metal-organic compounds

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[N,N'-Bis(3-methoxy-2-oxidobenzy]idene)cvclohexane-1.2-diaminium- $\kappa^4 O, O', O'', O'''$]tris(nitrato- $\kappa^2 O, O'$)europium(III) methanol monosolvate

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Key indicators: single-crystal X-ray study; T = 291 K; mean σ (C–C) = 0.005 Å; R factor = 0.025; wR factor = 0.068; data-to-parameter ratio = 13.3.

In the title mononuclear salen-type complex, $[Eu(NO_3)_3]$ -(C₂₂H₂₆N₂O₄)]·CH₃OH, the Eu^{III} ion is ten-coordinated by three bidentate nitrate counter-ions and one organic salentype ligand, which acts in a bis-bidentate chelating mode through its phenolate and methoxy O atoms. The protonated imine groups are involved in intramolecular N-H···O hydrogen bonds to the phenolate O atomss, emphasizing the zwitterionic nature of the ligand. An O-H···O hydrogen bond links the complex and solvent molecules.

Related literature

For the synthesis of the salen-type ligand, see: Mohamed et al. (2003); Aslantas et al. (2007). For the synthesis of lanthanide complexes with a similar ligand, see: Yang et al. (2006, 2008).



Experimental

Crystal data [Eu(NO3)3(C22H26N2O4)]·CH4O

 $M_r = 752.48$

Triclinic, P1	
a = 9.7718 (4) Å	
b = 12.8560 (6) Å	
c = 13.0567 (6) Å	
$\alpha = 78.798 \ (1)^{\circ}$	
$\beta = 68.492 \ (1)^{\circ}$	
$\gamma = 81.671 \ (1)^{\circ}$	

Data collection

Rigaku R-AXIS RAPID	8377 measured reflections
diffractometer	5185 independent reflections
Absorption correction: multi-scan	4683 reflections with $I > 2\sigma(I)$
(ABSCOR; Higashi, 1995)	$R_{\rm int} = 0.011$
$T_{\min} = 0.476, \ T_{\max} = 0.670$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$	390 parameters
$wR(F^2) = 0.068$	H-atom parameters constrained
S = 1.02	$\Delta \rho_{\rm max} = 0.76 \text{ e } \text{\AA}^{-3}$
5185 reflections	$\Delta \rho_{\rm min} = -0.72 \ {\rm e} \ {\rm \AA}^{-3}$

V = 1492.09 (12) Å³

 $0.40 \times 0.22 \times 0.20 \text{ mm}$

Mo $K\alpha$ radiation

 $\mu = 2.18 \text{ mm}^{-1}$ T = 291 K

7 - 2

Table 1 Hydrogen-bond geometry (Å, °).

$D - H \cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdot \cdot \cdot A$
$N1-H1N\cdotsO1$ $N2-H2N\cdotsO3$ $O1M-H1O\cdotsO13$	0.86	1.88	2.575 (3)	137
	0.86	1.88	2.593 (3)	139
	0.85	2.18	2.993 (6)	160

Data collection: RAPID-AUTO (Rigaku, 1998); cell refinement: RAPID-AUTO; data reduction: RAPID-AUTO; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2295).

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[N,N'-Bis(3-methoxy-2-oxidobenzylidene)cyclohexane-1,2-diaminium- $<math>\kappa^4 O, O', O'', O'''$]tris(nitrato- $\kappa^2 O, O'$)europium(III) methanol monosolvate

Peng-Fei Yan, Yan Bao, Guang-Ming Li, Jing-Ya Li and Peng Chen

S1. Comment

We present here the crystal structure of the title compound. As shown in Fig. 1, the Eu(III) ion is ten-coordinated by three bidentate nitrate counterions and one ligand that utilizes two hydroxyl oxygen atoms and two methoxy oxygen atoms, while the nitrogen atoms remain protonated (Yang *et al.*, 2006, 2008). The Eu—O bond lengths are in the range of 2.493 (3)–2.604 (3) Å. See Yang *et al.* 2006 and Yang *et al.* (2008) for the synthesis of lanthanide complex with *N*,*N*'-bis-(5-bromo-3-methoxysalicylidene)phenylene-1,2-cyclohexanediamine ligand.

S2. Experimental

To a CH_2Cl_2 solution (5 ml) of H_2L (0.0368 g, 0.1 mmol) under stirring was slowly added a MeCN (5 ml)/MeOH (5 ml) solution of $Eu(NO_3)_3$ $^{\circ}6H_2O$ (0.0446 g, 0.1 mmol) at room temperature. The diethyl ether was allowed to diffuse slowly into the filtrate at room temperature. The light yellow crystals were obtained within one week. [$(Eu(H_2L)(NO_3)_3]\dot{C}H_3OH$ Elemental Anal. Calc. for $C_{23}H_{30}N_5O_{14}Eu$: C, 36.71; H, 4.02; N, 9.31 %, Found: C, 36.78; H, 4.11; N, 9.32 wt%.

S3. Refinement

All H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93-0.97 Å, N-H = 0.86 Å, O-H = 0.85 and $U_{iso}(H) = 1.2 U_{eq}(C, N)$ or $U_{iso}(H) = 1.5 U_{eq}(O, C_{methyl})$.



Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids.

[*N*,*N*'-Bis(3-methoxy-2-oxidobenzylidene)cyclohexane-1,2-diaminium- $\kappa^4 O, O', O'', O'''$]tris(nitrato- $\kappa^2 O, O'$)europium(III) methanol monosolvate

Crystal data

$[Eu(NO_3)_3(C_{22}H_{26}N_2O_4)] \cdot CH_4O$ $M_r = 752.48$ Triclinic, $P\overline{1}$ Hall symbol: -P 1 a = 9.7718 (4) Å b = 12.8560 (6) Å c = 13.0567 (6) Å a = 78.798 (1)° $\beta = 68.492$ (1)° $\gamma = 81.671$ (1)° V = 1492.09 (12) Å ³	Z = 2 F(000) = 756 $D_x = 1.675 \text{ Mg m}^{-3}$ Mo K α radiation, $\lambda = 0.71073 \text{ Å}$ Cell parameters from 21234 reflections $\theta = 2.5-28.3^{\circ}$ $\mu = 2.18 \text{ mm}^{-1}$ T = 291 K Block, colorless $0.40 \times 0.22 \times 0.20 \text{ mm}$
Data collection	
Rigaku R-AXIS RAPID diffractometer Radiation source: fine-focus sealed tube Graphite monochromator ω scans	Absorption correction: multi-scan (<i>ABSCOR</i> ; Higashi, 1995) $T_{min} = 0.476$, $T_{max} = 0.670$ 8377 measured reflections 5185 independent reflections 4683 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.011$	$k = -14 \rightarrow 15$
$\theta_{\rm max} = 25.0^\circ, \theta_{\rm min} = 2.7^\circ$	$l = 0 \rightarrow 15$
$h = -10 \rightarrow 11$	

Refinement	
Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.025$	Hydrogen site location: inferred from
$wR(F^2) = 0.068$	neighbouring sites
S = 1.02	H-atom parameters constrained
5185 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.6408P]$
390 parameters	where $P = (F_{o}^{2} + 2F_{c}^{2})/3$
0 restraints	$(\Delta/\sigma)_{\rm max} = 0.002$
Primary atom site location: structure-invariant	$\Delta \rho_{\rm max} = 0.76 \text{ e } \text{\AA}^{-3}$
direct methods	$\Delta ho_{ m min} = -0.72 \ m e \ { m \AA}^{-3}$

Special details

D C

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. 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 $F^2 > 2sigma(F^2)$ 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.

	x	У	Ζ	$U_{ m iso}$ */ $U_{ m eq}$
Eu1	0.334321 (18)	0.769873 (11)	0.296926 (12)	0.04613 (7)
01	0.2712 (3)	0.64308 (17)	0.45525 (18)	0.0583 (6)
O2	0.2513 (3)	0.83055 (17)	0.5057 (2)	0.0604 (6)
O3	0.4470 (3)	0.60529 (17)	0.24850 (18)	0.0577 (6)
O4	0.5486 (3)	0.77389 (17)	0.10733 (18)	0.0545 (6)
O5	0.5635 (3)	0.7515 (2)	0.3459 (2)	0.0717 (7)
O6	0.6825 (5)	0.8845 (3)	0.3390 (4)	0.1204 (15)
07	0.4880 (3)	0.9107 (2)	0.2920 (2)	0.0691 (7)
08	0.1193 (4)	0.9066 (3)	0.3312 (2)	0.0882 (10)
O9	0.0929 (4)	1.0474 (3)	0.2154 (3)	0.0978 (11)
O10	0.2917 (3)	0.9440 (2)	0.1769 (3)	0.0772 (8)
O11	0.1063 (4)	0.6753 (3)	0.3153 (3)	0.0897 (10)
O12	0.0405 (4)	0.6683 (3)	0.1759 (3)	0.1056 (12)
O13	0.2334 (3)	0.7438 (2)	0.1540 (2)	0.0750 (8)
N1	0.2408 (3)	0.4421 (2)	0.5122 (2)	0.0457 (6)
H1N	0.2568	0.4975	0.4618	0.055*
N2	0.4602 (3)	0.4004 (2)	0.3056 (2)	0.0462 (6)
H2N	0.4272	0.4635	0.3204	0.055*
N3	0.5814 (4)	0.8503 (3)	0.3258 (3)	0.0734 (10)
N4	0.1652 (4)	0.9683 (2)	0.2404 (3)	0.0624 (8)
N5	0.1231 (4)	0.6948 (3)	0.2153 (3)	0.0671 (9)
C1	0.2144 (3)	0.7496 (2)	0.5949 (3)	0.0440 (7)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\hat{A}^2)

C2	0.1654 (4)	0.7602 (3)	0.7053 (3)	0.0505 (8)
H2	0.1561	0.8268	0.7261	0.061*
C3	0.1291 (4)	0.6694 (3)	0.7874 (3)	0.0568 (9)
H3	0.0949	0.6766	0.8624	0.068*
C4	0.1438 (4)	0.5718 (3)	0.7579 (3)	0.0505 (8)
H4	0.1201	0.5127	0.8129	0.061*
C5	0.1950 (3)	0.5590 (2)	0.6442 (2)	0.0410(7)
C6	0.2288 (3)	0.6498(2)	0.5614 (2)	0.0424 (7)
C7	0.2065 (3)	0.4573 (2)	0.6140(2)	0.0433 (7)
H7	0.1886	0.3986	0.6700	0.052*
C8	0 2548 (3)	0.3397(2)	0.4746(3)	0.0426(7)
H8	0.2302	0.2848	0.5407	0.051*
C9	0.1478(4)	0.3382(3)	0.4160(3)	0.0567 (9)
НОА	0.1680	0.3928	0.3509	0.068*
HOR	0.0478	0.3532	0.4655	0.068*
C10	0.0478 0.1627 (4)	0.3332 0.2281 (3)	0.3806 (4)	0.0680 (10)
	0.1027(4) 0.1324	0.2281 (3)	0.3800 (4)	0.0000 (10)
	0.1324	0.1748	0.4403	0.082*
	0.0977 0.2100 (4)	0.2291 0.1084 (2)	0.3392 0.3004 (2)	0.082°
	0.3190 (4)	0.1964 (3)	0.3094 (3)	0.0393 (9)
HIIA	0.3442	0.2457	0.2388	0.071*
HIIB CI2	0.3260	0.1205	0.2943	$0.0/1^{*}$
C12	0.4284 (4)	0.2050 (3)	0.3651 (3)	0.0527(8)
HI2A	0.5277	0.1914	0.3138	0.063*
HI2B	0.4123	0.1502	0.4299	0.063*
C13	0.4150 (3)	0.3129 (2)	0.4009 (3)	0.0447 (7)
H13	0.4784	0.3087	0.4451	0.054*
C14	0.5425 (3)	0.3944 (3)	0.2032 (3)	0.0473 (7)
H14	0.5730	0.3275	0.1817	0.057*
C15	0.5883 (3)	0.4856 (3)	0.1224 (3)	0.0463 (7)
C16	0.6903 (4)	0.4733 (3)	0.0152 (3)	0.0574 (9)
H16	0.7188	0.4057	-0.0046	0.069*
C17	0.7467 (4)	0.5591 (3)	-0.0591 (3)	0.0615 (9)
H17	0.8148	0.5496	-0.1289	0.074*
C18	0.7038 (4)	0.6623 (3)	-0.0320 (3)	0.0568 (9)
H18	0.7449	0.7205	-0.0833	0.068*
C19	0.6014 (4)	0.6770 (3)	0.0700 (3)	0.0471 (7)
C20	0.5417 (4)	0.5890 (3)	0.1506 (3)	0.0460 (7)
C21	0.6066 (5)	0.8667 (3)	0.0304 (4)	0.0790 (13)
H21A	0.7119	0.8618	0.0104	0.119*
H21B	0.5643	0.9291	0.0646	0.119*
H21C	0.5823	0.8713	-0.0354	0.119*
C22	0.2283 (5)	0.9369 (3)	0.5309 (4)	0.0775 (12)
H22A	0.1264	0.9517	0.5740	0.116*
H22B	0.2548	0.9864	0.4628	0.116*
H22C	0.2886	0.9439	0.5727	0.116*
O1M	0.3110 (9)	0.8258 (6)	-0.0896 (5)	0.190 (3)
H1O	0.3098	0.7939	-0.0258	0.285*
C2M	0.204 (2)	0.9063 (7)	-0.0741 (11)	0.303 (11)

H2MA	0.1879	0.9313	-0.1431	0.363*
H2MB	0.2391	0.9638	-0.0555	0.363*
H2MC	0.1121	0.8885	-0.0171	0.454*

Atomic displacement parameters $(Å^2)$

	U^{11}	<i>U</i> ²²	U^{33}	U^{12}	U^{13}	U^{23}
Eu1	0.05387 (12)	0.03330 (10)	0.03868 (10)	-0.00222 (7)	-0.00491 (7)	0.00021 (6)
01	0.0848 (18)	0.0388 (12)	0.0358 (12)	-0.0081 (11)	-0.0029 (12)	-0.0032 (9)
O2	0.0812 (18)	0.0363 (12)	0.0516 (14)	-0.0032 (11)	-0.0097 (13)	-0.0063 (10)
O3	0.0671 (15)	0.0384 (12)	0.0412 (12)	-0.0019 (11)	0.0092 (11)	-0.0029 (10)
O4	0.0574 (14)	0.0414 (12)	0.0456 (13)	-0.0078 (10)	-0.0013 (11)	0.0068 (10)
O5	0.0819 (19)	0.0623 (17)	0.0714 (18)	-0.0032 (14)	-0.0359 (16)	0.0056 (14)
O6	0.136 (3)	0.121 (3)	0.136 (3)	-0.057 (3)	-0.091 (3)	0.027 (3)
O7	0.0823 (19)	0.0531 (15)	0.0707 (17)	-0.0138 (14)	-0.0300 (16)	0.0060 (13)
08	0.085 (2)	0.090 (2)	0.0584 (17)	0.0272 (17)	-0.0053 (16)	-0.0025 (16)
O9	0.105 (3)	0.076 (2)	0.097 (2)	0.0413 (19)	-0.037 (2)	-0.0086 (17)
O10	0.0685 (18)	0.0566 (16)	0.081 (2)	0.0073 (14)	-0.0117 (16)	0.0106 (14)
O11	0.089 (2)	0.112 (3)	0.0550 (18)	-0.0422 (19)	-0.0033 (16)	0.0015 (17)
O12	0.092 (2)	0.138 (3)	0.095 (3)	-0.055 (2)	-0.018 (2)	-0.033 (2)
O13	0.0728 (18)	0.092 (2)	0.0536 (16)	-0.0344 (16)	-0.0083 (14)	-0.0040 (14)
N1	0.0498 (15)	0.0358 (13)	0.0431 (15)	-0.0063 (11)	-0.0094 (12)	0.0018 (11)
N2	0.0430 (15)	0.0362 (13)	0.0502 (16)	-0.0009 (11)	-0.0060 (12)	-0.0074 (11)
N3	0.086 (3)	0.077 (2)	0.059 (2)	-0.023 (2)	-0.032 (2)	0.0085 (18)
N4	0.075 (2)	0.0479 (17)	0.063 (2)	0.0108 (16)	-0.0255 (18)	-0.0141 (15)
N5	0.067 (2)	0.066 (2)	0.061 (2)	-0.0219 (17)	-0.0061 (18)	-0.0137 (16)
C1	0.0397 (17)	0.0422 (17)	0.0463 (18)	-0.0026 (13)	-0.0117 (14)	-0.0052 (14)
C2	0.0482 (19)	0.0532 (19)	0.051 (2)	0.0022 (15)	-0.0163 (16)	-0.0171 (16)
C3	0.059 (2)	0.070 (2)	0.0387 (18)	-0.0025 (18)	-0.0134 (16)	-0.0120 (17)
C4	0.0498 (19)	0.058 (2)	0.0388 (17)	-0.0090 (15)	-0.0123 (15)	0.0011 (15)
C5	0.0355 (16)	0.0433 (16)	0.0389 (16)	-0.0029 (13)	-0.0090 (13)	-0.0023 (13)
C6	0.0381 (16)	0.0440 (17)	0.0391 (16)	-0.0029 (13)	-0.0071 (13)	-0.0053 (13)
C7	0.0385 (16)	0.0468 (18)	0.0374 (16)	-0.0062 (13)	-0.0082 (13)	0.0022 (13)
C8	0.0455 (17)	0.0353 (15)	0.0413 (17)	-0.0070 (13)	-0.0111 (14)	0.0023 (13)
C9	0.0453 (19)	0.063 (2)	0.058 (2)	-0.0027 (16)	-0.0167 (17)	-0.0048 (17)
C10	0.061 (2)	0.073 (3)	0.081 (3)	-0.019 (2)	-0.030 (2)	-0.016 (2)
C11	0.068 (2)	0.051 (2)	0.065 (2)	-0.0102 (17)	-0.0254 (19)	-0.0117 (17)
C12	0.055 (2)	0.0382 (17)	0.060 (2)	-0.0003 (15)	-0.0181 (17)	-0.0044 (15)
C13	0.0435 (17)	0.0397 (16)	0.0484 (18)	-0.0045 (13)	-0.0156 (14)	-0.0006 (13)
C14	0.0428 (18)	0.0460 (18)	0.0505 (19)	-0.0011 (14)	-0.0125 (15)	-0.0107 (15)
C15	0.0415 (17)	0.0490 (18)	0.0417 (17)	0.0014 (14)	-0.0074 (14)	-0.0094 (14)
C16	0.054 (2)	0.064 (2)	0.0452 (19)	0.0036 (17)	-0.0063 (16)	-0.0146 (17)
C17	0.054 (2)	0.078 (3)	0.0376 (18)	0.0034 (19)	-0.0008 (16)	-0.0121 (18)
C18	0.049 (2)	0.068 (2)	0.0409 (18)	-0.0081 (17)	-0.0058 (15)	0.0029 (16)
C19	0.0440 (18)	0.0487 (18)	0.0402 (17)	-0.0019 (14)	-0.0079 (14)	-0.0016 (14)
C20	0.0439 (18)	0.0467 (18)	0.0389 (17)	-0.0031 (14)	-0.0070 (14)	-0.0020 (14)
C21	0.088 (3)	0.049 (2)	0.066 (3)	-0.015 (2)	0.004 (2)	0.0148 (19)
C22	0.106 (3)	0.0391 (19)	0.074 (3)	-0.003 (2)	-0.015 (2)	-0.0109 (18)

O1M	0.290 (9)	0.176 (6)	0.106 (4)	-0.019 (6)	-0.063 (5)	-0.038 (4)
C2M	0.67 (3)	0.119 (7)	0.301 (15)	-0.125 (12)	-0.39 (2)	0.051 (8)

Geometric parameters (Å, °)

Geometric parameters (A, ⁺)				
Eu1—O1	2.315 (2)	С5—С7	1.415 (4)	
Eu1—O3	2.329 (2)	C5—C6	1.415 (4)	
Eu1—O7	2.492 (3)	С7—Н7	0.9300	
Eu1—O8	2.500 (3)	C8—C9	1.509 (5)	
Eu1—O13	2.501 (3)	C8—C13	1.537 (4)	
Eu1—O5	2.513 (3)	C8—H8	0.9800	
Eu1—O10	2.547 (3)	C9—C10	1.544 (5)	
Eu1—O4	2.588 (2)	С9—Н9А	0.9700	
Eu1—O11	2.603 (3)	С9—Н9В	0.9700	
Eu1—O2	2.778 (2)	C10—C11	1.506 (5)	
Eu1—N3	2.930 (4)	C10—H10A	0.9700	
Eu1—N4	2.954 (3)	C10—H10B	0.9700	
O1—C6	1.310 (4)	C11—C12	1.516 (5)	
O2—C1	1.378 (4)	C11—H11A	0.9700	
O2—C22	1.436 (4)	C11—H11B	0.9700	
O3—C20	1.309 (4)	C12—C13	1.521 (4)	
O4—C19	1.386 (4)	C12—H12A	0.9700	
O4—C21	1.438 (4)	C12—H12B	0.9700	
O5—N3	1.271 (4)	C13—H13	0.9800	
O6—N3	1.216 (5)	C14—C15	1.419 (5)	
O7—N3	1.260 (4)	C14—H14	0.9300	
O8—N4	1.259 (4)	C15—C16	1.413 (5)	
O9—N4	1.214 (4)	C15—C20	1.416 (4)	
O10—N4	1.245 (4)	C16—C17	1.352 (5)	
O11—N5	1.233 (4)	C16—H16	0.9300	
O12—N5	1.219 (5)	C17—C18	1.405 (5)	
O13—N5	1.252 (4)	C17—H17	0.9300	
N1—C7	1.293 (4)	C18—C19	1.370 (5)	
N1—C8	1.464 (4)	C18—H18	0.9300	
N1—H1N	0.8600	C19—C20	1.416 (4)	
N2—C14	1.293 (4)	C21—H21A	0.9600	
N2—C13	1.485 (4)	C21—H21B	0.9600	
N2—H2N	0.8600	C21—H21C	0.9600	
C1—C2	1.370 (4)	C22—H22A	0.9600	
C1—C6	1.406 (4)	C22—H22B	0.9600	
C2—C3	1.411 (5)	C22—H22C	0.9600	
С2—Н2	0.9300	O1M—C2M	1.346 (15)	
C3—C4	1.355 (5)	O1M—H1O	0.8506	
С3—Н3	0.9300	C2M—H2MA	0.9600	
C4—C5	1.418 (4)	C2M—H2MB	0.9599	
C4—H4	0.9300	C2M—H2MC	0.9600	
O1—Eu1—O3	71.40 (8)	011—N5—Eu1	60.2 (2)	

O1—Eu1—O7	117.10 (9)	O13—N5—Eu1	55.6 (2)
O3—Eu1—O7	119.72 (9)	C2C1O2	126.3 (3)
O1—Eu1—O8	104.34 (9)	C2C1C6	121.3 (3)
O3—Eu1—O8	151.65 (12)	O2—C1—C6	112.3 (3)
O7—Eu1—O8	87.60 (11)	C1—C2—C3	119.6 (3)
01—Eu1—013	113.51 (9)	C1—C2—H2	120.2
03—Fu1—013	76 75 (10)	C3-C2-H2	120.2
07—Fu1—013	129 39 (9)	C4-C3-C2	120.6(3)
08 - Fu1 - 013	79.81 (11)	C4-C3-H3	119.7
$01 - F_{11} - 05$	79.38 (9)	C2_C3_H3	119.7
$O_3 = E_{\rm H} 1 = O_5$	75.58 (5)	$C_2 C_3 C_4 C_5$	119.7 120.7(3)
05 - Eu1 - 05	50.82 (9)	$C_3 = C_4 = C_3$	120.7 (3)
$0^{9} = E_{11} = 0^{5}$	120.07(11)	C_{5} C_{4} H_{4}	119.0
$0_0 = E_{11} = 0_5$	130.97(11) 144.46(10)	C_{3} C_{4} H_{4}	119.0 120.5(2)
OI_{3} Eul= O_{3}	144.40(10)	$C_{1} = C_{2} = C_{0}$	120.3(3)
OI = EuI = OI0	154.01 (9)	$C/-C_{5}-C_{4}$	120.4 (3)
03—Eu1—010	130.94 (9)	06-05-04	119.0 (3)
O'/—Eul—Ol0	67.06 (10)	01	119.7 (3)
08—Eu1—O10	49.69 (10)	01	121.6 (3)
O13—Eu1—O10	67.59 (10)	C1—C6—C5	118.7 (3)
O5—Eu1—O10	115.53 (9)	N1—C7—C5	123.1 (3)
O1—Eu1—O4	132.42 (8)	N1—C7—H7	118.5
O3—Eu1—O4	63.86 (7)	С5—С7—Н7	118.5
O7—Eu1—O4	74.94 (9)	N1—C8—C9	111.3 (3)
O8—Eu1—O4	122.65 (8)	N1—C8—C13	110.4 (2)
O13—Eu1—O4	71.69 (8)	C9—C8—C13	111.8 (3)
O5—Eu1—O4	75.58 (9)	N1—C8—H8	107.7
O10—Eu1—O4	73.42 (8)	С9—С8—Н8	107.7
O1—Eu1—O11	68.80 (10)	С13—С8—Н8	107.7
O3—Eu1—O11	79.36 (11)	C8—C9—C10	109.9 (3)
O7—Eu1—O11	160.81 (11)	С8—С9—Н9А	109.7
O8—Eu1—O11	73.22 (12)	С10—С9—Н9А	109.7
O13—Eu1—O11	48.66 (9)	С8—С9—Н9В	109.7
O5—Eu1—O11	144.90 (10)	С10—С9—Н9В	109.7
O10—Eu1—O11	99.54 (11)	H9A—C9—H9B	108.2
04—Eu1—011	115.63 (9)	C11—C10—C9	111.5 (3)
01 - Eu1 - 02	60 11 (7)	C11—C10—H10A	109 3
03 - Eu1 - 02	126 21 (8)	C9-C10-H10A	109.3
07—Fu1—02	69 33 (8)	C_{11} C_{10} H_{10B}	109.3
08 - Fu1 - 02	67.85 (9)	C9-C10-H10B	109.3
013 Eu1 02	1/2 $1/2$ $1/2$ $1/2$ $1/2$ $1/2$ $1/2$ $1/2$ $1/2$ $1/2$ $1/2$	$H_{10A} = C_{10} = H_{10B}$	109.5
015 - Eu1 - 02	72.00(0)	$\begin{array}{cccc} 110 & -110 \\ 110 & -110 \\ 110 & -110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\ 110 \\$	100.0
03 - Eu1 - 02	102.24(0)	$C_{10} = C_{11} = C_{12}$	112.1(3)
$O_1 = Bu_1 = O_2$	102.24(7) 142.29(9)	$C_{10} = C_{11} = H_{11} A$	107.2
04—Eu1— 02	142.30(0)	C_{12} C_{11} C	109.2
O1 = Eu1 = O2	101.90 (9)		109.2
O1 - Eu1 - N3	98.04 (9) 08.70 (11)	UI2—UII—HIIB	109.2
U_3 —Eul—N $_3$	98.70(11)	HIIA—UII—HIIB	107.9
O/—Eul—N3	25.25 (9)	C11—C12—C13	112.5 (3)
O8—Eu1—N3	109.63 (12)	C11—C12—H12A	109.1

O13—Eu1—N3	143.40 (9)	C13—C12—H12A	109.1
O5—Eu1—N3	25.57 (9)	C11—C12—H12B	109.1
O10—Eu1—N3	91.20 (10)	C13—C12—H12B	109.1
O4—Eu1—N3	73.81 (9)	H12A—C12—H12B	107.8
O11—Eu1—N3	167.32 (10)	N2—C13—C12	113.2 (3)
O2—Eu1—N3	68.89 (9)	N2—C13—C8	108.9 (2)
O1—Eu1—N4	129.29 (9)	C12—C13—C8	109.6 (3)
O3—Eu1—N4	146.89 (9)	N2—C13—H13	108.3
O7—Eu1—N4	77.08 (9)	С12—С13—Н13	108.3
O8—Eu1—N4	24.95 (9)	C8—C13—H13	108.3
O13—Eu1—N4	71.13 (10)	N2—C14—C15	122.7 (3)
O5—Eu1—N4	127.65 (9)	N2—C14—H14	118.6
O10—Eu1—N4	24.77 (9)	C15—C14—H14	118.6
O4—Eu1—N4	97.84 (8)	C16—C15—C20	119.4 (3)
011—Eu1—N4	85.35 (11)	C16—C15—C14	119.8 (3)
O2—Eu1—N4	85.53 (8)	C20—C15—C14	120.7 (3)
N3—Eu1—N4	102.20 (10)	C17—C16—C15	120.6 (3)
C6	132.23 (19)	C17—C16—H16	119.7
C1 - O2 - C22	116.9 (3)	C15—C16—H16	119.7
C1—O2—Eu1	115.61 (18)	C16-C17-C18	120.9 (3)
C22—O2—Eu1	127.2 (2)	С16—С17—Н17	119.6
C20—O3—Eu1	125.56 (19)	С18—С17—Н17	119.6
C19—O4—C21	116.2 (3)	C19—C18—C17	119.9 (3)
C19—O4—Eu1	116.49 (18)	С19—С18—Н18	120.1
C21—O4—Eu1	126.9 (2)	С17—С18—Н18	120.1
N3—O5—Eu1	95.9 (2)	C18—C19—O4	126.0 (3)
N3—O7—Eu1	97.2 (2)	C18—C19—C20	120.9 (3)
N4—O8—Eu1	98.1 (2)	O4—C19—C20	113.1 (3)
N4—O10—Eu1	96.2 (2)	O3—C20—C15	122.1 (3)
N5—O11—Eu1	95.5 (2)	O3—C20—C19	119.5 (3)
N5—O13—Eu1	100.0 (2)	C15—C20—C19	118.4 (3)
C7—N1—C8	126.2 (3)	C15—C20—Eu1	156.5 (2)
C7—N1—H1N	116.9	C19—C20—Eu1	84.60 (19)
C8—N1—H1N	116.9	O4—C21—H21A	109.5
C14—N2—C13	128.7 (3)	O4—C21—H21B	109.5
C14—N2—H2N	115.6	H21A—C21—H21B	109.5
C13—N2—H2N	115.6	O4—C21—H21C	109.5
O6—N3—O7	122.0 (4)	H21A—C21—H21C	109.5
O6—N3—O5	121.9 (4)	H21B—C21—H21C	109.5
O7—N3—O5	116.1 (3)	O2—C22—H22A	109.5
O6—N3—Eu1	179.0 (4)	O2—C22—H22B	109.5
O7—N3—Eu1	57.55 (19)	H22A—C22—H22B	109.5
O5—N3—Eu1	58.57 (19)	O2—C22—H22C	109.5
O9—N4—O10	121.2 (4)	H22A—C22—H22C	109.5
O9—N4—O8	123.0 (4)	H22B—C22—H22C	109.5
O10—N4—O8	115.8 (3)	C2M—O1M—H1O	107.9
O9—N4—Eu1	177.3 (3)	O1M—C2M—H2MA	108.5
O10—N4—Eu1	58.99 (17)	O1M—C2M—H2MB	108.6

O8—N4—Eu1	56.91 (17)	H2MA—C2M—H2MB	107.6
O12—N5—O11	123.7 (4)	O1M—C2M—H2MC	114.8
O12—N5—O13	120.6 (4)	H2MA—C2M—H2MC	108.6
O11—N5—O13	115.8 (4)	H2MB—C2M—H2MC	108.6
O12—N5—Eu1	176.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A	
N1—H1 <i>N</i> …O1	0.86	1.88	2.575 (3)	137	
N2—H2 <i>N</i> ···O3	0.86	1.88	2.593 (3)	139	
01 <i>M</i> —H1 <i>O</i> …O13	0.85	2.18	2.993 (6)	160	