C(8)	H(20)[6)]	2.941
C(8)	H(23)[7)]	3.422	C(8)	H(24)[2)]	3.330
C(9)	H(3)[6)]	3.437	C(9)	H(5)[2)]	3.130
C(9)	H(7)	2.503	C(9)	H(8)	3.263
C(9)	H(9)	2.080	C(9)	H(20)[6)]	3.374
C(10)	H(8)	2.723	C(10)	H(11)	2.055
C(10)	H(12)	2.042	C(10)	H(13)	2.059
C(10)	H(14)	2.067	C(10)	H(15)	2.057
C(10)	H(16)	2.064	C(10)	H(17)	2.065
C(10)	H(18)	2.058	C(10)	H(19)	2.075
C(11)	H(1)[3)]	3.508	C(11)	H(7)[2)]	3.408
C(11)	H(14)	2.710	C(11)	H(15)	3.335
C(11)	H(16)	2.703	C(11)	H(17)	2.629
C(11)	H(18)	3.299	C(11)	H(19)	2.673
C(12)	H(4)[1)]	3.477	C(12)	H(11)	2.720
C(12)	H(12)	3.335	C(12)	H(13)	2.694
C(12)	H(17)	3.315	C(12)	H(18)	2.672
C(12)	H(18)[8)]	3.455	C(12)	H(19)	2.639
C(12)	H(21)[9)]	3.359	C(13)	H(8)	2.439
C(13)	H(11)	3.309	C(13)	H(12)	2.639
C(13)	H(13)	2.638	C(13)	H(14)	2.631
C(13)	H(15)	2.674	C(13)	H(15)[8)]	3.458
C(13)	H(16)	3.317	C(13)	H(21)[9)]	3.296
C(14)	H(2)	3.504	C(14)	H(7)	2.462
C(14)	H(8)[6)]	3.358	C(14)	H(20)	2.080
C(14)	H(21)	3.346	C(14)	H(23)	3.312
C(14)	H(23)[4)]	3.168	C(14)	H(24)	2.113
C(15)	H(7)	2.586	C(15)	H(8)[2)]	3.591
C(15)	H(9)[2)]	3.526	C(15)	H(12)[6)]	3.242
C(15)	H(17)[6)]	3.329	C(15)	H(21)	2.106
C(15)	H(22)	3.358	C(15)	H(23)[4)]	3.049
C(15)	H(24)	3.351	C(16)	H(14)[10)]	3.411

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(16)	H(17)$^{6)}$	2.986	C(16)	H(19)$^{10)}$	3.307
C(16)	H(20)	2.087	C(16)	H(22)	2.145
C(16)	H(23)	3.296	C(16)	H(23)$^{4)}$	2.957
C(16)	H(24)$^{4)}$	3.445	C(17)	H(2)$^{11)}$	3.381
C(17)	H(3)$^{11)}$	3.430	C(17)	H(9)$^{12)}$	3.209
C(17)	H(17)$^{6)}$	3.215	C(17)	H(20)	3.318
C(17)	H(21)	2.086	C(17)	H(23)	2.077
C(17)	H(23)$^{4)}$	3.010	C(17)	H(24)	3.338
C(17)	H(24)$^{4)}$	3.031	C(18)	H(8)$^{6)}$	3.311
C(18)	H(9)$^{12)}$	3.153	C(18)	H(21)	3.320
C(18)	H(22)	2.024	C(18)	H(23)$^{4)}$	3.103
C(18)	H(24)	2.105	C(19)	H(2)	3.291
C(19)	H(6)	3.366	C(19)	H(8)$^{6)}$	3.163
C(19)	H(9)$^{6)}$	3.231	C(19)	H(20)	3.310
C(19)	H(22)	3.282	C(19)	H(23)	2.078
C(19)	H(23)$^{4)}$	3.167	H(1)	Al(1)	2.436
H(1)	O(1)	3.318	H(1)	C(2)	3.572
H(1)	C(2)$^{1)}$	3.572	H(1)	C(11)$^{3)}$	3.508
H(1)	H(2)	1.551	H(1)	H(3)	1.551
H(1)	H(4)	3.446	H(1)	H(4)$^{1)}$	2.909
H(1)	H(5)$^{1)}$	3.359	H(1)	H(11)$^{3)}$	3.180
H(1)	H(13)$^{3)}$	3.085	H(1)	H(16)$^{1)}$	3.385
H(1)	H(22)$^{4)}$	3.425	H(2)	Al(1)	2.438
H(2)	N(1)	3.416	H(2)	C(2)	3.534
H(2)	C(14)	3.504	H(2)	C(17)$^{4)}$	3.381
H(2)	C(19)	3.291	H(2)	H(1)	1.551
H(2)	H(3)	1.551	H(2)	H(6)	3.516
H(2)	H(13)$^{3)}$	3.386	H(2)	H(14)$^{1)}$	3.394
H(2)	H(16)$^{1)}$	3.540	H(2)	H(22)$^{4)}$	2.735
H(2)	H(23)$^{4)}$	3.256	H(2)	H(24)	3.019
H(3)	Al(1)	2.432	H(3)	O(1)	3.201
H(3)	N(1)	3.259	H(3)	C(2)$^{13)}$	3.455
H(3)	C(8)$^{2)}$	3.285	H(3)	C(9)$^{2)}$	3.437
H(3)	C(17)$^{4)}$	3.430	H(3)	H(1)	1.551
H(3)	H(2)	1.551	H(3)	H(5)$^{13)}$	3.236
H(3)	H(6)$^{13)}$	2.898	H(3)	H(9)$^{2)}$	2.574
H(3)	H(10)$^{2)}$	2.918	H(3)	H(22)$^{4)}$	2.514

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(3)	H(23)[4)]	3.378	H(4)	Al(1)	2.452
H(4)	Al(1)[1)]	3.491	H(4)	O(1)	3.396
H(4)	O(1)[1)]	3.483	H(4)	C(1)	3.503
H(4)	C(1)[1)]	3.593	H(4)	C(2)[1)]	3.519
H(4)	C(12)[1)]	3.477	H(4)	H(1)	3.446
H(4)	H(1)[1)]	2.909	H(4)	H(4)[1)]	2.847
H(4)	H(5)	1.551	H(4)	H(6)	1.551
H(4)	H(11)[1)]	3.415	H(4)	H(14)[1)]	3.428
H(4)	H(16)	3.447	H(4)	H(16)[1)]	2.713
H(5)	Al(1)	2.445	H(5)	O(1)	3.110
H(5)	N(1)	3.376	H(5)	C(8)[6)]	3.478
H(5)	C(9)[6)]	3.130	H(5)	H(1)[1)]	3.359
H(5)	H(3)[5)]	3.236	H(5)	H(4)	1.551
H(5)	H(6)	1.551	H(5)	H(9)[6)]	3.247
H(5)	H(10)[6)]	2.593	H(5)	H(16)	3.433
H(5)	H(24)	3.582	H(6)	Al(1)	2.445
H(6)	N(1)	3.343	H(6)	C(8)[6)]	3.451
H(6)	C(19)	3.366	H(6)	H(2)	3.516
H(6)	H(3)[5)]	2.898	H(6)	H(4)	1.551
H(6)	H(5)	1.551	H(6)	H(9)[6)]	2.837
H(6)	H(10)[6)]	3.380	H(6)	H(14)[1)]	3.227
H(6)	H(16)[1)]	3.251	H(6)	H(22)[11)]	2.806
H(6)	H(24)	2.432	H(7)	N(1)	1.968
H(7)	C(3)	3.342	H(7)	C(3)[6)]	3.201
H(7)	C(4)	2.047	H(7)	C(4)[6)]	3.519
H(7)	C(6)[6)]	3.132	H(7)	C(7)[6)]	3.345
H(7)	C(9)	2.503	H(7)	C(11)[6)]	3.408
H(7)	C(14)	2.462	H(7)	C(15)	2.586
H(7)	H(10)	2.244	H(7)	H(11)[6)]	3.390
H(7)	H(12)[6)]	2.753	H(7)	H(20)	2.284
H(8)	C(3)	3.298	H(8)	C(6)	2.060
H(8)	C(8)	2.052	H(8)	C(9)	3.263
H(8)	C(10)	2.723	H(8)	C(13)	2.439
H(8)	C(14)[2)]	3.358	H(8)	C(15)[6)]	3.591
H(8)	C(18)[2)]	3.311	H(8)	C(19)[2)]	3.163
H(8)	H(9)	2.362	H(8)	H(17)	2.246
H(8)	H(18)	2.158	H(8)	H(19)	3.370

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(8)	H(20)[6)]	3.166	H(8)	H(21)[6)]	3.536
H(8)	H(23)[7)]	3.337	H(8)	H(23)[2)]	3.592
H(8)	H(24)[2)]	3.455	H(9)	C(1)[6)]	3.442
H(9)	C(2)[2)]	3.436	H(9)	C(4)	3.338
H(9)	C(6)	3.363	H(9)	C(7)	2.110
H(9)	C(9)	2.080	H(9)	C(15)[6)]	3.526
H(9)	C(17)[7)]	3.209	H(9)	C(18)[7)]	3.153
H(9)	C(19)[2)]	3.231	H(9)	H(3)[6)]	2.574
H(9)	H(5)[2)]	3.247	H(9)	H(6)[2)]	2.837
H(9)	H(8)	2.362	H(9)	H(10)	2.413
H(9)	H(20)[6)]	3.304	H(9)	H(22)[7)]	2.645
H(9)	H(23)[7)]	2.698	H(9)	H(24)[2)]	2.774
H(10)	O(1)[6)]	3.027	H(10)	N(1)[6)]	3.553
H(10)	C(2)[2)]	3.380	H(10)	C(3)	3.344
H(10)	C(3)[6)]	3.228	H(10)	C(4)	2.081
H(10)	C(4)[6)]	3.495	H(10)	C(5)	2.563
H(10)	C(5)[6)]	3.575	H(10)	C(7)	3.313
H(10)	C(8)	2.073	H(10)	H(3)[6)]	2.918
H(10)	H(5)[2)]	2.593	H(10)	H(6)[2)]	3.380
H(10)	H(7)	2.244	H(10)	H(9)	2.413
H(10)	H(11)[6)]	3.298	H(11)	O(1)	2.350
H(11)	C(3)	2.831	H(11)	C(6)	2.702
H(11)	C(10)	2.055	H(11)	C(12)	2.720
H(11)	C(13)	3.309	H(11)	H(1)[3)]	3.180
H(11)	H(4)[1)]	3.415	H(11)	H(7)[2)]	3.390
H(11)	H(10)[2)]	3.298	H(11)	H(12)	1.551
H(11)	H(13)	1.551	H(11)	H(14)	3.006
H(11)	H(15)	3.575	H(11)	H(16)	2.557
H(11)	H(17)	3.509	H(11)	H(19)	3.540
H(12)	O(1)	3.451	H(12)	C(3)	3.371
H(12)	C(6)	2.700	H(12)	C(7)	3.525
H(12)	C(10)	2.042	H(12)	C(12)	3.335
H(12)	C(13)	2.639	H(12)	C(15)[2)]	3.242
H(12)	H(7)[2)]	2.753	H(12)	H(11)	1.551
H(12)	H(13)	1.551	H(12)	H(14)	3.562
H(12)	H(15)[13)]	3.416	H(12)	H(16)	3.570
H(12)	H(17)	2.437	H(12)	H(18)	3.494

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	H(19)	2.942	H(12)	H(19)[14)]	3.488
H(12)	H(20)[2)]	2.968	H(13)	C(1)[3)]	3.542
H(13)	C(6)	3.347	H(13)	C(10)	2.059
H(13)	C(12)	2.694	H(13)	C(13)	2.638
H(13)	H(1)[3)]	3.085	H(13)	H(2)[3)]	3.386
H(13)	H(11)	1.551	H(13)	H(12)	1.551
H(13)	H(14)	2.536	H(13)	H(15)	3.559
H(13)	H(16)	2.957	H(13)	H(17)	2.873
H(13)	H(17)[14)]	3.438	H(13)	H(18)	3.516
H(13)	H(19)	2.483	H(13)	H(19)[14)]	3.466
H(13)	H(21)[9)]	3.136	H(13)	H(22)[9)]	3.084
H(14)	C(6)	3.331	H(14)	C(10)	2.067
H(14)	C(11)	2.710	H(14)	C(13)	2.631
H(14)	C(16)[9)]	3.411	H(14)	H(2)[1)]	3.394
H(14)	H(4)[1)]	3.428	H(14)	H(6)[1)]	3.227
H(14)	H(11)	3.006	H(14)	H(12)	3.562
H(14)	H(13)	2.536	H(14)	H(15)	1.551
H(14)	H(16)	1.551	H(14)	H(17)	3.497
H(14)	H(18)	2.911	H(14)	H(18)[8)]	3.000
H(14)	H(19)	2.428	H(14)	H(21)[9)]	2.497
H(14)	H(22)[9)]	3.495	H(15)	O(1)	3.479
H(15)	C(3)	3.297	H(15)	C(6)	2.654
H(15)	C(7)	3.453	H(15)	C(10)	2.057
H(15)	C(11)	3.335	H(15)	C(13)	2.674
H(15)	C(13)[8)]	3.458	H(15)	H(11)	3.575
H(15)	H(12)[5)]	3.416	H(15)	H(13)	3.559
H(15)	H(14)	1.551	H(15)	H(16)	1.551
H(15)	H(17)	3.547	H(15)	H(18)	2.509
H(15)	H(18)[8)]	3.038	H(15)	H(19)	2.919
H(15)	H(19)[8)]	3.017	H(15)	H(20)[6)]	3.231
H(15)	H(21)[6)]	3.474	H(16)	Al(1)	3.530
H(16)	O(1)	2.412	H(16)	C(2)[1)]	3.342
H(16)	C(3)	2.816	H(16)	C(6)	2.701
H(16)	C(10)	2.064	H(16)	C(11)	2.703
H(16)	C(13)	3.317	H(16)	H(1)[1)]	3.385
H(16)	H(2)[1)]	3.540	H(16)	H(4)	3.447
H(16)	H(4)[1)]	2.713	H(16)	H(5)	3.433

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(16)	H(6)[1)]	3.251	H(16)	H(11)	2.557
H(16)	H(12)	3.570	H(16)	H(13)	2.957
H(16)	H(14)	1.551	H(16)	H(15)	1.551
H(16)	H(18)	3.548	H(16)	H(19)	3.505
H(17)	C(6)	2.750	H(17)	C(7)	2.789
H(17)	C(10)	2.065	H(17)	C(11)	2.629
H(17)	C(12)	3.315	H(17)	C(15)[2)]	3.329
H(17)	C(16)[2)]	2.986	H(17)	C(17)[2)]	3.215
H(17)	H(8)	2.246	H(17)	H(11)	3.509
H(17)	H(12)	2.437	H(17)	H(13)	2.873
H(17)	H(13)[14)]	3.438	H(17)	H(14)	3.497
H(17)	H(15)	3.547	H(17)	H(18)	1.551
H(17)	H(19)	1.551	H(17)	H(19)[14)]	3.313
H(17)	H(21)[2)]	3.149	H(17)	H(22)[2)]	3.579
H(18)	C(6)	2.686	H(18)	C(7)	2.677
H(18)	C(10)	2.058	H(18)	C(11)	3.299
H(18)	C(12)	2.672	H(18)	C(12)[8)]	3.455
H(18)	H(8)	2.158	H(18)	H(12)	3.494
H(18)	H(13)	3.516	H(18)	H(14)	2.911
H(18)	H(14)[8)]	3.000	H(18)	H(15)	2.509
H(18)	H(15)[8)]	3.038	H(18)	H(16)	3.548
H(18)	H(17)	1.551	H(18)	H(18)[8)]	3.458
H(18)	H(19)	1.551	H(18)	H(19)[8)]	3.413
H(18)	H(21)[6)]	2.987	H(19)	C(6)	3.361
H(19)	C(10)	2.075	H(19)	C(11)	2.673
H(19)	C(12)	2.639	H(19)	C(16)[9)]	3.307
H(19)	H(8)	3.370	H(19)	H(11)	3.540
H(19)	H(12)	2.942	H(19)	H(12)[14)]	3.488
H(19)	H(13)	2.483	H(19)	H(13)[14)]	3.466
H(19)	H(14)	2.428	H(19)	H(15)	2.919
H(19)	H(15)[8)]	3.017	H(19)	H(16)	3.505
H(19)	H(17)	1.551	H(19)	H(17)[14)]	3.313
H(19)	H(18)	1.551	H(19)	H(18)[8)]	3.413
H(19)	H(19)[14)]	3.388	H(19)	H(21)[9)]	2.418
H(20)	N(1)	2.644	H(20)	C(5)	2.651
H(20)	C(6)[2)]	3.245	H(20)	C(7)[2)]	2.855
H(20)	C(8)[2)]	2.941	H(20)	C(9)[2)]	3.374

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(20)	C(14)	2.080	H(20)	C(16)	2.087
H(20)	C(17)	3.318	H(20)	C(19)	3.310
H(20)	H(7)	2.284	H(20)	H(8)[2)]	3.166
H(20)	H(9)[2)]	3.304	H(20)	H(12)[6)]	2.968
H(20)	H(15)[2)]	3.231	H(20)	H(21)	2.430
H(20)	H(23)[4)]	3.565	H(21)	C(12)[10)]	3.359
H(21)	C(13)[10)]	3.296	H(21)	C(14)	3.346
H(21)	C(15)	2.106	H(21)	C(17)	2.086
H(21)	C(18)	3.320	H(21)	H(8)[2)]	3.536
H(21)	H(13)[10)]	3.136	H(21)	H(14)[10)]	2.497
H(21)	H(15)[2)]	3.474	H(21)	H(17)[6)]	3.149
H(21)	H(18)[2)]	2.987	H(21)	H(19)[10)]	2.418
H(21)	H(20)	2.430	H(21)	H(22)	2.479
H(21)	H(23)[4)]	3.508	H(21)	H(24)[4)]	3.551
H(22)	C(1)[11)]	3.021	H(22)	C(15)	3.358
H(22)	C(16)	2.145	H(22)	C(18)	2.024
H(22)	C(19)	3.282	H(22)	H(1)[11)]	3.425
H(22)	H(2)[11)]	2.735	H(22)	H(3)[11)]	2.514
H(22)	H(6)[4)]	2.806	H(22)	H(9)[12)]	2.645
H(22)	H(13)[10)]	3.084	H(22)	H(14)[10)]	3.495
H(22)	H(17)[6)]	3.579	H(22)	H(21)	2.479
H(22)	H(23)	2.307	H(22)	H(23)[4)]	3.546
H(22)	H(24)[4)]	2.809	H(23)	C(8)[12)]	3.422
H(23)	C(14)	3.312	H(23)	C(14)[11)]	3.168
H(23)	C(15)[11)]	3.049	H(23)	C(16)	3.296
H(23)	C(16)[11)]	2.957	H(23)	C(17)	2.077
H(23)	C(17)[11)]	3.010	H(23)	C(18)[11)]	3.103
H(23)	C(19)	2.078	H(23)	C(19)[11)]	3.167
H(23)	H(2)[11)]	3.256	H(23)	H(3)[11)]	3.378
H(23)	H(8)[12)]	3.337	H(23)	H(8)[6)]	3.592
H(23)	H(9)[12)]	2.698	H(23)	H(20)[11)]	3.565
H(23)	H(21)[11)]	3.508	H(23)	H(22)	2.307
H(23)	H(22)[11)]	3.546	H(23)	H(24)	2.397
H(24)	Al(1)	2.987	H(24)	N(1)	2.645
H(24)	C(1)	3.501	H(24)	C(2)	3.065
H(24)	C(8)[6)]	3.330	H(24)	C(14)	2.113
H(24)	C(15)	3.351	H(24)	C(16)[11)]	3.445

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(24)	C(17)	3.338	H(24)	C(17)[11)]	3.031
H(24)	C(18)	2.105	H(24)	H(2)	3.019
H(24)	H(5)	3.582	H(24)	H(6)	2.432
H(24)	H(8)[6)]	3.455	H(24)	H(9)[6)]	2.774
H(24)	H(21)[11)]	3.551	H(24)	H(22)[11)]	2.809
H(24)	H(23)	2.397			

Symmetry Operators:

(1) -X+1,-Y,-Z+1
(2) -X+1/2,Y+1/2,-Z+1/2
(3) -X+1,-Y+1,-Z+1
(4) -X+1/2+1,Y+1/2,-Z+1/2
(5) X,Y-1,Z
(6) -X+1/2,Y+1/2-1,-Z+1/2
(7) X-1,Y,Z
(8) -X,-Y,-Z+1
(9) X+1/2-1,-Y+1/2,Z+1/2
(10) X+1/2,-Y+1/2,Z+1/2-1
(11) -X+1/2+1,Y+1/2-1,-Z+1/2
(12) X+1,Y,Z
(13) X,Y+1,Z
(14) -X,-Y+1,-Z+1

X-ray Structure Report for $Me_2Al[O\text{-}2\text{-}^{t}Bu\text{-}6\text{-}\{(C_6F_5)N{=}CH\}C_6H_3]$ (**2g**)

April 12, 2007

Experimental

Data Collection

A yellow block crystal of $C_{19}H_{19}F_5NOAl$ having approximate dimensions of 0.36 x 0.24 x 0.18 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 420 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 17.7482(6) Å
b = 15.6029(6) Å
c = 14.4394(5) Å
V = 3998.6(2) Å^3

For Z = 8 and F.W. = 399.34, the calculated density is 1.327 g/cm^3. The systematic absences of:

0kl: l ± 2n
h0l: l ± 2n
hk0: h+k ± 2n

uniquely determine the space group to be:

Pccn (#56)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 5.0^o step, at χ=45.0^o and ϕ = 120.0^o. The exposure rate was 180.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 5.0^o step, at χ=45.0^o and ϕ = 270.0^o. The exposure rate was 180.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 35873 reflections that were collected, 4559 were unique (R_{int} = 0.044); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.537 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.581 to 0.973. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2154 observed reflections and 263 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0352$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.0816$$

The standard deviation of an observation of unit weight[4] was 1.02. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.14 and -0.18 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{19}F_5NOAl$
Formula Weight	399.34
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.36 X 0.24 X 0.18 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 420.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 17.7482(6) Å b = 15.6029(6) Å c = 14.4394(5) Å V = 3998.6(2) Å^3
Space Group	Pccn (#56)
Z value	8
D_{calc}	1.327 g/cm^3
F_{000}	1648.00
μ(MoKα)	1.537 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=120.0)	130.0 - 190.0^{o}
Exposure Rate	180.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=270.0)	0.0 - 160.0^{o}
Exposure Rate	180.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 35873 Unique: 4559 (R_{int} = 0.044)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.581 - 0.973)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0006Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	2154
No. Variables	263
Reflection/Parameter Ratio	8.19
Residuals: R1 (I>2.00σ(I))	0.0352
Residuals: wR2 (I>2.00σ(I))	0.0816
Goodness of Fit Indicator	1.019
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.14 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.18 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.84228(4)	0.03619(5)	0.29047(5)	3.698(16)
F(1)	0.67698(8)	0.19804(10)	0.29928(11)	5.97(4)
F(2)	0.52840(9)	0.16606(13)	0.28553(13)	8.20(5)
F(3)	0.46918(8)	0.02225(13)	0.36306(13)	8.48(5)
F(4)	0.55909(10)	-0.08707(12)	0.45583(13)	7.98(5)
F(5)	0.70816(9)	-0.05554(11)	0.47045(11)	6.25(4)
O(1)	0.92481(8)	0.08163(11)	0.33934(12)	4.33(4)
N(1)	0.77600(9)	0.08578(12)	0.38668(13)	3.54(4)
C(1)	0.81113(13)	0.09012(18)	0.17470(17)	5.16(7)
C(2)	0.83875(15)	-0.08790(17)	0.2984(2)	5.78(7)
C(3)	0.80077(12)	0.13522(14)	0.45248(16)	3.49(5)
C(4)	0.87727(12)	0.16023(14)	0.46738(15)	3.41(5)
C(5)	0.89028(14)	0.21412(16)	0.54385(18)	4.67(6)
C(6)	0.96161(15)	0.24061(19)	0.56299(19)	5.66(7)
C(7)	1.02092(14)	0.21271(17)	0.50765(18)	4.90(6)
C(8)	1.01224(12)	0.15922(15)	0.43184(16)	3.61(5)
C(9)	0.93765(12)	0.13261(15)	0.41062(16)	3.36(5)
C(10)	0.69614(12)	0.07118(16)	0.38431(16)	3.56(5)
C(11)	0.64887(13)	0.12684(17)	0.33835(18)	4.25(6)
C(12)	0.57268(15)	0.1113(2)	0.3317(2)	5.16(7)
C(13)	0.54329(14)	0.0393(2)	0.3709(2)	5.51(7)
C(14)	0.58852(16)	-0.0164(2)	0.4178(2)	5.21(7)
C(15)	0.66438(14)	-0.00056(18)	0.42436(18)	4.34(6)
C(16)	1.08025(12)	0.12799(17)	0.37557(18)	4.22(6)
C(17)	1.07252(14)	0.15345(18)	0.27410(17)	4.97(7)
C(18)	1.08690(15)	0.03040(18)	0.3847(2)	6.51(8)
C(19)	1.15462(14)	0.1675(2)	0.4101(2)	6.63(8)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.8502	0.0839	0.1302	6.22
H(2)	0.7666	0.0635	0.1524	6.24
H(3)	0.8018	0.1493	0.1851	6.22
H(4)	0.8614	-0.1120	0.2448	6.97
H(5)	0.7879	-0.1065	0.3026	6.97
H(6)	0.8656	-0.1059	0.3519	6.98
H(7)	0.7643	0.1582	0.4982	4.16
H(8)	0.8460	0.2313	0.5816	5.60
H(9)	0.9688	0.2756	0.6187	6.78
H(10)	1.0708	0.2322	0.5235	5.86
H(11)	1.1159	0.1352	0.2410	5.96
H(12)	1.0676	0.2139	0.2690	5.97
H(13)	1.0291	0.1266	0.2489	5.97
H(14)	1.1302	0.0110	0.3522	7.81
H(15)	1.0432	0.0044	0.3592	7.81
H(16)	1.0912	0.0153	0.4482	7.83
H(17)	1.1954	0.1479	0.3732	7.95
H(18)	1.1628	0.1516	0.4728	7.95
H(19)	1.1512	0.2281	0.4058	7.98

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0410(3)	0.0532(4)	0.0464(4)	-0.0032(3)	-0.0002(3)	-0.0107(3)
F(1)	0.0696(9)	0.0574(9)	0.0999(12)	0.0079(7)	0.0016(8)	0.0067(9)
F(2)	0.0652(10)	0.1251(15)	0.1214(15)	0.0335(10)	-0.0207(10)	-0.0100(12)
F(3)	0.0431(8)	0.1673(19)	0.1120(14)	-0.0259(10)	0.0032(9)	-0.0433(13)
F(4)	0.0993(12)	0.1056(14)	0.0985(13)	-0.0505(11)	0.0276(11)	-0.0044(11)
F(5)	0.0812(10)	0.0803(11)	0.0758(10)	-0.0023(9)	0.0046(8)	0.0217(9)
O(1)	0.0414(8)	0.0655(11)	0.0577(10)	-0.0034(8)	-0.0014(8)	-0.0245(9)
N(1)	0.0386(10)	0.0493(12)	0.0468(11)	-0.0039(9)	-0.0011(9)	-0.0047(10)
C(1)	0.0551(15)	0.078(2)	0.0624(17)	-0.0031(14)	0.0032(13)	-0.0029(15)
C(2)	0.0731(18)	0.0654(19)	0.0809(19)	-0.0039(15)	0.0077(16)	-0.0133(16)
C(3)	0.0483(14)	0.0416(13)	0.0427(14)	0.0017(11)	0.0003(11)	-0.0017(12)
C(4)	0.0445(13)	0.0445(13)	0.0408(14)	-0.0026(10)	-0.0039(11)	-0.0043(11)
C(5)	0.0588(16)	0.0632(17)	0.0556(16)	-0.0072(13)	0.0015(13)	-0.0190(14)
C(6)	0.0703(17)	0.085(2)	0.0595(18)	-0.0146(16)	-0.0048(15)	-0.0285(17)
C(7)	0.0508(15)	0.0727(18)	0.0626(17)	-0.0124(13)	-0.0122(13)	-0.0075(15)
C(8)	0.0441(13)	0.0470(14)	0.0459(14)	-0.0007(11)	-0.0086(11)	0.0017(12)
C(9)	0.0443(13)	0.0412(13)	0.0423(14)	-0.0001(11)	-0.0062(11)	0.0015(12)
C(10)	0.0388(12)	0.0492(15)	0.0473(14)	-0.0007(11)	0.0015(11)	-0.0101(12)
C(11)	0.0482(15)	0.0504(16)	0.0630(17)	-0.0010(12)	0.0035(13)	-0.0092(14)
C(12)	0.0447(15)	0.081(2)	0.0702(19)	0.0134(15)	-0.0072(14)	-0.0176(17)
C(13)	0.0391(14)	0.100(2)	0.071(2)	-0.0117(17)	0.0051(14)	-0.0304(19)
C(14)	0.0611(18)	0.075(2)	0.0620(18)	-0.0269(16)	0.0144(15)	-0.0144(16)
C(15)	0.0555(16)	0.0600(16)	0.0494(15)	-0.0011(14)	0.0027(13)	-0.0032(14)
C(16)	0.0401(13)	0.0596(17)	0.0608(16)	0.0013(11)	-0.0090(12)	0.0032(14)
C(17)	0.0527(15)	0.0746(19)	0.0615(18)	0.0038(13)	0.0031(13)	-0.0001(15)
C(18)	0.0669(18)	0.066(2)	0.115(2)	0.0259(15)	0.0051(18)	0.0144(18)
C(19)	0.0447(15)	0.123(2)	0.084(2)	-0.0042(16)	-0.0110(14)	-0.009(2)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.7737(16)	Al(1)	N(1)	1.9780(19)
Al(1)	C(1)	1.951(2)	Al(1)	C(2)	1.941(2)
F(1)	C(11)	1.342(3)	F(2)	C(12)	1.338(3)
F(3)	C(13)	1.347(2)	F(4)	C(14)	1.338(3)
F(5)	C(15)	1.335(3)	O(1)	C(9)	1.321(2)
N(1)	C(3)	1.300(2)	N(1)	C(10)	1.436(2)
C(3)	C(4)	1.429(3)	C(4)	C(5)	1.407(3)
C(4)	C(9)	1.416(3)	C(5)	C(6)	1.360(3)
C(6)	C(7)	1.391(3)	C(7)	C(8)	1.385(3)
C(8)	C(9)	1.421(3)	C(8)	C(16)	1.534(3)
C(10)	C(11)	1.378(3)	C(10)	C(15)	1.380(3)
C(11)	C(12)	1.377(3)	C(12)	C(13)	1.362(4)
C(13)	C(14)	1.364(4)	C(14)	C(15)	1.372(3)
C(16)	C(17)	1.524(3)	C(16)	C(18)	1.533(3)
C(16)	C(19)	1.540(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(3)	H(7)	0.991	C(5)	H(8)	0.993
C(6)	H(9)	0.981	C(7)	H(10)	0.964
C(17)	H(11)	0.950	C(17)	H(12)	0.950
C(17)	H(13)	0.950	C(18)	H(14)	0.950
C(18)	H(15)	0.950	C(18)	H(16)	0.950
C(19)	H(17)	0.950	C(19)	H(18)	0.950
C(19)	H(19)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	93.17(7)	O(1)	Al(1)	C(1)	113.73(10)
O(1)	Al(1)	C(2)	113.71(10)	N(1)	Al(1)	C(1)	105.34(9)
N(1)	Al(1)	C(2)	109.26(10)	C(1)	Al(1)	C(2)	118.12(12)
Al(1)	O(1)	C(9)	133.89(14)	Al(1)	N(1)	C(3)	122.97(14)
Al(1)	N(1)	C(10)	120.55(14)	C(3)	N(1)	C(10)	116.44(18)
N(1)	C(3)	C(4)	126.4(2)	C(3)	C(4)	C(5)	115.9(2)
C(3)	C(4)	C(9)	123.3(2)	C(5)	C(4)	C(9)	120.8(2)
C(4)	C(5)	C(6)	119.6(2)	C(5)	C(6)	C(7)	119.5(2)
C(6)	C(7)	C(8)	123.9(2)	C(7)	C(8)	C(9)	116.8(2)
C(7)	C(8)	C(16)	121.5(2)	C(9)	C(8)	C(16)	121.7(2)
O(1)	C(9)	C(4)	120.25(18)	O(1)	C(9)	C(8)	120.32(19)
C(4)	C(9)	C(8)	119.4(2)	N(1)	C(10)	C(11)	120.8(2)
N(1)	C(10)	C(15)	121.5(2)	C(11)	C(10)	C(15)	117.7(2)
F(1)	C(11)	C(10)	119.9(2)	F(1)	C(11)	C(12)	118.8(2)
C(10)	C(11)	C(12)	121.4(2)	F(2)	C(12)	C(11)	119.9(2)
F(2)	C(12)	C(13)	120.6(2)	C(11)	C(12)	C(13)	119.5(2)
F(3)	C(13)	C(12)	120.2(2)	F(3)	C(13)	C(14)	119.4(2)
C(12)	C(13)	C(14)	120.5(2)	F(4)	C(14)	C(13)	120.0(2)
F(4)	C(14)	C(15)	120.2(2)	C(13)	C(14)	C(15)	119.8(2)
F(5)	C(15)	C(10)	119.5(2)	F(5)	C(15)	C(14)	119.3(2)
C(10)	C(15)	C(14)	121.2(2)	C(8)	C(16)	C(17)	110.82(19)
C(8)	C(16)	C(18)	109.3(2)	C(8)	C(16)	C(19)	112.1(2)
C(17)	C(16)	C(18)	110.4(2)	C(17)	C(16)	C(19)	106.5(2)
C(18)	C(16)	C(19)	107.7(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.2	Al(1)	C(1)	H(2)	109.8
Al(1)	C(1)	H(3)	109.4	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.5	Al(1)	C(2)	H(5)	109.9
Al(1)	C(2)	H(6)	109.0	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
N(1)	C(3)	H(7)	118.7	C(4)	C(3)	H(7)	114.9
C(4)	C(5)	H(8)	117.5	C(6)	C(5)	H(8)	122.9
C(5)	C(6)	H(9)	117.2	C(7)	C(6)	H(9)	123.2
C(6)	C(7)	H(10)	117.3	C(8)	C(7)	H(10)	118.7
C(16)	C(17)	H(11)	109.4	C(16)	C(17)	H(12)	110.0
C(16)	C(17)	H(13)	109.0	H(11)	C(17)	H(12)	109.5
H(11)	C(17)	H(13)	109.5	H(12)	C(17)	H(13)	109.5
C(16)	C(18)	H(14)	109.7	C(16)	C(18)	H(15)	109.1
C(16)	C(18)	H(16)	109.6	H(14)	C(18)	H(15)	109.5
H(14)	C(18)	H(16)	109.5	H(15)	C(18)	H(16)	109.5
C(16)	C(19)	H(17)	110.0	C(16)	C(19)	H(18)	109.6
C(16)	C(19)	H(19)	108.8	H(17)	C(19)	H(18)	109.5
H(17)	C(19)	H(19)	109.5	H(18)	C(19)	H(19)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(3)	2.22(19)	O(1)	Al(1)	N(1)	C(10)	179.51(17)
N(1)	Al(1)	O(1)	C(9)	-2.6(2)	C(1)	Al(1)	O(1)	C(9)	105.6(2)
C(2)	Al(1)	O(1)	C(9)	-115.3(2)	C(1)	Al(1)	N(1)	C(3)	-113.45(19)
C(1)	Al(1)	N(1)	C(10)	63.85(19)	C(2)	Al(1)	N(1)	C(3)	118.71(19)
C(2)	Al(1)	N(1)	C(10)	-64.00(19)	Al(1)	O(1)	C(9)	C(4)	1.7(3)
Al(1)	O(1)	C(9)	C(8)	-179.21(17)	Al(1)	N(1)	C(3)	C(4)	-1.2(3)
Al(1)	N(1)	C(10)	C(11)	-90.8(2)	Al(1)	N(1)	C(10)	C(15)	86.5(2)
C(3)	N(1)	C(10)	C(11)	86.7(2)	C(3)	N(1)	C(10)	C(15)	-96.1(2)
C(10)	N(1)	C(3)	C(4)	-178.6(2)	N(1)	C(3)	C(4)	C(5)	-179.7(2)
N(1)	C(3)	C(4)	C(9)	-0.5(3)	C(3)	C(4)	C(5)	C(6)	179.8(2)
C(3)	C(4)	C(9)	O(1)	0.5(3)	C(3)	C(4)	C(9)	C(8)	-178.6(2)
C(5)	C(4)	C(9)	O(1)	179.6(2)	C(5)	C(4)	C(9)	C(8)	0.5(3)
C(9)	C(4)	C(5)	C(6)	0.6(3)	C(4)	C(5)	C(6)	C(7)	-1.2(3)
C(5)	C(6)	C(7)	C(8)	0.8(4)	C(6)	C(7)	C(8)	C(9)	0.3(3)
C(6)	C(7)	C(8)	C(16)	-177.7(2)	C(7)	C(8)	C(9)	O(1)	179.96(15)
C(7)	C(8)	C(9)	C(4)	-0.9(3)	C(7)	C(8)	C(16)	C(17)	-122.4(2)
C(7)	C(8)	C(16)	C(18)	115.7(2)	C(7)	C(8)	C(16)	C(19)	-3.6(3)
C(9)	C(8)	C(16)	C(17)	59.6(2)	C(9)	C(8)	C(16)	C(18)	-62.2(2)
C(9)	C(8)	C(16)	C(19)	178.4(2)	C(16)	C(8)	C(9)	O(1)	-2.0(3)
C(16)	C(8)	C(9)	C(4)	177.1(2)	N(1)	C(10)	C(11)	F(1)	-3.5(3)
N(1)	C(10)	C(11)	C(12)	176.8(2)	N(1)	C(10)	C(15)	F(5)	3.5(3)
N(1)	C(10)	C(15)	C(14)	-176.6(2)	C(11)	C(10)	C(15)	F(5)	-179.2(2)
C(11)	C(10)	C(15)	C(14)	0.7(3)	C(15)	C(10)	C(11)	F(1)	179.2(2)
C(15)	C(10)	C(11)	C(12)	-0.6(3)	F(1)	C(11)	C(12)	F(2)	1.2(3)
F(1)	C(11)	C(12)	C(13)	179.9(2)	C(10)	C(11)	C(12)	F(2)	-179.1(2)
C(10)	C(11)	C(12)	C(13)	-0.3(4)	F(2)	C(12)	C(13)	F(3)	0.4(4)
F(2)	C(12)	C(13)	C(14)	179.9(2)	C(11)	C(12)	C(13)	F(3)	-178.4(2)
C(11)	C(12)	C(13)	C(14)	1.1(4)	F(3)	C(13)	C(14)	F(4)	-0.4(4)
F(3)	C(13)	C(14)	C(15)	178.5(2)	C(12)	C(13)	C(14)	F(4)	-179.9(2)
C(12)	C(13)	C(14)	C(15)	-1.0(4)	F(4)	C(14)	C(15)	F(5)	-1.1(4)
F(4)	C(14)	C(15)	C(10)	178.9(2)	C(13)	C(14)	C(15)	F(5)	179.96(18)
C(13)	C(14)	C(15)	C(10)	0.0(3)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Al(1)	F(3)[1)]	3.2892(18)	F(1)	F(1)[2)]	3.057(2)
F(1)	C(3)[2)]	3.438(2)	F(1)	C(4)[2)]	3.422(2)
F(2)	C(6)[3)]	3.422(3)	F(2)	C(8)[2)]	3.524(3)
F(2)	C(17)[2)]	3.342(3)	F(3)	Al(1)[4)]	3.2892(18)
F(3)	F(4)[5)]	2.848(2)	F(3)	O(1)[4)]	3.433(2)
F(3)	C(1)[4)]	3.353(2)	F(3)	C(2)[4)]	3.441(3)
F(3)	C(14)[5)]	3.327(3)	F(4)	F(3)[5)]	2.848(2)
F(4)	C(6)[6)]	3.209(3)	F(4)	C(7)[6)]	3.240(3)
F(4)	C(13)[5)]	3.181(3)	F(4)	C(14)[5)]	3.578(3)
F(4)	C(17)[4)]	3.486(3)	F(5)	C(19)[7)]	3.458(3)
O(1)	F(3)[1)]	3.433(2)	C(1)	F(3)[1)]	3.353(2)
C(2)	F(3)[1)]	3.441(3)	C(3)	F(1)[2)]	3.438(2)
C(4)	F(1)[2)]	3.422(2)	C(6)	F(2)[8)]	3.422(3)
C(6)	F(4)[9)]	3.209(3)	C(7)	F(4)[9)]	3.240(3)
C(8)	F(2)[2)]	3.524(3)	C(13)	F(4)[5)]	3.181(3)
C(14)	F(3)[5)]	3.327(3)	C(14)	F(4)[5)]	3.578(3)
C(14)	C(17)[4)]	3.511(3)	C(17)	F(2)[2)]	3.342(3)
C(17)	F(4)[1)]	3.486(3)	C(17)	C(14)[1)]	3.511(3)
C(19)	F(5)[7)]	3.458(3)			

Symmetry Operators:

(1) X+1/2,-Y,-Z+1/2
(2) -X+1/2+1,-Y+1/2,Z
(3) -X+1/2+1,Y,Z+1/2-1
(4) X+1/2-1,-Y,-Z+1/2
(5) -X+1,-Y,-Z+1
(6) X+1/2-1,Y+1/2-1,-Z+1
(7) -X+2,-Y,-Z+1
(8) -X+1/2+1,Y,Z+1/2
(9) X+1/2,Y+1/2,-Z+1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F(1)	H(3)[1)]	2.921	F(1)	H(8)[2)]	3.211
F(2)	H(4)[3)]	3.112	F(2)	H(9)[2)]	2.953
F(2)	H(12)[1)]	2.543	F(2)	H(13)[1)]	3.434
F(2)	H(15)[3)]	3.393	F(3)	H(1)[3)]	2.686
F(3)	H(4)[3)]	2.836	F(3)	H(13)[3)]	3.023
F(3)	H(15)[3)]	3.492	F(4)	H(9)[4)]	2.885
F(4)	H(10)[4)]	2.843	F(4)	H(11)[3)]	3.108
F(4)	H(13)[3)]	3.067	F(5)	H(1)[5)]	3.336
F(5)	H(2)[5)]	3.248	F(5)	H(17)[6)]	3.179
F(5)	H(18)[6)]	2.857	O(1)	H(16)[6)]	3.432
C(1)	H(7)[2)]	3.069	C(1)	H(8)[7)]	3.155
C(1)	H(9)[7)]	3.588	C(1)	H(14)[3)]	3.599
C(2)	H(18)[6)]	3.451	C(3)	H(2)[5)]	3.318
C(3)	H(7)[1)]	3.487	C(3)	H(16)[6)]	3.354
C(4)	H(16)[6)]	3.050	C(5)	H(1)[8)]	3.462
C(5)	H(3)[8)]	3.342	C(5)	H(7)[1)]	3.455
C(5)	H(16)[6)]	3.597	C(6)	H(1)[8)]	3.514
C(6)	H(12)[8)]	3.590	C(6)	H(13)[8)]	3.597
C(7)	H(6)[6)]	3.309	C(9)	H(16)[6)]	3.122
C(11)	H(14)[3)]	3.507	C(12)	H(14)[3)]	3.426
C(12)	H(15)[3)]	3.337	C(13)	H(11)[3)]	3.419
C(13)	H(13)[3)]	3.124	C(13)	H(15)[3)]	3.392
C(14)	H(11)[3)]	2.988	C(14)	H(13)[3)]	3.140
C(15)	H(1)[5)]	3.262	C(15)	H(11)[3)]	3.295
C(17)	H(9)[7)]	3.106	C(18)	H(2)[9)]	3.550
C(19)	H(6)[6)]	3.587	H(1)	F(3)[9)]	2.686
H(1)	F(5)[2)]	3.336	H(1)	C(5)[7)]	3.462
H(1)	C(6)[7)]	3.514	H(1)	C(15)[2)]	3.262
H(1)	H(7)[2)]	3.018	H(1)	H(8)[7)]	2.968
H(1)	H(9)[7)]	3.044	H(2)	F(5)[2)]	3.248
H(2)	C(3)[2)]	3.318	H(2)	C(18)[3)]	3.550
H(2)	H(7)[2)]	2.728	H(2)	H(8)[2)]	3.448
H(2)	H(14)[3)]	2.685	H(2)	H(17)[3)]	3.550
H(3)	F(1)[1)]	2.921	H(3)	C(5)[7)]	3.342
H(3)	H(7)[2)]	2.947	H(3)	H(8)[2)]	3.279
H(3)	H(8)[7)]	2.514	H(3)	H(9)[7)]	3.329
H(4)	F(2)[9)]	3.112	H(4)	F(3)[9)]	2.836

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(4)	H(12)[10)]	3.001	H(4)	H(17)[3)]	3.449
H(4)	H(19)[10)]	3.316	H(5)	H(11)[3)]	3.149
H(5)	H(17)[3)]	3.090	H(5)	H(18)[6)]	3.432
H(6)	C(7)[6)]	3.309	H(6)	C(19)[6)]	3.587
H(6)	H(10)[6)]	2.898	H(6)	H(12)[10)]	3.515
H(6)	H(16)[6)]	3.305	H(6)	H(18)[6)]	2.678
H(7)	C(1)[5)]	3.069	H(7)	C(3)[1)]	3.487
H(7)	C(5)[1)]	3.455	H(7)	H(1)[5)]	3.018
H(7)	H(2)[5)]	2.728	H(7)	H(3)[5)]	2.947
H(7)	H(7)[1)]	2.909	H(7)	H(8)[1)]	2.874
H(8)	F(1)[5)]	3.211	H(8)	C(1)[8)]	3.155
H(8)	H(1)[8)]	2.968	H(8)	H(2)[5)]	3.448
H(8)	H(3)[5)]	3.279	H(8)	H(3)[8)]	2.514
H(8)	H(7)[1)]	2.874	H(8)	H(8)[1)]	3.458
H(9)	F(2)[5)]	2.953	H(9)	F(4)[11)]	2.885
H(9)	C(1)[8)]	3.588	H(9)	C(17)[8)]	3.106
H(9)	H(1)[8)]	3.044	H(9)	H(3)[8)]	3.329
H(9)	H(11)[8)]	3.446	H(9)	H(12)[8)]	2.794
H(9)	H(13)[8)]	2.648	H(10)	F(4)[11)]	2.843
H(10)	H(6)[6)]	2.898	H(11)	F(4)[9)]	3.108
H(11)	C(13)[9)]	3.419	H(11)	C(14)[9)]	2.988
H(11)	C(15)[9)]	3.295	H(11)	H(5)[9)]	3.149
H(11)	H(9)[7)]	3.446	H(12)	F(2)[1)]	2.543
H(12)	C(6)[7)]	3.590	H(12)	H(4)[12)]	3.001
H(12)	H(6)[12)]	3.515	H(12)	H(9)[7)]	2.794
H(13)	F(2)[1)]	3.434	H(13)	F(3)[9)]	3.023
H(13)	F(4)[9)]	3.067	H(13)	C(6)[7)]	3.597
H(13)	C(13)[9)]	3.124	H(13)	C(14)[9)]	3.140
H(13)	H(9)[7)]	2.648	H(14)	C(1)[9)]	3.599
H(14)	C(11)[9)]	3.507	H(14)	C(12)[9)]	3.426
H(14)	H(2)[9)]	2.685	H(15)	F(2)[9)]	3.393
H(15)	F(3)[9)]	3.492	H(15)	C(12)[9)]	3.337
H(15)	C(13)[9)]	3.392	H(16)	O(1)[6)]	3.432
H(16)	C(3)[6)]	3.354	H(16)	C(4)[6)]	3.050
H(16)	C(5)[6)]	3.597	H(16)	C(9)[6)]	3.122
H(16)	H(6)[6)]	3.305	H(16)	H(16)[6)]	3.597
H(17)	F(5)[6)]	3.179	H(17)	H(2)[9)]	3.550

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(17)	H(4)[9)]	3.449	H(17)	H(5)[9)]	3.090
H(17)	H(19)[13)]	3.373	H(18)	F(5)[6)]	2.857
H(18)	C(2)[6)]	3.451	H(18)	H(5)[6)]	3.432
H(18)	H(6)[6)]	2.678	H(19)	H(4)[12)]	3.316
H(19)	H(17)[13)]	3.373	H(19)	H(19)[13)]	3.573

Symmetry Operators:

(1) -X+1/2+1,-Y+1/2,Z
(2) -X+1/2+1,Y,Z+1/2-1
(3) X+1/2-1,-Y,-Z+1/2
(4) X+1/2-1,Y+1/2-1,-Z+1
(5) -X+1/2+1,Y,Z+1/2
(6) -X+2,-Y,-Z+1
(7) X,-Y+1/2,Z+1/2-1
(8) X,-Y+1/2,Z+1/2
(9) X+1/2,-Y,-Z+1/2
(10) -X+2,Y+1/2-1,-Z+1/2
(11) X+1/2,Y+1/2,-Z+1
(12) -X+2,Y+1/2,-Z+1/2
(13) -X+1/2+2,-Y+1/2,Z

X-ray Structure Report for $Me_2Al[\mu_2\text{-}O\text{-}2\text{-}\{(2,6\text{-}^{i}Pr_2C_6H_3)N{=}CH\}C_6H_4](AlMe_3)$ (**3a**)

April 8, 2007

Experimental

Data Collection

A colorless block crystal of $C_{150}H_{222}O_6Al_{12}N_6$ having approximate dimensions of 0.40 x 0.24 x 0.14 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 0 oscillations that were exposed for 0 seconds. The crystal-to-detector distance was 0.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a R-centered trigonal cell (laue class: -3) with dimensions:

a = 38.5672(11) Å
c = 9.3498(3) Å
V = 12044.0(6) Å^3

For Z = 3 and F.W. = 2529.22, the calculated density is 1.046 g/cm^3. Based on the systematic absences of:

hkil: -h+k+l ± 3n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

R-3 (#148)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.8^o. A total of 0 oscillation images were collected. The crystal-to-detector distance was 0.00 mm. Readout was performed in the 0.000 mm pixel mode.

Data Reduction

Of the 58936 reflections that were collected, 6067 were unique (R_{int} = 0.040); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.226 cm^{-1}. was applied which resulted in transmission factors ranging from 0.818 to 0.983. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[1] and expanded using Fourier techniques[2]. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 3181 observed reflections and 299 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0399$$

$$wR2 = [\, \Sigma \, (\, w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.1007$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.29 and -0.18 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

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(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2006). 9009 New Trails Dr. The Woodlands TX 77381 USA.

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{150}H_{222}O_6Al_{12}N_6$
Formula Weight	2529.22
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.40 X 0.24 X 0.14 mm
Crystal System	trigonal
Lattice Type	R-centered
Detector Position	0.00 mm
Pixel Size	0.000 mm
Lattice Parameters	a = 38.5672(11) Å c = 9.3498(3) Å V = 12044.0(6) Å^3
Space Group	R-3 (#148)
Z value	3
D_{calc}	1.046 g/cm^3
F_{000}	4104.00
μ(MoKα)	1.226 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	0 mm x 0 mm
Data Images	0 exposures
Detector Position	0.00 mm
Pixel Size	0.000 mm
$2\theta_{max}$	54.8^{o}
No. of Reflections Measured	Total: 58936 Unique: 6067 (R_{int} = 0.040)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.818 - 0.983)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF99 PATTY)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0011Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.8^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	3181
No. Variables	299
Reflection/Parameter Ratio	10.64
Residuals: R1 (I>2.00σ(I))	0.0399
Residuals: wR2 (I>2.00σ(I))	0.1007
Goodness of Fit Indicator	1.013
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.29 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.18 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
Al(1)	0.43311(2)	0.14048(2)	0.08157(7)	3.178(15)	
Al(2)	0.52212(2)	0.16992(2)	-0.08220(7)	3.801(17)	
O(1)	0.48729(5)	0.17610(4)	0.05822(14)	3.28(3)	
N(1)	0.43975(5)	0.13668(5)	0.29048(17)	2.75(3)	
C(1)	0.41306(8)	0.08921(8)	-0.0135(2)	4.64(6)	
C(2)	0.40500(8)	0.16993(8)	0.0538(2)	4.83(7)	
C(3)	0.50143(7)	0.21004(7)	0.1427(2)	3.05(5)	
C(4)	0.52389(8)	0.24787(7)	0.0842(2)	4.02(5)	
C(5)	0.53956(8)	0.28118(8)	0.1708(2)	4.61(6)	
C(6)	0.53343(8)	0.27775(8)	0.3176(2)	4.47(6)	
C(7)	0.51030(7)	0.24045(7)	0.3763(2)	3.66(5)	
C(8)	0.49295(6)	0.20580(6)	0.2906(2)	2.79(4)	
C(9)	0.46616(7)	0.16792(7)	0.3578(2)	2.88(4)	
C(10)	0.49526(9)	0.16890(10)	-0.2620(2)	5.68(7)	
C(11)	0.57697(8)	0.21576(9)	-0.0607(2)	5.32(7)	
C(12)	0.52117(10)	0.12018(9)	-0.0310(3)	6.10(8)	
C(13)	0.41376(6)	0.10128(6)	0.3758(2)	3.03(4)	
C(14)	0.38146(7)	0.09989(7)	0.4503(2)	3.64(5)	
C(15)	0.35704(8)	0.06494(8)	0.5282(2)	4.70(6)	
C(16)	0.36488(9)	0.03366(9)	0.5321(2)	5.18(6)	
C(17)	0.39714(8)	0.03626(7)	0.4589(2)	4.53(6)	
C(18)	0.42207(7)	0.06987(7)	0.3770(2)	3.53(5)	
C(19)	0.37287(8)	0.13421(8)	0.4529(2)	4.33(6)	
C(20)	0.38166(10)	0.15348(9)	0.6012(3)	6.21(8)	
C(21)	0.32986(10)	0.12074(12)	0.4099(3)	7.44(10)	
C(22)	0.45842(8)	0.07346(8)	0.3012(2)	4.25(6)	
C(23)	0.49429(9)	0.09047(11)	0.4018(3)	6.96(9)	
C(24)	0.45046(11)	0.03365(9)	0.2362(4)	7.59(10)	
C(25)	0.6667	0.3333	0.003(3)	19.4(9)	0.220
C(26)	0.6667	0.3333	0.104(4)	20.0(10)	0.200
C(27)	0.6667	0.3333	0.3333	24.5(27)	0.080
C(29)	0.6871(6)	0.3431(10)	0.2230(19)	21.6(9)	1/2

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
H(1)	0.4335	0.0823	-0.0152	5.59	
H(2)	0.3905	0.0693	0.0371	5.60	
H(3)	0.4055	0.0910	-0.1087	5.59	
H(4)	0.4185	0.1946	0.1039	6.19	
H(5)	0.4042	0.1750	-0.0453	6.20	
H(6)	0.3785	0.1546	0.0893	6.20	
H(7)	0.5281	0.2490	-0.0281	4.68	
H(8)	0.5568	0.3103	0.1354	5.30	
H(9)	0.5497	0.3055	0.3842	5.18	
H(10)	0.5042	0.2352	0.4879	4.35	
H(11)	0.4698	0.1660	0.4666	3.52	
H(12)	0.4688	0.1467	-0.2617	6.97	
H(13)	0.4944	0.1930	-0.2704	6.99	
H(14)	0.5096	0.1666	-0.3406	6.97	
H(15)	0.5958	0.2078	-0.0871	6.40	
H(16)	0.5803	0.2372	-0.1203	6.39	
H(17)	0.5812	0.2243	0.0362	6.39	
H(18)	0.5420	0.1188	-0.0800	7.91	
H(19)	0.5250	0.1198	0.0693	7.90	
H(20)	0.4961	0.0979	-0.0570	7.91	
H(21)	0.3324	0.0632	0.5916	5.27	
H(22)	0.3455	0.0087	0.5981	5.55	
H(23)	0.4001	0.0097	0.4540	5.22	
H(24)	0.3939	0.1575	0.3704	5.44	
H(25)	0.3788	0.1766	0.6005	7.43	
H(26)	0.3635	0.1347	0.6684	7.45	
H(27)	0.4083	0.1609	0.6274	7.45	
H(28)	0.3258	0.1431	0.4057	9.64	
H(29)	0.3246	0.1082	0.3187	9.65	
H(30)	0.3123	0.1021	0.4787	9.65	
H(31)	0.4636	0.0927	0.2192	5.44	
H(32)	0.5166	0.0915	0.3548	8.56	
H(33)	0.5008	0.1167	0.4306	8.57	
H(34)	0.4876	0.0737	0.4836	8.57	
H(35)	0.4718	0.0378	0.1746	9.80	
H(36)	0.4479	0.0157	0.3106	9.82	
H(37)	0.4262	0.0225	0.1832	9.82	

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} and occupancy (continued)

atom	x	y	z	B_{eq}	occ

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0477(4)	0.0428(4)	0.0326(3)	0.0244(3)	-0.0053(2)	-0.0041(2)
Al(2)	0.0561(4)	0.0623(5)	0.0321(3)	0.0341(4)	0.0026(3)	-0.0015(3)
O(1)	0.0516(9)	0.0410(9)	0.0307(7)	0.0223(8)	0.0030(6)	-0.0016(6)
N(1)	0.0365(10)	0.0376(10)	0.0325(9)	0.0200(9)	0.0009(7)	0.0028(8)
C(1)	0.0636(17)	0.0555(16)	0.0563(15)	0.0289(14)	-0.0081(13)	-0.0127(12)
C(2)	0.0707(19)	0.0692(18)	0.0553(15)	0.0438(16)	-0.0162(13)	-0.0092(13)
C(3)	0.0429(13)	0.0409(13)	0.0347(11)	0.0228(11)	-0.0013(9)	-0.0009(9)
C(4)	0.0612(16)	0.0439(15)	0.0431(13)	0.0229(13)	0.0067(11)	0.0092(11)
C(5)	0.0630(17)	0.0376(15)	0.0668(17)	0.0193(13)	0.0029(13)	0.0075(12)
C(6)	0.0599(17)	0.0413(15)	0.0618(16)	0.0202(13)	-0.0050(13)	-0.0071(12)
C(7)	0.0499(14)	0.0457(15)	0.0417(12)	0.0226(12)	-0.0021(10)	-0.0050(10)
C(8)	0.0383(12)	0.0383(12)	0.0316(10)	0.0207(10)	-0.0025(9)	0.0000(9)
C(9)	0.0390(12)	0.0419(13)	0.0310(10)	0.0222(11)	-0.0007(9)	0.0008(9)
C(10)	0.080(2)	0.102(2)	0.0382(13)	0.0483(19)	0.0004(13)	-0.0020(14)
C(11)	0.0630(18)	0.088(2)	0.0509(15)	0.0377(16)	0.0115(13)	0.0012(14)
C(12)	0.079(2)	0.083(2)	0.088(2)	0.0542(19)	0.0141(16)	0.0026(17)
C(13)	0.0371(12)	0.0372(13)	0.0353(10)	0.0145(11)	-0.0011(9)	0.0053(9)
C(14)	0.0416(14)	0.0521(15)	0.0402(12)	0.0201(12)	0.0026(10)	0.0022(11)
C(15)	0.0493(16)	0.0607(18)	0.0568(15)	0.0187(14)	0.0125(12)	0.0104(13)
C(16)	0.0592(18)	0.0538(18)	0.0618(16)	0.0119(15)	0.0088(13)	0.0198(13)
C(17)	0.0611(17)	0.0416(15)	0.0616(15)	0.0199(13)	-0.0030(13)	0.0117(12)
C(18)	0.0459(14)	0.0415(14)	0.0440(12)	0.0198(12)	-0.0044(10)	0.0046(10)
C(19)	0.0511(16)	0.0644(17)	0.0564(15)	0.0343(14)	0.0111(12)	0.0036(13)
C(20)	0.085(2)	0.070(2)	0.080(2)	0.0383(18)	0.0045(17)	-0.0140(16)
C(21)	0.078(2)	0.112(2)	0.115(2)	0.064(2)	-0.020(2)	-0.025(2)
C(22)	0.0572(16)	0.0514(16)	0.0645(16)	0.0357(14)	0.0037(13)	0.0113(12)
C(23)	0.0582(19)	0.113(2)	0.100(2)	0.0478(19)	0.0042(16)	0.034(2)
C(24)	0.115(3)	0.070(2)	0.126(2)	0.063(2)	0.033(2)	0.0111(19)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.8522(16)	Al(1)	N(1)	1.9849(17)
Al(1)	C(1)	1.941(2)	Al(1)	C(2)	1.940(3)
Al(2)	O(1)	1.9761(19)	Al(2)	C(10)	1.965(2)
Al(2)	C(11)	1.975(2)	Al(2)	C(12)	1.960(3)
O(1)	C(3)	1.386(2)	N(1)	C(9)	1.288(2)
N(1)	C(13)	1.462(2)	C(3)	C(4)	1.384(3)
C(3)	C(8)	1.412(2)	C(4)	C(5)	1.377(3)
C(5)	C(6)	1.387(3)	C(6)	C(7)	1.372(3)
C(7)	C(8)	1.407(3)	C(8)	C(9)	1.445(2)
C(13)	C(14)	1.405(3)	C(13)	C(18)	1.399(4)
C(14)	C(15)	1.402(3)	C(14)	C(19)	1.517(4)
C(15)	C(16)	1.383(5)	C(16)	C(17)	1.379(4)
C(17)	C(18)	1.395(3)	C(18)	C(22)	1.514(4)
C(19)	C(20)	1.529(3)	C(19)	C(21)	1.524(4)
C(22)	C(23)	1.524(4)	C(22)	C(24)	1.533(4)
C(25)	C(26)	0.94(5)	C(26)	C(29)	1.31(4)
C(26)	C(29)[1)]	1.31(4)	C(26)	C(29)[2)]	1.31(4)
C(27)	C(29)	1.238(19)	C(27)	C(29)[1)]	1.24(2)
C(27)	C(29)[2)]	1.24(2)	C(27)	C(29)[3)]	1.238(19)
C(27)	C(29)[4)]	1.24(2)	C(27)	C(29)[5)]	1.24(2)
C(29)	C(29)[1)]	1.18(4)	C(29)	C(29)[2)]	1.18(3)

Symmetry Operators:

(1) -Y+1,X-Y,Z (2) -X+Y+1,-X+1,Z
(3) -X+1/3+1,-Y+2/3,-Z+2/3 (4) Y+1/3,-X+Y+2/3,-Z+2/3
(5) X-Y+1/3,X+2/3-1,-Z+2/3

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(4)	H(7)	1.061	C(5)	H(8)	1.032
C(6)	H(9)	1.122	C(7)	H(10)	1.067
C(9)	H(11)	1.034	C(10)	H(12)	0.950
C(10)	H(13)	0.950	C(10)	H(14)	0.950
C(11)	H(15)	0.950	C(11)	H(16)	0.950
C(11)	H(17)	0.950	C(12)	H(18)	0.950
C(12)	H(19)	0.950	C(12)	H(20)	0.950
C(15)	H(21)	1.094	C(16)	H(22)	1.072
C(17)	H(23)	1.088	C(19)	H(24)	1.154
C(20)	H(25)	0.950	C(20)	H(26)	0.950
C(20)	H(27)	0.950	C(21)	H(28)	0.950
C(21)	H(29)	0.950	C(21)	H(30)	0.950
C(22)	H(31)	1.014	C(23)	H(32)	0.950
C(23)	H(33)	0.950	C(23)	H(34)	0.950
C(24)	H(35)	0.950	C(24)	H(36)	0.950
C(24)	H(37)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	91.80(6)	O(1)	Al(1)	C(1)	115.38(11)
O(1)	Al(1)	C(2)	107.66(9)	N(1)	Al(1)	C(1)	112.07(10)
N(1)	Al(1)	C(2)	107.79(10)	C(1)	Al(1)	C(2)	118.71(12)
O(1)	Al(2)	C(10)	100.81(12)	O(1)	Al(2)	C(11)	108.22(10)
O(1)	Al(2)	C(12)	105.94(11)	C(10)	Al(2)	C(11)	115.04(11)
C(10)	Al(2)	C(12)	115.97(14)	C(11)	Al(2)	C(12)	109.80(15)
Al(1)	O(1)	Al(2)	124.87(7)	Al(1)	O(1)	C(3)	113.49(15)
Al(2)	O(1)	C(3)	121.49(13)	Al(1)	N(1)	C(9)	118.88(13)
Al(1)	N(1)	C(13)	123.54(11)	C(9)	N(1)	C(13)	117.34(16)
O(1)	C(3)	C(4)	120.93(18)	O(1)	C(3)	C(8)	119.34(18)
C(4)	C(3)	C(8)	119.7(2)	C(3)	C(4)	C(5)	120.2(2)
C(4)	C(5)	C(6)	121.2(2)	C(5)	C(6)	C(7)	119.1(2)
C(6)	C(7)	C(8)	121.3(2)	C(3)	C(8)	C(7)	118.35(18)
C(3)	C(8)	C(9)	123.39(18)	C(7)	C(8)	C(9)	118.22(18)
N(1)	C(9)	C(8)	124.38(18)	N(1)	C(13)	C(14)	119.2(2)
N(1)	C(13)	C(18)	117.8(2)	C(14)	C(13)	C(18)	123.07(19)
C(13)	C(14)	C(15)	116.8(2)	C(13)	C(14)	C(19)	123.45(19)
C(15)	C(14)	C(19)	119.7(2)	C(14)	C(15)	C(16)	121.2(2)
C(15)	C(16)	C(17)	120.4(2)	C(16)	C(17)	C(18)	121.2(3)
C(13)	C(18)	C(17)	117.3(2)	C(13)	C(18)	C(22)	121.30(19)
C(17)	C(18)	C(22)	121.2(2)	C(14)	C(19)	C(20)	110.3(2)
C(14)	C(19)	C(21)	111.9(2)	C(20)	C(19)	C(21)	110.1(2)
C(18)	C(22)	C(23)	110.5(2)	C(18)	C(22)	C(24)	112.4(2)
C(23)	C(22)	C(24)	111.1(3)	C(25)	C(26)	C(29)	148.5(14)
C(25)	C(26)	C(29)$^{1)}$	148.5(18)	C(25)	C(26)	C(29)$^{2)}$	148.5(16)
C(29)	C(26)	C(29)$^{1)}$	54(2)	C(29)	C(26)	C(29)$^{2)}$	54(2)
C(29)$^{1)}$	C(26)	C(29)$^{2)}$	54(2)	C(29)	C(27)	C(29)$^{1)}$	57.2(19)
C(29)	C(27)	C(29)$^{2)}$	57.2(14)	C(29)	C(27)	C(29)$^{3)}$	180(2)
C(29)	C(27)	C(29)$^{4)}$	122.8(19)	C(29)	C(27)	C(29)$^{5)}$	122.8(14)
C(29)$^{1)}$	C(27)	C(29)$^{2)}$	57(2)	C(29)$^{1)}$	C(27)	C(29)$^{3)}$	123(2)
C(29)$^{1)}$	C(27)	C(29)$^{4)}$	180(2)	C(29)$^{1)}$	C(27)	C(29)$^{5)}$	123(2)
C(29)$^{2)}$	C(27)	C(29)$^{3)}$	122.8(14)	C(29)$^{2)}$	C(27)	C(29)$^{4)}$	123(2)
C(29)$^{2)}$	C(27)	C(29)$^{5)}$	180(2)	C(29)$^{3)}$	C(27)	C(29)$^{4)}$	57.2(19)
C(29)$^{3)}$	C(27)	C(29)$^{5)}$	57.2(14)	C(29)$^{4)}$	C(27)	C(29)$^{5)}$	57(2)
C(26)	C(29)	C(27)	114.9(19)	C(26)	C(29)	C(29)$^{1)}$	63.1(17)
C(26)	C(29)	C(29)$^{2)}$	63.1(16)	C(27)	C(29)	C(29)$^{1)}$	61.4(15)
C(27)	C(29)	C(29)$^{2)}$	61.4(14)	C(29)$^{1)}$	C(29)	C(29)$^{2)}$	60(2)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle

Symmetry Operators:

(1) -Y+1,X-Y,Z
(2) -X+Y+1,-X+1,Z
(3) -X+1/3+1,-Y+2/3,-Z+2/3
(4) Y+1/3,-X+Y+2/3,-Z+2/3
(5) X-Y+1/3,X+2/3-1,-Z+2/3

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.3	Al(1)	C(1)	H(2)	109.5
Al(1)	C(1)	H(3)	109.6	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.4	Al(1)	C(2)	H(5)	109.7
Al(1)	C(2)	H(6)	109.3	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
C(3)	C(4)	H(7)	115.9	C(5)	C(4)	H(7)	123.9
C(4)	C(5)	H(8)	124.7	C(6)	C(5)	H(8)	114.0
C(5)	C(6)	H(9)	118.1	C(7)	C(6)	H(9)	122.6
C(6)	C(7)	H(10)	123.8	C(8)	C(7)	H(10)	114.9
N(1)	C(9)	H(11)	119.0	C(8)	C(9)	H(11)	116.5
Al(2)	C(10)	H(12)	109.5	Al(2)	C(10)	H(13)	109.1
Al(2)	C(10)	H(14)	109.7	H(12)	C(10)	H(13)	109.5
H(12)	C(10)	H(14)	109.5	H(13)	C(10)	H(14)	109.5
Al(2)	C(11)	H(15)	109.5	Al(2)	C(11)	H(16)	109.7
Al(2)	C(11)	H(17)	109.3	H(15)	C(11)	H(16)	109.5
H(15)	C(11)	H(17)	109.5	H(16)	C(11)	H(17)	109.5
Al(2)	C(12)	H(18)	109.5	Al(2)	C(12)	H(19)	109.2
Al(2)	C(12)	H(20)	109.7	H(18)	C(12)	H(19)	109.5
H(18)	C(12)	H(20)	109.5	H(19)	C(12)	H(20)	109.5
C(14)	C(15)	H(21)	119.1	C(16)	C(15)	H(21)	119.6
C(15)	C(16)	H(22)	116.2	C(17)	C(16)	H(22)	123.3
C(16)	C(17)	H(23)	117.9	C(18)	C(17)	H(23)	120.3
C(14)	C(19)	H(24)	107.0	C(20)	C(19)	H(24)	109.2
C(21)	C(19)	H(24)	108.1	C(19)	C(20)	H(25)	110.3
C(19)	C(20)	H(26)	109.2	C(19)	C(20)	H(27)	108.9
H(25)	C(20)	H(26)	109.5	H(25)	C(20)	H(27)	109.5
H(26)	C(20)	H(27)	109.5	C(19)	C(21)	H(28)	110.3
C(19)	C(21)	H(29)	109.2	C(19)	C(21)	H(30)	108.9
H(28)	C(21)	H(29)	109.5	H(28)	C(21)	H(30)	109.5
H(29)	C(21)	H(30)	109.5	C(18)	C(22)	H(31)	104.8
C(23)	C(22)	H(31)	110.3	C(24)	C(22)	H(31)	107.5
C(22)	C(23)	H(32)	110.0	C(22)	C(23)	H(33)	109.4
C(22)	C(23)	H(34)	108.9	H(32)	C(23)	H(33)	109.5
H(32)	C(23)	H(34)	109.5	H(33)	C(23)	H(34)	109.5
C(22)	C(24)	H(35)	110.3	C(22)	C(24)	H(36)	109.6
C(22)	C(24)	H(37)	108.5	H(35)	C(24)	H(36)	109.5

Table 7. Bond angles involving hydrogens (^{o}) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(35)	C(24)	H(37)	109.5	H(36)	C(24)	H(37)	109.5

Table 8. Torsion Angles(O)

atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(9)	34.3(2)
N(1)	Al(1)	O(1)	Al(2)	128.13(12)
C(1)	Al(1)	O(1)	Al(2)	12.60(16)
C(2)	Al(1)	O(1)	Al(2)	-122.55(13)
C(1)	Al(1)	N(1)	C(9)	152.7(2)
C(2)	Al(1)	N(1)	C(9)	-74.9(2)
C(10)	Al(2)	O(1)	Al(1)	59.49(14)
C(11)	Al(2)	O(1)	Al(1)	-179.42(13)
C(12)	Al(2)	O(1)	Al(1)	-61.72(15)
Al(1)	O(1)	C(3)	C(4)	-129.8(2)
Al(2)	O(1)	C(3)	C(4)	45.9(3)
Al(1)	N(1)	C(9)	C(8)	-2.7(4)
Al(1)	N(1)	C(13)	C(18)	82.8(2)
C(9)	N(1)	C(13)	C(18)	-102.9(2)
O(1)	C(3)	C(4)	C(5)	-176.4(2)
O(1)	C(3)	C(8)	C(9)	-7.5(4)
C(4)	C(3)	C(8)	C(9)	172.9(2)
C(3)	C(4)	C(5)	C(6)	0.2(4)
C(5)	C(6)	C(7)	C(8)	0.3(4)
C(6)	C(7)	C(8)	C(9)	-174.7(2)
C(7)	C(8)	C(9)	N(1)	158.7(2)
N(1)	C(13)	C(14)	C(19)	-2.9(3)
N(1)	C(13)	C(18)	C(22)	4.2(2)
C(14)	C(13)	C(18)	C(22)	-176.9(2)
C(18)	C(13)	C(14)	C(19)	178.3(2)
C(13)	C(14)	C(19)	C(20)	-109.7(2)
C(15)	C(14)	C(19)	C(20)	68.6(2)
C(19)	C(14)	C(15)	C(16)	-177.7(2)
C(15)	C(16)	C(17)	C(18)	-1.4(3)
C(16)	C(17)	C(18)	C(22)	177.6(2)
C(13)	C(18)	C(22)	C(24)	-145.1(2)
C(17)	C(18)	C(22)	C(24)	39.4(3)
C(25)	C(26)	C(29)	C(29)$^{2)}$	-146(2)
C(25)	C(26)	C(29)$^{1)}$	C(29)$^{2)}$	145.9(18)
C(25)	C(26)	C(29)$^{2)}$	C(29)$^{1)}$	-146(2)
C(29)	C(26)	C(29)$^{1)}$	C(29)$^{2)}$	-68(2)
C(29)$^{1)}$	C(26)	C(29)	C(29)$^{2)}$	68(2)

atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(13)	-151.5(2)
N(1)	Al(1)	O(1)	C(3)	-56.35(17)
C(1)	Al(1)	O(1)	C(3)	-171.88(17)
C(2)	Al(1)	O(1)	C(3)	52.97(18)
C(1)	Al(1)	N(1)	C(13)	-33.1(2)
C(2)	Al(1)	N(1)	C(13)	99.3(2)
C(10)	Al(2)	O(1)	C(3)	-115.69(19)
C(11)	Al(2)	O(1)	C(3)	5.4(2)
C(12)	Al(2)	O(1)	C(3)	123.10(19)
Al(1)	O(1)	C(3)	C(8)	50.7(3)
Al(2)	O(1)	C(3)	C(8)	-133.6(2)
Al(1)	N(1)	C(13)	C(14)	-96.1(2)
C(9)	N(1)	C(13)	C(14)	78.2(3)
C(13)	N(1)	C(9)	C(8)	-177.2(2)
O(1)	C(3)	C(8)	C(7)	174.9(2)
C(4)	C(3)	C(8)	C(7)	-4.7(4)
C(8)	C(3)	C(4)	C(5)	3.1(4)
C(4)	C(5)	C(6)	C(7)	-1.9(5)
C(6)	C(7)	C(8)	C(3)	3.0(4)
C(3)	C(8)	C(9)	N(1)	-18.8(4)
N(1)	C(13)	C(14)	C(15)	178.81(18)
N(1)	C(13)	C(18)	C(17)	179.89(18)
C(14)	C(13)	C(18)	C(17)	-1.3(3)
C(18)	C(13)	C(14)	C(15)	-0.0(2)
C(13)	C(14)	C(15)	C(16)	0.7(3)
C(13)	C(14)	C(19)	C(21)	127.4(2)
C(15)	C(14)	C(19)	C(21)	-54.3(3)
C(14)	C(15)	C(16)	C(17)	0.0(3)
C(16)	C(17)	C(18)	C(13)	2.0(3)
C(13)	C(18)	C(22)	C(23)	90.1(3)
C(17)	C(18)	C(22)	C(23)	-85.3(3)
C(25)	C(26)	C(29)	C(29)$^{1)}$	146(2)
C(25)	C(26)	C(29)$^{1)}$	C(29)	-146(2)
C(25)	C(26)	C(29)$^{2)}$	C(29)	146(2)
C(29)	C(26)	C(29)$^{1)}$	C(27)	-34(2)
C(29)$^{1)}$	C(26)	C(29)	C(27)	34(2)
C(29)	C(26)	C(29)$^{2)}$	C(27)	34(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(29)	C(26)	C(29)$^{2)}$	C(29)$^{1)}$	68(2)
C(29)$^{2)}$	C(26)	C(29)	C(29)$^{1)}$	-68(2)
C(29)$^{1)}$	C(26)	C(29)$^{2)}$	C(29)	-68(2)
C(29)$^{2)}$	C(26)	C(29)$^{1)}$	C(29)	68(2)
C(29)	C(27)	C(29)$^{1)}$	C(29)$^{2)}$	69(2)
C(29)$^{1)}$	C(27)	C(29)	C(29)$^{2)}$	-69(2)
C(29)	C(27)	C(29)$^{2)}$	C(29)$^{1)}$	-69(2)
C(29)$^{2)}$	C(27)	C(29)	C(29)$^{1)}$	69(2)
C(29)$^{3)}$	C(27)	C(29)	C(26)	-67(196)
C(29)$^{3)}$	C(27)	C(29)	C(29)$^{2)}$	-102(73)
C(29)$^{4)}$	C(27)	C(29)	C(29)$^{1)}$	-180(2)
C(29)$^{5)}$	C(27)	C(29)	C(26)	-145(2)
C(29)$^{5)}$	C(27)	C(29)	C(29)$^{2)}$	-180(3)
C(29)$^{1)}$	C(27)	C(29)$^{2)}$	C(29)	69(2)
C(29)$^{2)}$	C(27)	C(29)$^{1)}$	C(29)	-69(2)
C(29)$^{3)}$	C(27)	C(29)$^{1)}$	C(26)	-145(2)
C(29)$^{3)}$	C(27)	C(29)$^{1)}$	C(29)$^{2)}$	-111(2)
C(29)$^{4)}$	C(27)	C(29)$^{1)}$	C(29)	147(210)
C(29)$^{5)}$	C(27)	C(29)$^{1)}$	C(26)	145.3(17)
C(29)$^{5)}$	C(27)	C(29)$^{1)}$	C(29)$^{2)}$	-180.0(16)
C(29)$^{3)}$	C(27)	C(29)$^{2)}$	C(26)	145(2)
C(29)$^{3)}$	C(27)	C(29)$^{2)}$	C(29)$^{1)}$	111(2)
C(29)$^{4)}$	C(27)	C(29)$^{2)}$	C(29)	-111(2)
C(29)$^{5)}$	C(27)	C(29)$^{2)}$	C(26)	70(161)
C(29)$^{5)}$	C(27)	C(29)$^{2)}$	C(29)$^{1)}$	35(202)
C(29)$^{5)}$	C(27)	C(29)$^{3)}$	C(26)$^{3)}$	-35(2)
C(26)	C(29)	C(29)$^{1)}$	C(29)$^{2)}$	72.9(17)
C(26)	C(29)	C(29)$^{2)}$	C(29)$^{1)}$	-73.0(19)
C(27)	C(29)	C(29)$^{1)}$	C(29)$^{2)}$	-71.7(16)
C(27)	C(29)	C(29)$^{2)}$	C(29)$^{1)}$	71.7(18)
C(29)$^{1)}$	C(29)	C(29)$^{2)}$	C(27)	-71.7(18)
C(29)$^{2)}$	C(29)	C(29)$^{1)}$	C(27)	71.7(16)
C(29)$^{2)}$	C(26)	C(29)	C(27)	-34(2)
C(29)$^{1)}$	C(26)	C(29)$^{2)}$	C(27)	-34(2)
C(29)$^{2)}$	C(26)	C(29)$^{1)}$	C(27)	34.1(18)
C(29)	C(27)	C(29)$^{1)}$	C(26)	35(2)
C(29)$^{1)}$	C(27)	C(29)	C(26)	-35(2)
C(29)	C(27)	C(29)$^{2)}$	C(26)	-35(2)
C(29)$^{2)}$	C(27)	C(29)	C(26)	35(2)
C(29)	C(27)	C(29)$^{3)}$	C(26)$^{3)}$	-113(195)
C(29)$^{3)}$	C(27)	C(29)	C(29)$^{1)}$	-32(260)
C(29)$^{4)}$	C(27)	C(29)	C(26)	145(2)
C(29)$^{4)}$	C(27)	C(29)	C(29)$^{2)}$	111(2)
C(29)$^{5)}$	C(27)	C(29)	C(29)$^{1)}$	-111(2)
C(29)$^{1)}$	C(27)	C(29)$^{2)}$	C(26)	35(2)
C(29)$^{2)}$	C(27)	C(29)$^{1)}$	C(26)	-34.7(17)
C(29)$^{1)}$	C(27)	C(29)$^{3)}$	C(26)$^{3)}$	-145(2)
C(29)$^{3)}$	C(27)	C(29)$^{1)}$	C(29)	180(2)
C(29)$^{4)}$	C(27)	C(29)$^{1)}$	C(26)	-178(203)
C(29)$^{4)}$	C(27)	C(29)$^{1)}$	C(29)$^{2)}$	-143(112)
C(29)$^{5)}$	C(27)	C(29)$^{1)}$	C(29)	111(2)
C(29)$^{2)}$	C(27)	C(29)$^{3)}$	C(26)$^{3)}$	145(2)
C(29)$^{3)}$	C(27)	C(29)$^{2)}$	C(29)	180(3)
C(29)$^{4)}$	C(27)	C(29)$^{2)}$	C(26)	-145(2)
C(29)$^{4)}$	C(27)	C(29)$^{2)}$	C(29)$^{1)}$	180.0(16)
C(29)$^{5)}$	C(27)	C(29)$^{2)}$	C(29)	105(104)
C(29)$^{4)}$	C(27)	C(29)$^{3)}$	C(26)$^{3)}$	35(2)
C(26)	C(29)	C(29)$^{1)}$	C(27)	145(2)
C(26)	C(29)	C(29)$^{2)}$	C(27)	-145(2)
C(27)	C(29)	C(29)$^{1)}$	C(26)	-145(2)
C(27)	C(29)	C(29)$^{2)}$	C(26)	145(2)
C(29)$^{1)}$	C(29)	C(29)$^{2)}$	C(26)	73.0(19)
C(29)$^{2)}$	C(29)	C(29)$^{1)}$	C(26)	-72.9(17)

Symmetry Operators:

(1) -Y+1,X-Y,Z
(2) -X+Y+1,-X+1,Z
(3) -X+1/3+1,-Y+2/3,-Z+2/3
(4) Y+1/3,-X+Y+2/3,-Z+2/3
(5) X-Y+1/3,X+2/3-1,-Z+2/3

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
C(25)	C(25)[1)]	3.18(4)			

Symmetry Operators:

(1) -X+1/3+1,-Y+2/3,-Z+2/3-1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
C(1)	H(21)[1)]	3.199	C(1)	H(23)[2)]	3.274
C(2)	H(28)[3)]	3.172	C(2)	H(32)[4)]	3.453
C(2)	H(34)[4)]	3.380	C(2)	H(36)[2)]	3.186
C(3)	H(9)[5)]	3.108	C(4)	H(9)[5)]	3.420
C(4)	H(14)[6)]	3.551	C(4)	H(15)[6)]	3.257
C(5)	H(14)[6)]	3.140	C(5)	H(33)[4)]	3.534
C(6)	H(9)[5)]	3.553	C(6)	H(17)[4)]	3.130
C(6)	H(33)[4)]	3.401	C(7)	H(8)[5)]	3.388
C(7)	H(9)[5)]	3.280	C(7)	H(17)[4)]	3.353
C(7)	H(19)[4)]	3.403	C(7)	H(32)[4)]	3.371
C(8)	H(9)[5)]	3.079	C(8)	H(32)[4)]	3.529
C(9)	H(14)[7)]	3.293	C(10)	H(8)[8)]	3.455
C(10)	H(10)[9)]	3.353	C(10)	H(11)[9)]	2.703
C(10)	H(27)[9)]	3.374	C(10)	H(33)[9)]	3.577
C(11)	H(7)[8)]	2.978	C(11)	H(9)[5)]	3.208
C(11)	H(10)[5)]	3.265	C(12)	H(10)[5)]	3.369
C(12)	H(25)[5)]	3.322	C(12)	H(27)[5)]	3.571
C(15)	H(22)[2)]	3.206	C(15)	H(37)[10)]	3.555
C(16)	H(2)[11)]	3.535	C(16)	H(22)[2)]	2.864
C(17)	H(2)[11)]	3.234	C(17)	H(22)[2)]	3.028
C(17)	H(29)[11)]	3.282	C(18)	H(22)[2)]	3.485
C(20)	H(5)[7)]	3.413	C(20)	H(18)[4)]	3.442
C(20)	H(35)[4)]	3.432	C(21)	H(5)[3)]	3.565
C(21)	H(23)[2)]	3.580	C(21)	H(34)[2)]	3.382
C(23)	H(4)[5)]	2.949	C(23)	H(29)[11)]	3.598
C(24)	H(6)[11)]	3.384	C(24)	H(21)[1)]	3.414
C(24)	H(25)[5)]	3.466	C(24)	H(26)[1)]	3.147
C(29)	H(8)[12)]	3.502	C(29)	H(9)[13)]	3.470
H(1)	H(21)[1)]	2.778	H(1)	H(30)[1)]	3.326
H(2)	C(16)[2)]	3.535	H(2)	C(17)[2)]	3.234
H(2)	H(21)[1)]	2.971	H(2)	H(22)[1)]	2.995
H(2)	H(22)[2)]	3.313	H(2)	H(23)[2)]	2.502
H(2)	H(36)[2)]	3.567	H(2)	H(37)[2)]	3.555
H(3)	H(21)[1)]	3.344	H(3)	H(22)[1)]	3.589
H(3)	H(23)[2)]	3.483	H(3)	H(26)[9)]	3.540
H(3)	H(37)[2)]	3.348	H(4)	C(23)[4)]	2.949
H(4)	H(28)[3)]	3.349	H(4)	H(32)[4)]	2.584

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(4)	H(33)[4)]	3.222	H(4)	H(34)[4)]	2.602
H(5)	C(20)[9)]	3.413	H(5)	C(21)[3)]	3.565
H(5)	H(18)[6)]	3.048	H(5)	H(25)[9)]	3.463
H(5)	H(26)[9)]	3.100	H(5)	H(27)[9)]	3.126
H(5)	H(28)[3)]	2.795	H(5)	H(36)[2)]	3.165
H(6)	C(24)[2)]	3.384	H(6)	H(23)[2)]	3.277
H(6)	H(28)[3)]	2.878	H(6)	H(32)[4)]	3.576
H(6)	H(34)[4)]	3.486	H(6)	H(36)[2)]	2.486
H(7)	C(11)[6)]	2.978	H(7)	H(14)[6)]	3.567
H(7)	H(15)[6)]	2.269	H(7)	H(16)[6)]	3.038
H(7)	H(18)[6)]	3.528	H(8)	C(7)[4)]	3.388
H(8)	C(10)[6)]	3.455	H(8)	C(29)[14)]	3.502
H(8)	H(10)[4)]	3.193	H(8)	H(13)[6)]	3.436
H(8)	H(14)[6)]	2.762	H(8)	H(16)[6)]	3.299
H(9)	C(3)[4)]	3.108	H(9)	C(4)[4)]	3.420
H(9)	C(6)[4)]	3.553	H(9)	C(7)[4)]	3.280
H(9)	C(8)[4)]	3.079	H(9)	C(11)[4)]	3.208
H(9)	C(29)[13)]	3.470	H(9)	H(15)[4)]	3.575
H(9)	H(17)[4)]	2.317	H(10)	C(10)[7)]	3.353
H(10)	C(11)[4)]	3.265	H(10)	C(12)[4)]	3.369
H(10)	H(8)[5)]	3.193	H(10)	H(13)[7)]	2.698
H(10)	H(14)[7)]	3.187	H(10)	H(15)[4)]	2.940
H(10)	H(17)[4)]	2.893	H(10)	H(18)[4)]	3.260
H(10)	H(19)[4)]	2.820	H(10)	H(32)[4)]	3.589
H(11)	C(10)[7)]	2.703	H(11)	H(12)[7)]	2.642
H(11)	H(13)[7)]	2.654	H(11)	H(14)[7)]	2.360
H(12)	H(11)[9)]	2.642	H(12)	H(27)[9)]	2.845
H(12)	H(33)[9)]	3.546	H(13)	H(8)[8)]	3.436
H(13)	H(10)[9)]	2.698	H(13)	H(11)[9)]	2.654
H(13)	H(15)[6)]	2.877	H(13)	H(18)[6)]	3.104
H(13)	H(27)[9)]	3.059	H(14)	C(4)[8)]	3.551
H(14)	C(5)[8)]	3.140	H(14)	C(9)[9)]	3.293
H(14)	H(7)[8)]	3.567	H(14)	H(8)[8)]	2.762
H(14)	H(10)[9)]	3.187	H(14)	H(11)[9)]	2.360
H(14)	H(33)[9)]	2.784	H(15)	C(4)[8)]	3.257
H(15)	H(7)[8)]	2.269	H(15)	H(9)[5)]	3.575
H(15)	H(10)[5)]	2.940	H(15)	H(13)[8)]	2.877

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(15)	H(16)[8)]	2.941	H(16)	H(7)[8)]	3.038
H(16)	H(8)[8)]	3.299	H(16)	H(15)[6)]	2.941
H(17)	C(6)[5)]	3.130	H(17)	C(7)[5)]	3.353
H(17)	H(9)[5)]	2.317	H(17)	H(10)[5)]	2.893
H(18)	C(20)[5)]	3.442	H(18)	H(5)[8)]	3.048
H(18)	H(7)[8)]	3.528	H(18)	H(10)[5)]	3.260
H(18)	H(13)[8)]	3.104	H(18)	H(25)[5)]	3.037
H(18)	H(27)[5)]	2.976	H(19)	C(7)[5)]	3.403
H(19)	H(10)[5)]	2.820	H(19)	H(25)[5)]	3.091
H(19)	H(27)[5)]	3.378	H(20)	H(25)[5)]	3.287
H(20)	H(30)[1)]	2.843	H(21)	C(1)[10)]	3.199
H(21)	C(24)[10)]	3.414	H(21)	H(1)[10)]	2.778
H(21)	H(2)[10)]	2.971	H(21)	H(3)[10)]	3.344
H(21)	H(35)[10)]	3.407	H(21)	H(37)[10)]	2.613
H(22)	C(15)[11)]	3.206	H(22)	C(16)[11)]	2.864
H(22)	C(17)[11)]	3.028	H(22)	C(18)[11)]	3.485
H(22)	H(2)[11)]	3.313	H(22)	H(2)[10)]	2.995
H(22)	H(3)[10)]	3.589	H(22)	H(22)[11)]	3.200
H(22)	H(22)[2)]	3.200	H(22)	H(23)[11)]	3.310
H(23)	C(1)[11)]	3.274	H(23)	C(21)[11)]	3.580
H(23)	H(2)[11)]	2.502	H(23)	H(3)[11)]	3.483
H(23)	H(6)[11)]	3.277	H(23)	H(22)[2)]	3.310
H(23)	H(29)[11)]	2.693	H(24)	H(32)[4)]	2.892
H(25)	C(12)[4)]	3.322	H(25)	C(24)[4)]	3.466
H(25)	H(5)[7)]	3.463	H(25)	H(18)[4)]	3.037
H(25)	H(19)[4)]	3.091	H(25)	H(20)[4)]	3.287
H(25)	H(25)[15)]	3.427	H(25)	H(26)[15)]	3.477
H(25)	H(28)[15)]	3.542	H(25)	H(30)[15)]	3.600
H(25)	H(32)[4)]	3.503	H(25)	H(35)[4)]	2.538
H(26)	C(24)[10)]	3.147	H(26)	H(3)[7)]	3.540
H(26)	H(5)[7)]	3.100	H(26)	H(25)[15)]	3.477
H(26)	H(35)[10)]	2.788	H(26)	H(36)[10)]	3.307
H(26)	H(37)[10)]	2.850	H(27)	C(10)[7)]	3.374
H(27)	C(12)[4)]	3.571	H(27)	H(5)[7)]	3.126
H(27)	H(12)[7)]	2.845	H(27)	H(13)[7)]	3.059
H(27)	H(18)[4)]	2.976	H(27)	H(19)[4)]	3.378
H(28)	C(2)[3)]	3.172	H(28)	H(4)[3)]	3.349

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(28)	H(5)[3)]	2.795	H(28)	H(6)[3)]	2.878
H(28)	H(25)[15)]	3.542	H(28)	H(34)[2)]	3.446
H(28)	H(35)[4)]	3.101	H(28)	H(36)[4)]	3.208
H(29)	C(17)[2)]	3.282	H(29)	C(23)[2)]	3.598
H(29)	H(23)[2)]	2.693	H(29)	H(34)[2)]	2.688
H(29)	H(36)[2)]	3.210	H(30)	H(1)[10)]	3.326
H(30)	H(20)[10)]	2.843	H(30)	H(25)[15)]	3.600
H(30)	H(34)[2)]	3.543	H(30)	H(35)[10)]	3.446
H(32)	C(2)[5)]	3.453	H(32)	C(7)[5)]	3.371
H(32)	C(8)[5)]	3.529	H(32)	H(4)[5)]	2.584
H(32)	H(6)[5)]	3.576	H(32)	H(10)[5)]	3.589
H(32)	H(24)[5)]	2.892	H(32)	H(25)[5)]	3.503
H(33)	C(5)[5)]	3.534	H(33)	C(6)[5)]	3.401
H(33)	C(10)[7)]	3.577	H(33)	H(4)[5)]	3.222
H(33)	H(12)[7)]	3.546	H(33)	H(14)[7)]	2.784
H(34)	C(2)[5)]	3.380	H(34)	C(21)[11)]	3.382
H(34)	H(4)[5)]	2.602	H(34)	H(6)[5)]	3.486
H(34)	H(28)[11)]	3.446	H(34)	H(29)[11)]	2.688
H(34)	H(30)[11)]	3.543	H(35)	C(20)[5)]	3.432
H(35)	H(21)[1)]	3.407	H(35)	H(25)[5)]	2.538
H(35)	H(26)[1)]	2.788	H(35)	H(28)[5)]	3.101
H(35)	H(30)[1)]	3.446	H(36)	C(2)[11)]	3.186
H(36)	H(2)[11)]	3.567	H(36)	H(5)[11)]	3.165
H(36)	H(6)[11)]	2.486	H(36)	H(26)[1)]	3.307
H(36)	H(28)[5)]	3.208	H(36)	H(29)[11)]	3.210
H(37)	C(15)[1)]	3.555	H(37)	H(2)[11)]	3.555
H(37)	H(3)[11)]	3.348	H(37)	H(21)[1)]	2.613
H(37)	H(26)[1)]	2.850			

Symmetry Operators:

(1) -X+Y+2/3,-X+1/3,Z+1/3-1
(2) -Y+1/3,X-Y+2/3-1,Z+2/3-1
(3) -X+2/3,-Y+1/3,-Z+1/3
(4) Y+1/3,-X+Y+2/3,-Z+2/3
(5) X-Y+1/3,X+2/3-1,-Z+2/3
(6) Y+1/3,-X+Y+2/3,-Z+2/3-1
(7) X,Y,Z+1
(8) X-Y+1/3,X+2/3-1,-Z+2/3-1
(9) X,Y,Z-1
(10) -Y+1/3,X-Y+2/3-1,Z+2/3
(11) -X+Y+2/3,-X+1/3,Z+1/3
(12) -X+Y+1,-X+1,Z
(13) -X+1/3+1,-Y+2/3,-Z+2/3
(14) -Y+1,X-Y,Z
(15) -X+2/3,-Y+1/3,-Z+1/3+1

X-ray Structure Report for $Me_2Al[\mu_2\text{-}O\text{-}2\text{-}(^tBuN{=}CH)C_6H_4](AlMe_3)$ (**3b**)

February 22, 2007

Experimental

Data Collection

A colorless block crystal of $C_{16}H_{29}NOAl_2$ having approximate dimensions of 0.30 x 0.16 x 0.12 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 240 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 11.1926(6) Å
b = 14.3616(7) Å β = 108.4732(18)^o
c = 12.9141(8) Å
V = 1968.89(19) Å^3

For Z = 4 and F.W. = 305.37, the calculated density is 1.030 g/cm^3. The systematic absences of:

h0l: h+l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 5.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 180.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 5.0^o step, at χ=45.0^o and ϕ = 180.0^o. The exposure rate was 180.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 19033 reflections that were collected, 4466 were unique (R_{int} = 0.045); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.446 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.762 to 0.983. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2129 observed reflections and 210 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0419$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1141$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.17 and -0.20 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) PATTY: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., Garcia-Granda, S., Gould, R.O., Smits, J.M.M. and Smykalla, C. (1992). The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2006). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{16}H_{29}NOAl_2$
Formula Weight	305.37
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.30 X 0.16 X 0.12 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 240.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 11.1926(6) Å b = 14.3616(7) Å c = 12.9141(8) Å β = 108.4732(18) ^{o} V = 1968.89(19) $Å^3$
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.030 g/cm^3
F_{000}	664.00
μ(MoKα)	1.446 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	180.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0^{o}
Exposure Rate	180.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 19033 Unique: 4466 (R_{int} = 0.045)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.762 - 0.983)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF99 PATTY)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0017Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	2129
No. Variables	210
Reflection/Parameter Ratio	10.14
Residuals: R1 (I>2.00σ(I))	0.0419
Residuals: wR2 (I>2.00σ(I))	0.1141
Goodness of Fit Indicator	1.012
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.17 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.20 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Al(1)	0.26191(7)	0.53881(5)	0.14759(6)	3.666(18)
Al(2)	0.54026(8)	0.65368(6)	0.21157(8)	4.59(2)
O(1)	0.43448(15)	0.54535(12)	0.21211(14)	3.95(4)
N(1)	0.23178(18)	0.53295(14)	0.29108(16)	3.40(4)
C(1)	0.2154(2)	0.4163(2)	0.0838(2)	5.33(7)
C(2)	0.2008(2)	0.6474(2)	0.0570(2)	5.41(7)
C(3)	0.4817(2)	0.46937(18)	0.2792(2)	3.86(6)
C(4)	0.5843(2)	0.4205(2)	0.2678(2)	4.86(7)
C(5)	0.6315(2)	0.3456(2)	0.3380(3)	5.57(8)
C(6)	0.5794(2)	0.3206(2)	0.4180(2)	5.39(7)
C(7)	0.4781(2)	0.36940(19)	0.4282(2)	4.51(6)
C(8)	0.4258(2)	0.44356(17)	0.3574(2)	3.64(5)
C(9)	0.3120(2)	0.48667(18)	0.3676(2)	3.84(6)
C(10)	0.1130(2)	0.5700(2)	0.3089(2)	4.07(6)
C(11)	0.0047(2)	0.5422(2)	0.2077(2)	5.40(8)
C(12)	0.0901(3)	0.5315(2)	0.4100(2)	6.00(9)
C(13)	0.1267(2)	0.6752(2)	0.3183(2)	5.31(7)
C(14)	0.4569(3)	0.7552(2)	0.2631(3)	8.09(10)
C(15)	0.7067(2)	0.6286(2)	0.3207(3)	6.73(9)
C(16)	0.5391(4)	0.6613(4)	0.0603(3)	10.37(16)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.1267	0.4133	0.0501	6.32
H(2)	0.2563	0.4052	0.0308	6.32
H(3)	0.2407	0.3704	0.1395	6.32
H(4)	0.1132	0.6409	0.0196	6.40
H(5)	0.2147	0.7014	0.1017	6.39
H(6)	0.2448	0.6532	0.0054	6.39
H(7)	0.6191	0.4420	0.1976	5.67
H(8)	0.6986	0.3017	0.3286	6.18
H(9)	0.6208	0.2650	0.4694	5.77
H(10)	0.4273	0.3444	0.4870	4.94
H(11)	0.2994	0.4673	0.4492	4.43
H(12)	0.0155	0.5720	0.1456	6.36
H(13)	0.0050	0.4766	0.1984	6.36
H(14)	-0.0732	0.5609	0.2159	6.36
H(15)	0.1554	0.5521	0.4728	7.42
H(16)	0.0112	0.5527	0.4136	7.44
H(17)	0.0901	0.4654	0.4074	7.42
H(18)	0.0516	0.7018	0.3247	6.36
H(19)	0.1955	0.6905	0.3811	6.35
H(20)	0.1419	0.6989	0.2550	6.35
H(21)	0.3934	0.7818	0.2032	8.97
H(22)	0.4198	0.7310	0.3142	8.98
H(23)	0.5169	0.8016	0.2974	8.97
H(24)	0.6947	0.6028	0.3844	8.08
H(25)	0.7516	0.5856	0.2911	8.08
H(26)	0.7534	0.6848	0.3391	8.08
H(27)	0.4603	0.6394	0.0135	13.04
H(28)	0.5509	0.7241	0.0427	13.04
H(29)	0.6049	0.6240	0.0510	13.05

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al(1)	0.0444(4)	0.0478(4)	0.0442(4)	0.0041(3)	0.0100(3)	0.0054(3)
Al(2)	0.0588(5)	0.0589(5)	0.0607(6)	-0.0079(4)	0.0247(4)	0.0041(4)
O(1)	0.0437(9)	0.0470(10)	0.0589(11)	0.0047(8)	0.0154(8)	0.0089(9)
N(1)	0.0414(11)	0.0413(11)	0.0442(12)	0.0004(9)	0.0103(9)	0.0020(10)
C(1)	0.0683(18)	0.072(2)	0.0585(19)	0.0035(16)	0.0145(15)	-0.0095(16)
C(2)	0.0661(18)	0.075(2)	0.0604(19)	0.0113(15)	0.0144(15)	0.0194(16)
C(3)	0.0405(14)	0.0427(15)	0.0537(17)	0.0028(11)	0.0011(12)	-0.0017(13)
C(4)	0.0469(15)	0.0597(18)	0.073(2)	0.0093(14)	0.0119(14)	-0.0051(16)
C(5)	0.0502(16)	0.0520(19)	0.094(2)	0.0160(14)	0.0002(17)	-0.0123(18)
C(6)	0.0611(18)	0.0458(17)	0.075(2)	0.0076(15)	-0.0106(17)	0.0020(16)
C(7)	0.0536(16)	0.0442(15)	0.0577(17)	-0.0017(13)	-0.0050(13)	0.0069(13)
C(8)	0.0442(13)	0.0411(15)	0.0452(15)	0.0019(11)	0.0032(12)	0.0018(12)
C(9)	0.0477(14)	0.0442(15)	0.0470(16)	-0.0028(12)	0.0052(12)	0.0031(12)
C(10)	0.0469(14)	0.0590(18)	0.0473(16)	0.0056(12)	0.0128(12)	0.0040(13)
C(11)	0.0443(15)	0.088(2)	0.069(2)	0.0037(15)	0.0122(14)	-0.0014(17)
C(12)	0.067(2)	0.101(2)	0.068(2)	0.0101(18)	0.0319(17)	0.0149(19)
C(13)	0.0660(19)	0.062(2)	0.073(2)	0.0177(15)	0.0209(16)	-0.0007(16)
C(14)	0.097(2)	0.066(2)	0.120(3)	0.0015(19)	0.001(2)	-0.021(2)
C(15)	0.0574(18)	0.088(2)	0.110(2)	-0.0115(18)	0.0264(18)	0.001(2)
C(16)	0.134(3)	0.188(5)	0.091(3)	-0.038(3)	0.062(2)	0.014(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Al(1)	O(1)	1.8477(16)	Al(1)	N(1)	1.988(2)
Al(1)	C(1)	1.943(3)	Al(1)	C(2)	1.939(3)
Al(2)	O(1)	1.9564(19)	Al(2)	C(14)	1.957(4)
Al(2)	C(15)	1.981(3)	Al(2)	C(16)	1.952(4)
O(1)	C(3)	1.389(3)	N(1)	C(9)	1.289(2)
N(1)	C(10)	1.515(3)	C(3)	C(4)	1.393(4)
C(3)	C(8)	1.397(4)	C(4)	C(5)	1.397(4)
C(5)	C(6)	1.386(5)	C(6)	C(7)	1.374(4)
C(7)	C(8)	1.405(3)	C(8)	C(9)	1.459(4)
C(10)	C(11)	1.526(3)	C(10)	C(12)	1.513(4)
C(10)	C(13)	1.519(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(2)	H(4)	0.950
C(2)	H(5)	0.950	C(2)	H(6)	0.950
C(4)	H(7)	1.137	C(5)	H(8)	1.018
C(6)	H(9)	1.048	C(7)	H(10)	1.141
C(9)	H(11)	1.141	C(11)	H(12)	0.950
C(11)	H(13)	0.950	C(11)	H(14)	0.950
C(12)	H(15)	0.950	C(12)	H(16)	0.950
C(12)	H(17)	0.950	C(13)	H(18)	0.950
C(13)	H(19)	0.950	C(13)	H(20)	0.950
C(14)	H(21)	0.950	C(14)	H(22)	0.950
C(14)	H(23)	0.950	C(15)	H(24)	0.950
C(15)	H(25)	0.950	C(15)	H(26)	0.950
C(16)	H(27)	0.950	C(16)	H(28)	0.950
C(16)	H(29)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Al(1)	N(1)	92.50(8)	O(1)	Al(1)	C(1)	109.79(10)
O(1)	Al(1)	C(2)	110.48(11)	N(1)	Al(1)	C(1)	104.78(12)
N(1)	Al(1)	C(2)	117.70(12)	C(1)	Al(1)	C(2)	118.45(12)
O(1)	Al(2)	C(14)	103.71(14)	O(1)	Al(2)	C(15)	106.66(12)
O(1)	Al(2)	C(16)	103.65(16)	C(14)	Al(2)	C(15)	109.59(16)
C(14)	Al(2)	C(16)	116.4(2)	C(15)	Al(2)	C(16)	115.41(19)
Al(1)	O(1)	Al(2)	125.88(9)	Al(1)	O(1)	C(3)	112.21(15)
Al(2)	O(1)	C(3)	121.05(13)	Al(1)	N(1)	C(9)	116.99(19)
Al(1)	N(1)	C(10)	122.87(14)	C(9)	N(1)	C(10)	119.7(2)
O(1)	C(3)	C(4)	119.6(2)	O(1)	C(3)	C(8)	119.3(2)
C(4)	C(3)	C(8)	121.0(2)	C(3)	C(4)	C(5)	118.3(3)
C(4)	C(5)	C(6)	121.4(3)	C(5)	C(6)	C(7)	119.6(2)
C(6)	C(7)	C(8)	120.8(3)	C(3)	C(8)	C(7)	118.8(2)
C(3)	C(8)	C(9)	123.8(2)	C(7)	C(8)	C(9)	117.3(2)
N(1)	C(9)	C(8)	124.4(2)	N(1)	C(10)	C(11)	106.0(2)
N(1)	C(10)	C(12)	113.5(2)	N(1)	C(10)	C(13)	106.9(2)
C(11)	C(10)	C(12)	109.7(2)	C(11)	C(10)	C(13)	111.2(2)
C(12)	C(10)	C(13)	109.5(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Al(1)	C(1)	H(1)	109.6	Al(1)	C(1)	H(2)	109.4
Al(1)	C(1)	H(3)	109.4	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
Al(1)	C(2)	H(4)	109.8	Al(1)	C(2)	H(5)	109.3
Al(1)	C(2)	H(6)	109.4	H(4)	C(2)	H(5)	109.5
H(4)	C(2)	H(6)	109.5	H(5)	C(2)	H(6)	109.5
C(3)	C(4)	H(7)	116.6	C(5)	C(4)	H(7)	124.9
C(4)	C(5)	H(8)	122.7	C(6)	C(5)	H(8)	115.4
C(5)	C(6)	H(9)	117.6	C(7)	C(6)	H(9)	122.8
C(6)	C(7)	H(10)	120.2	C(8)	C(7)	H(10)	118.6
N(1)	C(9)	H(11)	125.0	C(8)	C(9)	H(11)	110.3
C(10)	C(11)	H(12)	109.0	C(10)	C(11)	H(13)	109.5
C(10)	C(11)	H(14)	109.8	H(12)	C(11)	H(13)	109.5
H(12)	C(11)	H(14)	109.5	H(13)	C(11)	H(14)	109.5
C(10)	C(12)	H(15)	109.1	C(10)	C(12)	H(16)	109.8
C(10)	C(12)	H(17)	109.4	H(15)	C(12)	H(16)	109.5
H(15)	C(12)	H(17)	109.5	H(16)	C(12)	H(17)	109.5
C(10)	C(13)	H(18)	109.8	C(10)	C(13)	H(19)	109.4
C(10)	C(13)	H(20)	109.2	H(18)	C(13)	H(19)	109.5
H(18)	C(13)	H(20)	109.5	H(19)	C(13)	H(20)	109.5
Al(2)	C(14)	H(21)	109.7	Al(2)	C(14)	H(22)	109.0
Al(2)	C(14)	H(23)	109.8	H(21)	C(14)	H(22)	109.5
H(21)	C(14)	H(23)	109.5	H(22)	C(14)	H(23)	109.5
Al(2)	C(15)	H(24)	109.1	Al(2)	C(15)	H(25)	109.2
Al(2)	C(15)	H(26)	110.0	H(24)	C(15)	H(25)	109.5
H(24)	C(15)	H(26)	109.5	H(25)	C(15)	H(26)	109.5
Al(2)	C(16)	H(27)	109.2	Al(2)	C(16)	H(28)	109.6
Al(2)	C(16)	H(29)	109.6	H(27)	C(16)	H(28)	109.5
H(27)	C(16)	H(29)	109.5	H(28)	C(16)	H(29)	109.5

Table 8. Torsion Angles(O)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(1)	Al(1)	N(1)	C(9)	-36.71(18)	O(1)	Al(1)	N(1)	C(10)	150.72(18)
N(1)	Al(1)	O(1)	Al(2)	-111.23(13)	N(1)	Al(1)	O(1)	C(3)	58.20(18)
C(1)	Al(1)	O(1)	Al(2)	142.09(15)	C(1)	Al(1)	O(1)	C(3)	-48.5(2)
C(2)	Al(1)	O(1)	Al(2)	9.64(18)	C(2)	Al(1)	O(1)	C(3)	179.06(19)
C(1)	Al(1)	N(1)	C(9)	74.5(2)	C(1)	Al(1)	N(1)	C(10)	-98.05(19)
C(2)	Al(1)	N(1)	C(9)	-151.45(19)	C(2)	Al(1)	N(1)	C(10)	36.0(2)
C(14)	Al(2)	O(1)	Al(1)	51.47(17)	C(14)	Al(2)	O(1)	C(3)	-117.1(2)
C(15)	Al(2)	O(1)	Al(1)	167.14(16)	C(15)	Al(2)	O(1)	C(3)	-1.4(2)
C(16)	Al(2)	O(1)	Al(1)	-70.6(2)	C(16)	Al(2)	O(1)	C(3)	120.8(2)
Al(1)	O(1)	C(3)	C(4)	130.2(2)	Al(1)	O(1)	C(3)	C(8)	-50.2(2)
Al(2)	O(1)	C(3)	C(4)	-59.8(2)	Al(2)	O(1)	C(3)	C(8)	119.8(2)
Al(1)	N(1)	C(9)	C(8)	3.7(3)	Al(1)	N(1)	C(10)	C(11)	40.6(2)
Al(1)	N(1)	C(10)	C(12)	161.14(18)	Al(1)	N(1)	C(10)	C(13)	-78.0(2)
C(9)	N(1)	C(10)	C(11)	-131.7(2)	C(9)	N(1)	C(10)	C(12)	-11.2(3)
C(9)	N(1)	C(10)	C(13)	109.6(2)	C(10)	N(1)	C(9)	C(8)	176.5(2)
O(1)	C(3)	C(4)	C(5)	178.6(2)	O(1)	C(3)	C(8)	C(7)	-177.0(2)
O(1)	C(3)	C(8)	C(9)	5.7(3)	C(4)	C(3)	C(8)	C(7)	2.7(3)
C(4)	C(3)	C(8)	C(9)	-174.7(2)	C(8)	C(3)	C(4)	C(5)	-1.0(3)
C(3)	C(4)	C(5)	C(6)	-0.7(4)	C(4)	C(5)	C(6)	C(7)	0.8(4)
C(5)	C(6)	C(7)	C(8)	0.9(4)	C(6)	C(7)	C(8)	C(3)	-2.6(3)
C(6)	C(7)	C(8)	C(9)	174.9(2)	C(3)	C(8)	C(9)	N(1)	20.2(3)
C(7)	C(8)	C(9)	N(1)	-157.2(2)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
C(7)	C(9)[1)]	3.581(3)	C(9)	C(7)[1)]	3.581(3)

Symmetry Operators:

(1) -X+1,-Y+1,-Z+1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
C(1)	H(4)[1)]	3.587	C(1)	H(9)[2)]	3.014
C(1)	H(12)[1)]	3.261	C(1)	H(19)[3)]	3.380
C(1)	H(22)[3)]	3.516	C(1)	H(29)[4)]	3.102
C(2)	H(1)[1)]	3.591	C(2)	H(10)[5)]	3.141
C(2)	H(23)[6)]	3.412	C(4)	H(18)[3)]	3.528
C(5)	H(15)[7)]	3.183	C(5)	H(18)[3)]	3.186
C(5)	H(19)[7)]	3.568	C(5)	H(20)[3)]	3.593
C(6)	H(11)[7)]	3.549	C(6)	H(15)[7)]	3.390
C(6)	H(18)[3)]	3.460	C(6)	H(19)[7)]	2.994
C(6)	H(20)[3)]	3.267	C(6)	H(22)[7)]	3.534
C(7)	H(4)[3)]	3.564	C(7)	H(5)[3)]	3.178
C(7)	H(11)[7)]	3.423	C(7)	H(20)[3)]	3.373
C(7)	H(22)[7)]	3.471	C(7)	H(24)[7)]	3.567
C(8)	H(11)[7)]	3.532	C(9)	H(24)[7)]	3.473
C(11)	H(1)[1)]	3.249	C(11)	H(2)[1)]	3.590
C(11)	H(23)[3)]	3.464	C(11)	H(25)[8)]	3.392
C(12)	H(16)[9)]	3.094	C(12)	H(17)[9)]	3.557
C(12)	H(24)[7)]	3.538	C(13)	H(3)[5)]	3.138
C(13)	H(9)[7)]	3.366	C(13)	H(28)[10)]	3.572
C(14)	H(3)[5)]	3.313	C(14)	H(4)[11)]	3.547
C(14)	H(10)[7)]	3.390	C(14)	H(13)[5)]	3.226
C(15)	H(8)[12)]	3.509	C(15)	H(10)[7)]	3.305
C(15)	H(11)[7)]	3.295	C(15)	H(14)[13)]	3.311
C(15)	H(16)[13)]	3.413	C(16)	H(2)[4)]	3.041
C(16)	H(7)[4)]	3.563	C(16)	H(8)[12)]	3.474
H(1)	C(2)[1)]	3.591	H(1)	C(11)[1)]	3.249
H(1)	H(4)[1)]	2.663	H(1)	H(9)[2)]	2.756
H(1)	H(12)[1)]	2.533	H(1)	H(13)[1)]	3.460
H(1)	H(14)[1)]	3.315	H(1)	H(22)[3)]	3.282
H(1)	H(23)[3)]	3.324	H(2)	C(11)[1)]	3.590
H(2)	C(16)[4)]	3.041	H(2)	H(9)[2)]	2.853
H(2)	H(12)[1)]	3.187	H(2)	H(14)[1)]	3.232
H(2)	H(19)[3)]	3.272	H(2)	H(27)[4)]	3.457
H(2)	H(28)[4)]	3.210	H(2)	H(29)[4)]	2.177
H(3)	C(13)[3)]	3.138	H(3)	C(14)[3)]	3.313
H(3)	H(9)[2)]	2.921	H(3)	H(18)[3)]	3.285
H(3)	H(19)[3)]	2.715	H(3)	H(20)[3)]	2.918

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(3)	H(21)[3)]	3.152	H(3)	H(22)[3)]	2.876
H(3)	H(23)[3)]	3.384	H(3)	H(29)[4)]	3.425
H(4)	C(1)[1)]	3.587	H(4)	C(7)[5)]	3.564
H(4)	C(14)[6)]	3.547	H(4)	H(1)[1)]	2.663
H(4)	H(9)[5)]	3.208	H(4)	H(10)[5)]	2.955
H(4)	H(13)[1)]	3.188	H(4)	H(22)[6)]	3.386
H(4)	H(23)[6)]	2.849	H(5)	C(7)[5)]	3.178
H(5)	H(10)[5)]	2.628	H(6)	H(10)[5)]	3.374
H(6)	H(23)[6)]	3.131	H(6)	H(26)[6)]	3.188
H(7)	C(16)[4)]	3.563	H(7)	H(27)[4)]	2.839
H(7)	H(29)[4)]	3.520	H(8)	C(15)[14)]	3.509
H(8)	C(16)[14)]	3.474	H(8)	H(15)[7)]	3.318
H(8)	H(18)[3)]	3.210	H(8)	H(19)[7)]	3.558
H(8)	H(25)[14)]	3.588	H(8)	H(26)[14)]	2.921
H(8)	H(28)[14)]	2.985	H(8)	H(29)[14)]	3.406
H(9)	C(1)[15)]	3.014	H(9)	C(13)[7)]	3.366
H(9)	H(1)[15)]	2.756	H(9)	H(2)[15)]	2.853
H(9)	H(3)[15)]	2.921	H(9)	H(4)[3)]	3.208
H(9)	H(12)[3)]	3.283	H(9)	H(15)[7)]	3.542
H(9)	H(19)[7)]	2.418	H(9)	H(20)[3)]	3.542
H(9)	H(22)[7)]	2.970	H(10)	C(2)[3)]	3.141
H(10)	C(14)[7)]	3.390	H(10)	C(15)[7)]	3.305
H(10)	H(4)[3)]	2.955	H(10)	H(5)[3)]	2.628
H(10)	H(6)[3)]	3.374	H(10)	H(22)[7)]	2.813
H(10)	H(23)[7)]	3.382	H(10)	H(24)[7)]	2.576
H(10)	H(26)[7)]	3.491	H(11)	C(6)[7)]	3.549
H(11)	C(7)[7)]	3.423	H(11)	C(8)[7)]	3.532
H(11)	C(15)[7)]	3.295	H(11)	H(21)[3)]	3.596
H(11)	H(24)[7)]	2.354	H(12)	C(1)[1)]	3.261
H(12)	H(1)[1)]	2.533	H(12)	H(2)[1)]	3.187
H(12)	H(9)[5)]	3.283	H(13)	C(14)[3)]	3.226
H(13)	H(1)[1)]	3.460	H(13)	H(4)[1)]	3.188
H(13)	H(21)[3)]	3.134	H(13)	H(23)[3)]	2.528
H(14)	C(15)[8)]	3.311	H(14)	H(1)[1)]	3.315
H(14)	H(2)[1)]	3.232	H(14)	H(25)[8)]	2.474
H(14)	H(26)[8)]	3.380	H(15)	C(5)[7)]	3.183
H(15)	C(6)[7)]	3.390	H(15)	H(8)[7)]	3.318

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(15)	H(9)[7)]	3.542	H(15)	H(16)[9)]	3.103
H(15)	H(17)[9)]	3.564	H(15)	H(24)[7)]	3.034
H(15)	H(25)[7)]	3.505	H(16)	C(12)[9)]	3.094
H(16)	C(15)[8)]	3.413	H(16)	H(15)[9)]	3.103
H(16)	H(16)[9)]	2.773	H(16)	H(17)[9)]	2.891
H(16)	H(24)[8)]	3.516	H(16)	H(25)[8)]	2.873
H(16)	H(26)[8)]	3.330	H(16)	H(28)[10)]	3.574
H(17)	C(12)[9)]	3.557	H(17)	H(15)[9)]	3.564
H(17)	H(16)[9)]	2.891	H(17)	H(21)[3)]	3.031
H(17)	H(23)[3)]	3.457	H(17)	H(24)[7)]	3.144
H(18)	C(4)[5)]	3.528	H(18)	C(5)[5)]	3.186
H(18)	C(6)[5)]	3.460	H(18)	H(3)[5)]	3.285
H(18)	H(8)[5)]	3.210	H(18)	H(26)[8)]	3.410
H(18)	H(28)[10)]	3.012	H(19)	C(1)[5)]	3.380
H(19)	C(5)[7)]	3.568	H(19)	C(6)[7)]	2.994
H(19)	H(2)[5)]	3.272	H(19)	H(3)[5)]	2.715
H(19)	H(8)[7)]	3.558	H(19)	H(9)[7)]	2.418
H(19)	H(28)[10)]	3.258	H(20)	C(5)[5)]	3.593
H(20)	C(6)[5)]	3.267	H(20)	C(7)[5)]	3.373
H(20)	H(3)[5)]	2.918	H(20)	H(9)[5)]	3.542
H(21)	H(3)[5)]	3.152	H(21)	H(11)[5)]	3.596
H(21)	H(13)[5)]	3.134	H(21)	H(17)[5)]	3.031
H(22)	C(1)[5)]	3.516	H(22)	C(6)[7)]	3.534
H(22)	C(7)[7)]	3.471	H(22)	H(1)[5)]	3.282
H(22)	H(3)[5)]	2.876	H(22)	H(4)[11)]	3.386
H(22)	H(9)[7)]	2.970	H(22)	H(10)[7)]	2.813
H(23)	C(2)[11)]	3.412	H(23)	C(11)[5)]	3.464
H(23)	H(1)[5)]	3.324	H(23)	H(3)[5)]	3.384
H(23)	H(4)[11)]	2.849	H(23)	H(6)[11)]	3.131
H(23)	H(10)[7)]	3.382	H(23)	H(13)[5)]	2.528
H(23)	H(17)[5)]	3.457	H(24)	C(7)[7)]	3.567
H(24)	C(9)[7)]	3.473	H(24)	C(12)[7)]	3.538
H(24)	H(10)[7)]	2.576	H(24)	H(11)[7)]	2.354
H(24)	H(15)[7)]	3.034	H(24)	H(16)[13)]	3.516
H(24)	H(17)[7)]	3.144	H(25)	C(11)[13)]	3.392
H(25)	H(8)[12)]	3.588	H(25)	H(14)[13)]	2.474
H(25)	H(15)[7)]	3.505	H(25)	H(16)[13)]	2.873

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(26)	H(6)[11)]	3.188	H(26)	H(8)[12)]	2.921
H(26)	H(10)[7)]	3.491	H(26)	H(14)[13)]	3.380
H(26)	H(16)[13)]	3.330	H(26)	H(18)[13)]	3.410
H(27)	H(2)[4)]	3.457	H(27)	H(7)[4)]	2.839
H(28)	C(13)[16)]	3.572	H(28)	H(2)[4)]	3.210
H(28)	H(8)[12)]	2.985	H(28)	H(16)[16)]	3.574
H(28)	H(18)[16)]	3.012	H(28)	H(19)[16)]	3.258
H(29)	C(1)[4)]	3.102	H(29)	H(2)[4)]	2.177
H(29)	H(3)[4)]	3.425	H(29)	H(7)[4)]	3.520
H(29)	H(8)[12)]	3.406			

Symmetry Operators:

(1) -X,-Y+1,-Z
(2) X+1/2-1,-Y+1/2,Z+1/2-1
(3) -X+1/2,Y+1/2-1,-Z+1/2
(4) -X+1,-Y+1,-Z
(5) -X+1/2,Y+1/2,-Z+1/2
(6) X+1/2-1,-Y+1/2+1,Z+1/2-1
(7) -X+1,-Y+1,-Z+1
(8) X-1,Y,Z
(9) -X,-Y+1,-Z+1
(10) X+1/2-1,-Y+1/2+1,Z+1/2
(11) X+1/2,-Y+1/2+1,Z+1/2
(12) -X+1/2+1,Y+1/2,-Z+1/2
(13) X+1,Y,Z
(14) -X+1/2+1,Y+1/2-1,-Z+1/2
(15) X+1/2,-Y+1/2,Z+1/2
(16) X+1/2,-Y+1/2+1,Z+1/2-1