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Tris(4-methylbenzyl)(1,4,7-trimethyl-1,4,7-triazacyclononane)lanthanum(III)

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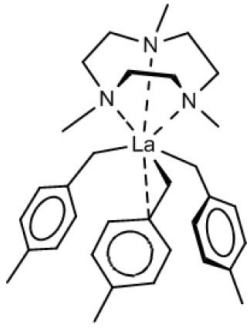
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.012 Å; R factor = 0.048; wR factor = 0.109; data-to-parameter ratio = 18.4.

The title compound, $[La(C_8H_9)_3(C_9H_{21}N_3)]$, incorporating a *fac*-$\kappa^3N$ ligand and formed by reaction of $La(CH_2C_6H_4\text{-}4\text{-}Me)_3(THF)_3$ (THF is tetrahydrofuran) and 1,4,7-trimethyl-1,4,7-triazacyclononane (Me_3-TACN), was synthesized in THF solution. In the crystal structure, the La atom is seven-coordinated by three N atoms from a TACN ligand and four C atoms from three benzyl ligands, one of which is bound η^2.

Related literature

For related literature, see: Hitchcock *et al.* (1988); Harder (2005); Bambirra *et al.* (2006)



Experimental

Crystal data

$[La(C_8H_9)_3(C_9H_{21}N_3)]$
M_r = 625.67
Orthorhombic, $Pna2_1$
a = 16.094 (2) Å
b = 13.472 (1) Å
c = 14.488 (1) Å
V = 3141.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.38 mm^{-1}
T = 100 (1) K
0.22 × 0.15 × 0.06 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006))
T_{min} = 0.758, T_{max} = 0.920
23512 measured reflections
6249 independent reflections
4265 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.109
S = 0.97
6249 reflections
340 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}
Absolute structure: Flack (1983), 3077 Friedel pairs
Flack parameter: 0.05 (2)

Table 1
Selected geometric parameters (Å, °).

La—N1	2.717 (7)	La—C12	3.111 (7)
La—N2	2.806 (7)	La—C18	2.605 (9)
La—N3	2.761 (6)	La—C19	3.165 (7)
La—C10	2.641 (7)	La—C26	2.662 (7)
La—C11	2.959 (7)	La—C27	3.744 (7)
N1—La—N2	63.4 (2)	C18—La—C26	117.6 (3)
N1—La—N3	64.94 (19)	La—C10—C11	87.9 (4)
N2—La—N3	63.6 (2)	La—C18—C19	98.8 (5)
C10—La—C18	105.1 (2)	La—C26—C27	127.7 (5)
C10—La—C26	115.9 (2)		

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

This project was supported by NRSC-C.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2078).

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supplementary materials

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Tris(4-methylbenzyl)(1,4,7-trimethyl-1,4,7-triazacyclononane)lanthanum(III)

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Comment

Homoleptic trialkyl complexes of the type $M(CH_2SiMe_3)_3(THF)_n$ (n = 2, 3) of the group 3 metals and lanthanides are valuable starting materials for organo-rare-earth-metal chemistry. These, however, are only available for the small to medium sized metals (*M* = Sc—Sm). For the larger metals such as neodymium and lanthanum they can not be isolated. Nevertheless, trialkyl complexes of lanthanum have been reported. Examples are $La[CH(SiMe_3)_2]_3$ with relatively large alkyls and $La(CH_2C_6H_4\text{-}2\text{-}NMe_2)_3$ with an internal Lewis base. Recently we have described the synthesis, structures and reactivity studies of $La(CH_2C_6H_4\text{-}4\text{-}R)_3(THF)_3$ (*R* = H, Me). Here we report the molecular structure of (Me_3—TACN)$La(CH_2C_6H_4$-4-Me$)_3$ (Me_3—TACN = 1,4.7-trimethyl-l,4,7-triazacyclononane) (I) that has been generated by addition of Me_3—TACN to a solution of $La(CH_2C_6H_4\text{-}4\text{-}Me)_3(THF)_3$ in THF. Compound I could be isolated as yellow-orange crystals that are thermally much more stable than its precursor. The asymmetric unit of I contains one formula unit of the title compound shown in Figure 1. A s expected the TACN ligand is facially coordinated to the metal center as opposed to the structure of $La(CH_2C_6H_4\text{-}2\text{-}NMe_2)_3$ (Harder, 2005) were the coordination of one of the aminobenzyl ligands is "upside-down". It appears that the *fac*-κ^3 coordination of the TACN ligand increases steric congestion around the metal center compared to $La(CH_2C_6H_4\text{-}4\text{-}Me)_3(THF)_3$. This is evidenced by the bonding mode of the benzyl ligands, of which two are clearly η^1 bound to La and one that is bound in a η^2-fashion. The former have rather large La—C—C bond angles of 98.8 (5)° and 127.7 (5)°, while the latter has a acute angle of 87.9 (4)°.

Experimental

All preparations were performed under an inert nitrogen atmosphere, using standard Schlenk and glovebox techniques. Solid $La(CH_2Ph\text{-}4\text{-}Me)_3[(THF)_3]$ (134.0 mg, 200.0 µmol) was reacted with a solution of [Me_3—TACN] (34.0 mg, 200 µmol) in THF (2 ml). The formed red solution was left to stand over night at −30 °C, after which time yellow-orange crystals of the title compound deposit (90 mg, 72%)·1H NMR (500 MHz, THF-d_8, 20°C): δ 6.66 (t, $^3J_{HH}$ = 8.1 Hz, 6 H, *m*-Ar), 6.14 (d, $^3J_{HH}$ = 8.1 Hz, 6 H, *o*-Ar), 2.70 (m, 6 H, NCH_2), 2.60 (m, 6 H, NCH_2), 2.37 (s, 9H, NMe), 2.10 (s, 9 H, Me), 1.29 (s, 6 H, $LaCH_2$). ^{13}C NMR (125.7 MHz, THF-d_8, 20°C): δ 150.7 (Ar C_{ipso}), 131.8 (d, $^1J_{CH}$ = 153.3 Hz, Ar CH), 125.9 (Ar CMe), 123.6 (d, $^1J_{CH}$ = 152.0 Hz, Ar CH), 68.7 (t, $^1J_{CH}$ = 125.0 Hz, $LaCH_2$), 56.6 (t, $^1J_{CH}$ = 134.8 Hz, NCH_2), 48.2 (q, $^1J_{CH}$ = 134.8 Hz, NMe), 21.8 (q, $^1J_{CH}$ = 125.2 Hz, ArMe). Anal. Calcd for $C_{33}H_{48}N_3La$: C 63.35; H 7.73; N 6.72. Found: C, 63.20; H, 7.68; N, 6.45.

Refinement

The structure was solved by Patterson methods and extension of the model was accomplished by direct methods applied to difference structure factors using the program *DIRDIF*. The positional and anisotropic displacement parameters for the

non-hydrogen atoms were refined. The hydrogen atoms were generated by geometrical considerations, constrained to idealized geometries, and allowed to ride on their carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms. The methyl-groups were refined as rigid groups, which were allowed to rotate freely.

Figures

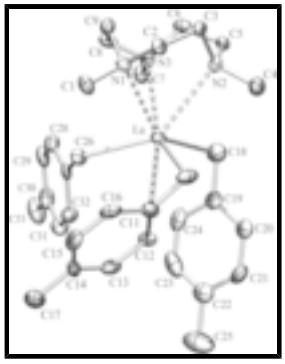


Fig. 1. Perspective *ORTEP* drawing of I. Displacement ellipsoids for non-H are represented at the 50% probability level. The H-atoms have been omitted to improve clarity.

tris(4-methylbenzyl)(1,4,7-trimethyl-1,4,7-triazacyclononane)lanthanum(III)

Crystal data

$[La(C_8H_9)_3(C_9H_{21}N_3)]$	The final unit cell was obtained from the xyz centroids of 5692 reflections after integration using the SAINTPLUS software package (Bruker, 2006). Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the finalatomic coordinates of the structure did not yield extra symmetry elements [Le Page, Y. (1987). J. Appl. Cryst. 20, 264–269; Le Page, Y. (1988). J. Appl. Cryst. 21, 983–984; Spek, A. L. (1988). J. Appl. Cryst. 21, 578–579].
M_r = 625.67	D_x = 1.323 Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 5692 reflections
a = 16.094 (2) Å	θ = 2.4–27.2°
b = 13.472 (1) Å	μ = 1.38 mm^{-1}
c = 14.488 (1) Å	T = 100 (1) K
V = 3141.3 (5) Å^3	Platelet, orange
Z = 4	0.22 × 0.15 × 0.06 mm
F_{000} = 1296	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	6249 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	4265 reflections with $I > 2\sigma(I)$
Monochromator: parallel mounted graphite	R_{int} = 0.093

Detector resolution: 66.06 pixels mm^{-1}

T = 100(1) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2006))

T_{min} = 0.758, T_{max} = 0.920

23512 measured reflections

θ_{max} = 26.4º

θ_{min} = 2.5º

h = −20→20

k = −16→16

l = −18→17

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.048

$wR(F^2)$ = 0.109

S = 0.97

6249 reflections

340 parameters

1 restraint

Primary atom site location: heavy-atom method

Secondary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 1.47 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.60 e $Å^{-3}$

Extinction correction: none

Absolute structure: Flack & Bernardinelli (1999, 2000), 2909 Friedel pairs

Flack parameter: 0.05 (2)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2$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}
La	0.42537 (2)	0.58635 (2)	0.50450 (4)	0.0211 (1)
N1	0.5365 (4)	0.6802 (5)	0.3935 (5)	0.026 (2)
N2	0.5294 (4)	0.7266 (6)	0.5891 (5)	0.027 (3)
N3	0.5890 (4)	0.5310 (5)	0.5298 (4)	0.025 (2)
C1	0.4963 (4)	0.6822 (6)	0.3013 (5)	0.029 (3)
C2	0.5419 (5)	0.7849 (6)	0.4269 (6)	0.024 (3)
C3	0.5758 (4)	0.7908 (5)	0.5244 (5)	0.023 (3)
C4	0.4736 (4)	0.7884 (6)	0.6465 (5)	0.034 (3)
C5	0.5867 (5)	0.6692 (7)	0.6479 (6)	0.030 (3)

C6	0.6392 (5)	0.5957 (6)	0.5938 (6)	0.027 (3)
C7	0.5891 (4)	0.4283 (5)	0.5652 (5)	0.032 (3)
C8	0.6227 (5)	0.5317 (6)	0.4373 (6)	0.028 (3)
C9	0.6199 (5)	0.6331 (7)	0.3894 (6)	0.027 (3)
C10	0.3210 (4)	0.7356 (5)	0.4818 (5)	0.030 (3)
C11	0.2747 (4)	0.6733 (6)	0.4198 (5)	0.026 (3)
C12	0.2381 (4)	0.5834 (5)	0.4513 (5)	0.023 (2)
C13	0.1999 (4)	0.5183 (6)	0.3904 (6)	0.032 (3)
C14	0.1981 (6)	0.5344 (8)	0.2965 (7)	0.032 (3)
C15	0.2339 (5)	0.6229 (7)	0.2639 (5)	0.032 (3)
C16	0.2709 (4)	0.6885 (6)	0.3221 (5)	0.031 (3)
C17	0.1569 (5)	0.4621 (8)	0.2326 (6)	0.055 (4)
C18	0.4079 (4)	0.5330 (7)	0.6762 (6)	0.039 (3)
C19	0.3228 (4)	0.4991 (6)	0.6723 (5)	0.030 (3)
C20	0.2566 (5)	0.5528 (7)	0.7110 (5)	0.033 (3)
C21	0.1759 (6)	0.5205 (9)	0.7018 (7)	0.037 (3)
C22	0.1554 (5)	0.4338 (7)	0.6528 (6)	0.040 (3)
C23	0.2200 (6)	0.3807 (7)	0.6145 (6)	0.046 (3)
C24	0.3022 (5)	0.4114 (6)	0.6238 (5)	0.036 (3)
C25	0.0657 (6)	0.4015 (9)	0.6432 (8)	0.072 (4)
C26	0.4194 (4)	0.4447 (5)	0.3766 (5)	0.026 (2)
C27	0.4138 (4)	0.3371 (5)	0.3910 (5)	0.021 (2)
C28	0.4841 (5)	0.2750 (6)	0.3899 (5)	0.031 (3)
C29	0.4791 (5)	0.1733 (6)	0.4079 (5)	0.034 (3)
C30	0.4029 (5)	0.1277 (7)	0.4265 (6)	0.038 (3)
C31	0.3335 (5)	0.1875 (6)	0.4267 (5)	0.035 (3)
C32	0.3384 (4)	0.2881 (6)	0.4098 (5)	0.029 (3)
C33	0.3974 (6)	0.0178 (6)	0.4438 (7)	0.055 (4)
H1	0.53078	0.72056	0.25841	0.0434*
H1'	0.44138	0.71308	0.30625	0.0434*
H1"	0.49031	0.61416	0.27820	0.0434*
H2	0.48600	0.81541	0.42505	0.0293*
H2'	0.57850	0.82323	0.38504	0.0293*
H3	0.63494	0.77068	0.52416	0.0279*
H3'	0.57277	0.86035	0.54611	0.0279*
H4	0.43657	0.74540	0.68233	0.0510*
H4'	0.44036	0.83164	0.60649	0.0510*
H4"	0.50685	0.82916	0.68859	0.0510*
H5	0.55402	0.63267	0.69473	0.0359*
H5'	0.62387	0.71578	0.68081	0.0359*
H6	0.68080	0.63280	0.55709	0.0319*
H6'	0.66970	0.55285	0.63775	0.0319*
H7	0.55386	0.38668	0.52597	0.0471*
H7'	0.56762	0.42763	0.62852	0.0471*
H7"	0.64602	0.40238	0.56477	0.0471*
H8	0.59143	0.48335	0.39933	0.0336*
H8'	0.68119	0.50912	0.43990	0.0336*
H9	0.66090	0.67770	0.41886	0.0327*
H9'	0.63612	0.62481	0.32392	0.0327*

H10	0.29025	0.75419	0.53844	0.0358*
H10'	0.34600	0.79464	0.45204	0.0358*
H12	0.23978	0.56764	0.51519	0.0272*
H13	0.17403	0.46042	0.41431	0.0385*
H15	0.23209	0.63725	0.19969	0.0384*
H16	0.29529	0.74670	0.29695	0.0372*
H17	0.18329	0.39693	0.23883	0.0829*
H17'	0.16258	0.48543	0.16883	0.0829*
H17"	0.09784	0.45682	0.24834	0.0829*
H18	0.44667	0.47909	0.69341	0.0472*
H18'	0.41477	0.58965	0.71908	0.0472*
H20	0.26771	0.61225	0.74396	0.0393*
H21	0.13254	0.55797	0.72952	0.0446*
H23	0.20818	0.32177	0.58104	0.0551*
H24	0.34527	0.37269	0.59700	0.0434*
H25	0.06367	0.33119	0.62588	0.1081*
H25'	0.03851	0.44133	0.59537	0.1081*
H25"	0.03692	0.41096	0.70214	0.1081*
H26	0.37140	0.46186	0.33690	0.0314*
H26'	0.46948	0.45477	0.33802	0.0314*
H28	0.53683	0.30329	0.37646	0.0374*
H29	0.52835	0.13446	0.40751	0.0415*
H31	0.28070	0.15862	0.43891	0.0414*
H32	0.28870	0.32608	0.41078	0.0344*
H33	0.35714	−0.01173	0.40115	0.0816*
H33'	0.37950	0.00605	0.50751	0.0816*
H33"	0.45208	−0.01251	0.43400	0.0816*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
La	0.0182 (1)	0.0200 (2)	0.0252 (2)	0.0003 (2)	−0.0014 (4)	0.0005 (4)
N1	0.024 (4)	0.022 (4)	0.031 (4)	−0.004 (3)	0.000 (3)	−0.003 (3)
N2	0.025 (4)	0.033 (5)	0.022 (4)	−0.003 (4)	0.004 (3)	−0.002 (4)
N3	0.025 (3)	0.024 (4)	0.025 (5)	0.001 (2)	−0.002 (2)	−0.001 (3)
C1	0.036 (4)	0.026 (5)	0.025 (4)	−0.005 (3)	−0.007 (3)	−0.002 (4)
C2	0.029 (4)	0.014 (5)	0.030 (5)	−0.002 (3)	0.002 (4)	−0.006 (4)
C3	0.023 (3)	0.016 (3)	0.030 (8)	−0.001 (3)	0.007 (3)	−0.007 (3)
C4	0.030 (4)	0.034 (5)	0.038 (5)	0.005 (4)	−0.001 (4)	−0.009 (4)
C5	0.024 (5)	0.039 (6)	0.027 (5)	−0.011 (4)	0.003 (4)	−0.011 (4)
C6	0.018 (4)	0.031 (6)	0.031 (5)	0.001 (4)	−0.008 (3)	0.009 (4)
C7	0.037 (4)	0.026 (5)	0.032 (5)	0.004 (3)	−0.002 (3)	0.002 (4)
C8	0.020 (4)	0.022 (5)	0.042 (6)	−0.003 (4)	−0.005 (4)	0.001 (4)
C9	0.028 (5)	0.028 (6)	0.026 (5)	−0.001 (4)	0.004 (4)	−0.003 (4)
C10	0.018 (3)	0.024 (4)	0.048 (8)	0.005 (3)	0.001 (3)	0.005 (4)
C11	0.018 (3)	0.026 (5)	0.034 (5)	0.008 (3)	−0.006 (3)	−0.001 (4)
C12	0.017 (3)	0.028 (4)	0.023 (4)	0.005 (3)	−0.008 (3)	0.013 (4)
C13	0.020 (4)	0.027 (5)	0.049 (6)	0.002 (3)	0.004 (4)	0.000 (4)

C14	0.027 (5)	0.043 (7)	0.026 (5)	0.000 (4)	−0.001 (4)	−0.014 (5)
C15	0.032 (4)	0.050 (6)	0.014 (4)	0.011 (4)	−0.001 (3)	0.011 (4)
C16	0.016 (4)	0.036 (5)	0.042 (5)	0.000 (3)	0.003 (4)	0.008 (4)
C17	0.031 (5)	0.082 (8)	0.052 (6)	−0.006 (5)	−0.002 (4)	−0.019 (6)
C18	0.035 (5)	0.050 (6)	0.033 (5)	0.002 (4)	−0.004 (4)	0.009 (4)
C19	0.034 (4)	0.038 (5)	0.019 (4)	−0.001 (4)	0.001 (3)	0.012 (4)
C20	0.039 (5)	0.041 (6)	0.019 (5)	−0.003 (4)	0.000 (4)	0.008 (4)
C21	0.029 (5)	0.053 (7)	0.030 (6)	0.011 (5)	0.006 (4)	0.002 (5)
C22	0.043 (5)	0.045 (6)	0.031 (5)	−0.006 (4)	−0.004 (4)	0.016 (4)
C23	0.075 (7)	0.034 (6)	0.029 (5)	−0.007 (5)	−0.014 (5)	0.007 (4)
C24	0.051 (5)	0.035 (5)	0.023 (4)	0.017 (4)	0.003 (4)	0.010 (4)
C25	0.055 (6)	0.090 (9)	0.071 (8)	−0.026 (6)	−0.017 (5)	0.038 (6)
C26	0.027 (4)	0.016 (4)	0.035 (4)	0.001 (3)	−0.001 (3)	−0.003 (3)
C27	0.028 (4)	0.019 (4)	0.017 (4)	0.004 (3)	−0.003 (3)	−0.005 (3)
C28	0.037 (4)	0.028 (5)	0.029 (4)	−0.001 (3)	0.003 (3)	0.000 (4)
C29	0.050 (5)	0.025 (5)	0.028 (5)	0.008 (4)	−0.009 (4)	−0.007 (4)
C30	0.059 (6)	0.025 (5)	0.031 (5)	0.000 (4)	0.001 (4)	0.001 (4)
C31	0.039 (5)	0.028 (5)	0.037 (5)	−0.005 (4)	0.001 (4)	0.005 (4)
C32	0.026 (4)	0.025 (5)	0.035 (5)	0.002 (3)	0.000 (3)	0.001 (4)
C33	0.087 (7)	0.032 (6)	0.045 (6)	0.003 (5)	−0.013 (5)	0.002 (5)

Geometric parameters (Å, °)

La—N1	2.717 (7)	C1—H1'	0.9800
La—N2	2.806 (7)	C1—H1"	0.9800
La—N3	2.761 (6)	C2—H2	0.9900
La—C10	2.641 (7)	C2—H2'	0.9900
La—C11	2.959 (7)	C3—H3	0.9900
La—C12	3.111 (7)	C3—H3'	0.9900
La—C18	2.605 (9)	C4—H4	0.9800
La—C19	3.165 (7)	C4—H4'	0.9800
La—C26	2.662 (7)	C4—H4"	0.9800
La—C27	3.744 (7)	C5—H5	0.9900
N1—C1	1.485 (10)	C5—H5'	0.9900
N1—C2	1.494 (11)	C6—H6	0.9900
N1—C9	1.486 (11)	C6—H6'	0.9900
N2—C3	1.478 (10)	C7—H7	0.9800
N2—C4	1.480 (10)	C7—H7'	0.9800
N2—C5	1.475 (11)	C7—H7"	0.9800
N3—C6	1.507 (10)	C8—H8	0.9900
N3—C7	1.476 (9)	C8—H8'	0.9900
N3—C8	1.446 (10)	C9—H9	0.9900
C2—C3	1.516 (11)	C9—H9'	0.9900
C5—C6	1.520 (12)	C10—H10	0.9900
C8—C9	1.533 (12)	C10—H10'	0.9900
C10—C11	1.438 (10)	C12—H12	0.9500
C11—C12	1.422 (10)	C13—H13	0.9500
C11—C16	1.432 (10)	C15—H15	0.9500
C12—C13	1.388 (11)	C16—H16	0.9500

C13—C14	1.378 (13)	C17—H17	0.9800
C14—C15	1.406 (14)	C17—H17'	0.9800
C14—C17	1.499 (14)	C17—H17"	0.9800
C15—C16	1.359 (11)	C18—H18	0.9900
C18—C19	1.445 (9)	C18—H18'	0.9900
C19—C20	1.405 (11)	C20—H20	0.9500
C19—C24	1.414 (11)	C21—H21	0.9500
C20—C21	1.376 (13)	C23—H23	0.9500
C21—C22	1.406 (15)	C24—H24	0.9500
C22—C23	1.379 (13)	C25—H25	0.9800
C22—C25	1.514 (13)	C25—H25'	0.9800
C23—C24	1.393 (13)	C25—H25"	0.9800
C26—C27	1.467 (10)	C26—H26	0.9900
C27—C28	1.407 (10)	C26—H26'	0.9900
C27—C32	1.408 (9)	C28—H28	0.9500
C28—C29	1.397 (11)	C29—H29	0.9500
C29—C30	1.398 (12)	C31—H31	0.9500
C30—C31	1.377 (12)	C32—H32	0.9500
C30—C33	1.504 (12)	C33—H33	0.9800
C31—C32	1.380 (11)	C33—H33'	0.9800
C1—H1	0.9800	C33—H33"	0.9800
La···H24	3.4300	H3···C12^{i}	2.7800
N1···N2	2.904 (10)	H3···H10^{i}	2.5300
N1···N3	2.942 (9)	H3'···H4'	2.3400
N1···C3	2.493 (10)	H3'···H4"	2.3600
N1···C8	2.516 (11)	H3'···C12^{i}	3.0900
N2···N1	2.904 (10)	H4···C18	2.9000
N2···N3	2.933 (10)	H4···H5	2.4300
N2···C2	2.486 (11)	H4···H18'	2.1900
N2···C6	2.497 (11)	H4'···C10	2.9400
N3···N1	2.942 (9)	H4'···H3'	2.3400
N3···N2	2.933 (10)	H4'···C17^{ii}	2.9800
N3···C5	2.529 (11)	H4"···H3'	2.3600
N3···C9	2.505 (11)	H4"···H5'	2.4300
C3···C12^{i}	3.289 (9)	H4"···H17"^{ii}	2.5600
C3···C11^{i}	3.575 (9)	H4"···H21^{i}	2.6000
C4···C17^{ii}	3.383 (12)	H5···C18	2.7200
C5···C27^{iii}	3.523 (11)	H5···H4	2.4300
C9···C16^{i}	3.554 (11)	H5···H18'	2.3400
C10···C12	2.486 (9)	H5···C26^{iii}	2.8700
C10···C4	3.497 (10)	H5···C27^{iii}	2.9200
C10···C16	2.531 (10)	H5···H26'^{iii}	2.4200
C11···C3^{iv}	3.575 (9)	H5'···H3	2.3900
C12···C24	3.561 (10)	H5'···H4"	2.4300
C12···C3^{iv}	3.289 (9)	H6···C3	2.7600
C16···C9^{iv}	3.554 (11)	H6···C9	2.6200

C17···C4^{v}	3.383 (12)	H6···H3	2.0500
C18···C5	3.437 (11)	H6···H8'	2.3800
C24···C32	3.565 (11)	H6···H9	2.1200
C24···C12	3.561 (10)	H6···C10^{i}	3.0700
C26···C1	3.600 (10)	H6···H10^{i}	2.3400
C26···C8	3.585 (10)	H6'···H7'	2.3600
C27···C5^{vi}	3.523 (11)	H6'···H7"	2.3200
C32···C24	3.565 (11)	H7···C27	3.0600
C1···H18^{vi}	2.8300	H7···C28	2.7200
C1···H7'^{vi}	3.0800	H7···H8	2.3300
C2···H33"^{vii}	3.0900	H7···H28	2.4600
C3···H9	2.5600	H7'···C18	3.0200
C3···H6	2.7600	H7'···H6'	2.3600
C4···H17"^{ii}	2.9400	H7'···H18	2.2700
C4···H18'	3.0300	H7'···C1^{iii}	3.0800
C6···H3	2.5700	H7'···H1"^{iii}	2.4300
C6···H9	2.7900	H7"···H6'	2.3200
C7···H18	3.0300	H7"···H8'	2.3800
C8···H26'	3.0400	H8···C26	2.8400
C9···H6	2.6200	H8···H7	2.3300
C9···H3	2.7000	H8···H26'	2.1900
C10···H4'	2.9400	H8'···H6	2.3800
C10···H2	2.9800	H8'···H7"	2.3800
C10···H3^{iv}	3.0600	H9···C3	2.5600
C10···H9^{iv}	2.9700	H9···C6	2.7900
C10···H6^{iv}	3.0700	H9···H2'	2.4200
C11···H10'	2.0500	H9···H3	2.0200
C11···H10	2.0500	H9···H6	2.1200
C11···H9^{iv}	2.7200	H9···C10^{i}	2.9700
C11···H3^{iv}	2.8100	H9···C11^{i}	2.7200
C12···H3^{iv}	2.7800	H9···C16^{i}	2.8900
C12···H2'^{iv}	3.0200	H9'···H1	2.3300
C12···H3'^{iv}	3.0900	H9'···H1"	2.4400
C13···H32	2.9700	H9'···C18^{vi}	3.1000
C13···H26	2.9700	H9'···C19^{vi}	2.8400
C13···H2'^{iv}	2.9000	H10···H3^{iv}	2.5300
C14···H2'^{iv}	3.0000	H10···H6^{iv}	2.3400
C14···H26	3.0100	H10'···H2	2.3000
C16···H1'	2.7700	H10'···H16	2.4800
C16···H9^{iv}	2.8900	H12···C19	2.8000
C17···H4'^{v}	2.9800	H12···C20	2.8600
C18···H26"^{iii}	3.0700	H12···C21	2.9600
C18···H1"^{iii}	2.9700	H12···C22	3.0100

C18···H9'^{iii}	3.1000	H12···C23	2.9200
C18···H5	2.7200	H12···C24	2.8100
C18···H7'	3.0200	H13···H32	2.5900
C18···H4	2.9000	H15···H17'	2.3700
C19···H12	2.8000	H16···H1'	2.4000
C19···H9'^{iii}	2.8400	H16···H10'	2.4800
C20···H12	2.8600	H17'···H15	2.3700
C21···H12	2.9600	H17'···H33"^{ix}	2.4500
C21···H33^{ii}	2.9700	H17"···C4^{v}	2.9400
C22···H12	3.0100	H17"···H4"^{v}	2.5600
C23···H12	2.9200	H18···C7	3.0300
C24···H12	2.8100	H18···H7'	2.2700
C26···H1"	2.9200	H18···H24	2.5800
C26···H5^{vi}	2.8700	H18···C1^{iii}	2.8300
C26···H8	2.8400	H18···H1"^{iii}	2.0300
C27···H7	3.0600	H18'···C4	3.0300
C27···H5^{vi}	2.9200	H18'···H4	2.1900
C28···H7	2.7200	H18'···H5	2.3400
C30···H21^{v}	3.0600	H18'···H20	2.4100
C32···H24	2.9400	H20···H18'	2.4100
C33···H2^{viii}	3.0900	H21···H25"	2.5400
H1···H2'	2.4200	H21···C30^{ii}	3.0600
H1···H9'	2.3300	H21···H4"^{iv}	2.6000
H1'···C16	2.7700	H23···H25	2.4200
H1'···H2	2.3200	H24···La	3.4300
H1'···H16	2.4000	H24···C32	2.9400
H1"···C26	2.9200	H24···H18	2.5800
H1"···H9'	2.4400	H25···H23	2.4200
H1"···H26'	2.3400	H25"···H21	2.5400
H1"···C18^{vi}	2.9700	H26···C13	2.9700
H1"···H7'^{vi}	2.4300	H26···C14	3.0100
H1"···H18^{vi}	2.0300	H26···H32	2.5000
H2···C10	2.9800	H26'···C8	3.0400
H2···C33^{vii}	3.0900	H26'···H1"	2.3400
H2···H1'	2.3200	H26'···H8	2.1900
H2···H10'	2.3000	H26'···H28	2.3800
H2···H33"^{vii}	2.3900	H26'···C18^{vi}	3.0700
H2'···H1	2.4200	H26'···H5^{vi}	2.4200
H2'···H9	2.4200	H28···H7	2.4600
H2'···C12^{i}	3.0200	H28···H26'	2.3800
H2'···C13^{i}	2.9000	H29···H33"	2.3600
H2'···C14^{i}	3.0000	H32···C13	2.9700
H3···C6	2.5700	H32···H13	2.5900
H3···C9	2.7000	H32···H26	2.5000

H3···H5'	2.3900	H33···C21^{v}	2.9700
H3···H6	2.0500	H33'···H17''^{x}	2.4500
H3···H9	2.0200	H33"···C2^{viii}	3.0900
H3···C10^{i}	3.0600	H33"···H2^{viii}	2.3900
H3···C11^{i}	2.8100	H33"···H29	2.3600
N1—La—N2	63.4 (2)	N2—C3—H3'	109.00
N1—La—N3	64.94 (19)	C2—C3—H3	109.00
N1—La—C10	89.5 (2)	C2—C3—H3'	109.00
N1—La—C11	96.3 (2)	H3—C3—H3'	108.00
N1—La—C18	139.9 (2)	N2—C4—H4	110.00
N1—La—C26	86.9 (2)	N2—C4—H4'	109.00
N2—La—N3	63.6 (2)	N2—C4—H4"	109.00
N2—La—C10	85.5 (2)	H4—C4—H4'	109.00
N2—La—C11	113.8 (2)	H4—C4—H4"	110.00
N2—La—C18	80.4 (2)	H4'—C4—H4"	109.00
N2—La—C26	143.9 (2)	N2—C5—H5	109.00
N3—La—C10	146.0 (2)	N2—C5—H5'	109.00
N3—La—C11	160.62 (19)	C6—C5—H5	109.00
N3—La—C18	84.36 (19)	C6—C5—H5'	109.00
N3—La—C26	86.17 (19)	H5—C5—H5'	108.00
C10—La—C11	29.0 (2)	N3—C6—H6	109.00
C10—La—C18	105.1 (2)	N3—C6—H6'	109.00
C10—La—C26	115.9 (2)	C5—C6—H6	109.00
C11—La—C18	114.6 (2)	C5—C6—H6'	109.00
C11—La—C26	88.0 (2)	H6—C6—H6'	108.00
C18—La—C26	117.6 (3)	N3—C7—H7	110.00
La—N1—C1	104.7 (4)	N3—C7—H7'	109.00
La—N1—C2	106.6 (5)	N3—C7—H7"	109.00
La—N1—C9	114.8 (5)	H7—C7—H7'	109.00
C1—N1—C2	107.4 (6)	H7—C7—H7"	109.00
C1—N1—C9	111.4 (6)	H7'—C7—H7"	109.00
C2—N1—C9	111.3 (6)	N3—C8—H8	109.00
La—N2—C3	114.7 (5)	N3—C8—H8'	109.00
La—N2—C4	105.2 (4)	C9—C8—H8	109.00
La—N2—C5	105.8 (5)	C9—C8—H8'	109.00
C3—N2—C4	109.5 (6)	H8—C8—H8'	108.00
C3—N2—C5	110.9 (6)	N1—C9—H9	109.00
C4—N2—C5	110.5 (6)	N1—C9—H9'	109.00
La—N3—C6	115.9 (5)	C8—C9—H9	109.00
La—N3—C7	107.5 (4)	C8—C9—H9'	109.00
La—N3—C8	103.5 (4)	H9—C9—H9'	108.00
C6—N3—C7	109.1 (6)	La—C10—H10	114.00
C6—N3—C8	111.4 (6)	La—C10—H10'	114.00
C7—N3—C8	109.1 (6)	C11—C10—H10	114.00
N1—C2—C3	111.9 (6)	C11—C10—H10'	114.00
N2—C3—C2	112.2 (6)	H10—C10—H10'	111.00
N2—C5—C6	113.1 (7)	C11—C12—H12	119.00
N3—C6—C5	113.3 (6)	C13—C12—H12	119.00

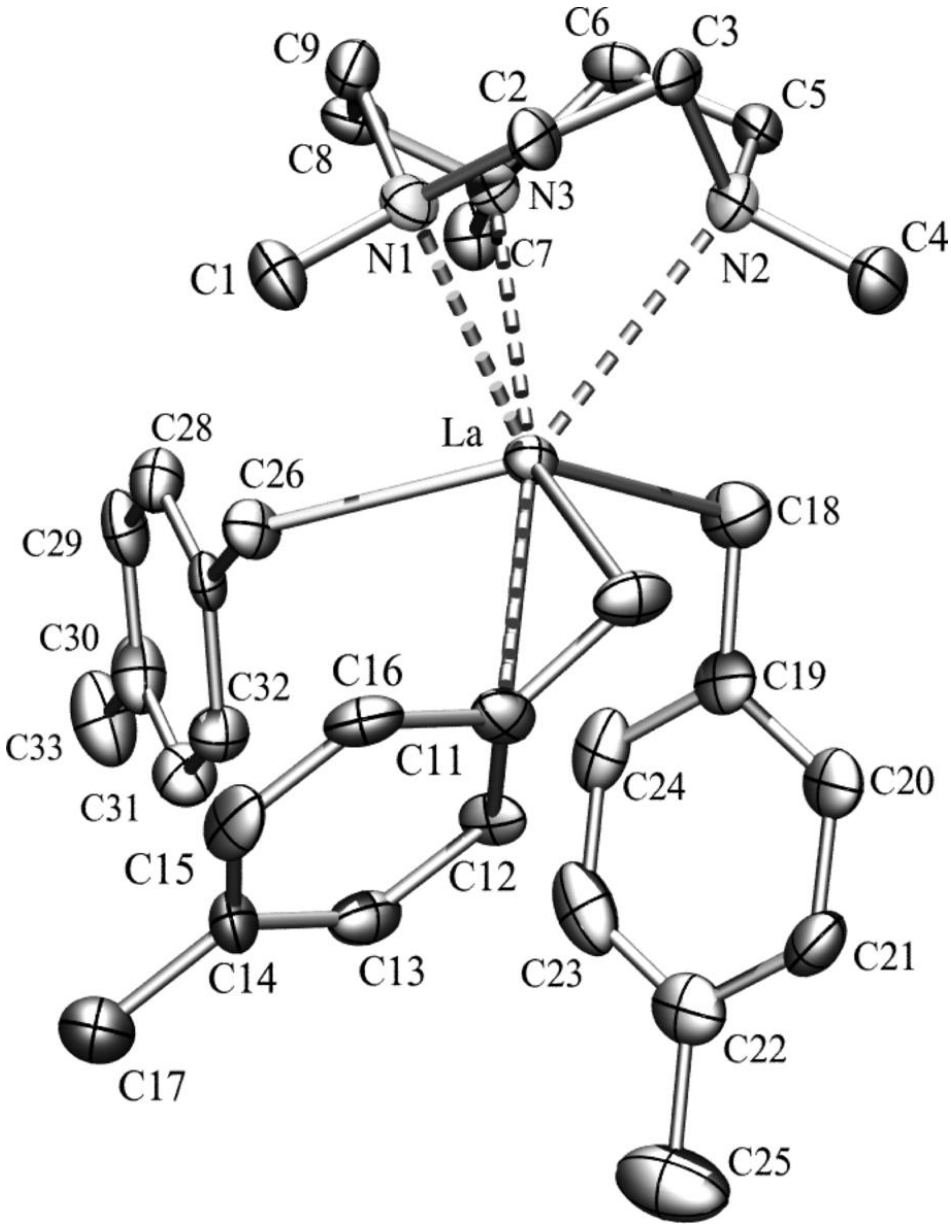
N3—C8—C9	114.5 (7)	C12—C13—H13	119.00
N1—C9—C8	112.9 (7)	C14—C13—H13	119.00
La—C10—C11	87.9 (4)	C14—C15—H15	119.00
La—C11—C10	63.1 (4)	C16—C15—H15	119.00
La—C11—C12	82.5 (4)	C11—C16—H16	119.00
La—C11—C16	120.1 (4)	C15—C16—H16	119.00
C10—C11—C12	120.8 (6)	C14—C17—H17	110.00
C10—C11—C16	123.8 (7)	C14—C17—H17'	109.00
C12—C11—C16	114.9 (7)	C14—C17—H17"	109.00
C11—C12—C13	121.2 (7)	H17—C17—H17'	110.00
C12—C13—C14	122.5 (8)	H17—C17—H17"	110.00
C13—C14—C15	117.2 (8)	H17'—C17—H17"	109.00
C13—C14—C17	121.1 (9)	La—C18—H18	112.00
C15—C14—C17	121.7 (8)	La—C18—H18'	112.00
C14—C15—C16	121.5 (7)	C19—C18—H18	112.00
C11—C16—C15	122.6 (7)	C19—C18—H18'	112.00
La—C18—C19	98.8 (5)	H18—C18—H18'	110.00
C18—C19—C20	122.7 (7)	C19—C20—H20	119.00
C18—C19—C24	120.4 (7)	C21—C20—H20	120.00
C20—C19—C24	116.8 (7)	C20—C21—H21	119.00
C19—C20—C21	121.0 (8)	C22—C21—H21	119.00
C20—C21—C22	122.2 (9)	C22—C23—H23	119.00
C21—C22—C23	117.2 (8)	C24—C23—H23	119.00
C21—C22—C25	120.6 (8)	C19—C24—H24	119.00
C23—C22—C25	122.2 (9)	C23—C24—H24	119.00
C22—C23—C24	121.6 (8)	C22—C25—H25	109.00
C19—C24—C23	121.3 (7)	C22—C25—H25'	109.00
La—C26—C27	127.7 (5)	C22—C25—H25"	109.00
C26—C27—C28	122.4 (6)	H25—C25—H25'	109.00
C26—C27—C32	122.9 (6)	H25—C25—H25"	109.00
C28—C27—C32	114.6 (6)	H25'—C25—H25"	110.00
C27—C28—C29	122.3 (7)	La—C26—H26	105.00
C28—C29—C30	121.2 (8)	La—C26—H26'	105.00
C29—C30—C31	117.1 (8)	C27—C26—H26	105.00
C29—C30—C33	121.1 (8)	C27—C26—H26'	105.00
C31—C30—C33	121.9 (8)	H26—C26—H26'	106.00
C30—C31—C32	121.9 (7)	C27—C28—H28	119.00
C27—C32—C31	123.0 (7)	C29—C28—H28	119.00
N1—C1—H1	109.00	C28—C29—H29	119.00
N1—C1—H1'	110.00	C30—C29—H29	119.00
N1—C1—H1"	109.00	C30—C31—H31	119.00
H1—C1—H1'	109.00	C32—C31—H31	119.00
H1—C1—H1"	109.00	C27—C32—H32	118.00
H1'—C1—H1"	109.00	C31—C32—H32	119.00
N1—C2—H2	109.00	C30—C33—H33	109.00
N1—C2—H2'	109.00	C30—C33—H33'	109.00
C3—C2—H2	109.00	C30—C33—H33"	109.00
C3—C2—H2'	109.00	H33—C33—H33'	110.00
H2—C2—H2'	108.00	H33—C33—H33"	109.00

N2—C3—H3	109.00	H33'—C33—H33"	109.00
N2—La—N1—C1	−150.7 (5)	N2—La—C18—C19	147.4 (5)
N2—La—N1—C2	−37.0 (5)	N3—La—C18—C19	−148.5 (5)
N2—La—N1—C9	86.7 (6)	C10—La—C18—C19	64.8 (6)
N3—La—N1—C1	137.7 (5)	C11—La—C18—C19	35.5 (6)
N3—La—N1—C2	−108.7 (5)	C26—La—C18—C19	−65.8 (6)
N3—La—N1—C9	15.1 (5)	N1—La—C26—C27	141.4 (6)
C10—La—N1—C1	−65.5 (5)	N2—La—C26—C27	108.2 (6)
C10—La—N1—C2	48.2 (5)	N3—La—C26—C27	76.4 (5)
C10—La—N1—C9	172.0 (5)	C10—La—C26—C27	−130.7 (5)
C11—La—N1—C1	−37.2 (5)	C11—La—C26—C27	−122.2 (6)
C11—La—N1—C2	76.5 (5)	C18—La—C26—C27	−5.2 (6)
C11—La—N1—C9	−159.8 (5)	La—N1—C2—C3	63.5 (6)
C18—La—N1—C1	−178.7 (5)	C1—N1—C2—C3	175.3 (6)
C18—La—N1—C2	−65.0 (6)	C9—N1—C2—C3	−62.4 (8)
C18—La—N1—C9	58.8 (7)	La—N1—C9—C8	8.8 (8)
C26—La—N1—C1	50.4 (5)	C1—N1—C9—C8	−110.1 (8)
C26—La—N1—C2	164.1 (5)	C2—N1—C9—C8	130.0 (7)
C26—La—N1—C9	−72.1 (5)	La—N2—C3—C2	14.2 (7)
N1—La—N2—C3	12.5 (4)	C4—N2—C3—C2	−103.8 (7)
N1—La—N2—C4	132.9 (5)	C5—N2—C3—C2	134.0 (7)
N1—La—N2—C5	−110.1 (5)	La—N2—C5—C6	60.3 (7)
N3—La—N2—C3	86.2 (5)	C3—N2—C5—C6	−64.7 (9)
N3—La—N2—C4	−153.4 (5)	C4—N2—C5—C6	173.6 (7)
N3—La—N2—C5	−36.4 (5)	La—N3—C6—C5	11.0 (8)
C10—La—N2—C3	−79.2 (5)	C7—N3—C6—C5	−110.4 (7)
C10—La—N2—C4	41.2 (5)	C8—N3—C6—C5	129.0 (7)
C10—La—N2—C5	158.2 (5)	La—N3—C8—C9	61.3 (7)
C11—La—N2—C3	−72.5 (5)	C6—N3—C8—C9	−63.9 (8)
C11—La—N2—C4	47.9 (5)	C7—N3—C8—C9	175.5 (6)
C11—La—N2—C5	164.8 (5)	N1—C2—C3—N2	−53.0 (8)
C18—La—N2—C3	174.7 (5)	N2—C5—C6—N3	−50.1 (9)
C18—La—N2—C4	−64.9 (5)	N3—C8—C9—N1	−50.4 (9)
C18—La—N2—C5	52.0 (5)	La—C10—C11—C12	61.8 (6)
C26—La—N2—C3	50.2 (7)	La—C10—C11—C16	−109.7 (7)
C26—La—N2—C4	170.6 (4)	La—C11—C12—C13	−121.6 (6)
C26—La—N2—C5	−72.4 (6)	C10—C11—C12—C13	−174.0 (6)
N1—La—N3—C6	84.9 (5)	C16—C11—C12—C13	−1.8 (10)
N1—La—N3—C7	−152.8 (5)	La—C11—C16—C15	97.0 (7)
N1—La—N3—C8	−37.4 (4)	C10—C11—C16—C15	173.0 (7)
N2—La—N3—C6	13.5 (5)	C12—C11—C16—C15	1.1 (10)
N2—La—N3—C7	135.8 (5)	C11—C12—C13—C14	2.8 (11)
N2—La—N3—C8	−108.8 (5)	C12—C13—C14—C15	−2.7 (13)
C10—La—N3—C6	40.2 (7)	C12—C13—C14—C17	179.5 (8)
C10—La—N3—C7	162.5 (4)	C13—C14—C15—C16	1.9 (13)
C10—La—N3—C8	−82.1 (6)	C17—C14—C15—C16	179.7 (8)
C18—La—N3—C6	−68.6 (5)	C14—C15—C16—C11	−1.2 (12)
C18—La—N3—C7	53.8 (4)	La—C18—C19—C20	−106.7 (7)
C18—La—N3—C8	169.2 (5)	La—C18—C19—C24	69.7 (8)

C26—La—N3—C6	173.2 (5)	C18—C19—C20—C21	176.7 (8)
C26—La—N3—C7	−64.5 (4)	C24—C19—C20—C21	0.2 (12)
C26—La—N3—C8	50.9 (5)	C18—C19—C24—C23	−176.1 (8)
N1—La—C10—C11	104.1 (4)	C20—C19—C24—C23	0.5 (11)
N2—La—C10—C11	167.4 (4)	C19—C20—C21—C22	−0.8 (14)
N3—La—C10—C11	143.6 (4)	C20—C21—C22—C23	0.7 (14)
C18—La—C10—C11	−113.8 (4)	C20—C21—C22—C25	−179.2 (9)
C26—La—C10—C11	17.8 (5)	C21—C22—C23—C24	0.0 (13)
N1—La—C11—C10	−77.4 (4)	C25—C22—C23—C24	179.9 (9)
N1—La—C11—C12	152.4 (4)	C22—C23—C24—C19	−0.6 (13)
N1—La—C11—C16	37.9 (6)	La—C26—C27—C28	−96.6 (8)
N2—La—C11—C10	−13.7 (5)	La—C26—C27—C32	81.4 (8)
N2—La—C11—C12	−143.9 (4)	C26—C27—C28—C29	176.9 (7)
N2—La—C11—C16	101.6 (6)	C32—C27—C28—C29	−1.2 (11)
C10—La—C11—C12	−130.2 (6)	C26—C27—C32—C31	−177.5 (7)
C10—La—C11—C16	115.3 (8)	C28—C27—C32—C31	0.6 (11)
C18—La—C11—C10	76.4 (5)	C27—C28—C29—C30	1.1 (12)
C18—La—C11—C12	−53.8 (5)	C28—C29—C30—C31	−0.3 (12)
C18—La—C11—C16	−168.4 (6)	C28—C29—C30—C33	178.6 (8)
C26—La—C11—C10	−164.0 (4)	C29—C30—C31—C32	−0.3 (12)
C26—La—C11—C12	65.8 (4)	C33—C30—C31—C32	−179.3 (8)
C26—La—C11—C16	−48.8 (6)	C30—C31—C32—C27	0.2 (12)
N1—La—C18—C19	172.5 (4)		

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*; (ii) −*x*+1/2, *y*+1/2, *z*+1/2; (iii) −*x*+1, −*y*+1, *z*+1/2; (iv) *x*−1/2, −*y*+3/2, *z*; (v) −*x*+1/2, *y*−1/2, *z*−1/2; (vi) −*x*+1, −*y*+1, *z*−1/2; (vii) *x*, *y*+1, *z*; (viii) *x*, *y*−1, *z*; (ix) −*x*+1/2, *y*+1/2, *z*−1/2; (x) −*x*+1/2, *y*−1/2, *z*+1/2.

Fig. 1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