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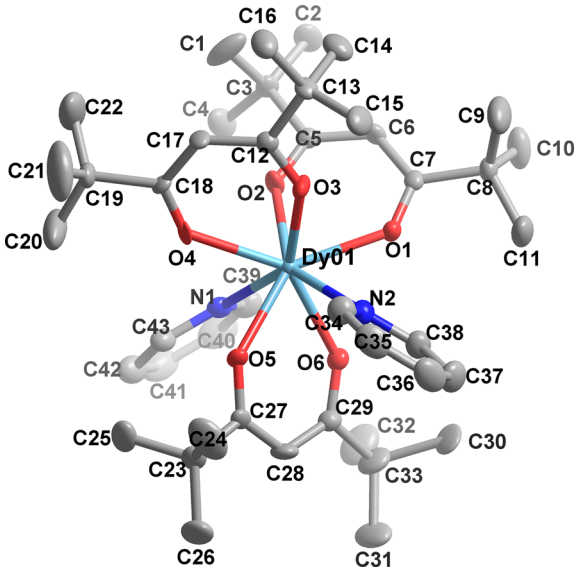
Crystal structure of dipyridine-k^1*N*-tris(2,2,6,6-tetramethyl-5-oxohept-3-en-3-olato-k^2*O*,*O*′) dysprosium(III), $DyC_{43}H_{67}O_6N_2$



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Abstract

$DyC_{43}H_{67}O_6N_2$, triclinic, $P\bar{1}$ (no. 2), a = 10.384(3) Å, b = 13.502(4) Å, c = 16.972(5) Å, α = 94.903(4)°, β = 98.580(4)°, γ = 105.417(4)°, V = 2248.6(11) Å^3, Z = 2, $R_{gt}(F)$ = 0.0519, $wR_{ref}(F^2)$ = 0.1489, T = 150 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.30 × 0.30 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.71 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II
θ_{max}, completeness:	25.0°, 98 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25,176, 7762, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 7453
$N(param)_{refined}$:	517
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

1 Source of materials

A mixture of $Dy(thd)_3$ (thd = 2,2,6,6–Tetramethyl-3,5-heptadione, 350 mg, 0.5 mmol) [5] and NH_4F (37 mg, 1 mmol) were placed in a 30 mL scintillation flask, then 8 mL pyridine was added. The flask was sealed and heated at 120 °C for 72 h. Thereafter, the solution was then cooled to room temperature and filtered. Light yellow block crystals were isolated by evaporation (yield 30 %, based on Dy).

2 Experimental details

Absorption corrections were used by using multi-scan program [1]. Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program and refined with the ShelXL [4] refinement package. The H atoms were fixed, fixed U_{iso} were set to 1.2 times of all C(H) groups.

3 Comment

Due to the unique magnetic properties, the dysprosium-based compounds are widely used in single-molecule magnets (SMMs) to implement large-scale information storage and processing. Some Dy–SMMs, such as $[Dy(Cpttt)_2]^+$ [6], $[(Cp^{iPr5})Dy(Cp^*)]^+$ [7] and $(Cp^{iPr5})_2Dy_2I_3$ [8], with brilliant performance have shocked this field. In this work, we report the single crystal structure of a dysprosium(III) complex, which provides a basis for studying Dy–SMMs.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1825 (12)	0.5262 (8)	0.6059 (7)	0.057 (3)
H1A	0.2444	0.5303	0.5672	0.085*
H1B	0.1174	0.5650	0.5902	0.085*
H1C	0.1334	0.4535	0.6065	0.085*
C2	0.3526 (9)	0.6816 (6)	0.6868 (6)	0.037 (2)
H2A	0.4081	0.7101	0.7399	0.056*
H2B	0.2947	0.7259	0.6712	0.056*
H2C	0.4120	0.6790	0.6474	0.056*
C3	0.2641 (8)	0.5728 (6)	0.6899 (5)	0.0246 (16)
C4	0.1672 (10)	0.5780 (8)	0.7481 (7)	0.047 (3)
H4A	0.1103	0.5080	0.7504	0.071*
H4B	0.1093	0.6212	0.7297	0.071*
H4C	0.2194	0.6081	0.8017	0.071*
C5	0.3452 (7)	0.4969 (6)	0.7178 (4)	0.0201 (15)
C6	0.4845 (8)	0.5222 (6)	0.7269 (5)	0.0234 (16)
H6	0.5312	0.5882	0.7149	0.028*
C7	0.5625 (7)	0.4556 (6)	0.7530 (4)	0.0198 (15)
C8	0.7176 (7)	0.4902 (6)	0.7577 (5)	0.0253 (17)
C9	0.7441 (10)	0.4812 (10)	0.6713 (6)	0.053 (3)
H9A	0.7058	0.4092	0.6461	0.080*
H9B	0.8421	0.5027	0.6719	0.080*
H9C	0.7012	0.5260	0.6408	0.080*
C10	0.7825 (10)	0.6017 (8)	0.7968 (7)	0.053 (3)
H10A	0.7501	0.6486	0.7630	0.079*
H10B	0.8814	0.6178	0.8026	0.079*
H10C	0.7577	0.6105	0.8499	0.079*
C11	0.7833 (9)	0.4191 (8)	0.8042 (7)	0.050 (3)
H11A	0.7627	0.4214	0.8587	0.075*
H11B	0.8819	0.4422	0.8070	0.075*
H11C	0.7475	0.3479	0.7767	0.075*
C12	0.2598 (8)	0.2296 (6)	0.5538 (4)	0.0223 (16)
C13	0.3451 (8)	0.2515 (6)	0.4874 (4)	0.0255 (17)
C14	0.4137 (9)	0.3677 (7)	0.5003 (5)	0.038 (2)
H14A	0.3445	0.4049	0.4928	0.057*
H14B	0.4745	0.3850	0.4614	0.057*
H14C	0.4663	0.3880	0.5550	0.057*
C15	0.4529 (9)	0.1932 (8)	0.4975 (6)	0.041 (2)
H15A	0.5055	0.2120	0.5523	0.061*
H15B	0.5137	0.2118	0.4588	0.061*
H15C	0.4086	0.1184	0.4880	0.061*
C16	0.2618 (9)	0.2203 (8)	0.4022 (5)	0.038 (2)
H16A	0.2141	0.1461	0.3945	0.057*
H16B	0.3226	0.2347	0.3630	0.057*
H16C	0.1956	0.2600	0.3945	0.057*
C17	0.1199 (7)	0.2102 (6)	0.5385 (4)	0.0231 (16)
H17	0.0767	0.2051	0.4843	0.028*
C18	0.0378 (7)	0.1975 (6)	0.5973 (4)	0.0188 (15)
C19	−0.1167 (8)	0.1774 (7)	0.5718 (5)	0.0318 (18)
C20	−0.1878 (11)	0.1577 (14)	0.6402 (7)	0.083 (4)
H20A	−0.1695	0.0976	0.6631	0.125*
H20B	−0.2857	0.1439	0.6218	0.125*
H20C	−0.1555	0.2186	0.6813	0.125*
C21	−0.1728 (12)	0.0847 (13)	0.5069 (10)	0.095 (5)
H21A	−0.1393	0.1022	0.4574	0.142*
H21B	−0.2722	0.0661	0.4967	0.142*
H21C	−0.1431	0.0259	0.5247	0.142*
C22	−0.1427 (12)	0.2685 (11)	0.5345 (10)	0.082 (4)
H22A	−0.1021	0.3316	0.5728	0.123*
H22B	−0.2409	0.2581	0.5203	0.123*
H22C	−0.1023	0.2753	0.4859	0.123*
C23	0.0838 (8)	−0.0634 (6)	0.8368 (5)	0.0307 (19)
C24	0.1142 (12)	−0.1277 (7)	0.7673 (8)	0.061 (3)
H24A	0.1105	−0.0921	0.7195	0.091*
H24B	0.0466	−0.1958	0.7559	0.091*
H24C	0.2050	−0.1366	0.7819	0.091*
C25	−0.0597 (9)	−0.0530 (7)	0.8122 (7)	0.045 (2)
H25A	−0.0833	−0.0143	0.8567	0.067*
H25B	−0.1246	−0.1222	0.7991	0.067*
H25C	−0.0629	−0.0160	0.7650	0.067*
C26	0.0852 (11)	−0.1198 (8)	0.9114 (7)	0.058 (3)
H26A	0.1741	−0.1321	0.9260	0.086*
H26B	0.0145	−0.1863	0.8997	0.086*
H26C	0.0680	−0.0772	0.9562	0.086*
C27	0.1840 (7)	0.0453 (6)	0.8479 (5)	0.0218 (16)
C28	0.2725 (9)	0.0860 (6)	0.9217 (5)	0.0308 (18)
H28	0.2662	0.0445	0.9641	0.037*
C29	0.3688 (8)	0.1831 (6)	0.9369 (5)	0.0253 (17)
C30[a]	0.6141 (18)	0.2505 (18)	1.0062 (10)	0.044 (4)
H30A[a]	0.6308	0.1896	0.9786	0.066*
H30B[a]	0.6761	0.2725	1.0580	0.066*
H30C[a]	0.6293	0.3070	0.9730	0.066*
C30A[a]	0.353 (3)	0.203 (2)	1.0855 (14)	0.078 (7)
H30D[a]	0.2775	0.2315	1.0685	0.117*
H30E[a]	0.4008	0.2369	1.1391	0.117*
H30F[a]	0.3169	0.1281	1.0873	0.117*
C31[a]	0.451 (3)	0.1389 (19)	1.0781 (12)	0.060 (5)
H31A[a]	0.3573	0.1124	1.0864	0.090*
H31B[a]	0.5103	0.1696	1.1298	0.090*
H31C[a]	0.4809	0.0819	1.0545	0.090*
C31A[a]	0.545 (3)	0.164 (3)	1.0408 (17)	0.088 (7)
H31D[a]	0.4962	0.0905	1.0282	0.131*
H31E[a]	0.5828	0.1807	1.0984	0.131*
H31F[a]	0.6192	0.1807	1.0101	0.131*
C32[a]	0.429 (3)	0.312 (2)	1.0546 (15)	0.062 (5)
H32A[a]	0.4486	0.3662	1.0197	0.093*
H32B[a]	0.4845	0.3369	1.1081	0.093*
H32C[a]	0.3323	0.2949	1.0590	0.093*
C32A[a]	0.496 (3)	0.341 (2)	1.0368 (18)	0.080 (8)
H32D[a]	0.5723	0.3713	1.0102	0.119*
H32E[a]	0.5220	0.3642	1.0948	0.119*
H32F[a]	0.4176	0.3646	1.0154	0.119*
C33	0.4585 (11)	0.2215 (8)	1.0211 (5)	0.045 (2)
C34	0.4016 (9)	0.0449 (7)	0.6851 (5)	0.0340 (19)
H34	0.3228	0.0389	0.6464	0.041*
C35	0.4646 (11)	−0.0331 (7)	0.6807 (6)	0.045 (2)
H35	0.4288	−0.0917	0.6407	0.054*
C36	0.5801 (10)	−0.0242 (7)	0.7355 (7)	0.046 (3)
H36	0.6259	−0.0764	0.7339	0.056*
C37	0.6278 (10)	0.0616 (8)	0.7924 (6)	0.043 (2)
H37	0.7075	0.0699	0.8310	0.051*
C38	0.5582 (8)	0.1356 (6)	0.7928 (5)	0.0302 (18)

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H38	0.5923	0.1948	0.8323	0.036*
C39	0.2034 (10)	0.3953 (7)	0.9003 (5)	0.037 (2)
H39	0.2884	0.4390	0.8937	0.044*
C40	0.1430 (12)	0.4257 (8)	0.9615 (6)	0.048 (3)
H40	0.1847	0.4892	0.9957	0.058*
C41	0.0203 (11)	0.3616 (9)	0.9719 (6)	0.049 (3)
H41	−0.0231	0.3794	1.0142	0.059*
C42	−0.0374 (10)	0.2719 (8)	0.9202 (6)	0.042 (2)
H42	−0.1225	0.2272	0.9255	0.051*
C43	0.0287 (8)	0.2473 (7)	0.8606 (5)	0.0290 (18)
H43	−0.0126	0.1846	0.8253	0.035*
Dy01	0.29504 (3)	0.25154 (3)	0.75313 (2)	0.01750 (12)
N1	0.1483 (7)	0.3067 (5)	0.8492 (4)	0.0262 (14)
N2	0.4457 (7)	0.1281 (5)	0.7406 (4)	0.0263 (14)
O1	0.5118 (5)	0.3668 (4)	0.7715 (3)	0.0234 (11)
O2	0.2725 (5)	0.4109 (4)	0.7299 (4)	0.0313 (13)
O3	0.3274 (5)	0.2356 (5)	0.6238 (3)	0.0299 (13)
O4	0.0817 (5)	0.2027 (4)	0.6716 (3)	0.0218 (11)
O5	0.1819 (5)	0.0937 (4)	0.7879 (3)	0.0200 (11)
O6	0.3906 (5)	0.2453 (4)	0.8856 (3)	0.0229 (11)

[a]Occupancy: 0.5.

The title compound crystallizes in the triclinic space group $P\bar{1}$ with two formula units in the unit cell. This compound is composed of three thd ligands, two pyridine and one Dy metal atom. The dysprosium center is octahedrally coordinated by six oxygen atoms of the thd ligands and two nitrogen atoms of the pyridine ring. The distances of Dy—O bonds are in range of 2.271–2.336 Å. The distances of Dy—N bonds are 2.587 and 2.583 Å. In addition, the N—Dy—N angle is 140.3°. All geometric parameters are in the expected ranges [9, 10].

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