Supplementary information:

X-ray Experimental for $Al_{10}O_6{}^{i}Bu_{16}(\mu\text{-}H)_2$

X-ray diffraction data were collected at 100 K on an Enraf-Nonius CAD4 diffractometer equipped with an Oxford Cryostream chiller, MoKα (λ=0.71073 Å) radiation and a graphite monochromator. Data reduction included corrections for background, Lorentz, polarization, and absorption effects. Absorption corrections were based on ψ-scans. The structure was solved by direct methods and refined using the MolEN programs.(Fair, 1990) Refinement was by full-matrix least squares, with neutral-atom scattering factors and anomalous dispersion corrections. Weights were $w=4F_o^2[\sigma^2(I)+(0.02F_o^2)^2]^{-1}$. The central C atom of one isobutyl group (C14) was found to be disordered into two sites, which were assigned fixed occupancies of 58/42%, based on prior refinement of this variable. All non-hydrogen atoms were refined anisotropically, except for the disordered C14. The bridging hydride H atom was located in a difference map and individually refined. All other H atoms were placed in calculated positions and treated as riding, except for those in the disordered isobutyl group, which were not included in the model. Crystal data, final R values, and other details are included in Table 2.

The compound crystallizes on an inversion center in the space group P-1. Al1-O1-Al2-O2 are found to be coplanar up to a maximum deviation of 0.020 (1) Å. The shortest Al–Al distance (Al1-Al2) is found to be 2.616 (1) Å. The three coordinate Al sites are found to be planar up to a maximum deviation of 0.0304 (6) Å. The following planes have all co-planar atoms:

(1) Al1-Al2-Al4-Al5 (maximum deviation is 0.0234(5) Å);

(2) Al4-Al5-Al4'-Al5' (max. dev. 0.0000(5) Å); and

(3) Al1-Al2-Al3-Al1'-Al2'-Al3' (max. dev. 0.0644(5) Å)

and exhibit dihedral angles of:

planes (1) and (2) = 88.63(5)°;

planes (1) and (3) = 90.93(4)°; and

planes (2) and (3) = 87.68(4)°.

C.K. Fair, "MolEN, An Interactive Structure Solution Procedure", Delft, The Netherlands, 1990.

Table 2. Crystal data and X-ray Data Collection Parameters for $Al_{10}O_6{}^{i}Bu_{16}(\mu\text{-}H)_2$

Formula	$C_{64}H_{146}O_6Al_{10}$
Color/shape	colorless fragment
Formula weight	1281.7
Space group	Triclinic, P-1
Temp., K	100
Cell constants	
a, Å	13.443(5)
b, Å	14.180(5)
c, Å	14.278(6)
α, deg.	98.27(3)
β, deg.	115.92(3)
γ, deg.	112.25(3)
Cell volume, Å^3	2103(4)
Formula units/unit cell	1
D_{calc}, g cm^{-3}	1.012
μ_{calc}, cm^{-1}	1.5
Diffractometer / scan	Enraf-Nonius CAD4 /ω-2θ
Radiation, graphite monochr.	MoKα (λ= 0.71073 Å)
Crystal dimensions, mm	0.22 x 0.30 x 0.55
Reflections measured	10,006
R_{int}	0.039
Independent reflections	9613
2θ range, deg	5< 2θ <55
Range of *h,k,l*	±17, 18, ±18
Reflections observed	6413
Criterion for observed	I>1σ(I)
Data/parameters	6413/364
R (obs)	0.063
Max resid. peaks (eÅ^{-3})	0.69, -0.16

Table 3. Coordinates and equivalent isotropic thermal parameters for $Al_{10}O_6{}^{i}Bu_{16}(\mu\text{-}H)_2$

Atom	Occ	x	y	z	U_{eq}(Å^2)
Al1	1	0.68523(7)	0.62469(6)	0.61326(6)	0.0127(3)
Al2	1	0.59321(7)	0.41229(6)	0.55513(6)	0.0131(3)
Al3	1	0.64576(8)	0.81507(7)	0.57520(7)	0.0192(3)
Al4	1	0.68895(7)	0.50876(7)	0.39658(7)	0.0175(3)
Al5	1	0.59560(7)	0.52526(7)	0.77595(7)	0.0156(3)
O1	1	0.6238(2)	0.5224(1)	0.6636(1)	0.0141(7)
O2	1	0.6616(2)	0.5161(1)	0.5093(1)	0.0147(7)
O3	1	0.5750(2)	0.6705(1)	0.5446(1)	0.0128(7)
C1	1	0.8485(2)	0.7611(2)	0.7085(2)	0.017(1)
C2	1	0.9715(2)	0.7534(3)	0.7583(3)	0.022(1)
C3	1	0.9841(3)	0.7041(3)	0.8476(3)	0.028(1)
C4	1	1.0884(3)	0.8653(3)	0.8070(3)	0.029(2)
C5	1	0.6572(2)	0.3125(2)	0.5967(2)	0.019(1)
C6	1	0.7960(2)	0.3677(2)	0.6953(2)	0.021(1)
C7	1	0.8271(3)	0.2837(3)	0.7387(3)	0.031(1)
C8	1	0.8879(3)	0.4300(3)	0.6642(3)	0.028(1)
C9	1	0.6456(3)	0.8909(2)	0.6995(2)	0.021(1)
C10	1	0.7305(3)	1.0158(2)	0.7608(2)	0.022(1)
C11	1	0.7098(3)	1.0575(3)	0.8514(3)	0.031(1)
C12	1	0.8698(3)	1.0515(3)	0.8098(3)	0.034(2)
C13	1	0.6974(3)	0.8597(2)	0.4737(2)	0.031(1)
C14a	0.58	0.6301(5)	0.9052(5)	0.3916(4)	0.028(1)
C14b	0.42	0.7122(7)	0.9665(6)	0.4591(6)	0.029(2)
C15	1	0.6133(4)	0.9930(3)	0.4425(3)	0.070(2)
C16	1	0.7110(4)	0.9531(4)	0.3395(3)	0.141(2)
C17	1	0.6550(3)	0.3580(2)	0.3390(2)	0.024(1)
C18	1	0.6979(3)	0.3242(3)	0.2641(3)	0.030(1)
C19	1	0.8405(3)	0.3675(3)	0.3313(3)	0.050(2)
C20	1	0.6260(3)	0.2000(3)	0.2016(3)	0.048(2)
C21	1	0.8360(3)	0.6481(3)	0.4371(2)	0.023(1)
C22	1	0.8276(3)	0.6937(3)	0.3443(3)	0.029(1)
C23	1	0.9455(4)	0.8043(3)	0.3887(3)	0.047(2)
C24	1	0.8061(3)	0.6127(3)	0.2472(3)	0.042(1)

C25	1	0.5965(2)	0.4019(2)	0.8247(2)	0.021(1)
C26	1	0.6879(3)	0.4240(2)	0.9476(2)	0.027(1)
C27	1	0.6672(3)	0.3172(3)	0.9686(3)	0.045(1)
C28	1	0.8255(3)	0.4932(3)	0.9853(3)	0.033(1)
C29	1	0.6867(3)	0.6798(2)	0.8764(2)	0.019(1)
C30	1	0.7030(3)	0.7058(2)	0.9912(2)	0.022(1)
C31	1	0.5776(3)	0.6437(3)	0.9831(3)	0.033(1)
C32	1	0.7638(3)	0.8284(3)	1.0532(3)	0.032(1)
H1Al	1	0.563(2)	0.512(2)	0.301(2)	0.016(9)

Table 4. Selected Distances in $Al_{10}O_6{}^{i}Bu_{16}(\mu\text{-}H)_2$

Atom 1	Atom 2	d / Å	Atom 1	Atom 2	d / Å
Al1	Al2	2.616(1)	C6	C7	1.529(5)
Al1	O1	1.793(2)	C6	C8	1.508(5)
Al1	O2	1.797(2)	C9	C10	1.535(4)
Al1	O3	1.795(2)	C10	C11	1.521(6)
Al1	C1	1.955(2)	C10	C12	1.513(5)
Al2	O1	1.806(2)	C13	C14a	1.532(7)
Al2	O2	1.795(2)	C13	C14b	1.51(1)
Al2	$O3^{i}$	1.792(2)	C14a	C14b	1.002(6)
Al2	C5	1.952(4)	C14a	C15	1.491(9)
Al3	O3	1.788(2)	C14a	C16	1.577(9)
Al3	C9	1.938(4)	C14b	C15	1.45(1)
Al3	C13	1.948(4)	C14b	C16	1.68(1)
Al4	O2	1.799(3)	C17	C18	1.518(6)
Al4	C17	1.968(4)	C18	C19	1.517(5)
Al4	C21	1.965(3)	C18	C20	1.528(5)
Al4	H1Al	1.67(3)	C21	C22	1.536(5)
Al5	O1	1.801(3)	C22	C23	1.525(5)
Al5	C25	1.977(4)	C22	C24	1.503(6)
Al5	C29	1.970(3)	C25	C26	1.534(4)
Al5	$H1Al^{i}$	1.72(3)	C26	C27	1.532(5)
C1	C2	1.543(5)	C26	C28	1.508(5)
C2	C3	1.517(6)	C29	C30	1.524(5)
C2	C4	1.520(4)	C30	C31	1.518(5)
C5	C6	1.535(3)	C30	C32	1.523(5)

Symmetry code (i) 1-x, 1-y, 1-z

Table 4. Selected Bond Angles in $Al_{10}O_6{}^{i}Bu_{16}(\mu\text{-}H)_2$ (in degrees).

Atom 1	Atom 2	Atom 3	angle/°	Atom 1	Atom 2	Atom 3	angle/°
Al2	Al1	O1	43.56(7)	C1	C2	C3	110.6(3)
Al2	Al1	O2	43.21(8)	C1	C2	C4	111.2(3)
Al2	Al1	O3	116.70(6)	C3	C2	C4	110.1(2)
Al2	Al1	C1	139.7(1)	Al2	C5	C6	115.4(2)
O1	Al1	O2	86.7(1)	C5	C6	C7	111.2(2)
O1	Al1	O3	111.8(1)	C5	C6	C8	111.6(3)
O1	Al1	C1	123.7(1)	C7	C6	C8	110.4(3)
O2	Al1	O3	108.66(9)	Al3	C9	C10	120.6(3)
O2	Al1	C1	121.8(1)	C9	C10	C11	111.3(3)
O3	Al1	C1	103.5(1)	C9	C10	C12	111.8(3)
Al1	Al2	O1	43.19(7)	C11	C10	C12	110.6(3)
Al1	Al2	O2	43.27(8)	Al3	C13	C14a	121.4(4)
Al1	Al2	$O3^{i}$	115.74(9)	Al3	C13	C14b	120.3(5)
Al1	Al2	C5	137.10(7)	C14a	C13	C14b	38.5(3)
O1	Al2	O2	86.4(1)	C13	C14a	C14b	69.5(6)
O1	Al2	$O3^{i}$	109.5(1)	C13	C14a	C15	114.6(4)
O1	Al2	C5	119.3(1)	C13	C14a	C16	107.9(5)
O2	Al2	$O3^{i}$	109.8(1)	C14b	C14a	C15	67.8(7)
O2	Al2	C5	123.3(1)	C14b	C14a	C16	77.9(7)
O3	$Al2^{i}$	C5	107.1(1)	C15	C14a	C16	108.6(5)
O3	Al3	C9	111.3(1)	C13	C14b	C14a	72.0(6)
O3	Al3	C13	114.0(1)	C13	C14b	C15	118.7(6)
C9	Al3	C13	134.6(1)	C13	C14b	C16	103.9(6)
O2	Al4	C17	105.7(1)	C14a	C14b	C15	72.4(7)
O2	Al4	C21	109.2(1)	C14a	C14b	C16	66.5(6)
O2	Al4	H1Al	100(1)	C15	C14b	C16	105.3(6)
C17	Al4	C21	128.8(2)	C14a	C15	C14b	39.8(3)
C17	Al4	H1Al	105 (1)	C14a	C16	C14b	35.6(3)
C21	Al4	H1Al	104 (1)	Al4	C17	C18	123.0(2)
O1	Al5	C25	111.5(1)	C17	C18	C19	110.7(3)
O1	Al5	C29	107.8(1)	C17	C18	C20	112.5(3)
O1	Al5	$H1Al^{i}$	97(1)	C19	C18	C20	110.5(3)
C25	Al5	C29	125.4(1)	Al4	C21	C22	117.3(2)
C25	Al5	$H1Al^{i}$	104(1)	C21	C22	C23	111.6(2)

C29	Al5	H1Al^{i}	106(1)	C21	C22	C24	110.9(3)
Al1	O1	Al2	93.3(1)	C23	C22	C24	110.8(4)
Al1	O1	Al5	134.0(1)	Al5	C25	C26	120.3(2)
Al2	O1	Al5	132.7(1)	C25	C26	C27	111.8(2)
Al1	O2	Al2	93.5(1)	C25	C26	C28	112.1(3)
Al1	O2	Al4	134.6(1)	C27	C26	C28	109.7(3)
Al2	O2	Al4	131.9(1)	Al5	C29	C30	119.2(2)
Al1	O3	Al2^{i}	127.5(1)	C29	C30	C31	111.7(2)
Al1	O3	Al3	114.85(8)	C29	C30	C32	112.3(3)
Al2	O3^{i}	Al3	117.6(1)	C31	C30	C32	110.1(3)
Al1	C1	C2	118.2(2)	Al4	H1Al	Al5^{i}	154(2)

Symmetry code (i) 1-x, 1-y, 1-z