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Hexakis{μ_2-4-[2-(diisopropylamino)ethylamino]pent-3-en-2-onato-$\kappa^3N,O:O$}tricalcium(II) hexane solvate

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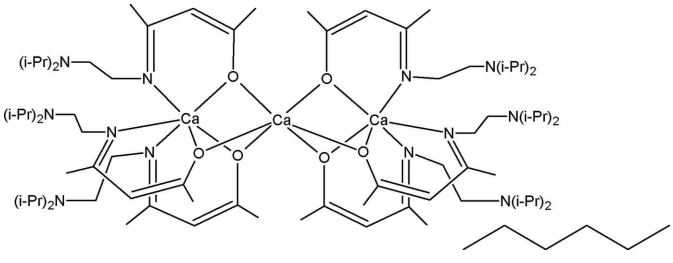
Key indicators: single-crystal X-ray study; T = 110 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.041; wR factor = 0.105; data-to-parameter ratio = 19.5.

The title compound, $[Ca_3(C_{13}H_{25}N_2O)_6]\cdot C_6H_{14}$, is a trinuclear complex with all Ca atoms presenting octahedral coordination. The central Ca atom (as well as the hexane solvent molecule) is located on a crystallographic twofold rotation axis and is coordinated by six bridging O atoms, with Ca—O distances ranging from 2.344 (1) to 2.351 (1) Å. The terminal Ca atoms are coordinated by three bridging O atoms, with Ca—O distances ranging from 2.311 (1) to 2.335 (1) Å, and three N atoms of the β-ketoiminate ligands, with Ca—N distances ranging from 2.432 (1) to 2.452 (1) Å. One of the diisopropylamino groups is disordered over two positions, with site occupancy factors of *ca* 0.54 and 0.46.

Related literature

For related literature, see: Corazza *et al.* (1988); Deacon *et al.* (2004); Matthews *et al.* (2005); Pasko *et al.* (2005); Sanchez *et al.* (2002); Sarazin *et al.* (2006); Westerhausen (1991); Westerhausen *et al.* (2003).



‡ Deceased.

Experimental

Crystal data

$[Ca_3(C_{13}H_{25}N_2O)_6]\cdot C_6H_{14}$
M_r = 1558.51
Monoclinic, $C2/c$
a = 24.1444 (3) Å
b = 15.3089 (2) Å
c = 26.6679 (5) Å
β = 107.132 (1)°
V = 9419.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 110 (2) K
0.3 × 0.3 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
19153 measured reflections
10664 independent reflections
8196 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 1.03
10664 reflections
547 parameters
14 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ca1—O1	2.3348 (10)	Ca1—N5	2.4510 (12)
Ca1—O2	2.3107 (10)	Ca2—O1	2.3442 (10)
Ca1—O3	2.3136 (10)	Ca2—O2	2.3490 (10)
Ca1—N1	2.4323 (12)	Ca2—O3	2.3506 (10)
Ca1—N3	2.4522 (12)		

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2209).

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supplementary materials

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Hexakis{μ_2-4-[2-(diisopropylamino)ethylamino]pent-3-en-2-onato-$\kappa^3N,O:O$}tricalcium(II) hexane solvate

P. Richard, M. Eleter, L. G. Hubert-Pfalzgraf and S. Daniele

Comment

The title compound was obtained by reaction between the Ca silylamide $Ca[N(SiMe_3)_2]_2(THF)_2$ (Westerhausen, 1991) and the ketoimine. Its solid state structure is similar to that of the strontium derivative. However, ^{1}H NMR data in solution ($CDCl_3$ or C_6D_6) show the presence of two different molecular species. In concentrated solution (1.4 *M* in $CDCl_3$), one specie accounts for about 90% of the resonances while the amount of the second specie increases by dilution. These data suggest an equilibrium between a trinuclear form (I) having the solid state structure and a dimeric structure which could be similar to that reported for a tetrakis(β-ketoiminate)di-magnesium (Corazza *et al.*, 1988).

In the title compound (I), the Ca atoms are hexacoordinated. The ketoiminate ligands are linked to the two terminal Ca atoms in a "three blades propeller" mode while the central Ca atom is bonded to the six bridging oxygen atoms of the six ligands and exhibits a distorded trigonal antiprismatic geometry. This coordination scheme is similar to that reported for the Sr analog (Pasko *et al.*, 2005). An other very similar coordination scheme was also reported for a trimagnesium compound linked to a slightly different β-ketoiminate ligand (Matthews *et al.*, 2005). For the terminal Ca atoms, the Ca—N distances [Ca1—N1 2.4323 (12), Ca1—N3 2.4522 (12) and Ca1—N5 2.4510 (12) Å] are in good agreement with those observed for the few β-ketoiminate known (Sanchez *et al.*, 2002; Sarazin *et al.*, 2006 & Westerhausen *et al.*, 2003) while, due to the bridging nature of the O atoms, the Ca—O distances [Ca1—O1 2.335 (1), Ca1—O2 2.311 (1) and Ca1—O3 2.314 (1) Å] are longer than those reported. The Ca—O distances for the central Ca atom [Ca2—O1 2.344 (1), Ca2—O2 2.349 (1) & Ca2—O3 2.351 (1) Å] are in good agreement with those observed for bridging O atoms (see for example Deacon *et al.*, 2004).

Experimental

Synthesis of calcium 4-(2-diisopropylamino-ethylamino)-pent-3-en-2-onate $[CaLiPr_2]_3$. A solution of 885 mg (3.91 mmol) of 4-(2-diisopropylamino-ethylamino)-pent-3-en-2-one in 2 ml of hexane was added to 989 mg (1.96 mmol) of $Ca[N(SiMe_3)_2]_2(THF)_2$ in 10 ml of hexane. After stirring for 24 h at rt, concentration gave a white crystalline solid (648 mg, 68%). FT—IR (cm^{-1}): 1618*m* (νC=O); 1589*m*, 1578 s, 1517*m* (νC=C); 498 s (νCa=N). ^{1}H NMR (250 MHz, $CDCl_3$); 1.01 [d, 72H, Me(iPr), 3 J = 6.65 Hz); 1.92, 1.99 [s, 36H, Me(acac)]; 2.59 (t, 12H, CH_2—$NiPr_2$, 3 J = 7.43 Hz); 3.00 [sep, 12H, CH(iPr)), 3 J = 6.71 Hz]; 3.2 [t, 12H, CH2—N(acac)]; 4.94 [s, 6H, CH(acac)] for B; 0.90 [d, 72H, Me(iPr), 3 J = 6.47 Hz); 1.73, 1.85 [s, 36H, Me(acac)]; 2.33 (t, 12H, CH_2—$NiPr_2$, 3 J = 5.84 Hz); 2.88 [sep, 12H, CH(iPr), 3 J = 6.64 Hz]; 3.22 [t, 12H, CH2—N(acac)]; 4.54 [s, 6H, CH(acac)] for (I).

Refinement

The complex as well as the solvate molecule are located on a twofold axis and only the half of these molecules are present in the asymetric unit. One of the di(isopropyl)-amino groups was found to be disordered over two positions [occupancies: 0.537:0.463]. The lenghts of equivalent bonds in both components of the disordered groups were restrained to be similar during refinement. All H atoms were placed in idealized positions (C—H=0.93 to 0.98 Å) and refined using a riding model [U_{iso}(H)=1.2U_{eq}(CH, CH_2) and 1.5U_{eq}(CH_3)]. Torsion angles of the methyl groups attached to sp^2 C were refined.

Figures

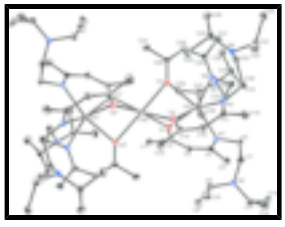


Fig. 1. Molecular view of (I) with the atom labelling scheme. Ellipsoids are drawn at the 30% probability level. Forclarity, the hexane solvate molecule is not shown and only one component of the disordered diethyl amino group is shown·[Symmetry code: (i) −*x*, *y*, −*z* + 1/2]

Hexakis{μ$_2$-4-[2-(diisopropylamino)ethylamino]pent-3-en-2-onato- κ^3*N*,*O*:*O*}tricalcium(II) hexane solvate

Crystal data

$[Ca_3(C_{13}H_{25}N_2O)_6]\cdot C_6H_{14}$	F_{000} = 3440
M_r = 1558.51	D_x = 1.099 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 9659 reflections
a = 24.1444 (3) Å	θ = 1–27.5º
b = 15.3089 (2) Å	μ = 0.23 mm^{-1}
c = 26.6679 (5) Å	*T* = 110 (2) K
β = 107.1320 (10)º	Prism, colourless
V = 9419.7 (3) Å^3	0.3 × 0.3 × 0.25 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	8196 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.035
Monochromator: graphite	θ_{max} = 27.5º
T = 110(2) K	θ_{min} = 2.1º
φ scans (κ = 0) + additional ω scans	*h* = −30→30
Absorption correction: none	*k* = −19→18
19153 measured reflections	*l* = −34→34
10664 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 5.2682P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
10664 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
547 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
14 restraints	Extinction correction: none
Primary atom site location: heavy-atom method	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.03612 (7)	0.01492 (10)	0.19167 (6)	0.0264 (3)	
H1A	0.0676	0.0495	0.2127	0.040*	
H1B	0.0508	−0.0279	0.1726	0.040*	
H1C	0.0171	−0.0140	0.2140	0.040*	
C2	−0.00666 (6)	0.07331 (9)	0.15352 (6)	0.0201 (3)	
C3	−0.02456 (6)	0.05069 (10)	0.10133 (6)	0.0224 (3)	
H3	−0.0098	−0.0018	0.0933	0.027*	
C4	−0.06277 (6)	0.09587 (10)	0.05709 (6)	0.0212 (3)	
C5	−0.07075 (7)	0.05060 (11)	0.00496 (6)	0.0306 (4)	
H5A	−0.1112	0.0392	−0.0112	0.046*	
H5B	−0.0498	−0.0036	0.0106	0.046*	
H5C	−0.0564	0.0874	−0.0175	0.046*	
C6	−0.12108 (6)	0.21628 (11)	0.01393 (6)	0.0261 (3)	
H6A	−0.1571	0.2383	0.0183	0.031*	
H6B	−0.1306	0.1766	−0.0157	0.031*	
C7	−0.08580 (7)	0.29214 (11)	0.00281 (7)	0.0302 (4)	
H7A	−0.1095	0.3259	−0.0265	0.036*	
H7B	−0.0744	0.3301	0.0332	0.036*	

N2	−0.0335 (4)	0.2609 (9)	−0.0097 (4)	0.0202 (16)	0.54
C8	0.01620 (13)	0.2779 (2)	0.03670 (14)	0.0250 (6)	0.54
H8	0.0034	0.2633	0.0673	0.030*	0.54
C9	0.0646 (3)	0.2142 (4)	0.0370 (3)	0.0333 (11)	0.54
H9A	0.0492	0.1560	0.0315	0.050*	0.54
H9B	0.0941	0.2172	0.0702	0.050*	0.54
H9C	0.0810	0.2292	0.0094	0.050*	0.54
C10	0.0389 (2)	0.3719 (3)	0.04501 (17)	0.0337 (9)	0.54
H10A	0.0077	0.4107	0.0452	0.051*	0.54
H10B	0.0546	0.3879	0.0171	0.051*	0.54
H10C	0.0687	0.3760	0.0780	0.051*	0.54
C11	−0.02796 (17)	0.2864 (2)	−0.06088 (13)	0.0274 (7)	0.54
H11	0.0129	0.2784	−0.0587	0.033*	0.54
C12	−0.0434 (3)	0.3802 (4)	−0.0801 (2)	0.0430 (13)	0.54
H12A	−0.0212	0.4205	−0.0544	0.064*	0.54
H12B	−0.0839	0.3901	−0.0854	0.064*	0.54
H12C	−0.0347	0.3887	−0.1127	0.064*	0.54
C13	−0.0619 (2)	0.2222 (3)	−0.10225 (16)	0.0387 (9)	0.54
H13A	−0.0518	0.1636	−0.0903	0.058*	0.54
H13B	−0.0526	0.2316	−0.1344	0.058*	0.54
H13C	−0.1027	0.2310	−0.1080	0.058*	0.54
N2X	−0.0405 (5)	0.2711 (11)	−0.0197 (4)	0.0208 (18)	0.46
C8X	0.02092 (15)	0.2937 (2)	0.00340 (18)	0.0322 (8)	0.46
H8X	0.0402	0.2847	−0.0238	0.039*	0.46
C9X	0.0489 (3)	0.2328 (5)	0.0481 (3)	0.051 (2)	0.46
H9X1	0.0415	0.1735	0.0365	0.077*	0.46
H9X2	0.0331	0.2433	0.0766	0.077*	0.46
H9X3	0.0900	0.2430	0.0596	0.077*	0.46
C10X	0.0314 (3)	0.3897 (4)	0.0215 (2)	0.0450 (14)	0.46
H10D	0.0131	0.4278	−0.0072	0.068*	0.46
H10E	0.0722	0.4013	0.0332	0.068*	0.46
H10F	0.0152	0.3998	0.0499	0.068*	0.46
C11X	−0.06212 (19)	0.2748 (3)	−0.07734 (16)	0.0252 (7)	0.46
H11X	−0.1033	0.2593	−0.0865	0.030*	0.46
C12X	−0.0600 (3)	0.3671 (4)	−0.0997 (2)	0.0349 (13)	0.46
H12D	−0.0773	0.4079	−0.0814	0.052*	0.46
H12E	−0.0810	0.3675	−0.1364	0.052*	0.46
H12F	−0.0204	0.3832	−0.0952	0.052*	0.46
C13X	−0.0344 (2)	0.2077 (4)	−0.1049 (2)	0.0426 (13)	0.46
H13D	−0.0355	0.1511	−0.0897	0.064*	0.46
H13E	0.0051	0.2238	−0.1007	0.064*	0.46
H13F	−0.0554	0.2060	−0.1415	0.064*	0.46
C14	0.03140 (7)	0.43725 (11)	0.18893 (8)	0.0356 (4)	
H14A	0.0389	0.4394	0.2263	0.053*	
H14B	0.0341	0.4950	0.1759	0.053*	
H14C	0.0594	0.4000	0.1805	0.053*	
C15	−0.02842 (6)	0.40173 (9)	0.16407 (6)	0.0221 (3)	
C16	−0.06666 (6)	0.44917 (10)	0.12539 (6)	0.0240 (3)	
H16	−0.0512	0.4992	0.1149	0.029*	

C17	−0.12707 (6)	0.43358 (10)	0.09841 (6)	0.0223 (3)
C18	−0.15555 (7)	0.50790 (11)	0.06224 (7)	0.0341 (4)
H18A	−0.1637	0.4894	0.0264	0.051*
H18B	−0.1299	0.5573	0.0684	0.051*
H18C	−0.1911	0.5239	0.0691	0.051*
C19	−0.21716 (6)	0.35616 (10)	0.08018 (6)	0.0228 (3)
H19A	−0.2268	0.2999	0.0631	0.027*
H19B	−0.2301	0.4013	0.0538	0.027*
C20	−0.24847 (6)	0.36647 (10)	0.12203 (6)	0.0224 (3)
H20A	−0.2888	0.3504	0.1071	0.027*
H20B	−0.2315	0.3266	0.1507	0.027*
C21	−0.29068 (6)	0.51364 (10)	0.11119 (6)	0.0244 (3)
H21	−0.2969	0.4976	0.0744	0.029*
C22	−0.27038 (8)	0.60871 (11)	0.11683 (7)	0.0349 (4)
H22A	−0.2335	0.6129	0.1101	0.052*
H22B	−0.2667	0.6286	0.1518	0.052*
H22C	−0.2982	0.6443	0.0921	0.052*
C23	−0.34947 (7)	0.50708 (11)	0.12156 (7)	0.0296 (4)
H23A	−0.3625	0.4475	0.1175	0.044*
H23B	−0.3770	0.5432	0.0970	0.044*
H23C	−0.3458	0.5263	0.1566	0.044*
C24	−0.23473 (7)	0.46046 (11)	0.20018 (6)	0.0267 (3)
H24	−0.2443	0.5201	0.2081	0.032*
C25	−0.17030 (7)	0.44731 (12)	0.22745 (7)	0.0344 (4)
H25A	−0.1482	0.4865	0.2127	0.052*
H25B	−0.1598	0.3881	0.2226	0.052*
H25C	−0.1623	0.4590	0.2643	0.052*
C26	−0.27080 (8)	0.39859 (13)	0.22335 (7)	0.0381 (4)
H26A	−0.3112	0.4076	0.2059	0.057*
H26B	−0.2630	0.4103	0.2601	0.057*
H26C	−0.2607	0.3392	0.2185	0.057*
C27	−0.12638 (7)	0.22155 (11)	0.30709 (6)	0.0272 (3)
H27A	−0.0917	0.1914	0.3261	0.041*
H27B	−0.1575	0.2054	0.3209	0.041*
H27C	−0.1201	0.2834	0.3108	0.041*
C28	−0.14193 (6)	0.19710 (9)	0.24988 (6)	0.0200 (3)
C29	−0.19634 (6)	0.16570 (10)	0.22532 (6)	0.0229 (3)
H29	−0.2209	0.1629	0.2464	0.027*
C30	−0.22169 (6)	0.13627 (9)	0.17216 (6)	0.0215 (3)
C31	−0.28271 (7)	0.10094 (12)	0.16156 (7)	0.0319 (4)
H31A	−0.3098	0.1416	0.1401	0.048*
H31B	−0.2913	0.0929	0.1942	0.048*
H31C	−0.2857	0.0459	0.1437	0.048*
C32	−0.22305 (6)	0.10384 (10)	0.08364 (6)	0.0232 (3)
H32A	−0.2188	0.1442	0.0570	0.028*
H32B	−0.2641	0.0960	0.0789	0.028*
C33	−0.19624 (7)	0.01598 (10)	0.07648 (6)	0.0252 (3)
H33A	−0.2093	0.0001	0.0396	0.030*
H33B	−0.1544	0.0223	0.0865	0.030*

C34	−0.26656 (7)	−0.09638 (10)	0.08128 (6)	0.0269 (3)
H34	−0.2935	−0.0493	0.0654	0.032*
C35	−0.29155 (8)	−0.14053 (12)	0.12123 (7)	0.0351 (4)
H35A	−0.2909	−0.1002	0.1490	0.053*
H35B	−0.2687	−0.1909	0.1356	0.053*
H35C	−0.3308	−0.1582	0.1043	0.053*
C36	−0.26749 (8)	−0.16028 (11)	0.03701 (7)	0.0367 (4)
H36A	−0.2518	−0.1324	0.0120	0.055*
H36B	−0.3067	−0.1779	0.0200	0.055*
H36C	−0.2446	−0.2107	0.0512	0.055*
C37	−0.16169 (7)	−0.11353 (11)	0.13201 (7)	0.0299 (4)
H37	−0.1782	−0.1655	0.1436	0.036*
C38	−0.12600 (8)	−0.14521 (12)	0.09636 (8)	0.0420 (5)
H38A	−0.1512	−0.1728	0.0658	0.063*
H38B	−0.0974	−0.1864	0.1151	0.063*
H38C	−0.1071	−0.0963	0.0859	0.063*
C39	−0.12278 (7)	−0.06952 (12)	0.18109 (7)	0.0376 (4)
H39A	−0.1458	−0.0505	0.2029	0.056*
H39B	−0.1040	−0.0201	0.1711	0.056*
H39C	−0.0941	−0.1103	0.2002	0.056*
C40	0.47431 (10)	0.18978 (15)	0.26053 (9)	0.0564 (6)
H40A	0.4784	0.1415	0.2849	0.068*
H40B	0.4748	0.2434	0.2801	0.068*
C41	0.41649 (11)	0.18220 (16)	0.21952 (10)	0.0638 (6)
H41A	0.4138	0.1253	0.2030	0.077*
H41B	0.4142	0.2260	0.1927	0.077*
C42	0.36531 (11)	0.19359 (19)	0.24107 (11)	0.0717 (7)
H42A	0.3299	0.1881	0.2130	0.108*
H42B	0.3670	0.2503	0.2568	0.108*
H42C	0.3667	0.1496	0.2670	0.108*
Ca1	−0.107689 (11)	0.224696 (18)	0.140247 (11)	0.01547 (7)
Ca2	0.0000	0.22675 (3)	0.2500	0.01720 (9)
O1	−0.02404 (4)	0.14111 (7)	0.17375 (4)	0.0220 (2)
O2	−0.03891 (4)	0.32665 (6)	0.18186 (4)	0.0243 (2)
O3	−0.10101 (4)	0.20875 (7)	0.22811 (4)	0.0230 (2)
N1	−0.08916 (5)	0.16878 (8)	0.06134 (5)	0.0203 (3)
N3	−0.15417 (5)	0.36245 (8)	0.10367 (5)	0.0199 (3)
N4	−0.24532 (5)	0.45532 (8)	0.14261 (5)	0.0211 (3)
N5	−0.19571 (5)	0.14134 (8)	0.13566 (5)	0.0205 (3)
N6	−0.21080 (5)	−0.05430 (8)	0.10712 (5)	0.0231 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0279 (8)	0.0246 (8)	0.0268 (8)	0.0088 (7)	0.0079 (7)	0.0032 (7)
C2	0.0179 (7)	0.0195 (7)	0.0243 (8)	0.0009 (6)	0.0084 (6)	0.0006 (6)
C3	0.0250 (8)	0.0184 (7)	0.0254 (8)	0.0009 (6)	0.0097 (6)	−0.0040 (6)
C4	0.0199 (7)	0.0239 (7)	0.0210 (8)	−0.0044 (6)	0.0078 (6)	−0.0037 (6)

C5	0.0334 (9)	0.0341 (9)	0.0243 (8)	0.0004 (7)	0.0088 (7)	−0.0089 (7)
C6	0.0210 (7)	0.0388 (9)	0.0191 (8)	0.0052 (7)	0.0067 (6)	0.0022 (7)
C7	0.0357 (9)	0.0300 (9)	0.0291 (9)	0.0100 (7)	0.0162 (7)	0.0094 (7)
N2	0.018 (3)	0.026 (4)	0.017 (3)	0.006 (2)	0.005 (2)	0.008 (2)
C8	0.0229 (16)	0.0235 (15)	0.0258 (17)	0.0013 (13)	0.0029 (13)	0.0006 (14)
C9	0.024 (3)	0.033 (2)	0.039 (3)	0.0037 (18)	0.0027 (17)	0.0006 (18)
C10	0.029 (2)	0.030 (2)	0.040 (3)	−0.0002 (16)	0.006 (2)	−0.0024 (19)
C11	0.0266 (17)	0.0329 (18)	0.0244 (17)	−0.0036 (15)	0.0101 (15)	0.0048 (14)
C12	0.053 (4)	0.041 (3)	0.035 (3)	−0.010 (2)	0.012 (2)	0.011 (2)
C13	0.057 (3)	0.040 (2)	0.0202 (18)	−0.007 (2)	0.012 (2)	−0.0016 (17)
N2X	0.016 (2)	0.016 (3)	0.025 (4)	0.005 (2)	−0.002 (2)	0.008 (3)
C8X	0.0245 (18)	0.032 (2)	0.036 (2)	−0.0019 (15)	0.0021 (16)	0.0067 (17)
C9X	0.033 (4)	0.040 (4)	0.064 (5)	−0.016 (3)	−0.013 (3)	0.016 (3)
C10X	0.045 (3)	0.033 (3)	0.046 (4)	−0.017 (2)	−0.003 (3)	0.003 (3)
C11X	0.024 (2)	0.032 (2)	0.0212 (19)	−0.0042 (17)	0.0091 (16)	0.0009 (17)
C12X	0.034 (3)	0.037 (3)	0.037 (4)	−0.002 (2)	0.014 (3)	0.010 (3)
C13X	0.052 (3)	0.045 (3)	0.039 (3)	0.006 (3)	0.026 (3)	−0.001 (2)
C14	0.0286 (9)	0.0257 (8)	0.0438 (11)	−0.0079 (7)	−0.0030 (8)	0.0064 (8)
C15	0.0229 (7)	0.0188 (7)	0.0240 (8)	−0.0014 (6)	0.0058 (6)	−0.0005 (6)
C16	0.0250 (8)	0.0203 (7)	0.0275 (8)	−0.0006 (6)	0.0088 (6)	0.0047 (6)
C17	0.0253 (8)	0.0233 (7)	0.0193 (7)	0.0055 (6)	0.0080 (6)	0.0033 (6)
C18	0.0297 (9)	0.0322 (9)	0.0388 (10)	0.0048 (7)	0.0074 (8)	0.0149 (8)
C19	0.0212 (7)	0.0228 (7)	0.0217 (8)	0.0046 (6)	0.0022 (6)	−0.0019 (6)
C20	0.0186 (7)	0.0218 (7)	0.0253 (8)	0.0025 (6)	0.0042 (6)	0.0005 (6)
C21	0.0254 (8)	0.0248 (8)	0.0216 (8)	0.0084 (6)	0.0051 (6)	0.0003 (6)
C22	0.0398 (10)	0.0262 (8)	0.0407 (10)	0.0090 (7)	0.0153 (8)	0.0039 (8)
C23	0.0226 (8)	0.0339 (9)	0.0298 (9)	0.0076 (7)	0.0035 (7)	−0.0038 (7)
C24	0.0269 (8)	0.0306 (8)	0.0205 (8)	0.0075 (7)	0.0038 (6)	−0.0038 (7)
C25	0.0292 (9)	0.0407 (10)	0.0278 (9)	0.0077 (8)	−0.0005 (7)	−0.0041 (8)
C26	0.0384 (10)	0.0504 (11)	0.0282 (9)	0.0082 (9)	0.0142 (8)	0.0058 (8)
C27	0.0299 (8)	0.0321 (8)	0.0224 (8)	−0.0029 (7)	0.0118 (7)	−0.0007 (7)
C28	0.0221 (7)	0.0192 (7)	0.0204 (7)	0.0032 (6)	0.0087 (6)	0.0029 (6)
C29	0.0233 (7)	0.0233 (7)	0.0255 (8)	0.0000 (6)	0.0125 (6)	−0.0003 (6)
C30	0.0187 (7)	0.0179 (7)	0.0279 (8)	0.0008 (6)	0.0069 (6)	0.0016 (6)
C31	0.0242 (8)	0.0374 (9)	0.0366 (10)	−0.0066 (7)	0.0128 (7)	−0.0051 (8)
C32	0.0230 (7)	0.0239 (7)	0.0210 (8)	−0.0053 (6)	0.0036 (6)	0.0002 (6)
C33	0.0299 (8)	0.0230 (8)	0.0231 (8)	−0.0057 (6)	0.0085 (6)	−0.0032 (6)
C34	0.0280 (8)	0.0236 (8)	0.0249 (8)	−0.0069 (6)	0.0013 (7)	−0.0014 (6)
C35	0.0347 (9)	0.0349 (9)	0.0336 (10)	−0.0143 (8)	0.0069 (8)	0.0010 (8)
C36	0.0478 (11)	0.0270 (8)	0.0298 (9)	−0.0089 (8)	0.0031 (8)	−0.0035 (7)
C37	0.0304 (8)	0.0251 (8)	0.0313 (9)	−0.0035 (7)	0.0046 (7)	0.0025 (7)
C38	0.0419 (10)	0.0336 (10)	0.0508 (12)	0.0071 (8)	0.0140 (9)	0.0006 (9)
C39	0.0293 (9)	0.0445 (10)	0.0333 (10)	−0.0067 (8)	0.0006 (7)	0.0039 (8)
C40	0.0796 (17)	0.0473 (12)	0.0545 (14)	−0.0031 (12)	0.0389 (13)	0.0011 (11)
C41	0.0844 (18)	0.0534 (14)	0.0609 (16)	−0.0170 (13)	0.0331 (14)	−0.0008 (12)
C42	0.0645 (16)	0.0838 (19)	0.0715 (18)	−0.0185 (14)	0.0270 (14)	−0.0046 (15)
Ca1	0.01479 (13)	0.01616 (14)	0.01496 (14)	0.00052 (11)	0.00360 (10)	0.00004 (11)
Ca2	0.01669 (19)	0.01833 (19)	0.0150 (2)	0.000	0.00218 (15)	0.000
O1	0.0220 (5)	0.0237 (5)	0.0185 (5)	0.0056 (4)	0.0030 (4)	−0.0033 (4)

O2	0.0258 (5)	0.0174 (5)	0.0238 (6)	−0.0037 (4)	−0.0020 (4)	0.0033 (4)
O3	0.0185 (5)	0.0346 (6)	0.0168 (5)	−0.0012 (4)	0.0066 (4)	0.0012 (4)
N1	0.0177 (6)	0.0253 (6)	0.0180 (6)	0.0001 (5)	0.0053 (5)	0.0008 (5)
N3	0.0184 (6)	0.0217 (6)	0.0195 (6)	0.0032 (5)	0.0055 (5)	0.0001 (5)
N4	0.0202 (6)	0.0211 (6)	0.0207 (7)	0.0047 (5)	0.0041 (5)	−0.0005 (5)
N5	0.0211 (6)	0.0186 (6)	0.0215 (6)	−0.0020 (5)	0.0058 (5)	−0.0003 (5)
N6	0.0239 (6)	0.0206 (6)	0.0228 (7)	−0.0057 (5)	0.0039 (5)	0.0005 (5)

Geometric parameters (Å, °)

C1—C2	1.510 (2)	C21—N4	1.4691 (19)
C1—H1A	0.9600	C21—C23	1.527 (2)
C1—H1B	0.9600	C21—C22	1.529 (2)
C1—H1C	0.9600	C21—H21	0.9800
C2—O1	1.2953 (17)	C22—H22A	0.9600
C2—C3	1.375 (2)	C22—H22B	0.9600
C3—C4	1.442 (2)	C22—H22C	0.9600
C3—H3	0.9300	C23—H23A	0.9600
C4—N1	1.3066 (19)	C23—H23B	0.9600
C4—C5	1.514 (2)	C23—H23C	0.9600
C5—H5A	0.9600	C24—N4	1.4824 (19)
C5—H5B	0.9600	C24—C25	1.524 (2)
C5—H5C	0.9600	C24—C26	1.534 (2)
C6—N1	1.4652 (19)	C24—H24	0.9800
C6—C7	1.520 (2)	C25—H25A	0.9600
C6—H6A	0.9700	C25—H25B	0.9600
C6—H6B	0.9700	C25—H25C	0.9600
C7—N2X	1.431 (12)	C26—H26A	0.9600
C7—N2	1.478 (9)	C26—H26B	0.9600
C7—H7A	0.9700	C26—H26C	0.9600
C7—H7B	0.9700	C27—C28	1.507 (2)
N2—C11	1.464 (9)	C27—H27A	0.9600
N2—C8	1.470 (10)	C27—H27B	0.9600
C8—C9	1.520 (5)	C27—H27C	0.9600
C8—C10	1.533 (5)	C28—O3	1.2974 (17)
C8—H8	0.9800	C28—C29	1.371 (2)
C9—H9A	0.9600	C29—C30	1.441 (2)
C9—H9B	0.9600	C29—H29	0.9300
C9—H9C	0.9600	C30—N5	1.3063 (19)
C10—H10A	0.9600	C30—C31	1.516 (2)
C10—H10B	0.9600	C31—H31A	0.9600
C10—H10C	0.9600	C31—H31B	0.9600
C11—C13	1.524 (5)	C31—H31C	0.9600
C11—C12	1.533 (7)	C32—N5	1.4663 (19)
C11—H11	0.9800	C32—C33	1.529 (2)
C12—H12A	0.9600	C32—H32A	0.9700
C12—H12B	0.9600	C32—H32B	0.9700
C12—H12C	0.9600	C33—N6	1.4552 (19)
C13—H13A	0.9600	C33—H33A	0.9700

C13—H13B	0.9600	C33—H33B	0.9700
C13—H13C	0.9600	C34—N6	1.4683 (19)
N2X—C8X	1.469 (12)	C34—C35	1.528 (2)
N2X—C11X	1.472 (11)	C34—C36	1.528 (2)
C8X—C9X	1.507 (8)	C34—H34	0.9800
C8X—C10X	1.545 (7)	C35—H35A	0.9600
C8X—H8X	0.9800	C35—H35B	0.9600
C9X—H9X1	0.9600	C35—H35C	0.9600
C9X—H9X2	0.9600	C36—H36A	0.9600
C9X—H9X3	0.9600	C36—H36B	0.9600
C10X—H10D	0.9600	C36—H36C	0.9600
C10X—H10E	0.9600	C37—N6	1.485 (2)
C10X—H10F	0.9600	C37—C39	1.525 (2)
C11X—C13X	1.527 (6)	C37—C38	1.538 (2)
C11X—C12X	1.539 (7)	C37—H37	0.9800
C11X—H11X	0.9800	C38—H38A	0.9600
C12X—H12D	0.9600	C38—H38B	0.9600
C12X—H12E	0.9600	C38—H38C	0.9600
C12X—H12F	0.9600	C39—H39A	0.9600
C13X—H13D	0.9600	C39—H39B	0.9600
C13X—H13E	0.9600	C39—H39C	0.9600
C13X—H13F	0.9600	C40—C41	1.503 (3)
C14—C15	1.502 (2)	C40—C40^{i}	1.505 (4)
C14—H14A	0.9600	C40—H40A	0.9700
C14—H14B	0.9600	C40—H40B	0.9700
C14—H14C	0.9600	C41—C42	1.519 (3)
C15—O2	1.2965 (17)	C41—H41A	0.9700
C15—C16	1.372 (2)	C41—H41B	0.9700
C16—C17	1.444 (2)	C42—H42A	0.9600
C16—H16	0.9300	C42—H42B	0.9600
C17—N3	1.2990 (19)	C42—H42C	0.9600
C17—C18	1.519 (2)	Ca1—O1	2.3348 (10)
C18—H18A	0.9600	Ca1—O2	2.3107 (10)
C18—H18B	0.9600	Ca1—O3	2.3136 (10)
C18—H18C	0.9600	Ca1—N1	2.4323 (12)
C19—N3	1.4675 (18)	Ca1—N3	2.4522 (12)
C19—C20	1.529 (2)	Ca1—N5	2.4510 (12)
C19—H19A	0.9700	Ca1—Ca2	3.2931 (3)
C19—H19B	0.9700	Ca2—O1	2.3442 (10)
C20—N4	1.4602 (19)	Ca2—O2	2.3490 (10)
C20—H20A	0.9700	Ca2—O3	2.3506 (10)
C20—H20B	0.9700		
C2—C1—H1A	109.5	C24—C25—H25A	109.5
C2—C1—H1B	109.5	C24—C25—H25B	109.5
H1A—C1—H1B	109.5	H25A—C25—H25B	109.5
C2—C1—H1C	109.5	C24—C25—H25C	109.5
H1A—C1—H1C	109.5	H25A—C25—H25C	109.5
H1B—C1—H1C	109.5	H25B—C25—H25C	109.5

O1—C2—C3	125.43 (13)	C24—C26—H26A	109.5
O1—C2—C1	115.79 (13)	C24—C26—H26B	109.5
C3—C2—C1	118.78 (13)	H26A—C26—H26B	109.5
C2—C3—C4	129.94 (14)	C24—C26—H26C	109.5
C2—C3—H3	115.0	H26A—C26—H26C	109.5
C4—C3—H3	115.0	H26B—C26—H26C	109.5
N1—C4—C3	123.34 (13)	C28—C27—H27A	109.5
N1—C4—C5	122.49 (14)	C28—C27—H27B	109.5
C3—C4—C5	114.16 (13)	H27A—C27—H27B	109.5
C4—C5—H5A	109.5	C28—C27—H27C	109.5
C4—C5—H5B	109.5	H27A—C27—H27C	109.5
H5A—C5—H5B	109.5	H27B—C27—H27C	109.5
C4—C5—H5C	109.5	O3—C28—C29	125.69 (14)
H5A—C5—H5C	109.5	O3—C28—C27	115.04 (13)
H5B—C5—H5C	109.5	C29—C28—C27	119.26 (13)
N1—C6—C7	111.40 (13)	C28—C29—C30	130.49 (13)
N1—C6—H6A	109.3	C28—C29—H29	114.8
C7—C6—H6A	109.3	C30—C29—H29	114.8
N1—C6—H6B	109.3	N5—C30—C29	123.81 (13)
C7—C6—H6B	109.3	N5—C30—C31	122.41 (14)
H6A—C6—H6B	108.0	C29—C30—C31	113.75 (13)
N2X—C7—N2	12.2 (8)	C30—C31—H31A	109.5
N2X—C7—C6	116.8 (7)	C30—C31—H31B	109.5
N2—C7—C6	111.2 (6)	H31A—C31—H31B	109.5
N2X—C7—H7A	97.2	C30—C31—H31C	109.5
N2—C7—H7A	109.4	H31A—C31—H31C	109.5
C6—C7—H7A	109.4	H31B—C31—H31C	109.5
N2X—C7—H7B	115.0	N5—C32—C33	111.92 (12)
N2—C7—H7B	109.4	N5—C32—H32A	109.2
C6—C7—H7B	109.4	C33—C32—H32A	109.2
H7A—C7—H7B	108.0	N5—C32—H32B	109.2
C11—N2—C8	117.5 (8)	C33—C32—H32B	109.2
C11—N2—C7	116.2 (6)	H32A—C32—H32B	107.9
C8—N2—C7	107.0 (7)	N6—C33—C32	113.53 (12)
N2—C8—C9	109.0 (6)	N6—C33—H33A	108.9
N2—C8—C10	117.2 (6)	C32—C33—H33A	108.9
C9—C8—C10	111.1 (4)	N6—C33—H33B	108.9
N2—C8—H8	106.3	C32—C33—H33B	108.9
C9—C8—H8	106.3	H33A—C33—H33B	107.7
C10—C8—H8	106.3	N6—C34—C35	111.27 (13)
C8—C9—H9A	109.5	N6—C34—C36	116.49 (14)
C8—C9—H9B	109.5	C35—C34—C36	109.67 (13)
H9A—C9—H9B	109.5	N6—C34—H34	106.2
C8—C9—H9C	109.5	C35—C34—H34	106.2
H9A—C9—H9C	109.5	C36—C34—H34	106.2
H9B—C9—H9C	109.5	C34—C35—H35A	109.5
C8—C10—H10A	109.5	C34—C35—H35B	109.5
C8—C10—H10B	109.5	H35A—C35—H35B	109.5
H10A—C10—H10B	109.5	C34—C35—H35C	109.5

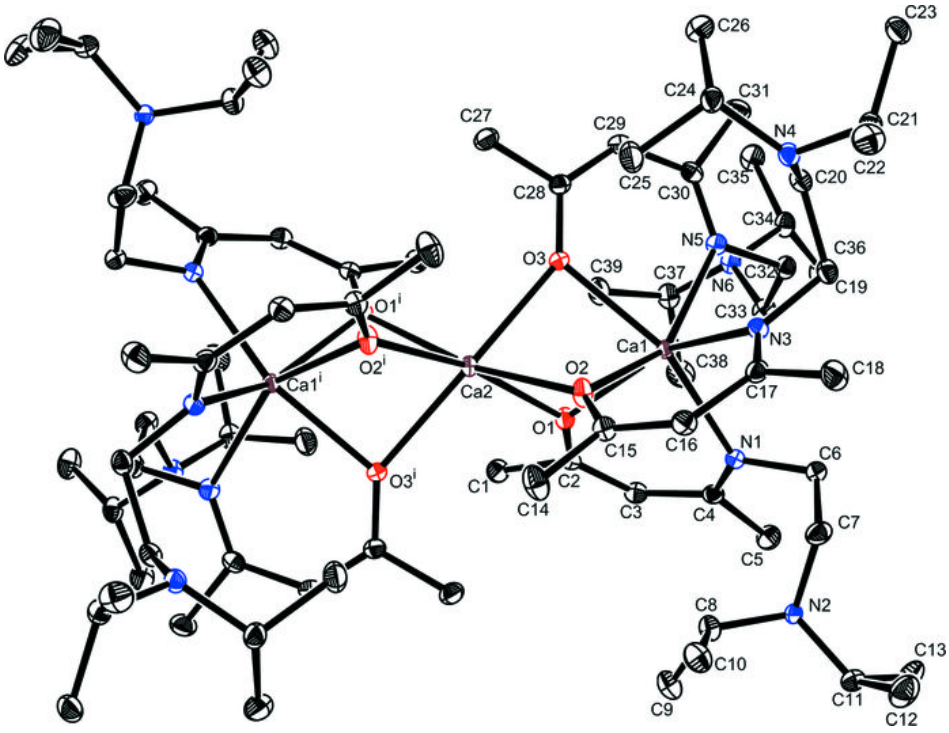
C8—C10—H10C	109.5	H35A—C35—H35C	109.5
H10A—C10—H10C	109.5	H35B—C35—H35C	109.5
H10B—C10—H10C	109.5	C34—C36—H36A	109.5
N2—C11—C13	109.0 (6)	C34—C36—H36B	109.5
N2—C11—C12	118.9 (6)	H36A—C36—H36B	109.5
C13—C11—C12	109.6 (3)	C34—C36—H36C	109.5
N2—C11—H11	106.2	H36A—C36—H36C	109.5
C13—C11—H11	106.2	H36B—C36—H36C	109.5
C12—C11—H11	106.2	N6—C37—C39	108.83 (13)
C11—C12—H12A	109.5	N6—C37—C38	115.81 (14)
C11—C12—H12B	109.5	C39—C37—C38	110.75 (14)
H12A—C12—H12B	109.5	N6—C37—H37	107.0
C11—C12—H12C	109.5	C39—C37—H37	107.0
H12A—C12—H12C	109.5	C38—C37—H37	107.0
H12B—C12—H12C	109.5	C37—C38—H38A	109.5
C11—C13—H13A	109.5	C37—C38—H38B	109.5
C11—C13—H13B	109.5	H38A—C38—H38B	109.5
H13A—C13—H13B	109.5	C37—C38—H38C	109.5
C11—C13—H13C	109.5	H38A—C38—H38C	109.5
H13A—C13—H13C	109.5	H38B—C38—H38C	109.5
H13B—C13—H13C	109.5	C37—C39—H39A	109.5
C7—N2X—C8X	124.9 (9)	C37—C39—H39B	109.5
C7—N2X—C11X	110.4 (7)	H39A—C39—H39B	109.5
C8X—N2X—C11X	115.6 (8)	C37—C39—H39C	109.5
N2X—C8X—C9X	110.4 (6)	H39A—C39—H39C	109.5
N2X—C8X—C10X	114.0 (7)	H39B—C39—H39C	109.5
C9X—C8X—C10X	110.3 (5)	C41—C40—C40^{i}	114.8 (3)
N2X—C8X—H8X	107.3	C41—C40—H40A	108.6
C9X—C8X—H8X	107.3	C40^{i}—C40—H40A	108.6
C10X—C8X—H8X	107.3	C41—C40—H40B	108.6
C8X—C9X—H9X1	109.5	C40^{i}—C40—H40B	108.6
C8X—C9X—H9X2	109.5	H40A—C40—H40B	107.5
H9X1—C9X—H9X2	109.5	C40—C41—C42	113.6 (2)
C8X—C9X—H9X3	109.5	C40—C41—H41A	108.8
H9X1—C9X—H9X3	109.5	C42—C41—H41A	108.8
H9X2—C9X—H9X3	109.5	C40—C41—H41B	108.8
C8X—C10X—H10D	109.5	C42—C41—H41B	108.8
C8X—C10X—H10E	109.5	H41A—C41—H41B	107.7
H10D—C10X—H10E	109.5	C41—C42—H42A	109.5
C8X—C10X—H10F	109.5	C41—C42—H42B	109.5
H10D—C10X—H10F	109.5	H42A—C42—H42B	109.5
H10E—C10X—H10F	109.5	C41—C42—H42C	109.5
N2X—C11X—C13X	113.9 (6)	H42A—C42—H42C	109.5
N2X—C11X—C12X	113.4 (7)	H42B—C42—H42C	109.5
C13X—C11X—C12X	111.2 (4)	O2—Ca1—O3	76.06 (4)
N2X—C11X—H11X	105.8	O2—Ca1—O1	76.59 (4)
C13X—C11X—H11X	105.8	O3—Ca1—O1	76.13 (4)
C12X—C11X—H11X	105.8	O2—Ca1—N1	111.34 (4)

C11X—C12X—H12D	109.5	O3—Ca1—N1	149.69 (4)
C11X—C12X—H12E	109.5	O1—Ca1—N1	77.25 (4)
H12D—C12X—H12E	109.5	O2—Ca1—N5	149.95 (4)
C11X—C12X—H12F	109.5	O3—Ca1—N5	78.25 (4)
H12D—C12X—H12F	109.5	O1—Ca1—N5	111.92 (4)
H12E—C12X—H12F	109.5	N1—Ca1—N5	98.70 (4)
C11X—C13X—H13D	109.5	O2—Ca1—N3	77.88 (4)
C11X—C13X—H13E	109.5	O3—Ca1—N3	112.38 (4)
H13D—C13X—H13E	109.5	O1—Ca1—N3	150.05 (4)
C11X—C13X—H13F	109.5	N1—Ca1—N3	97.92 (4)
H13D—C13X—H13F	109.5	N5—Ca1—N3	98.02 (4)
H13E—C13X—H13F	109.5	O2—Ca1—Ca2	45.51 (3)
C15—C14—H14A	109.5	O3—Ca1—Ca2	45.54 (2)
C15—C14—H14B	109.5	O1—Ca1—Ca2	45.38 (3)
H14A—C14—H14B	109.5	N1—Ca1—Ca2	118.30 (3)
C15—C14—H14C	109.5	N5—Ca1—Ca2	119.69 (3)
H14A—C14—H14C	109.5	N3—Ca1—Ca2	119.62 (3)
H14B—C14—H14C	109.5	O1—Ca2—O1^{ii}	111.99 (5)
O2—C15—C16	125.71 (14)	O1—Ca2—O2^{ii}	167.31 (4)
O2—C15—C14	115.18 (13)	O1^{ii}—Ca2—O2^{ii}	75.68 (4)
C16—C15—C14	119.11 (14)	O1—Ca2—O2	75.68 (4)
C15—C16—C17	130.12 (14)	O1^{ii}—Ca2—O2	167.31 (4)
C15—C16—H16	114.9	O2^{ii}—Ca2—O2	98.76 (5)
C17—C16—H16	114.9	O1—Ca2—O3	75.24 (3)
N3—C17—C16	123.50 (13)	O1^{ii}—Ca2—O3	97.10 (4)
N3—C17—C18	123.25 (14)	O2^{ii}—Ca2—O3	114.69 (4)
C16—C17—C18	113.24 (13)	O2—Ca2—O3	74.63 (4)
C17—C18—H18A	109.5	O1—Ca2—O3^{ii}	97.10 (3)
C17—C18—H18B	109.5	O1^{ii}—Ca2—O3^{ii}	75.24 (3)
H18A—C18—H18B	109.5	O2^{ii}—Ca2—O3^{ii}	74.63 (4)
C17—C18—H18C	109.5	O2—Ca2—O3^{ii}	114.69 (4)
H18A—C18—H18C	109.5	O3—Ca2—O3^{ii}	166.53 (6)
H18B—C18—H18C	109.5	O1—Ca2—Ca1	45.15 (2)
N3—C19—C20	110.76 (12)	O1^{ii}—Ca2—Ca1	133.99 (3)
N3—C19—H19A	109.5	O2^{ii}—Ca2—Ca1	136.46 (3)
C20—C19—H19A	109.5	O2—Ca2—Ca1	44.56 (2)
N3—C19—H19B	109.5	O3—Ca2—Ca1	44.63 (2)
C20—C19—H19B	109.5	O3^{ii}—Ca2—Ca1	135.18 (2)
H19A—C19—H19B	108.1	O1—Ca2—Ca1^{ii}	133.99 (3)
N4—C20—C19	112.84 (12)	O1^{ii}—Ca2—Ca1^{ii}	45.15 (2)
N4—C20—H20A	109.0	O2^{ii}—Ca2—Ca1^{ii}	44.56 (2)
C19—C20—H20A	109.0	O2—Ca2—Ca1^{ii}	136.46 (3)
N4—C20—H20B	109.0	O3—Ca2—Ca1^{ii}	135.18 (2)
C19—C20—H20B	109.0	O3^{ii}—Ca2—Ca1^{ii}	44.63 (2)
H20A—C20—H20B	107.8	Ca1—Ca2—Ca1	178.905 (17)

N4—C21—C23	115.67 (13)	C2—O1—Ca1	129.03 (9)
N4—C21—C22	111.04 (13)	C2—O1—Ca2	141.40 (9)
C23—C21—C22	109.44 (13)	Ca1—O1—Ca2	89.47 (3)
N4—C21—H21	106.7	C15—O2—Ca1	127.83 (9)
C23—C21—H21	106.7	C15—O2—Ca2	141.58 (9)
C22—C21—H21	106.7	Ca1—O2—Ca2	89.93 (4)
C21—C22—H22A	109.5	C28—O3—Ca1	129.24 (9)
C21—C22—H22B	109.5	C28—O3—Ca2	140.91 (9)
H22A—C22—H22B	109.5	Ca1—O3—Ca2	89.82 (3)
C21—C22—H22C	109.5	C4—N1—C6	119.69 (13)
H22A—C22—H22C	109.5	C4—N1—Ca1	126.46 (10)
H22B—C22—H22C	109.5	C6—N1—Ca1	112.81 (9)
C21—C23—H23A	109.5	C17—N3—C19	120.03 (12)
C21—C23—H23B	109.5	C17—N3—Ca1	125.30 (9)
H23A—C23—H23B	109.5	C19—N3—Ca1	114.12 (9)
C21—C23—H23C	109.5	C20—N4—C21	113.84 (12)
H23A—C23—H23C	109.5	C20—N4—C24	114.37 (12)
H23B—C23—H23C	109.5	C21—N4—C24	115.50 (12)
N4—C24—C25	109.12 (13)	C30—N5—C32	120.04 (12)
N4—C24—C26	116.05 (13)	C30—N5—Ca1	126.39 (10)
C25—C24—C26	110.30 (14)	C32—N5—Ca1	113.15 (9)
N4—C24—H24	107.0	C33—N6—C34	113.61 (12)
C25—C24—H24	107.0	C33—N6—C37	114.16 (12)
C26—C24—H24	107.0	C34—N6—C37	116.29 (12)

Symmetry codes: (i) $-x+1, y, -z+1/2$; (ii) $-x, y, -z+1/2$.

Fig. 1


C26
C23
C24
C31
C27
C29
N4
C21
C28
C25
C30
C35
C20
C22
C34
N5
C36
O3
C39
C37
N6
C32
C19
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N3
O1i
Ca1
O2i
C38
C18
Ca1i
Ca2
O2
C17
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C15
C16
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C6
C1
C2
C3
C4
C7
C14
C5
O3i
C8
N2
C13
C10
C11
C9
C12