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Structural characterization of three hydride-bridged sodium aluminate compounds

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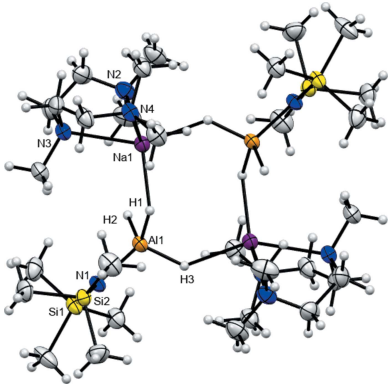
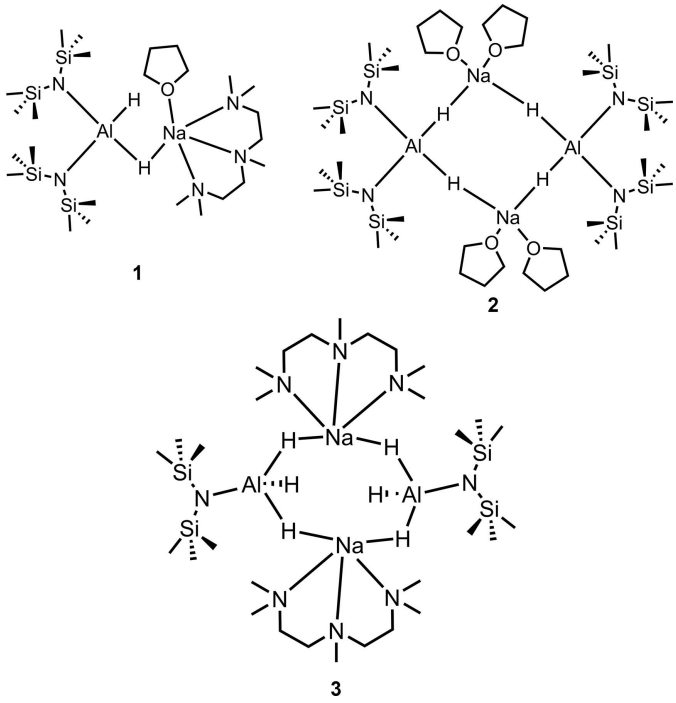
The synthesis and single-crystal structures of three hydride-bridged sodium aluminate compounds containing the utility amide HMDS [$N(SiMe_3)_2$ or $C_6H_{18}NSi_2$] are reported. Both bis[bis(trimethylsilyl)amido-2κN]-μ-hydrido-hydrido-2κH-(*N*,*N*,*N*′,*N*′′,*N*′′-pentamethyldiethylenetriamine-1$\kappa^3 N$,*N*′,*N*′′)-(tetrahydrofuran-1κO)aluminiumsodium, $[AlNa(C_6H_{18}NSi_2)_2H_2(C_4H_8O)(C_9H_{23}N_3)]$ or $(HMDS)_2Al(H)_2Na(THF)(PMDETA)$, (**1**) (THF = tetrahydrofuran, C_4H_8O; PMDETA = *N*,*N*,*N*′,*N*′′,*N*′′-pentamethyldiethylenetriamine, $C_9H_{23}N_3$) and tetrakis[bis(trimethylsilyl)amido]-3$\kappa^2 N$,4$\kappa^2 N$-tetra-μ-hydrido-tetrakis(tetrahydrofuran)-1$\kappa^2 O$,2$\kappa^2 O$-dialuminiumdisodium, $[Al_2Na_2(C_6H_{18}NSi_2)_4H_4(C_4H_8O)_4]$ or $[(HMDS)_2Al(H)_2Na(THF)_2]_2$ (**2**), are dihydrides. However, **1** is a dinuclear Al—H—Na monomer with one bridging and one terminal hydride ligand whilst in **2** all the hydride ligands bridge between Al and Na atoms to give a dimeric structure with a core $(AlHNaH)_2$ eight-membered ring. In contrast, the structure of bis[bis(trimethylsilyl)amido]-3κN,4κN-dihydrido-3κH,4κH-tetra-μ-hydrido-bis(*N*,*N*,*N*′,*N*′′,*N*′′-pentamethyldiethylenetriamine)-1$\kappa^3 N$,*N*′,*N*′′;-2$\kappa^3 N$,*N*′,*N*′′-dialuminiumdisodium, $[Al_2Na_2(C_6H_{18}NSi_2)_2H_6(C_9H_{23}N_3)_2]$ or $[(HMDS)Al(H)_3Na(PMDETA)]_2$ (**3**), also contains a $(AlHNaH)_2$ eight-membered ring but is a trihydride with two bridging and one terminal hydride ligand per Al centre. The $(AlHNaH)_2$ eight-membered rings of **2** and **3** differ in their structural details. That of **2** is based around a twofold axis and has a larger Al···Al intra-ring distance than that found in centrosymmetric **3**.

1. Chemical context

This work merges two topical areas of chemistry, namely sodium organometallic chemistry and molecular main-group hydride chemistry. Though relatively underdeveloped down the years, the former is currently receiving increased attention driven by the fact that sodium is much more earth-abundant and therefore more sustainable than lithium, an aspect exacerbated by lithium's rapidly escalating usage in battery technology (Yoshio *et al.*, 2009; Lu *et al.*, 2013; Wang *et al.*, 2015; Zhang, 2006). Recent progress has been reported in the use of sodium in cross-coupling catalysis in organic synthesis (Asako *et al.*, 2019) and in the reaction and solvation chemistry of sodium organoamides (Woltornist & Collum, 2021; Ma *et al.*, 2021). As evidenced by a recent 181 page review with the vast majority of studies covered appearing in this century, the latter chemistry is unquestionably a topic of great current interest (Roy *et al.*, 2021; Aldridge & Downs, 2001). Turning to molecular main-group hydrides, compounds have been developed that can mediate a myriad of catalytic reactions that previously were considered the preserve of transition-metal catalysts (Dando *et al.*, 1993; Liptrot *et al.*, 2015; Höllerhage *et al.*, 2021; Spielmann & Harder, 2007; Uhl, 2008).





This study focuses on sodium hydridoaluminates. Though less well known than its lithium congener ($LiAlH_4$), the parent sodium compound in this class, sodium aluminium hydride ($NaAlH_4$) has found use as a reductant or metallating agent (Zakharkin & Gavrilenko, 1962; Walker, 1976; Gavrilenko *et al.*, 1987; Eisler & Chivers, 2006), and has been considered for hydrogen-storage applications (Bogdanović *et al.*, 2000; Sheppard *et al.*, 2013; Fan *et al.*, 2009; Bogdanović *et al.*, 2007). Since our group has enjoyed success in synthesising lithium amido-hydridoaluminates that exhibit bimetallic cooperativity in performing catalytic hydroboration and metallation applications (Pollard *et al.*, 2018), here we set out to synthesize and crystallographically characterize a series of related sodium amido–hydridoaluminates. The structures obtained with just a single amide in the presence of THF and PMDETA are surprisingly diverse.

Table 1
Selected geometric parameters (Å, °) for **1**.

Al1—N2	1.883 (2)	Na1—N5	2.477 (3)
Al1—N1	1.885 (2)	Na1—N4	2.511 (3)
Al1—H1	1.59 (3)	Na1—N3	2.513 (2)
Al1—H2	1.54 (3)	Na1—H1	2.19 (3)
Na1—O1	2.334 (2)		
N2—Al1—N1	115.12 (10)	N5—Na1—N4	73.23 (9)
N2—Al1—H1	110.3 (11)	O1—Na1—N3	100.21 (8)
N1—Al1—H1	107.0 (10)	N5—Na1—N3	111.63 (9)
N2—Al1—H2	107.5 (11)	N4—Na1—N3	73.89 (9)
N1—Al1—H2	110.4 (11)	O1—Na1—H1	103.2 (7)
H1—Al1—H2	106.2 (15)	N5—Na1—H1	111.1 (8)
O1—Na1—N5	93.00 (9)	N4—Na1—H1	94.1 (8)
O1—Na1—N4	161.00 (9)	N3—Na1—H1	129.4 (7)

2. Structural commentary

As shown in Fig. 1, aluminate **1** exists as a hydride-bridged monomer with the four-coordinate aluminium centre in a slightly distorted tetrahedral geometry [bond-angle range 106.2 (15) to 115.12 (10)°; Table 1] and with the geometry of the five-coordinate sodium centre sitting near the centre of the continuum between trigonal–bipyramidal and square-pyramidal geometries (as shown by a τ_5 value of 0.527 using the method of Addison *et al.*, 1984). Bond lengths and angles are given in Table 1. Here the dihydride R_2AlH_2 unit consists of

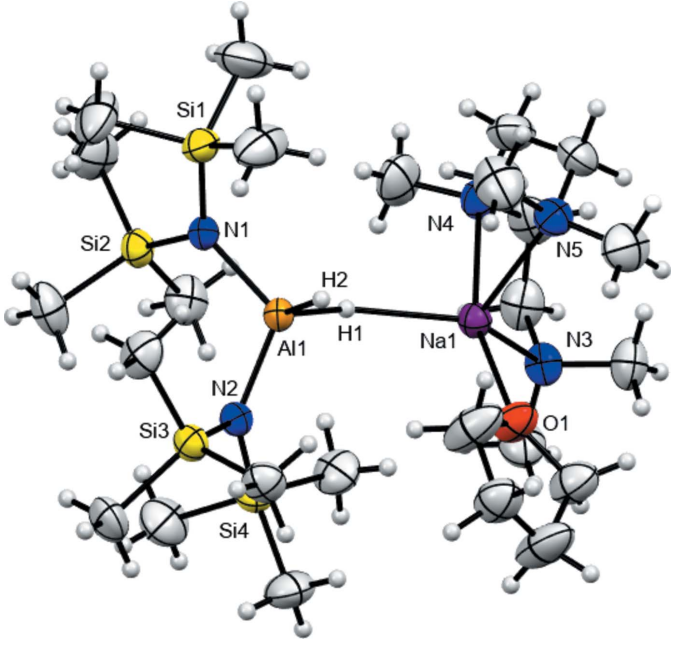


Figure 1
The molecular structure of **1** with non-H atoms shown as 50% probability ellipsoids. Hydrogen atoms are shown as small spheres of arbitrary size.

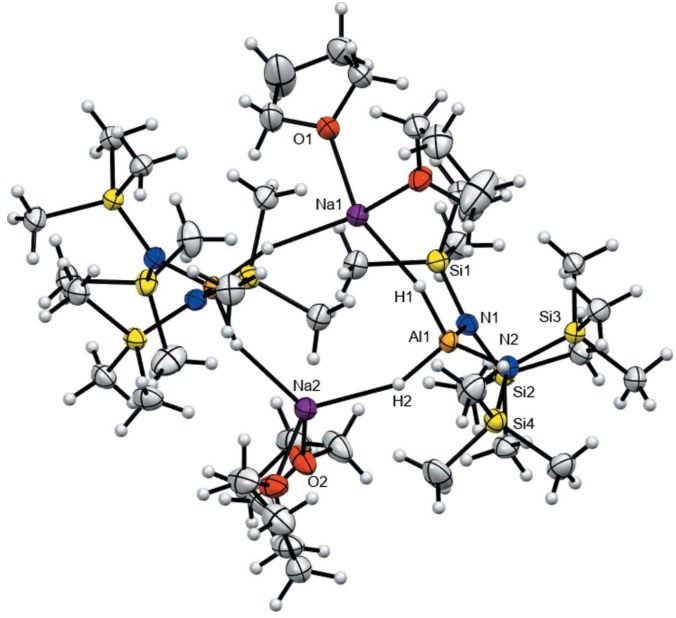


Figure 2
The molecular structure of **2** with non-H atoms shown as 50% probability ellipsoids. Hydrogen atoms are shown as small spheres of arbitrary size.

Table 2
Selected geometric parameters (Å, °) for **2**.

Al1—N2	1.872 (2)	Na1—O1	2.279 (2)
Al1—N1	1.876 (2)	Na1—H1	2.21 (3)
Al1—H1	1.55 (3)	Na2—O2	2.264 (2)
Al1—H2	1.53 (3)	Na2—O2^{i}	2.264 (2)
Na1—O1^{i}	2.279 (2)	Na2—H2	2.21 (3)
N2—Al1—N1	118.28 (11)	O1^{i}—Na1—O1	96.84 (12)
N2—Al1—H1	110.6 (12)	O1^{i}—Na1—H1	89.9 (8)
N1—Al1—H1	106.6 (12)	O1—Na1—H1	139.9 (8)
N2—Al1—H2	110.3 (13)	O2—Na2—O2^{i}	91.72 (13)
N1—Al1—H2	108.1 (13)	O2—Na2—H2	100.9 (9)
H1—Al1—H2	101.6 (18)	O2^{i}—Na2—H2	119.6 (9)

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

Table 3
Selected geometric parameters (Å, °) for **3**.

Al1—N1	1.8621 (12)	Na1—N4	2.4651 (14)
Al1—H1	1.59 (2)	Na1—N3	2.5046 (14)
Al1—H2	1.57 (3)	Na1—H1	2.25 (2)
Al1—H3	1.58 (2)	H3—Na1^{i}	2.20 (2)
Na1—N2	2.4639 (15)		
N1—Al1—Na1^{i}	138.06 (4)	N2—Na1—N4	109.15 (5)
N1—Al1—H1	115.9 (8)	N2—Na1—N3	74.35 (5)
N1—Al1—H2	111.6 (9)	N4—Na1—N3	74.05 (5)
H1—Al1—H2	102.9 (12)	N2—Na1—H1	154.8 (6)
N1—Al1—H3	112.5 (8)	N4—Na1—H1	93.8 (6)
H1—Al1—H3	105.5 (11)	N3—Na1—H1	103.3 (6)
H2—Al1—H3	107.7 (12)		

Symmetry code: (i) $-x, -y+1, -z+2$.

one terminal hydride ligand and one hydride ligand that bridges between Al and Na.

Aluminate **2** is also an R_2AlH_2 dihydride, but here both hydride ligands bridge between Al and Na centres to give the dimer shown in Fig. 2. The core feature is the eight-membered $(AlHNaH)_2$ ring highlighted in Fig. 3. This has crystallographically imposed twofold symmetry, with the 2 axis passing through both the Na1 and Na2 sites. The aluminium centre and both sodium centres occupy 4-coordinate sites with distorted tetrahedral geometries, with the sodium centres much more distorted than the aluminium [range of bond angles = 101.6 (18) to 118.28 (11)° for Al1 and 89.9 (8) to 139.9 (8) and 91.72 (13) to 121.3 (2)° for Na1 and Na2, respectively; Table 2]. Selected geometric parameters are given in Table 2.

Finally, the hydrido-rich $RAlH_3$ aluminate trihydride **3** also exists as a hydride-bridged dimer with the same skeleton of an eight-membered $(AlHNaH)_2$ ring as seen for **2**, see Figs. 4 and 5. Structure **3** thus features two bridging hydride ligands and one terminal hydride ligand per Al centre, see Table 3 for selected geometric parameters. Differences between the $(AlHNaH)_2$ rings of compounds **2** and **3** are that the ring of **3** is crystallographically centrosymmetric rather than having the twofold symmetry of **2**, and that the Al—H—Na angles of **2** are much closer to linear than the more bent angles found in **3** (compare 154.3 and 163.5° with 109.6 and 132.1°). The near linear and bent geometries result in very different Al···Al separation distances for the two compounds [compare 5.6436 (15) and 4.7666 (9) Å for **2** and **3**, respectively]. This greater distance is presumably related to the Al centres of **2** each bearing two bulky HMDS ligands whilst the Al centres of **3** each bear only one HMDS ligand. As with **1** and **2**, aluminate

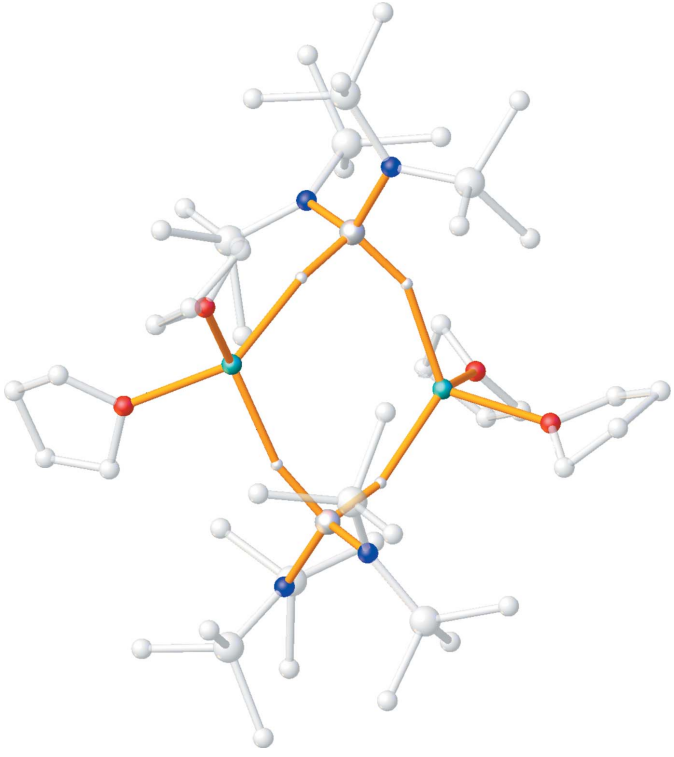


Figure 3
View of structure **2** highlighting the core $(AlHNaH)_2$ eight-membered ring.

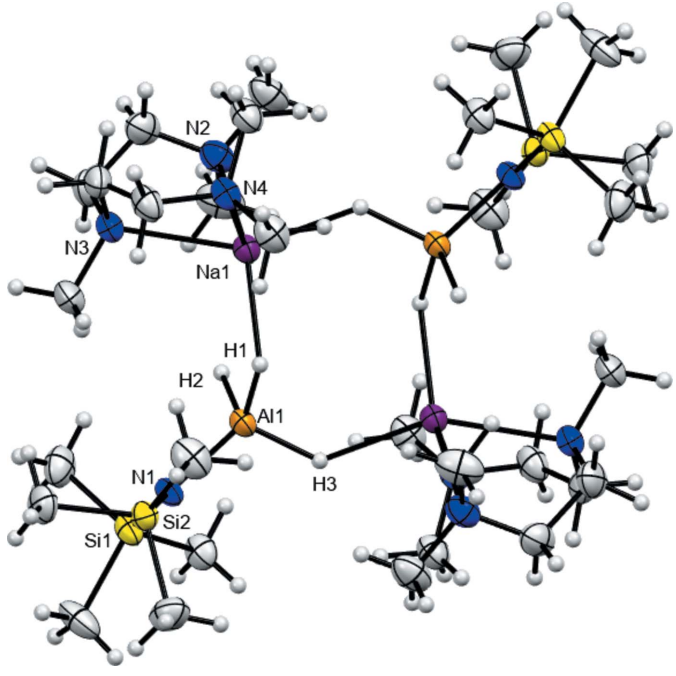


Figure 4
The molecular structure of **3** with non-H atoms shown as 50% probability ellipsoids. Hydrogen atoms are shown as small spheres of arbitrary size.

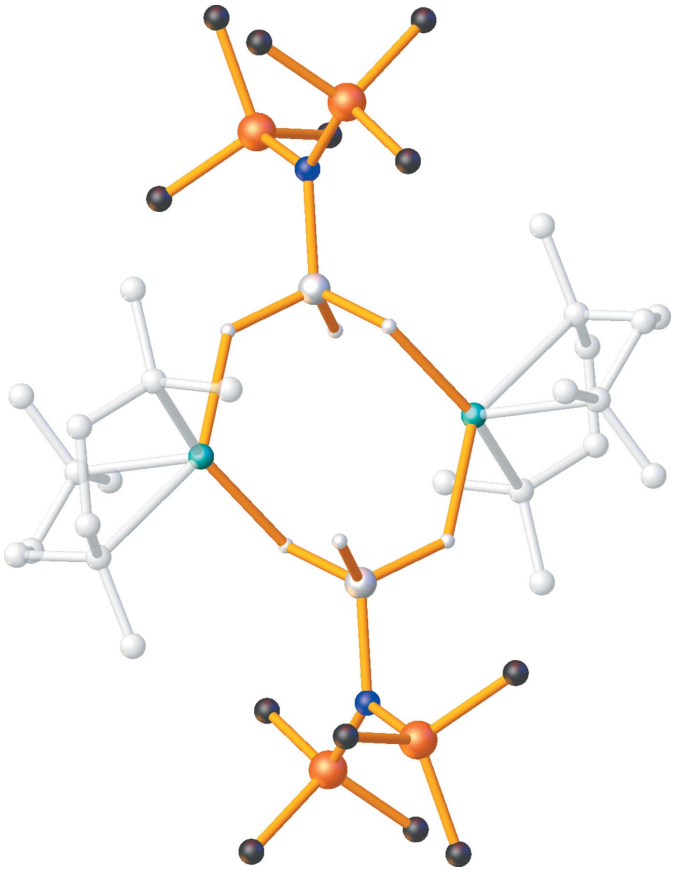


Figure 5
View of structure **3** highlighting the core $(AlHNaH)_2$ eight-membered ring. This core is a variation of that of structure **2** as highlighted in Fig. 3.

3 also contains a four-coordinate, distorted tetrahedral aluminium centre [bond angle range = 102.9 (12)–115.9 (8)°]. Like **1**, **3** also features a five-coordinate sodium centre, but here the geometry is closer to square-pyramidal as shown by a τ_5 value of 0.077. It is worth noting that Stalke previously reported an Li compound that is analogous with **3**. This contained the equivalent eight-membered ring but had two molecules of diethyl ether solvating each lithium centre rather than the chelated PMDETA found here on Na (Heine & Stalke, 1992).

For **1**, the bridging Al1—H1 bond may initially appear to be slightly longer than the terminal Al1—H2 bond [1.59 (3) and 1.54 (3) Å, respectively]. However, the large s.u. values and the general lack of accuracy of H atom positions derived from X-ray data should be kept in mind. Additionally, **3** does not show the expected difference in bridging *versus* terminal bond lengths, with all Al—H distances essentially equivalent [1.57 (3) Å for the terminal ligand and 1.59 (2) and 1.58 (2) Å for the bridging ligands], whilst the bridging hydrides of **2** are just as short as the terminal bond of **1** [1.53 (3) and 1.55 (3) Å]. Thus there is no reliable experimental evidence herein to support the notion that the bridging Al-hydride bonds should be longer than the terminal ones. All the Na—H bonds in the three compounds are bridging: they have a bond length range from 2.19 (3) Å in **1** to 2.25 (2) Å in **3**.

3. Supramolecular features

There are no intermolecular interactions significantly shorter than the sum of van der Waals radii. In all three compounds, the closest contacts are H···H contacts between methyl groups or between methyl and CH_2 groups. None of the hydride ligands show any significant intermolecular contacts, thus the monomer of **1** and the dimers of **2** and **3** can be described as discrete.

4. Database survey

A search of the Cambridge Structural Database (CSD version 2021.3, update of December 2021; Groom *et al.*, 2016) for $[NaXAlX']_2$ eight-membered rings similar to that of structures **2** and **3**, returned eleven hits; six of these had oxygen as the bridging atom (Muñoz *et al.*, 2014; Wu *et al.*, 2010; Huang *et al.*, 2009; Veith *et al.*, 2008; Nöth *et al.*, 2001), two had nitrogen (Eisler & Chivers, 2006; Böttcher *et al.*, 2001), one had fluorine (Hatop *et al.*, 2000), one had a mix of carbon and oxygen (Huang *et al.*, 2009) and the last hit had a mixture of nitrogen and carbon (Cortes-Llamas & Muñoz-Hernández, 2007).

Searching the CSD for the 'Na—H—Al' bridging unit returned just nine hits, with two of these results being $NaAlH_4$ with 15-crown-5 solvating the sodium atom (Sirsch *et al.*, 2010; Olbrich & Trzaska, 2005). This small number of structural precedents showcases the relative novelty of the newly reported structures. The Al—H bonds in these existing compounds have an average length of 1.54 Å for the terminal bonds and 1.57 Å for the bridging interactions; the Na—H bonds are all bridging and have an average length of 2.32 Å. This demonstrates that our results are generally in good agreement with those already reported in literature, although we note that the Na—H bonds reported herein are slightly shorter than the literature average.

There are 87 hits returned in the CSD when a search is carried out for the 'Na—HMDS' fragment; this reduces to 78 when only heterobimetallic compounds are considered and reduces to just two hits when 'H—Na—HMDS' is searched with one structure being a titanium-hydride compound (Stennett & Power, 2021), and the other a sodium-hydrido magnesiate (Liptrot *et al.*, 2014). When the CSD is searched for 'Al—HMDS' it returns 78 results; when only heterobimetallic species are considered this reduces to 46 hits (with 10 of these involving alkali metals) and 29 hits are returned when the 'H—Al—HMDS' fragment is searched. These relatively high numbers emphasise the importance of HMDS as a utility ligand in main-group chemistry (Mulvey & Robertson, 2013; Westerhausen, 1998).

5. Synthesis and crystallization

The synthesis of these new sodium aluminium hydride compounds was carried out by metallation *via* reaction of $NaAlH_4$ with the amine 1,1,1,3,3,3-hexamethyldisilazane in a 1:2 stoichiometric ratio in THF (tetrahydrofuran) solution. After three hours at 313 K, the reaction mixture was left at

Table 4
Experimental details.

	1	**2**	**3**
Crystal data			
Chemical formula	$[AlNa(C_6H_{18}NSi_2)_2H_2(C_4H_8O)(C_9H_{23}N_3)]$	$[Al_2Na_2(C_6H_{18}NSi_2)_4H_4(C_4H_8O)_4]$	$[Al_2Na_2(C_6H_{18}NSi_2)_2H_6(C_9H_{23}N_3)_2]$
M_r	618.18	1033.96	773.38
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$
Temperature (K)	200	100	123
a, b, c (Å)	11.9127 (9), 18.1837 (12), 18.4076 (13)	22.4151 (4), 17.2323 (2), 17.4649 (3)	9.2634 (5), 11.7188 (7), 12.6742 (7)
α, β, γ (°)	90, 94.420 (7), 90	90, 110.674 (2), 90	84.811 (5), 76.840 (5), 73.485 (5)
V (Å^3)	3975.5 (5)	6311.64 (19)	1283.97 (13)
Z	4	4	1
Radiation type	Mo $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	0.21	2.29	1.77
Crystal size (mm)	0.30 × 0.20 × 0.20	0.25 × 0.20 × 0.10	0.44 × 0.20 × 0.10
Data collection			
Diffractometer	Oxford Diffraction Gemini E	Rigaku Synergy-i	Oxford Diffraction Gemini S
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.763, 1.000	0.121, 1.000	0.256, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29795, 8563, 5685	40833, 6268, 5781	14891, 5082, 4511
R_{int}	0.063	0.063	0.028
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.639	0.620	0.620
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.155, 1.05	0.053, 0.160, 1.13	0.040, 0.112, 1.02
No. of reflections	8563	6268	5082
No. of parameters	359	293	231
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.24	0.36, −0.42	0.46, −0.21

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), SHELXT (Sheldrick, 2015*a*), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015*b*) and *Mercury* (Macrae *et al.*, 2020).

room temperature for 24 hours. Filtration of the grey suspension that formed resulted in a clear, homogeneous solution. Removal of the solvent *in vacuo* followed by re-suspension of the residue in hexane, then introduction of a stoichiometric quantity of the Lewis base donor ligand PMDETA (*N*,*N*,*N*′,*N*″,*N*″-pentamethyldiethylenetriamine), or THF, resulted in the formation of crystals suitable for single-crystal X-ray diffraction analysis. This analysis established their identities as the dihydrides $(HMDS)_2Al(H)(H)$-Na·(THF)(PMDETA), (**1**), and $[\{(HMDS)_2Al(H)(H)Na\cdot\cdot(THF)_2\}_2]$, (**2**), respectively. Attempting to repeat the synthesis of **1**, but reducing the room-temperature period from 24hours to 1 hour resulted in the formation of a trihydride product $[(HMDS)Al(H)(H)Na\cdot PMDETA]_2$, (**3**). The three crystalline products were obtained in yields of 56, 57 and 61%, respectively.

Compound **1**: ^{1}H NMR (400.03 MHz, d_8-toluene, 300 K): δ 0.48 (*s*, 36H, CH_3 of HMDS); 1.45 (*m*, J = 3.30 Hz, 8H, CH_2 of THF); 1.72 (*s*, 8H, CH_2 of PMDETA); 1.86 (*s*, 3H, CH_3 of PMDETA); 1.94 (*s*, 12H, CH_3 of PMDETA); 3.54 (*m*, J = 2.25 Hz, 8H, CH_2 of THF) ppm. ^{27}Al NMR (104.23 MHz, d_8-toluene, 300 K): δ 105.0 (*t*, J = 164.24) ppm. $^{13}C\{^1H\}$ NMR (100.59 MHz, d_8-toluene, 300 K): δ 6.3 (*s*, CH_3 of HMDS); 25.8 (*s*, CH_2 of THF); 43.6 (*s*, CH_3 of PMDETA); 45.4 (*s*, CH_3 of PMDETA); 54.2 (*s*, CH_2 of PMDETA); 57.0 (*s*, CH_2 of PMDETA); 67.8 (*s*, CH_2 of THF) ppm.

Compound **2**: ^{1}H NMR (400.03 MHz, C_6D_6, 300 K): δ 0.49 (*s*, 36H, CH_3 of HMDS); 1.38 (*m*, J = 3.47 Hz, 8H, CH_2 of THF); 3.50 (*m*, J = 2.48 Hz, 8H, CH_2 of THF) ppm. ^{27}Al NMR (104.23 MHz, C_6D_6, 300 K); δ 105.4 (*s*) ppm. ^{29}Si NMR (79.47 MHz, C_6D_6, 300 K); δ 300 K): δ −3.1 (*s*) ppm. $^{13}C\{^1H\}$ NMR (100.59 MHz, C_6D_6, 300 K); δ 6.3 (*s*, CH_3 of HMDS); 25.5 (*s*, CH_2 of THF); 68.3 (*s*, CH_2 of THF) ppm.

Compound **3**: ^{1}H NMR (400.13 MHz, d_8-toluene, 300 K): δ 0.45 (*s*, 18H, CH_3 of HMDS); 1.85 (*s*, 8H, CH_2 of PMDETA); 2.09 (*s*, 3H, CH_3 of PMDETA); 2.10 (*s*, 12H, CH_3 of PMDETA) ppm. ^{27}Al NMR (104.23 MHz, d_8-toluene, 300 K): δ 106.9 (*q*, J = 172.62 Hz) ppm. $^{13}C\{^1H\}$ NMR (100.59 MHz, d_8-toluene, 300 K): δ 5.5 (*s*, CH_3 of HMDS); 43.4 (*s*, CH_3 of PMDETA); 45.4 (*s*, CH_3 of PMDETA); 54.8 (*s*, CH_2 of PMDETA); 57.1 (*s*, CH_2 of PMDETA) ppm.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. For all structures, H atoms bound to C atoms were placed in the expected geometric positions and treated in riding modes. For CH_3 groups, C—H = 0.98 Å with $U_{iso}(H) = 1.25U_{eq}(C)$ and for CH_2 groups, C—H = 0.99 Å with $U_{iso}(H) = 1.2U_{eq}(C)$. All hydride H atoms were refined freely and isotropically.

The chosen crystal for structure **2** was treated as a two-component twin and was refined against a hklf 5 formatted reflection file. The twin matrix used was (0 −0.989 0.046 / 0 0.497 0.989 / −1 −0.004 −0.016) and the relative contributions for the twin components refined to 0.841 (4):0.159 (4).

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Structural characterization of three hydride-bridged sodium aluminate compounds

Alan R. Kennedy, Robert E. Mulvey and Michael T. Whitelaw

Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2021); cell refinement: *CrysAlis PRO* (Rigaku OD, 2021); data reduction: *CrysAlis PRO* (Rigaku OD, 2021). Program(s) used to solve structure: SHELXT (Sheldrick, 2015a) for (1), (2); *SHELXS* (Sheldrick, 2008) for (3). For all structures, program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELXL* (Sheldrick, 2015b).

Bis[bis(trimethylsilyl)amido-2κN]-μ-hydrido-hydrido-2κH-(*N*,*N*,*N*′,*N*′′,*N*′′-pentamethyldiethylenetriamine-1$\kappa^3 N$,*N*′,*N*′′)(tetrahydrofuran-1κO)aluminiumsodium (1)

Crystal data

$[AlNa(C_6H_{18}NSi_2)_2H_2(C_4H_8O)(C_9H_{23}N_3)]$
M_r = 618.18
Monoclinic, $P2_1/n$
a = 11.9127 (9) Å
b = 18.1837 (12) Å
c = 18.4076 (13) Å
β = 94.420 (7)°
V = 3975.5 (5) Å^3
Z = 4
$F(000)$ = 1368
D_x = 1.033 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5988 reflections
θ = 3.6–26.2°
μ = 0.21 mm^{-1}
T = 200 K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Gemini E diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan (CrysalisPro; Rigaku OD, 2021)
T_{min} = 0.763, T_{max} = 1.000
29795 measured reflections
8563 independent reflections
5685 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.0°, θ_{min} = 3.4°
h = −14→14
k = −21→23
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.155
S = 1.05
8563 reflections
359 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 1.3148P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Al1	0.63956 (7)	0.59440 (4)	0.80712 (4)	0.0331 (2)
Na1	0.55685 (8)	0.72504 (5)	0.94113 (5)	0.0348 (2)
Si1	0.44880 (7)	0.52445 (5)	0.70025 (4)	0.0448 (2)
Si2	0.60346 (7)	0.63621 (4)	0.64509 (4)	0.0381 (2)
Si3	0.85525 (7)	0.50748 (4)	0.80247 (4)	0.0397 (2)
Si4	0.88537 (7)	0.66751 (5)	0.82632 (5)	0.0437 (2)
O1	0.68222 (18)	0.67243 (11)	1.03006 (11)	0.0518 (5)
N1	0.56066 (18)	0.58335 (12)	0.71501 (11)	0.0343 (5)
N2	0.79757 (18)	0.59425 (11)	0.80660 (11)	0.0344 (5)
N3	0.5768 (2)	0.85845 (12)	0.97529 (13)	0.0423 (6)
N4	0.3864 (2)	0.78852 (14)	0.88184 (12)	0.0466 (6)
N5	0.3906 (2)	0.66856 (13)	0.99193 (13)	0.0438 (6)
C1	0.4592 (4)	0.4664 (2)	0.6168 (2)	0.0799 (12)
H1A	0.453165	0.497888	0.573465	0.120*
H1B	0.397962	0.430276	0.613477	0.120*
H1C	0.531733	0.440805	0.619912	0.120*
C2	0.4317 (3)	0.4567 (2)	0.7748 (2)	0.0759 (12)
H2A	0.499070	0.425762	0.781334	0.114*
H2B	0.365773	0.425688	0.762000	0.114*
H2C	0.421113	0.482991	0.820250	0.114*
C3	0.3115 (3)	0.5748 (3)	0.6918 (2)	0.0854 (13)
H3A	0.300833	0.600689	0.737434	0.128*
H3B	0.249910	0.539693	0.681527	0.128*
H3C	0.311754	0.610512	0.651871	0.128*
C4	0.4936 (3)	0.6539 (2)	0.56844 (18)	0.0660 (10)
H4A	0.472300	0.607375	0.544316	0.099*
H4B	0.523876	0.687488	0.533180	0.099*
H4C	0.427118	0.676149	0.587811	0.099*
C5	0.7243 (3)	0.59371 (19)	0.60198 (18)	0.0623 (10)
H5A	0.787843	0.587871	0.638592	0.093*
H5B	0.746598	0.625467	0.562570	0.093*
H5C	0.701865	0.545431	0.582120	0.093*
C6	0.6453 (3)	0.73071 (16)	0.67637 (18)	0.0565 (8)
H6A	0.580992	0.755089	0.696218	0.085*
H6B	0.669284	0.759156	0.635073	0.085*
H6C	0.707629	0.727456	0.714223	0.085*
C7	0.7618 (3)	0.44199 (16)	0.74853 (18)	0.0561 (8)

H7A	0.689997	0.437673	0.770779	0.084*
H7B	0.798140	0.393656	0.747910	0.084*
H7C	0.748243	0.460203	0.698514	0.084*
C8	0.9917 (3)	0.50652 (19)	0.75711 (18)	0.0598 (9)
H8A	0.978653	0.524917	0.707126	0.090*
H8B	1.020649	0.456114	0.756174	0.090*
H8C	1.046738	0.538037	0.784416	0.090*
C9	0.8824 (3)	0.46502 (17)	0.89489 (17)	0.0515 (8)
H9A	0.941049	0.493006	0.923096	0.077*
H9B	0.907408	0.414071	0.889685	0.077*
H9C	0.813040	0.465772	0.920227	0.077*
C10	0.8152 (3)	0.74902 (17)	0.8652 (2)	0.0608 (9)
H10A	0.750759	0.763914	0.832232	0.091*
H10B	0.869008	0.789767	0.871064	0.091*
H10C	0.789172	0.736068	0.912808	0.091*
C11	1.0014 (3)	0.6463 (2)	0.8979 (2)	0.0668 (10)
H11A	0.968960	0.628249	0.941836	0.100*
H11B	1.045002	0.691043	0.909527	0.100*
H11C	1.050798	0.608593	0.879638	0.100*
C12	0.9562 (3)	0.7009 (2)	0.7452 (2)	0.0727 (11)
H12A	1.007383	0.662841	0.729602	0.109*
H12B	0.998983	0.745648	0.758126	0.109*
H12C	0.899180	0.711685	0.705338	0.109*
C13	0.7129 (4)	0.59808 (19)	1.0185 (2)	0.0794 (13)
H13A	0.739248	0.592384	0.969087	0.095*
H13B	0.646853	0.565664	1.022377	0.095*
C14	0.8028 (3)	0.5774 (2)	1.0734 (2)	0.0657 (10)
H14A	0.866077	0.553911	1.050396	0.079*
H14B	0.774472	0.543060	1.109473	0.079*
C15	0.8383 (4)	0.6473 (2)	1.1081 (2)	0.0865 (14)
H15A	0.903577	0.668296	1.085061	0.104*
H15B	0.859096	0.640319	1.160768	0.104*
C16	0.7383 (3)	0.6958 (2)	1.0963 (2)	0.0650 (10)
H16A	0.688769	0.690686	1.136820	0.078*
H16B	0.761539	0.747928	1.093089	0.078*
C17	0.6899 (3)	0.89006 (19)	0.9743 (2)	0.0654 (10)
H17A	0.742078	0.862904	1.008247	0.098*
H17B	0.688110	0.941800	0.989076	0.098*
H17C	0.715079	0.886581	0.924999	0.098*
C18	0.5413 (3)	0.86402 (18)	1.04925 (17)	0.0578 (9)
H18A	0.467986	0.840096	1.051605	0.087*
H18B	0.535260	0.915954	1.062622	0.087*
H18C	0.596965	0.839744	1.083183	0.087*
C19	0.5011 (3)	0.89715 (17)	0.92279 (18)	0.0578 (9)
H19A	0.535441	0.898517	0.875505	0.069*
H19B	0.493323	0.948554	0.939388	0.069*
C20	0.3867 (3)	0.86384 (18)	0.91131 (19)	0.0594 (9)
H20A	0.351778	0.863152	0.958411	0.071*

H20B	0.339517	0.895394	0.877408	0.071*
C21	0.3929 (3)	0.7908 (2)	0.80280 (17)	0.0719 (11)
H21A	0.459166	0.819221	0.791375	0.108*
H21B	0.324847	0.814149	0.779988	0.108*
H21C	0.398900	0.740625	0.784108	0.108*
C22	0.2871 (3)	0.7477 (2)	0.89959 (19)	0.0603 (9)
H22A	0.275253	0.706168	0.865093	0.072*
H22B	0.220562	0.780358	0.892814	0.072*
C23	0.2948 (3)	0.71804 (19)	0.97632 (17)	0.0553 (8)
H23A	0.301165	0.759769	1.010931	0.066*
H23B	0.224432	0.691265	0.984341	0.066*
C24	0.4038 (3)	0.6565 (2)	1.07060 (19)	0.0701 (10)
H24A	0.469178	0.624809	1.082520	0.105*
H24B	0.336009	0.632814	1.086492	0.105*
H24C	0.415160	0.703849	1.095551	0.105*
C25	0.3733 (4)	0.5992 (2)	0.9539 (2)	0.0809 (12)
H25A	0.376104	0.607081	0.901433	0.121*
H25B	0.299568	0.579044	0.963589	0.121*
H25C	0.432434	0.564443	0.971050	0.121*
H1	0.598 (2)	0.6691 (15)	0.8409 (15)	0.052 (8)*
H2	0.607 (2)	0.5328 (15)	0.8589 (15)	0.052 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0318 (4)	0.0404 (5)	0.0275 (4)	0.0016 (3)	0.0042 (3)	−0.0022 (3)
Na1	0.0359 (6)	0.0337 (6)	0.0350 (5)	0.0033 (4)	0.0037 (4)	−0.0001 (4)
Si1	0.0394 (5)	0.0590 (5)	0.0357 (4)	−0.0115 (4)	0.0002 (4)	0.0008 (4)
Si2	0.0414 (5)	0.0422 (5)	0.0314 (4)	0.0027 (3)	0.0065 (3)	0.0047 (3)
Si3	0.0384 (4)	0.0408 (4)	0.0400 (4)	0.0079 (3)	0.0036 (3)	−0.0035 (3)
Si4	0.0397 (5)	0.0446 (5)	0.0469 (5)	−0.0087 (4)	0.0047 (4)	−0.0040 (4)
O1	0.0556 (13)	0.0492 (13)	0.0482 (12)	0.0099 (10)	−0.0120 (10)	0.0016 (10)
N1	0.0322 (12)	0.0434 (13)	0.0275 (11)	0.0004 (10)	0.0045 (9)	0.0001 (9)
N2	0.0336 (12)	0.0350 (12)	0.0349 (12)	0.0023 (9)	0.0032 (10)	−0.0022 (10)
N3	0.0510 (15)	0.0330 (13)	0.0439 (13)	0.0007 (11)	0.0094 (12)	0.0015 (10)
N4	0.0460 (15)	0.0570 (16)	0.0367 (13)	0.0075 (12)	0.0030 (11)	0.0063 (12)
N5	0.0441 (14)	0.0431 (14)	0.0447 (14)	−0.0069 (11)	0.0059 (11)	0.0040 (11)
C1	0.101 (3)	0.083 (3)	0.055 (2)	−0.034 (2)	0.007 (2)	−0.023 (2)
C2	0.073 (3)	0.090 (3)	0.062 (2)	−0.041 (2)	−0.0040 (19)	0.017 (2)
C3	0.039 (2)	0.120 (3)	0.096 (3)	−0.005 (2)	−0.003 (2)	0.011 (3)
C4	0.075 (3)	0.074 (2)	0.0466 (19)	0.0054 (19)	−0.0068 (18)	0.0160 (17)
C5	0.072 (2)	0.064 (2)	0.055 (2)	0.0109 (18)	0.0310 (18)	0.0117 (17)
C6	0.073 (2)	0.0442 (18)	0.0532 (19)	−0.0041 (16)	0.0105 (17)	0.0077 (15)
C7	0.070 (2)	0.0357 (17)	0.061 (2)	0.0084 (15)	−0.0011 (17)	−0.0102 (15)
C8	0.051 (2)	0.070 (2)	0.061 (2)	0.0158 (17)	0.0163 (17)	−0.0086 (17)
C9	0.0486 (19)	0.0513 (19)	0.0542 (19)	0.0080 (14)	0.0006 (15)	0.0055 (15)
C10	0.067 (2)	0.0426 (18)	0.072 (2)	−0.0112 (16)	0.0024 (19)	−0.0153 (16)
C11	0.050 (2)	0.076 (2)	0.072 (2)	−0.0141 (17)	−0.0112 (18)	−0.011 (2)

C12	0.069 (2)	0.079 (3)	0.073 (2)	−0.020 (2)	0.019 (2)	0.010 (2)
C13	0.095 (3)	0.052 (2)	0.084 (3)	0.0189 (19)	−0.041 (2)	−0.018 (2)
C14	0.063 (2)	0.067 (2)	0.065 (2)	0.0156 (18)	−0.0102 (19)	−0.0038 (19)
C15	0.080 (3)	0.081 (3)	0.091 (3)	0.026 (2)	−0.043 (2)	−0.029 (2)
C16	0.065 (2)	0.058 (2)	0.068 (2)	0.0061 (17)	−0.0147 (19)	−0.0146 (18)
C17	0.068 (2)	0.057 (2)	0.073 (2)	−0.0197 (18)	0.016 (2)	−0.0072 (18)
C18	0.078 (2)	0.0506 (19)	0.0462 (18)	0.0016 (17)	0.0128 (17)	−0.0060 (15)
C19	0.079 (3)	0.0402 (18)	0.0543 (19)	0.0095 (17)	0.0064 (18)	0.0056 (15)
C20	0.061 (2)	0.055 (2)	0.062 (2)	0.0246 (17)	0.0062 (18)	0.0155 (17)
C21	0.082 (3)	0.092 (3)	0.0418 (18)	0.011 (2)	0.0010 (18)	0.0126 (19)
C22	0.0409 (18)	0.077 (2)	0.062 (2)	0.0038 (16)	−0.0059 (16)	0.0061 (18)
C23	0.0389 (18)	0.071 (2)	0.0568 (19)	−0.0030 (16)	0.0111 (15)	0.0008 (17)
C24	0.071 (2)	0.084 (3)	0.056 (2)	−0.017 (2)	0.0095 (18)	0.0195 (19)
C25	0.087 (3)	0.059 (2)	0.099 (3)	−0.016 (2)	0.026 (3)	−0.014 (2)

Geometric parameters (Å, º)

Al1—N2	1.883 (2)	C7—H7A	0.9800
Al1—N1	1.885 (2)	C7—H7B	0.9800
Al1—H1	1.59 (3)	C7—H7C	0.9800
Al1—H2	1.54 (3)	C8—H8A	0.9800
Na1—O1	2.334 (2)	C8—H8B	0.9800
Na1—N5	2.477 (3)	C8—H8C	0.9800
Na1—N4	2.511 (3)	C9—H9A	0.9800
Na1—N3	2.513 (2)	C9—H9B	0.9800
Na1—H1	2.19 (3)	C9—H9C	0.9800
Si1—N1	1.715 (2)	C10—H10A	0.9800
Si1—C2	1.868 (3)	C10—H10B	0.9800
Si1—C3	1.871 (4)	C10—H10C	0.9800
Si1—C1	1.875 (4)	C11—H11A	0.9800
Si2—N1	1.715 (2)	C11—H11B	0.9800
Si2—C5	1.864 (3)	C11—H11C	0.9800
Si2—C6	1.868 (3)	C12—H12A	0.9800
Si2—C4	1.877 (3)	C12—H12B	0.9800
Si3—N2	1.725 (2)	C12—H12C	0.9800
Si3—C7	1.863 (3)	C13—C14	1.464 (5)
Si3—C9	1.873 (3)	C13—H13A	0.9900
Si3—C8	1.884 (3)	C13—H13B	0.9900
Si4—N2	1.715 (2)	C14—C15	1.470 (5)
Si4—C10	1.871 (3)	C14—H14A	0.9900
Si4—C12	1.872 (3)	C14—H14B	0.9900
Si4—C11	1.874 (4)	C15—C16	1.485 (5)
O1—C16	1.411 (4)	C15—H15A	0.9900
O1—C13	1.421 (4)	C15—H15B	0.9900
N3—C19	1.451 (4)	C16—H16A	0.9900
N3—C18	1.460 (4)	C16—H16B	0.9900
N3—C17	1.466 (4)	C17—H17A	0.9800
N4—C22	1.455 (4)	C17—H17B	0.9800

N4—C21	1.463 (4)	C17—H17C	0.9800
N4—C20	1.473 (4)	C18—H18A	0.9800
N5—C25	1.450 (4)	C18—H18B	0.9800
N5—C24	1.461 (4)	C18—H18C	0.9800
N5—C23	1.464 (4)	C19—C20	1.492 (5)
C1—H1A	0.9800	C19—H19A	0.9900
C1—H1B	0.9800	C19—H19B	0.9900
C1—H1C	0.9800	C20—H20A	0.9900
C2—H2A	0.9800	C20—H20B	0.9900
C2—H2B	0.9800	C21—H21A	0.9800
C2—H2C	0.9800	C21—H21B	0.9800
C3—H3A	0.9800	C21—H21C	0.9800
C3—H3B	0.9800	C22—C23	1.508 (4)
C3—H3C	0.9800	C22—H22A	0.9900
C4—H4A	0.9800	C22—H22B	0.9900
C4—H4B	0.9800	C23—H23A	0.9900
C4—H4C	0.9800	C23—H23B	0.9900
C5—H5A	0.9800	C24—H24A	0.9800
C5—H5B	0.9800	C24—H24B	0.9800
C5—H5C	0.9800	C24—H24C	0.9800
C6—H6A	0.9800	C25—H25A	0.9800
C6—H6B	0.9800	C25—H25B	0.9800
C6—H6C	0.9800	C25—H25C	0.9800

N2—Al1—N1	115.12 (10)	Si3—C8—H8A	109.5
N2—Al1—H1	110.3 (11)	Si3—C8—H8B	109.5
N1—Al1—H1	107.0 (10)	H8A—C8—H8B	109.5
N2—Al1—H2	107.5 (11)	Si3—C8—H8C	109.5
N1—Al1—H2	110.4 (11)	H8A—C8—H8C	109.5
H1—Al1—H2	106.2 (15)	H8B—C8—H8C	109.5
O1—Na1—N5	93.00 (9)	Si3—C9—H9A	109.5
O1—Na1—N4	161.00 (9)	Si3—C9—H9B	109.5
N5—Na1—N4	73.23 (9)	H9A—C9—H9B	109.5
O1—Na1—N3	100.21 (8)	Si3—C9—H9C	109.5
N5—Na1—N3	111.63 (9)	H9A—C9—H9C	109.5
N4—Na1—N3	73.89 (9)	H9B—C9—H9C	109.5
O1—Na1—H1	103.2 (7)	Si4—C10—H10A	109.5
N5—Na1—H1	111.1 (8)	Si4—C10—H10B	109.5
N4—Na1—H1	94.1 (8)	H10A—C10—H10B	109.5
N3—Na1—H1	129.4 (7)	Si4—C10—H10C	109.5
N1—Si1—C2	115.05 (14)	H10A—C10—H10C	109.5
N1—Si1—C3	111.78 (16)	H10B—C10—H10C	109.5
C2—Si1—C3	103.9 (2)	Si4—C11—H11A	109.5
N1—Si1—C1	112.45 (15)	Si4—C11—H11B	109.5
C2—Si1—C1	104.39 (19)	H11A—C11—H11B	109.5
C3—Si1—C1	108.6 (2)	Si4—C11—H11C	109.5
N1—Si2—C5	111.77 (13)	H11A—C11—H11C	109.5
N1—Si2—C6	111.84 (12)	H11B—C11—H11C	109.5

C5—Si2—C6	108.43 (16)	Si4—C12—H12A	109.5
N1—Si2—C4	115.45 (14)	Si4—C12—H12B	109.5
C5—Si2—C4	105.54 (17)	H12A—C12—H12B	109.5
C6—Si2—C4	103.18 (17)	Si4—C12—H12C	109.5
N2—Si3—C7	112.61 (13)	H12A—C12—H12C	109.5
N2—Si3—C9	112.24 (12)	H12B—C12—H12C	109.5
C7—Si3—C9	105.96 (15)	O1—C13—C14	108.9 (3)
N2—Si3—C8	112.84 (13)	O1—C13—H13A	109.9
C7—Si3—C8	104.96 (15)	C14—C13—H13A	109.9
C9—Si3—C8	107.70 (15)	O1—C13—H13B	109.9
N2—Si4—C10	114.41 (13)	C14—C13—H13B	109.9
N2—Si4—C12	112.82 (15)	H13A—C13—H13B	108.3
C10—Si4—C12	106.89 (17)	C13—C14—C15	104.5 (3)
N2—Si4—C11	113.19 (14)	C13—C14—H14A	110.9
C10—Si4—C11	102.75 (17)	C15—C14—H14A	110.9
C12—Si4—C11	105.92 (18)	C13—C14—H14B	110.9
C16—O1—C13	107.9 (2)	C15—C14—H14B	110.9
C16—O1—Na1	135.86 (19)	H14A—C14—H14B	108.9
C13—O1—Na1	116.29 (19)	C14—C15—C16	104.4 (3)
Si1—N1—Si2	120.23 (13)	C14—C15—H15A	110.9
Si1—N1—Al1	122.45 (12)	C16—C15—H15A	110.9
Si2—N1—Al1	117.31 (12)	C14—C15—H15B	110.9
Si4—N2—Si3	118.77 (13)	C16—C15—H15B	110.9
Si4—N2—Al1	126.25 (12)	H15A—C15—H15B	108.9
Si3—N2—Al1	113.78 (12)	O1—C16—C15	105.6 (3)
C19—N3—C18	112.2 (3)	O1—C16—H16A	110.6
C19—N3—C17	109.0 (3)	C15—C16—H16A	110.6
C18—N3—C17	108.4 (3)	O1—C16—H16B	110.6
C19—N3—Na1	105.05 (18)	C15—C16—H16B	110.6
C18—N3—Na1	105.78 (17)	H16A—C16—H16B	108.8
C17—N3—Na1	116.38 (19)	N3—C17—H17A	109.5
C22—N4—C21	110.1 (3)	N3—C17—H17B	109.5
C22—N4—C20	111.7 (3)	H17A—C17—H17B	109.5
C21—N4—C20	109.9 (3)	N3—C17—H17C	109.5
C22—N4—Na1	108.21 (18)	H17A—C17—H17C	109.5
C21—N4—Na1	110.0 (2)	H17B—C17—H17C	109.5
C20—N4—Na1	106.78 (18)	N3—C18—H18A	109.5
C25—N5—C24	110.4 (3)	N3—C18—H18B	109.5
C25—N5—C23	111.1 (3)	H18A—C18—H18B	109.5
C24—N5—C23	108.0 (3)	N3—C18—H18C	109.5
C25—N5—Na1	105.5 (2)	H18A—C18—H18C	109.5
C24—N5—Na1	114.1 (2)	H18B—C18—H18C	109.5
C23—N5—Na1	107.76 (17)	N3—C19—C20	114.3 (3)
Si1—C1—H1A	109.5	N3—C19—H19A	108.7
Si1—C1—H1B	109.5	C20—C19—H19A	108.7
H1A—C1—H1B	109.5	N3—C19—H19B	108.7
Si1—C1—H1C	109.5	C20—C19—H19B	108.7
H1A—C1—H1C	109.5	H19A—C19—H19B	107.6

H1B—C1—H1C	109.5	N4—C20—C19	113.9 (3)
Si1—C2—H2A	109.5	N4—C20—H20A	108.8
Si1—C2—H2B	109.5	C19—C20—H20A	108.8
H2A—C2—H2B	109.5	N4—C20—H20B	108.8
Si1—C2—H2C	109.5	C19—C20—H20B	108.8
H2A—C2—H2C	109.5	H20A—C20—H20B	107.7
H2B—C2—H2C	109.5	N4—C21—H21A	109.5
Si1—C3—H3A	109.5	N4—C21—H21B	109.5
Si1—C3—H3B	109.5	H21A—C21—H21B	109.5
H3A—C3—H3B	109.5	N4—C21—H21C	109.5
Si1—C3—H3C	109.5	H21A—C21—H21C	109.5
H3A—C3—H3C	109.5	H21B—C21—H21C	109.5
H3B—C3—H3C	109.5	N4—C22—C23	113.6 (3)
Si2—C4—H4A	109.5	N4—C22—H22A	108.8
Si2—C4—H4B	109.5	C23—C22—H22A	108.8
H4A—C4—H4B	109.5	N4—C22—H22B	108.8
Si2—C4—H4C	109.5	C23—C22—H22B	108.8
H4A—C4—H4C	109.5	H22A—C22—H22B	107.7
H4B—C4—H4C	109.5	N5—C23—C22	113.2 (3)
Si2—C5—H5A	109.5	N5—C23—H23A	108.9
Si2—C5—H5B	109.5	C22—C23—H23A	108.9
H5A—C5—H5B	109.5	N5—C23—H23B	108.9
Si2—C5—H5C	109.5	C22—C23—H23B	108.9
H5A—C5—H5C	109.5	H23A—C23—H23B	107.8
H5B—C5—H5C	109.5	N5—C24—H24A	109.5
Si2—C6—H6A	109.5	N5—C24—H24B	109.5
Si2—C6—H6B	109.5	H24A—C24—H24B	109.5
H6A—C6—H6B	109.5	N5—C24—H24C	109.5
Si2—C6—H6C	109.5	H24A—C24—H24C	109.5
H6A—C6—H6C	109.5	H24B—C24—H24C	109.5
H6B—C6—H6C	109.5	N5—C25—H25A	109.5
Si3—C7—H7A	109.5	N5—C25—H25B	109.5
Si3—C7—H7B	109.5	H25A—C25—H25B	109.5
H7A—C7—H7B	109.5	N5—C25—H25C	109.5
Si3—C7—H7C	109.5	H25A—C25—H25C	109.5
H7A—C7—H7C	109.5	H25B—C25—H25C	109.5
H7B—C7—H7C	109.5		

C2—Si1—N1—Si2	167.40 (19)	C8—Si3—N2—Al1	151.73 (15)
C3—Si1—N1—Si2	−74.4 (2)	N1—Al1—N2—Si4	112.19 (15)
C1—Si1—N1—Si2	48.1 (2)	N1—Al1—N2—Si3	−80.58 (14)
C2—Si1—N1—Al1	−13.7 (2)	C16—O1—C13—C14	−8.2 (5)
C3—Si1—N1—Al1	104.5 (2)	Na1—O1—C13—C14	172.3 (3)
C1—Si1—N1—Al1	−133.02 (19)	O1—C13—C14—C15	−11.0 (5)
C5—Si2—N1—Si1	−97.36 (19)	C13—C14—C15—C16	24.8 (5)
C6—Si2—N1—Si1	140.84 (16)	C13—O1—C16—C15	23.9 (4)
C4—Si2—N1—Si1	23.3 (2)	Na1—O1—C16—C15	−156.7 (3)
C5—Si2—N1—Al1	83.68 (18)	C14—C15—C16—O1	−30.4 (5)

C6—Si2—N1—Al1	−38.12 (19)	C18—N3—C19—C20	−67.6 (4)
C4—Si2—N1—Al1	−155.69 (16)	C17—N3—C19—C20	172.3 (3)
N2—Al1—N1—Si1	134.48 (13)	Na1—N3—C19—C20	46.9 (3)
N2—Al1—N1—Si2	−46.58 (16)	C22—N4—C20—C19	155.9 (3)
C10—Si4—N2—Si3	−156.81 (16)	C21—N4—C20—C19	−81.5 (3)
C12—Si4—N2—Si3	80.7 (2)	Na1—N4—C20—C19	37.8 (3)
C11—Si4—N2—Si3	−39.6 (2)	N3—C19—C20—N4	−62.2 (4)
C10—Si4—N2—Al1	9.8 (2)	C21—N4—C22—C23	158.3 (3)
C12—Si4—N2—Al1	−112.65 (19)	C20—N4—C22—C23	−79.3 (3)
C11—Si4—N2—Al1	127.09 (18)	Na1—N4—C22—C23	38.0 (3)
C7—Si3—N2—Si4	−158.62 (15)	C25—N5—C23—C22	−71.0 (4)
C9—Si3—N2—Si4	81.91 (17)	C24—N5—C23—C22	167.7 (3)
C8—Si3—N2—Si4	−40.01 (19)	Na1—N5—C23—C22	44.0 (3)
C7—Si3—N2—Al1	33.12 (18)	N4—C22—C23—N5	−58.8 (4)
C9—Si3—N2—Al1	−86.35 (16)		

Tetrakis[bis(trimethylsilyl)amido]-3κ^2N,4κ^2N-tetra-μ-hydrido-tetrakis(tetrahydrofuran)-1κ^2O,2κ^2O-dialuminiumdisodium (2)

Crystal data

$[Al_2Na_2(C_6H_{18}NSi_2)_4H_4(C_4H_8O)_4]$
M_r = 1033.96
Monoclinic, $C2/c$
a = 22.4151 (4) Å
b = 17.2323 (2) Å
c = 17.4649 (3) Å
β = 110.674 (2)°
V = 6311.64 (19) Å^3
Z = 4
$F(000)$ = 2272
D_x = 1.088 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 24656 reflections
θ = 2.7–72.5°
μ = 2.29 mm^{-1}
T = 100 K
Fragment, colourless
0.25 × 0.20 × 0.10 mm

Data collection

Rigaku Synergy-i diffractometer
Radiation source: microsource tube
ω scans
Absorption correction: multi-scan (CrysalisPro; Rigaku OD, 2021)
T_{min} = 0.121, T_{max} = 1.000
40833 measured reflections
6268 independent reflections
5781 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 72.8°, θ_{min} = 3.3°
h = −27→27
k = −21→21
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.160
S = 1.13
6268 reflections
293 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 17.678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a two-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Al1	0.48738 (4)	0.74554 (5)	0.40514 (5)	0.02599 (19)
Na1	0.500000	0.88645 (9)	0.250000	0.0325 (4)
Na2	0.500000	0.61172 (9)	0.250000	0.0341 (4)
Si1	0.62196 (4)	0.80971 (4)	0.46633 (5)	0.02963 (19)
Si2	0.58845 (4)	0.68549 (4)	0.56835 (5)	0.02967 (19)
Si3	0.41149 (4)	0.81457 (4)	0.50915 (5)	0.02863 (19)
Si4	0.34965 (4)	0.69261 (5)	0.38716 (5)	0.0320 (2)
O1	0.57142 (11)	0.97423 (12)	0.23571 (13)	0.0377 (5)
O2	0.57085 (12)	0.52024 (13)	0.31969 (13)	0.0460 (6)
N1	0.56860 (11)	0.74909 (13)	0.48689 (14)	0.0272 (5)
N2	0.41601 (11)	0.74821 (13)	0.43715 (14)	0.0278 (5)
C1	0.59865 (16)	0.91507 (18)	0.4541 (2)	0.0385 (7)
H1A	0.554713	0.920006	0.415707	0.058*
H1B	0.627356	0.943553	0.432883	0.058*
H1C	0.601733	0.936564	0.507309	0.058*
C2	0.70269 (15)	0.8121 (2)	0.5490 (2)	0.0436 (8)
H2A	0.698200	0.825640	0.601215	0.065*
H2B	0.729328	0.850928	0.535467	0.065*
H2C	0.722745	0.760917	0.553606	0.065*
C3	0.63416 (15)	0.7790 (2)	0.36960 (19)	0.0381 (7)
H3A	0.654264	0.727664	0.377494	0.057*
H3B	0.661732	0.816621	0.356140	0.057*
H3C	0.592847	0.776663	0.324765	0.057*
C4	0.66680 (16)	0.63313 (19)	0.5912 (2)	0.0436 (8)
H4A	0.676244	0.627973	0.540759	0.065*
H4B	0.663986	0.581467	0.613140	0.065*
H4C	0.700857	0.662756	0.631567	0.065*
C5	0.59591 (16)	0.73563 (19)	0.66691 (18)	0.0392 (7)
H5A	0.627146	0.777728	0.677101	0.059*
H5B	0.610032	0.698241	0.712042	0.059*
H5C	0.554421	0.757047	0.662961	0.059*
C6	0.52614 (16)	0.60790 (18)	0.54800 (19)	0.0384 (7)
H6A	0.484882	0.631662	0.541356	0.058*
H6B	0.537915	0.571569	0.594118	0.058*
H6C	0.523034	0.579928	0.497882	0.058*
C7	0.47767 (14)	0.88716 (17)	0.53522 (18)	0.0330 (6)
H7A	0.518695	0.860368	0.558584	0.050*
H7B	0.472896	0.924276	0.575190	0.050*

H7C	0.476106	0.914898	0.485563	0.050*
C8	0.33596 (15)	0.87377 (18)	0.4736 (2)	0.0367 (7)
H8A	0.333389	0.902770	0.424218	0.055*
H8B	0.336152	0.910169	0.516749	0.055*
H8C	0.299062	0.839154	0.461174	0.055*
C9	0.41473 (17)	0.76732 (18)	0.60776 (19)	0.0383 (7)
H9A	0.371958	0.749761	0.602880	0.057*
H9B	0.430228	0.804880	0.652582	0.057*
H9C	0.443697	0.722728	0.619133	0.057*
C10	0.29033 (17)	0.7455 (2)	0.2992 (2)	0.0489 (9)
H10A	0.273983	0.790570	0.319801	0.073*
H10B	0.254933	0.710651	0.270487	0.073*
H10C	0.311072	0.762878	0.261331	0.073*
C11	0.30752 (17)	0.6617 (2)	0.4580 (2)	0.0455 (8)
H11A	0.337589	0.634616	0.505253	0.068*
H11B	0.272266	0.626814	0.428917	0.068*
H11C	0.290734	0.707547	0.476773	0.068*
C12	0.36677 (19)	0.5996 (2)	0.3437 (3)	0.0561 (10)
H12A	0.378534	0.610788	0.295846	0.084*
H12B	0.328708	0.566634	0.327294	0.084*
H12C	0.402040	0.572692	0.385153	0.084*
C13	0.58808 (17)	1.04898 (18)	0.2724 (2)	0.0416 (7)
H13A	0.576298	1.053510	0.321741	0.050*
H13B	0.566101	1.090399	0.233316	0.050*
C14	0.65940 (19)	1.0549 (2)	0.2947 (2)	0.0547 (9)
H14A	0.682001	1.036790	0.351398	0.066*
H14B	0.672380	1.108948	0.289447	0.066*
C15	0.6732 (2)	1.0019 (3)	0.2329 (3)	0.0830 (16)
H15A	0.686751	1.032568	0.194063	0.100*
H15B	0.707222	0.964249	0.261184	0.100*
C16	0.61219 (17)	0.9610 (2)	0.1894 (2)	0.0478 (8)
H16A	0.592302	0.981604	0.133204	0.057*
H16B	0.619827	0.904706	0.185990	0.057*
C17	0.6185 (2)	0.5338 (2)	0.3979 (2)	0.0565 (10)
H17A	0.610593	0.502048	0.440596	0.068*
H17B	0.619886	0.589213	0.413246	0.068*
C18	0.68020 (19)	0.5094 (2)	0.3867 (2)	0.0559 (10)
H18A	0.712452	0.492711	0.439224	0.067*
H18B	0.697985	0.552445	0.363767	0.067*
C19	0.65973 (17)	0.4419 (2)	0.3265 (2)	0.0440 (8)
H19A	0.677828	0.447050	0.282646	0.053*
H19B	0.673445	0.391682	0.354859	0.053*
C20	0.58746 (18)	0.44804 (19)	0.2921 (2)	0.0466 (8)
H20A	0.571838	0.446523	0.231567	0.056*
H20B	0.568085	0.404319	0.311814	0.056*
H1	0.4847 (15)	0.8127 (18)	0.345 (2)	0.031 (8)*
H2	0.4846 (16)	0.675 (2)	0.352 (2)	0.040 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0297 (4)	0.0277 (4)	0.0201 (4)	0.0006 (3)	0.0082 (3)	0.0000 (3)
Na1	0.0435 (9)	0.0274 (8)	0.0309 (8)	0.000	0.0187 (7)	0.000
Na2	0.0473 (9)	0.0281 (8)	0.0283 (8)	0.000	0.0151 (7)	0.000
Si1	0.0308 (4)	0.0300 (4)	0.0274 (4)	−0.0012 (3)	0.0095 (3)	−0.0007 (3)
Si2	0.0366 (4)	0.0265 (4)	0.0225 (4)	0.0019 (3)	0.0061 (3)	0.0009 (3)
Si3	0.0357 (4)	0.0267 (4)	0.0257 (4)	−0.0001 (3)	0.0136 (3)	−0.0004 (3)
Si4	0.0328 (4)	0.0362 (4)	0.0271 (4)	−0.0059 (3)	0.0104 (3)	−0.0030 (3)
O1	0.0474 (12)	0.0336 (11)	0.0384 (11)	−0.0096 (9)	0.0230 (10)	−0.0095 (9)
O2	0.0650 (15)	0.0355 (12)	0.0296 (11)	0.0125 (11)	0.0068 (11)	−0.0034 (9)
N1	0.0304 (12)	0.0254 (11)	0.0233 (11)	0.0006 (9)	0.0066 (9)	−0.0015 (9)
N2	0.0311 (12)	0.0282 (12)	0.0251 (11)	−0.0005 (9)	0.0112 (9)	−0.0017 (9)
C1	0.0455 (17)	0.0330 (15)	0.0416 (17)	−0.0058 (13)	0.0211 (14)	−0.0023 (13)
C2	0.0349 (16)	0.051 (2)	0.0397 (17)	−0.0068 (14)	0.0070 (14)	−0.0024 (15)
C3	0.0376 (16)	0.0440 (17)	0.0347 (16)	0.0012 (13)	0.0153 (13)	0.0000 (13)
C4	0.0468 (18)	0.0362 (16)	0.0388 (17)	0.0084 (14)	0.0041 (14)	0.0016 (14)
C5	0.0502 (18)	0.0381 (16)	0.0264 (14)	0.0025 (14)	0.0098 (13)	0.0010 (12)
C6	0.0495 (18)	0.0304 (15)	0.0308 (15)	0.0004 (13)	0.0085 (13)	0.0058 (12)
C7	0.0410 (16)	0.0289 (14)	0.0304 (14)	0.0016 (12)	0.0140 (12)	−0.0028 (11)
C8	0.0416 (16)	0.0325 (15)	0.0406 (17)	0.0025 (12)	0.0202 (14)	0.0013 (13)
C9	0.0524 (19)	0.0352 (16)	0.0301 (15)	−0.0031 (14)	0.0181 (14)	−0.0023 (12)
C10	0.0423 (18)	0.067 (2)	0.0322 (16)	−0.0094 (16)	0.0064 (14)	0.0048 (16)
C11	0.0462 (18)	0.0499 (19)	0.0401 (17)	−0.0147 (15)	0.0149 (15)	0.0023 (15)
C12	0.052 (2)	0.053 (2)	0.065 (2)	−0.0180 (17)	0.0216 (19)	−0.0270 (19)
C13	0.058 (2)	0.0323 (16)	0.0363 (16)	−0.0070 (14)	0.0192 (15)	−0.0071 (13)
C14	0.060 (2)	0.057 (2)	0.045 (2)	−0.0206 (18)	0.0154 (17)	−0.0109 (17)
C15	0.059 (3)	0.118 (4)	0.082 (3)	−0.036 (3)	0.037 (2)	−0.047 (3)
C16	0.054 (2)	0.050 (2)	0.050 (2)	−0.0118 (16)	0.0316 (17)	−0.0158 (16)
C17	0.083 (3)	0.0425 (19)	0.0300 (16)	0.0085 (19)	0.0029 (17)	−0.0066 (14)
C18	0.064 (2)	0.045 (2)	0.044 (2)	−0.0110 (18)	0.0012 (17)	0.0013 (16)
C19	0.055 (2)	0.0398 (17)	0.0394 (17)	0.0007 (15)	0.0191 (15)	0.0029 (14)
C20	0.058 (2)	0.0321 (16)	0.0445 (18)	0.0053 (15)	0.0115 (16)	−0.0073 (14)

Geometric parameters (Å, º)

Al1—N2	1.872 (2)	C5—H5C	0.9800
Al1—N1	1.876 (2)	C6—H6A	0.9800
Al1—H1	1.55 (3)	C6—H6B	0.9800
Al1—H2	1.53 (3)	C6—H6C	0.9800
Na1—O1^{i}	2.279 (2)	C7—H7A	0.9800
Na1—O1	2.279 (2)	C7—H7B	0.9800
Na1—H1	2.21 (3)	C7—H7C	0.9800
Na2—O2	2.264 (2)	C8—H8A	0.9800
Na2—O2^{i}	2.264 (2)	C8—H8B	0.9800
Na2—H2	2.21 (3)	C8—H8C	0.9800
Si1—N1	1.719 (2)	C9—H9A	0.9800

Si1—C2	1.875 (3)	C9—H9B	0.9800
Si1—C3	1.880 (3)	C9—H9C	0.9800
Si1—C1	1.880 (3)	C10—H10A	0.9800
Si2—N1	1.725 (2)	C10—H10B	0.9800
Si2—C6	1.875 (3)	C10—H10C	0.9800
Si2—C5	1.880 (3)	C11—H11A	0.9800
Si2—C4	1.887 (3)	C11—H11B	0.9800
Si3—N2	1.729 (2)	C11—H11C	0.9800
Si3—C7	1.870 (3)	C12—H12A	0.9800
Si3—C9	1.883 (3)	C12—H12B	0.9800
Si3—C8	1.884 (3)	C12—H12C	0.9800
Si4—N2	1.725 (2)	C13—C14	1.509 (5)
Si4—C12	1.870 (4)	C13—H13A	0.9900
Si4—C10	1.875 (4)	C13—H13B	0.9900
Si4—C11	1.879 (3)	C14—C15	1.526 (6)
O1—C13	1.428 (4)	C14—H14A	0.9900
O1—C16	1.436 (4)	C14—H14B	0.9900
O2—C17	1.425 (4)	C15—C16	1.486 (5)
O2—C20	1.430 (4)	C15—H15A	0.9900
C1—H1A	0.9800	C15—H15B	0.9900
C1—H1B	0.9800	C16—H16A	0.9900
C1—H1C	0.9800	C16—H16B	0.9900
C2—H2A	0.9800	C17—C18	1.522 (6)
C2—H2B	0.9800	C17—H17A	0.9900
C2—H2C	0.9800	C17—H17B	0.9900
C3—H3A	0.9800	C18—C19	1.526 (5)
C3—H3B	0.9800	C18—H18A	0.9900
C3—H3C	0.9800	C18—H18B	0.9900
C4—H4A	0.9800	C19—C20	1.519 (5)
C4—H4B	0.9800	C19—H19A	0.9900
C4—H4C	0.9800	C19—H19B	0.9900
C5—H5A	0.9800	C20—H20A	0.9900
C5—H5B	0.9800	C20—H20B	0.9900

N2—Al1—N1	118.28 (11)	Si3—C7—H7A	109.5
N2—Al1—H1	110.6 (12)	Si3—C7—H7B	109.5
N1—Al1—H1	106.6 (12)	H7A—C7—H7B	109.5
N2—Al1—H2	110.3 (13)	Si3—C7—H7C	109.5
N1—Al1—H2	108.1 (13)	H7A—C7—H7C	109.5
H1—Al1—H2	101.6 (18)	H7B—C7—H7C	109.5
O1^{i}—Na1—O1	96.84 (12)	Si3—C8—H8A	109.5
O1^{i}—Na1—H1	89.9 (8)	Si3—C8—H8B	109.5
O1—Na1—H1	139.9 (8)	H8A—C8—H8B	109.5
O2—Na2—O2^{i}	91.72 (13)	Si3—C8—H8C	109.5
O2—Na2—H2	100.9 (9)	H8A—C8—H8C	109.5
O2^{i}—Na2—H2	119.6 (9)	H8B—C8—H8C	109.5
N1—Si1—C2	113.95 (14)	Si3—C9—H9A	109.5
N1—Si1—C3	110.44 (13)	Si3—C9—H9B	109.5

C2—Si1—C3	106.93 (15)	H9A—C9—H9B	109.5
N1—Si1—C1	115.13 (13)	Si3—C9—H9C	109.5
C2—Si1—C1	102.73 (16)	H9A—C9—H9C	109.5
C3—Si1—C1	107.01 (15)	H9B—C9—H9C	109.5
N1—Si2—C6	109.80 (13)	Si4—C10—H10A	109.5
N1—Si2—C5	112.13 (13)	Si4—C10—H10B	109.5
C6—Si2—C5	108.71 (15)	H10A—C10—H10B	109.5
N1—Si2—C4	115.34 (14)	Si4—C10—H10C	109.5
C6—Si2—C4	105.94 (15)	H10A—C10—H10C	109.5
C5—Si2—C4	104.53 (15)	H10B—C10—H10C	109.5
N2—Si3—C7	111.99 (12)	Si4—C11—H11A	109.5
N2—Si3—C9	112.70 (13)	Si4—C11—H11B	109.5
C7—Si3—C9	107.18 (14)	H11A—C11—H11B	109.5
N2—Si3—C8	113.25 (13)	Si4—C11—H11C	109.5
C7—Si3—C8	105.15 (14)	H11A—C11—H11C	109.5
C9—Si3—C8	106.02 (15)	H11B—C11—H11C	109.5
N2—Si4—C12	114.68 (14)	Si4—C12—H12A	109.5
N2—Si4—C10	112.34 (14)	Si4—C12—H12B	109.5
C12—Si4—C10	105.76 (19)	H12A—C12—H12B	109.5
N2—Si4—C11	111.61 (14)	Si4—C12—H12C	109.5
C12—Si4—C11	104.32 (18)	H12A—C12—H12C	109.5
C10—Si4—C11	107.54 (17)	H12B—C12—H12C	109.5
C13—O1—C16	106.2 (2)	O1—C13—C14	105.2 (3)
C13—O1—Na1	129.20 (19)	O1—C13—H13A	110.7
C16—O1—Na1	124.55 (19)	C14—C13—H13A	110.7
C17—O2—C20	105.6 (3)	O1—C13—H13B	110.7
C17—O2—Na2	123.2 (2)	C14—C13—H13B	110.7
C20—O2—Na2	129.9 (2)	H13A—C13—H13B	108.8
Si1—N1—Si2	124.23 (14)	C13—C14—C15	103.7 (3)
Si1—N1—Al1	114.88 (13)	C13—C14—H14A	111.0
Si2—N1—Al1	120.35 (13)	C15—C14—H14A	111.0
Si4—N2—Si3	118.29 (14)	C13—C14—H14B	111.0
Si4—N2—Al1	120.69 (13)	C15—C14—H14B	111.0
Si3—N2—Al1	120.60 (13)	H14A—C14—H14B	109.0
Si1—C1—H1A	109.5	C16—C15—C14	105.5 (3)
Si1—C1—H1B	109.5	C16—C15—H15A	110.6
H1A—C1—H1B	109.5	C14—C15—H15A	110.7
Si1—C1—H1C	109.5	C16—C15—H15B	110.6
H1A—C1—H1C	109.5	C14—C15—H15B	110.6
H1B—C1—H1C	109.5	H15A—C15—H15B	108.8
Si1—C2—H2A	109.5	O1—C16—C15	106.8 (3)
Si1—C2—H2B	109.5	O1—C16—H16A	110.4
H2A—C2—H2B	109.5	C15—C16—H16A	110.4
Si1—C2—H2C	109.5	O1—C16—H16B	110.4
H2A—C2—H2C	109.5	C15—C16—H16B	110.4
H2B—C2—H2C	109.5	H16A—C16—H16B	108.6
Si1—C3—H3A	109.5	O2—C17—C18	103.8 (3)
Si1—C3—H3B	109.5	O2—C17—H17A	111.0

H3A—C3—H3B	109.5	C18—C17—H17A	111.0
Si1—C3—H3C	109.5	O2—C17—H17B	111.0
H3A—C3—H3C	109.5	C18—C17—H17B	111.0
H3B—C3—H3C	109.5	H17A—C17—H17B	109.0
Si2—C4—H4A	109.5	C17—C18—C19	103.4 (3)
Si2—C4—H4B	109.5	C17—C18—H18A	111.1
H4A—C4—H4B	109.5	C19—C18—H18A	111.1
Si2—C4—H4C	109.5	C17—C18—H18B	111.1
H4A—C4—H4C	109.5	C19—C18—H18B	111.1
H4B—C4—H4C	109.5	H18A—C18—H18B	109.0
Si2—C5—H5A	109.5	C20—C19—C18	103.8 (3)
Si2—C5—H5B	109.5	C20—C19—H19A	111.0
H5A—C5—H5B	109.5	C18—C19—H19A	111.0
Si2—C5—H5C	109.5	C20—C19—H19B	111.0
H5A—C5—H5C	109.5	C18—C19—H19B	111.0
H5B—C5—H5C	109.5	H19A—C19—H19B	109.0
Si2—C6—H6A	109.5	O2—C20—C19	107.2 (3)
Si2—C6—H6B	109.5	O2—C20—H20A	110.3
H6A—C6—H6B	109.5	C19—C20—H20A	110.3
Si2—C6—H6C	109.5	O2—C20—H20B	110.3
H6A—C6—H6C	109.5	C19—C20—H20B	110.3
H6B—C6—H6C	109.5	H20A—C20—H20B	108.5

C2—Si1—N1—Si2	−8.5 (2)	C9—Si3—N2—Si4	74.85 (19)
C3—Si1—N1—Si2	111.87 (18)	C8—Si3—N2—Si4	−45.49 (19)
C1—Si1—N1—Si2	−126.83 (17)	C7—Si3—N2—Al1	8.37 (19)
C2—Si1—N1—Al1	−179.99 (15)	C9—Si3—N2—Al1	−112.58 (17)
C3—Si1—N1—Al1	−59.62 (17)	C8—Si3—N2—Al1	127.07 (16)
C1—Si1—N1—Al1	61.68 (18)	N1—Al1—N2—Si4	−141.53 (14)
C6—Si2—N1—Si1	−159.84 (16)	N1—Al1—N2—Si3	46.08 (18)
C5—Si2—N1—Si1	79.2 (2)	C16—O1—C13—C14	−36.7 (3)
C4—Si2—N1—Si1	−40.3 (2)	Na1—O1—C13—C14	139.8 (2)
C6—Si2—N1—Al1	11.22 (19)	O1—C13—C14—C15	27.3 (4)
C5—Si2—N1—Al1	−109.74 (17)	C13—C14—C15—C16	−8.4 (5)
C4—Si2—N1—Al1	130.78 (16)	C13—O1—C16—C15	31.3 (4)
N2—Al1—N1—Si1	−136.46 (13)	Na1—O1—C16—C15	−145.4 (3)
N2—Al1—N1—Si2	51.68 (18)	C14—C15—C16—O1	−13.2 (5)
C12—Si4—N2—Si3	−157.14 (19)	C20—O2—C17—C18	41.2 (4)
C10—Si4—N2—Si3	82.1 (2)	Na2—O2—C17—C18	−126.9 (3)
C11—Si4—N2—Si3	−38.8 (2)	O2—C17—C18—C19	−34.2 (4)
C12—Si4—N2—Al1	30.3 (2)	C17—C18—C19—C20	14.7 (4)
C10—Si4—N2—Al1	−90.49 (19)	C17—O2—C20—C19	−31.9 (4)
C11—Si4—N2—Al1	148.64 (17)	Na2—O2—C20—C19	135.1 (3)
C7—Si3—N2—Si4	−164.19 (14)	C18—C19—C20—O2	9.4 (4)

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.

Bis[bis(trimethylsilyl)amido]-3κN,4κN-dihydrido-3κH,4κH-tetra-μ-hydrido-bis(*N*,*N*,*N*′,*N*′′,*N*′′-pentamethyldiethylenetriamine)-1$\kappa^3 N$,*N*′,*N*′′;2$\kappa^3 N$,*N*′,*N*′′-dialuminiumdisodium (3)

Crystal data

$[Al_2Na_2(C_6H_{18}NSi_2)_2H_6(C_9H_{23}N_3)_2]$
M_r = 773.38
Triclinic, $P\overline{1}$
a = 9.2634 (5) Å
b = 11.7188 (7) Å
c = 12.6742 (7) Å
α = 84.811 (5)°
β = 76.840 (5)°
γ = 73.485 (5)°
V = 1283.97 (13) Å^3
Z = 1
$F(000)$ = 428
D_x = 1.000 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 6113 reflections
θ = 3.6–72.8°
μ = 1.77 mm^{-1}
T = 123 K
Fragment, colourless
0.44 × 0.20 × 0.10 mm

Data collection

Oxford Diffraction Gemini S diffractometer
Radiation source: sealed tube
ω scans
Absorption correction: multi-scan (CrysalisPro; Rigaku OD, 2021)
T_{min} = 0.256, T_{max} = 1.000
14891 measured reflections
5082 independent reflections
4511 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 73.1°, θ_{min} = 5.5°
h = −11→11
k = −11→14
l = −12→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.112
S = 1.02
5082 reflections
231 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.2717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Al1	0.15640 (5)	0.36454 (4)	0.86299 (3)	0.02993 (13)
Na1	0.09543 (6)	0.64121 (5)	0.89017 (4)	0.02873 (14)
Si1	0.42221 (5)	0.17657 (4)	0.74287 (3)	0.03074 (12)
Si2	0.14772 (5)	0.27561 (3)	0.63862 (3)	0.02898 (12)
N1	0.24175 (14)	0.27040 (11)	0.74157 (9)	0.0276 (3)
N2	0.26670 (16)	0.73937 (13)	0.94714 (12)	0.0381 (3)
N3	0.27235 (15)	0.68367 (12)	0.71937 (11)	0.0343 (3)
N4	−0.06952 (15)	0.78444 (12)	0.78285 (10)	0.0328 (3)

C1	0.5770 (2)	0.25579 (18)	0.70459 (16)	0.0483 (4)
H1A	0.579864	0.288836	0.630531	0.072*
H1B	0.676842	0.199529	0.708484	0.072*
H1C	0.555328	0.320433	0.754724	0.072*
C2	0.4294 (2)	0.10732 (15)	0.88131 (12)	0.0378 (3)
H2A	0.411673	0.169625	0.933088	0.057*
H2B	0.530737	0.050992	0.880031	0.057*
H2C	0.349417	0.065084	0.903260	0.057*
C3	0.4802 (2)	0.04831 (17)	0.64892 (15)	0.0527 (5)
H3A	0.410822	−0.002798	0.672664	0.079*
H3B	0.586164	0.002034	0.650097	0.079*
H3C	0.473813	0.078871	0.575073	0.079*
C4	0.0921 (3)	0.13492 (17)	0.63057 (16)	0.0502 (5)
H4A	0.184058	0.071871	0.600817	0.075*
H4B	0.017955	0.149016	0.583416	0.075*
H4C	0.045187	0.110525	0.703285	0.075*
C5	−0.0369 (2)	0.39697 (18)	0.65368 (16)	0.0487 (4)
H5A	−0.104651	0.386287	0.723284	0.073*
H5B	−0.087694	0.393827	0.594517	0.073*
H5C	−0.015061	0.474356	0.651117	0.073*
C6	0.2679 (2)	0.30358 (17)	0.50353 (12)	0.0426 (4)
H6A	0.296001	0.377881	0.504980	0.064*
H6B	0.208877	0.310035	0.447118	0.064*
H6C	0.361578	0.237430	0.487899	0.064*
C7	0.3711 (3)	0.63945 (18)	0.99332 (18)	0.0560 (5)
H7A	0.313825	0.609207	1.059452	0.084*
H7B	0.453733	0.666266	1.010827	0.084*
H7C	0.415752	0.575918	0.940628	0.084*
C8	0.1932 (2)	0.83010 (18)	1.02912 (16)	0.0502 (5)
H8A	0.120914	0.896553	0.999502	0.075*
H8B	0.272045	0.859085	1.049613	0.075*
H8C	0.137252	0.795444	1.093150	0.075*
C9	0.3473 (2)	0.79147 (16)	0.85071 (16)	0.0439 (4)
H9A	0.438541	0.808004	0.867603	0.053*
H9B	0.278316	0.868415	0.832065	0.053*
C10	0.39983 (19)	0.71209 (17)	0.75246 (16)	0.0450 (4)
H10A	0.454667	0.752530	0.690948	0.054*
H10B	0.473652	0.636979	0.769557	0.054*
C11	0.3315 (2)	0.57993 (16)	0.64911 (14)	0.0450 (4)
H11A	0.381879	0.510420	0.689467	0.067*
H11B	0.406052	0.596702	0.585539	0.067*
H11C	0.245788	0.563578	0.625543	0.067*
C12	0.1830 (2)	0.78566 (15)	0.66357 (13)	0.0402 (4)
H12A	0.235289	0.787619	0.586317	0.048*
H12B	0.180042	0.860256	0.695995	0.048*
C13	0.0192 (2)	0.77997 (16)	0.67094 (13)	0.0412 (4)
H13A	−0.033492	0.847476	0.628251	0.049*
H13B	0.022212	0.705336	0.638558	0.049*

C14	−0.1103 (2)	0.90356 (15)	0.82598 (14)	0.0418 (4)
H14A	−0.171600	0.904038	0.900064	0.063*
H14B	−0.170571	0.960760	0.780285	0.063*
H14C	−0.016190	0.925779	0.826504	0.063*
C15	−0.20954 (19)	0.74745 (17)	0.78988 (15)	0.0432 (4)
H15A	−0.181785	0.665877	0.764622	0.065*
H15B	−0.273938	0.801054	0.744417	0.065*
H15C	−0.266747	0.750616	0.865291	0.065*
H1	0.027 (3)	0.484 (2)	0.8479 (18)	0.057 (6)*
H2	0.281 (3)	0.411 (2)	0.899 (2)	0.067 (7)*
H3	0.080 (3)	0.296 (2)	0.9634 (18)	0.056 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Al1	0.0318 (2)	0.0301 (2)	0.0260 (2)	−0.00858 (17)	0.00097 (17)	−0.00846 (16)
Na1	0.0294 (3)	0.0304 (3)	0.0252 (3)	−0.0080 (2)	−0.0028 (2)	−0.0030 (2)
Si1	0.0321 (2)	0.0321 (2)	0.0228 (2)	−0.00108 (16)	−0.00370 (15)	−0.00400 (15)
Si2	0.0335 (2)	0.0311 (2)	0.0230 (2)	−0.00925 (16)	−0.00644 (15)	−0.00147 (15)
N1	0.0313 (6)	0.0284 (6)	0.0211 (5)	−0.0040 (5)	−0.0052 (4)	−0.0049 (4)
N2	0.0344 (7)	0.0400 (7)	0.0415 (7)	−0.0082 (6)	−0.0098 (6)	−0.0105 (6)
N3	0.0327 (7)	0.0318 (6)	0.0328 (6)	−0.0066 (5)	0.0036 (5)	−0.0059 (5)
N4	0.0312 (6)	0.0363 (7)	0.0276 (6)	−0.0036 (5)	−0.0056 (5)	−0.0046 (5)
C1	0.0310 (8)	0.0525 (10)	0.0522 (10)	−0.0063 (7)	0.0006 (7)	0.0053 (8)
C2	0.0414 (9)	0.0400 (8)	0.0291 (7)	−0.0039 (7)	−0.0116 (6)	0.0013 (6)
C3	0.0617 (12)	0.0434 (10)	0.0405 (9)	0.0130 (8)	−0.0141 (8)	−0.0172 (8)
C4	0.0692 (13)	0.0476 (10)	0.0451 (10)	−0.0302 (9)	−0.0165 (9)	−0.0012 (8)
C5	0.0395 (9)	0.0555 (11)	0.0491 (10)	−0.0008 (8)	−0.0188 (8)	−0.0063 (8)
C6	0.0528 (10)	0.0518 (10)	0.0231 (7)	−0.0163 (8)	−0.0065 (7)	0.0021 (7)
C7	0.0612 (12)	0.0476 (10)	0.0642 (13)	−0.0052 (9)	−0.0323 (10)	−0.0097 (9)
C8	0.0482 (10)	0.0535 (11)	0.0515 (10)	−0.0103 (8)	−0.0133 (8)	−0.0203 (9)
C9	0.0315 (8)	0.0439 (9)	0.0580 (11)	−0.0140 (7)	−0.0046 (7)	−0.0108 (8)
C10	0.0292 (8)	0.0486 (10)	0.0523 (10)	−0.0114 (7)	0.0057 (7)	−0.0101 (8)
C11	0.0474 (10)	0.0380 (9)	0.0401 (9)	−0.0077 (7)	0.0085 (7)	−0.0112 (7)
C12	0.0468 (9)	0.0357 (8)	0.0299 (7)	−0.0092 (7)	0.0051 (7)	0.0008 (6)
C13	0.0470 (9)	0.0447 (9)	0.0261 (7)	−0.0032 (7)	−0.0071 (7)	−0.0027 (6)
C14	0.0399 (9)	0.0393 (9)	0.0397 (9)	−0.0020 (7)	−0.0042 (7)	−0.0066 (7)
C15	0.0334 (8)	0.0513 (10)	0.0445 (9)	−0.0061 (7)	−0.0117 (7)	−0.0082 (8)

Geometric parameters (Å, º)

Al1—N1	1.8621 (12)	C3—H3C	0.9800
Al1—Na1^{i}	3.4586 (7)	C4—H4A	0.9800
Al1—H1	1.59 (2)	C4—H4B	0.9800
Al1—H2	1.57 (3)	C4—H4C	0.9800
Al1—H3	1.58 (2)	C5—H5A	0.9800
Na1—N2	2.4639 (15)	C5—H5B	0.9800
Na1—N4	2.4651 (14)	C5—H5C	0.9800

Na1—N3	2.5046 (14)	C6—H6A	0.9800
Na1—H1	2.25 (2)	C6—H6B	0.9800
H3—Na1^{i}	2.20 (2)	C6—H6C	0.9800
Si1—N1	1.7229 (13)	C7—H7A	0.9800
Si1—C2	1.8726 (16)	C7—H7B	0.9800
Si1—C1	1.8742 (19)	C7—H7C	0.9800
Si1—C3	1.8812 (17)	C8—H8A	0.9800
Si2—N1	1.7140 (12)	C8—H8B	0.9800
Si2—C5	1.8713 (18)	C8—H8C	0.9800
Si2—C6	1.8751 (17)	C9—C10	1.526 (2)
Si2—C4	1.8793 (18)	C9—H9A	0.9900
N2—C9	1.460 (2)	C9—H9B	0.9900
N2—C7	1.463 (2)	C10—H10A	0.9900
N2—C8	1.463 (2)	C10—H10B	0.9900
N3—C10	1.468 (2)	C11—H11A	0.9800
N3—C12	1.471 (2)	C11—H11B	0.9800
N3—C11	1.472 (2)	C11—H11C	0.9800
N4—C14	1.461 (2)	C12—C13	1.519 (3)
N4—C15	1.462 (2)	C12—H12A	0.9900
N4—C13	1.4656 (19)	C12—H12B	0.9900
C1—H1A	0.9800	C13—H13A	0.9900
C1—H1B	0.9800	C13—H13B	0.9900
C1—H1C	0.9800	C14—H14A	0.9800
C2—H2A	0.9800	C14—H14B	0.9800
C2—H2B	0.9800	C14—H14C	0.9800
C2—H2C	0.9800	C15—H15A	0.9800
C3—H3A	0.9800	C15—H15B	0.9800
C3—H3B	0.9800	C15—H15C	0.9800

N1—Al1—Na1^{i}	138.06 (4)	Si2—C4—H4B	109.5
N1—Al1—H1	115.9 (8)	H4A—C4—H4B	109.5
Na1^{i}—Al1—H1	81.2 (8)	Si2—C4—H4C	109.5
N1—Al1—H2	111.6 (9)	H4A—C4—H4C	109.5
Na1^{i}—Al1—H2	100.0 (9)	H4B—C4—H4C	109.5
H1—Al1—H2	102.9 (12)	Si2—C5—H5A	109.5
N1—Al1—H3	112.5 (8)	Si2—C5—H5B	109.5
Na1^{i}—Al1—H3	28.2 (8)	H5A—C5—H5B	109.5
H1—Al1—H3	105.5 (11)	Si2—C5—H5C	109.5
H2—Al1—H3	107.7 (12)	H5A—C5—H5C	109.5
N2—Na1—N4	109.15 (5)	H5B—C5—H5C	109.5
N2—Na1—N3	74.35 (5)	Si2—C6—H6A	109.5
N4—Na1—N3	74.05 (5)	Si2—C6—H6B	109.5
N2—Na1—Al1^{i}	98.40 (4)	H6A—C6—H6B	109.5
N4—Na1—Al1^{i}	101.98 (4)	Si2—C6—H6C	109.5
N3—Na1—Al1^{i}	169.57 (4)	H6A—C6—H6C	109.5
N2—Na1—H1	154.8 (6)	H6B—C6—H6C	109.5
N4—Na1—H1	93.8 (6)	N2—C7—H7A	109.5
N3—Na1—H1	103.3 (6)	N2—C7—H7B	109.5

Al1^{i}—Na1—H1	86.5 (6)	H7A—C7—H7B	109.5
N1—Si1—C2	110.28 (7)	N2—C7—H7C	109.5
N1—Si1—C1	112.43 (7)	H7A—C7—H7C	109.5
C2—Si1—C1	107.83 (9)	H7B—C7—H7C	109.5
N1—Si1—C3	113.92 (8)	N2—C8—H8A	109.5
C2—Si1—C3	105.48 (8)	N2—C8—H8B	109.5
C1—Si1—C3	106.49 (10)	H8A—C8—H8B	109.5
N1—Si2—C5	112.92 (7)	N2—C8—H8C	109.5
N1—Si2—C6	111.60 (7)	H8A—C8—H8C	109.5
C5—Si2—C6	106.10 (9)	H8B—C8—H8C	109.5
N1—Si2—C4	112.80 (8)	N2—C9—C10	113.56 (14)
C5—Si2—C4	105.06 (10)	N2—C9—H9A	108.9
C6—Si2—C4	107.88 (9)	C10—C9—H9A	108.9
Si2—N1—Si1	123.12 (7)	N2—C9—H9B	108.9
Si2—N1—Al1	122.84 (7)	C10—C9—H9B	108.9
Si1—N1—Al1	114.04 (6)	H9A—C9—H9B	107.7
C9—N2—C7	111.89 (15)	N3—C10—C9	113.27 (13)
C9—N2—C8	109.57 (15)	N3—C10—H10A	108.9
C7—N2—C8	108.88 (15)	C9—C10—H10A	108.9
C9—N2—Na1	108.11 (10)	N3—C10—H10B	108.9
C7—N2—Na1	102.07 (11)	C9—C10—H10B	108.9
C8—N2—Na1	116.18 (11)	H10A—C10—H10B	107.7
C10—N3—C12	111.69 (14)	N3—C11—H11A	109.5
C10—N3—C11	110.25 (13)	N3—C11—H11B	109.5
C12—N3—C11	110.07 (14)	H11A—C11—H11B	109.5
C10—N3—Na1	106.49 (10)	N3—C11—H11C	109.5
C12—N3—Na1	107.15 (9)	H11A—C11—H11C	109.5
C11—N3—Na1	111.10 (10)	H11B—C11—H11C	109.5
C14—N4—C15	109.51 (13)	N3—C12—C13	112.39 (14)
C14—N4—C13	111.49 (14)	N3—C12—H12A	109.1
C15—N4—C13	110.79 (13)	C13—C12—H12A	109.1
C14—N4—Na1	109.95 (10)	N3—C12—H12B	109.1
C15—N4—Na1	108.44 (10)	C13—C12—H12B	109.1
C13—N4—Na1	106.57 (9)	H12A—C12—H12B	107.9
Si1—C1—H1A	109.5	N4—C13—C12	112.54 (13)
Si1—C1—H1B	109.5	N4—C13—H13A	109.1
H1A—C1—H1B	109.5	C12—C13—H13A	109.1
Si1—C1—H1C	109.5	N4—C13—H13B	109.1
H1A—C1—H1C	109.5	C12—C13—H13B	109.1
H1B—C1—H1C	109.5	H13A—C13—H13B	107.8
Si1—C2—H2A	109.5	N4—C14—H14A	109.5
Si1—C2—H2B	109.5	N4—C14—H14B	109.5
H2A—C2—H2B	109.5	H14A—C14—H14B	109.5
Si1—C2—H2C	109.5	N4—C14—H14C	109.5
H2A—C2—H2C	109.5	H14A—C14—H14C	109.5
H2B—C2—H2C	109.5	H14B—C14—H14C	109.5
Si1—C3—H3A	109.5	N4—C15—H15A	109.5
Si1—C3—H3B	109.5	N4—C15—H15B	109.5

H3A—C3—H3B	109.5	H15A—C15—H15B	109.5
Si1—C3—H3C	109.5	N4—C15—H15C	109.5
H3A—C3—H3C	109.5	H15A—C15—H15C	109.5
H3B—C3—H3C	109.5	H15B—C15—H15C	109.5
Si2—C4—H4A	109.5		

C5—Si2—N1—Si1	−174.14 (9)	C7—N2—C9—C10	70.97 (19)
C6—Si2—N1—Si1	−54.71 (11)	C8—N2—C9—C10	−168.16 (15)
C4—Si2—N1—Si1	66.93 (12)	Na1—N2—C9—C10	−40.65 (17)
C5—Si2—N1—Al1	6.69 (12)	C12—N3—C10—C9	75.00 (19)
C6—Si2—N1—Al1	126.12 (9)	C11—N3—C10—C9	−162.28 (15)
C4—Si2—N1—Al1	−112.24 (10)	Na1—N3—C10—C9	−41.67 (17)
C2—Si1—N1—Si2	−137.16 (9)	N2—C9—C10—N3	59.5 (2)
C1—Si1—N1—Si2	102.47 (10)	C10—N3—C12—C13	−155.70 (13)
C3—Si1—N1—Si2	−18.79 (12)	C11—N3—C12—C13	81.48 (16)
C2—Si1—N1—Al1	42.07 (10)	Na1—N3—C12—C13	−39.45 (15)
C1—Si1—N1—Al1	−78.30 (10)	C14—N4—C13—C12	72.82 (17)
C3—Si1—N1—Al1	160.44 (9)	C15—N4—C13—C12	−164.94 (14)
Na1^{i}—Al1—N1—Si2	82.91 (9)	Na1—N4—C13—C12	−47.17 (16)
Na1^{i}—Al1—N1—Si1	−96.33 (8)	N3—C12—C13—N4	62.45 (18)

Symmetry code: (i) −*x*, −*y*+1, −*z*+2.