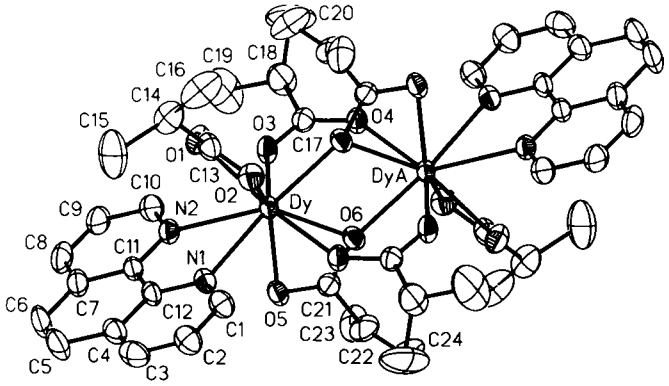
Z. Kristallogr. NCS 220 (2005) 469-470

Crystal structure of tetrakis(μ-isobutyrato-*O*,*O*')bis(isobutyrato-*O*,*O*')-bis(1,10-phenanthroline-*N*,*N*')didysprosium(III), $Dy_2(C_4H_7O_2)_6(C_{12}H_8N_2)_2$

P. Wang*, L.-P. Song, Z.-F. Li and D.-Q. Han

Jiangxi University of Science and Technology, School of Materials & Chemical Engineering, Ganzhou, Jiangxi, 341000 P. R. China

Received July 14, 2005, accepted and available on-line September 19, 2005; CCDC no. 1267/1597



Abstract

$C_{48}H_{58}Dy_2N_4O_{12}$, monoclinic, $P12_1/n1$ (no. 14), a = 12.6175(7) Å, b = 13.2833(9) Å, c = 15.765(1) Å, β = 109.977(3)°, V = 2483.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.066, T = 273 K.

Source of material

A solution obtained by dissolving 0.200 g (0.463 mmol) of Dy_2O_3 in 20 ml (36.5 %) HCl was evaporated to dryness. To the solid was added 25 ml of CH_3OH/H_2O (1:1 *v/v*) and to the resulting solution was added 0.5 ml isobutylic acid and 0.25 g (1.261 mmol) phenanthroline with stirring. This gave a colorless solution. After several days, pale yellow crystals were grown by slow evaporation at room temperature (yield 15 % based on the initial of Dy_2O_3).

Discussion

Being isotypic with $[M(phen)(C_5H_7O_2)_3]_2$ (M = La [1,2], Tb, Ho [3], $C_5H_7O_2$ = *trans*-2,3-dimethylacrylate), the title compound consists of a dinuclear centrosymmetric $[Dy(phen)L_3]_2$ molecule, in which two crystallographically equivalent Dy atoms are bridged by four L^- anions with two different coordination modes. Each Dy atom is coordinated by two N atoms from one chelating phen ligand and seven carboxyl oxygen atoms from five L^- anions, to form a distorted tricapped triangular prism. The Dy—O bond distances vary from 2.306(2) Å to 2.561(2) Å and the Dy—N bond length are 2.566(3) Å, 2.613(3) Å, respectively. The three crystallographically independent carboxylate groups exhibit different types of coordination to Dy: the two carboxylate oxygen atoms of one acid molecule chelate one Dy atom, and one of the other atoms (O6) coordinates to the second Dy atom; one acid molecule coordinates to one Dy atom through each carboxylate oxygen atom; the third acid molecule only chelates one Dy atom with two oxygen atoms. The C—O and C—C distances are usual, within the range of 1.24 Å – 1.27 Å and 1.40 Å – 1.55 Å, respectively. The chelating phen ligand exhibits nearly perfect coplanarity, each phen ligand is antiparallel to one partner of two neighboring phen ligands with the mean distance of 3.25 Å, but nonparallel to the other partner with a dihedral angle of 30° and a nearest distance of 3.56 Å, indicating that, apart from C–H···O bonds, weak π-π stacking interactions are responsible for the assembly of the dimers into layers parallel to (011). The three isobutyrato groups show rotational disorder which could only be characterized by split positions being equally occupied.

* Correspondence author (e-mail: wangp520@yahoo.com.cn)

Table 1. Data collection and handling.

Crystal:	pale yellow needle, size 0.131 × 0.175 × 0.332 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	30.49 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	26040, 5915
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4191
$N(param)_{refined}$:	300
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	4*e*		0.9394	0.2066	0.6284	0.066
H(2)	4*e*		0.8653	0.0700	0.6789	0.078
H(3)	4*e*		0.6967	0.0879	0.7044	0.081
H(5)	4*e*		0.5333	0.1960	0.7013	0.094
H(6)	4*e*		0.4450	0.3456	0.6692	0.098
H(8)	4*e*		0.4426	0.5293	0.6194	0.081
H(9)	4*e*		0.5326	0.6574	0.5761	0.078
H(10)	4*e*		0.7073	0.6284	0.5608	0.066
H(14A)	4*e*	0.5	1.0320	0.4992	0.8867	0.114
H(14B)	4*e*	0.5	1.0714	0.4964	0.8932	0.114
H(18A)	4*e*	0.5	0.8549	0.8201	0.4529	0.101
H(18B)	4*e*	0.5	0.8972	0.8313	0.4591	0.101
H(22A)	4*e*	0.5	0.8379	0.4158	0.2645	0.080
H(22B)	4*e*	0.5	0.7960	0.4662	0.2544	0.080
H(15A)	4*e*	0.5	0.8854	0.3930	0.8594	0.186
H(15B)	4*e*	0.5	0.9748	0.3773	0.9559	0.186
H(15C)	4*e*	0.5	0.9679	0.3009	0.8784	0.186
H(16A)	4*e*	0.5	1.2064	0.4568	0.8930	0.186
H(16B)	4*e*	0.5	1.1785	0.3426	0.8991	0.186
H(16C)	4*e*	0.5	1.1797	0.4165	0.9769	0.186
H(15D)	4*e*	0.5	0.8846	0.4614	0.8653	0.186
H(15E)	4*e*	0.5	0.9604	0.4110	0.9555	0.186
H(15F)	4*e*	0.5	0.9102	0.3457	0.8681	0.186
H(16D)	4*e*	0.5	1.1977	0.3759	0.8881	0.186
H(16E)	4*e*	0.5	1.1085	0.2914	0.8817	0.186
H(16F)	4*e*	0.5	1.1556	0.3567	0.9694	0.186
H(19A)	4*e*	0.5	0.7517	0.7727	0.5382	0.177
H(19B)	4*e*	0.5	0.7819	0.8876	0.5460	0.177

Table 2. Continued.

Atom	Site	Occ	x	y	z	U_{iso}
H(19C)	4e	0.5	0.8467	0.8137	0.6240	0.177
H(20A)	4e	0.5	1.0426	0.8538	0.5093	0.177
H(20B)	4e	0.5	1.0331	0.8668	0.6053	0.177
H(20C)	4e	0.5	0.9672	0.9396	0.5266	0.177
H(19D)	4e	0.5	0.7290	0.7782	0.4641	0.177
H(19E)	4e	0.5	0.7592	0.8886	0.4994	0.177
H(19F)	4e	0.5	0.7711	0.8003	0.5681	0.177
H(20D)	4e	0.5	1.0554	0.8451	0.5827	0.177
H(20E)	4e	0.5	0.9798	0.8453	0.6436	0.177
H(20F)	4e	0.5	0.9680	0.9320	0.5734	0.177
H(23A)	4e	0.5	0.7204	0.5519	0.2490	0.145

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(23B)	4e	0.5	0.6208	0.4778	0.2412	0.145
H(23C)	4e	0.5	0.6792	0.4763	0.1679	0.145
H(24A)	4e	0.5	0.7962	0.2612	0.3035	0.145
H(24B)	4e	0.5	0.7227	0.2888	0.2041	0.145
H(24C)	4e	0.5	0.6699	0.2932	0.2808	0.145
H(23D)	4e	0.5	0.6187	0.4696	0.2625	0.145
H(23E)	4e	0.5	0.6200	0.3519	0.2701	0.145
H(23F)	4e	0.5	0.6240	0.4034	0.1818	0.145
H(24D)	4e	0.5	0.9084	0.3272	0.2910	0.145
H(24E)	4e	0.5	0.8098	0.3159	0.1985	0.145
H(24F)	4e	0.5	0.8027	0.2614	0.2846	0.145

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Dy	4e		0.91688(1)	0.45451(1)	0.576946(9)	0.03236(8)	0.04375(9)	0.03650(8)	0.00052(7)	0.01740(6)	0.00048(7)
O(1)	4e		0.9449(2)	0.5121(2)	0.7279(2)	0.055(2)	0.081(2)	0.045(1)	0.008(1)	0.020(1)	-0.008(1)
O(2)	4e		1.0432(2)	0.3801(2)	0.7195(2)	0.054(1)	0.060(2)	0.049(1)	0.003(1)	0.018(1)	0.004(1)
O(3)	4e		0.8837(2)	0.6264(2)	0.5432(2)	0.053(1)	0.044(1)	0.064(1)	0.008(1)	0.038(1)	0.007(1)
O(4)	4e		0.9972(2)	0.6808(2)	0.4699(2)	0.054(1)	0.042(1)	0.060(1)	0.002(1)	0.034(1)	0.002(1)
O(5)	4e		0.7651(2)	0.4112(2)	0.4356(2)	0.038(1)	0.079(2)	0.050(1)	-0.013(1)	0.021(1)	-0.005(1)
O(6)	4e		0.9102(2)	0.4804(2)	0.4142(1)	0.034(1)	0.055(2)	0.044(1)	-0.005(1)	0.013(1)	0.000(1)
N(1)	4e		0.8211(2)	0.3031(2)	0.6217(2)	0.042(2)	0.053(2)	0.046(2)	-0.005(1)	0.020(1)	0.003(1)
N(2)	4e		0.7211(2)	0.4868(2)	0.5941(2)	0.037(2)	0.058(2)	0.047(2)	0.002(1)	0.020(1)	-0.002(1)
C(1)	4e		0.8712(3)	0.2141(3)	0.6380(2)	0.055(2)	0.058(2)	0.054(2)	-0.002(2)	0.022(2)	0.012(2)
C(2)	4e		0.8274(3)	0.1313(3)	0.6687(2)	0.073(3)	0.061(3)	0.060(2)	-0.006(2)	0.021(2)	0.013(2)
C(3)	4e		0.7275(3)	0.1421(3)	0.6836(2)	0.079(3)	0.070(3)	0.055(2)	-0.029(2)	0.023(2)	0.009(2)
C(4)	4e		0.6721(3)	0.2340(3)	0.6675(2)	0.049(2)	0.077(3)	0.046(2)	-0.017(2)	0.019(2)	0.005(2)
C(5)	4e		0.5663(3)	0.2486(4)	0.6804(3)	0.072(3)	0.097(3)	0.085(3)	-0.019(3)	0.053(2)	0.001(3)
C(6)	4e		0.5150(3)	0.3382(4)	0.6624(3)	0.053(2)	0.129(4)	0.081(3)	-0.024(3)	0.048(2)	-0.005(3)
C(7)	4e		0.5635(3)	0.4227(3)	0.6332(2)	0.044(2)	0.092(3)	0.049(2)	-0.008(2)	0.023(2)	-0.010(2)
C(8)	4e		0.5128(3)	0.5184(4)	0.6140(3)	0.039(2)	0.108(4)	0.062(2)	0.006(2)	0.025(2)	-0.014(2)
C(9)	4e		0.5654(3)	0.5939(3)	0.5879(3)	0.046(2)	0.081(3)	0.073(3)	0.008(2)	0.025(2)	-0.018(2)
C(10)	4e		0.6712(3)	0.5756(3)	0.5786(2)	0.047(2)	0.059(2)	0.062(2)	0.004(2)	0.024(2)	-0.004(2)
C(11)	4e		0.6681(3)	0.4103(3)	0.6204(2)	0.035(2)	0.065(2)	0.040(2)	-0.007(2)	0.016(2)	-0.005(2)
C(12)	4e		0.7216(3)	0.3139(3)	0.6370(2)	0.039(2)	0.066(2)	0.036(2)	-0.012(2)	0.018(2)	0.001(2)
C(13)	4e		1.0110(3)	0.4414(3)	0.7644(2)	0.046(2)	0.080(3)	0.042(2)	-0.010(2)	0.019(2)	0.005(2)
C(17)	4e		0.9269(2)	0.6943(2)	0.5109(2)	0.045(2)	0.045(2)	0.050(2)	0.004(2)	0.020(2)	-0.003(2)
C(21)	4e		0.8167(2)	0.4350(2)	0.3846(2)	0.037(2)	0.053(2)	0.039(2)	0.003(2)	0.011(2)	-0.003(2)
C(14)	4e		1.0419(2)	0.4311(3)	0.8668(3)	0.094(3)	0.143(5)	0.048(3)	0.006(3)	0.027(3)	0.019(3)
C(18)	4e		0.8958(2)	0.8028(3)	0.5161(3)	0.092(3)	0.052(3)	0.135(4)	0.001(2)	0.071(3)	-0.011(3)
C(22)	4e		0.7723(2)	0.4101(2)	0.2841(2)	0.059(2)	0.089(3)	0.041(2)	-0.007(2)	0.002(2)	-0.014(2)
C(15A)	4e	0.5	0.9604(4)	0.3703(7)	0.8924(4)	0.143(4)	0.160(7)	0.084(3)	0.029(4)	0.056(3)	0.043(4)
C(16A)	4e	0.5	1.1620(3)	0.4099(8)	0.9130(4)	0.143	0.160	0.084	0.029	0.056	0.043
C(15B)	4e	0.5	0.9403(3)	0.4105(8)	0.8911(4)	0.143	0.160	0.084	0.029	0.056	0.043
C(16B)	4e	0.5	1.1340(5)	0.3573(6)	0.9049(4)	0.143	0.160	0.084	0.029	0.056	0.043
C(19A)	4e	0.5	0.8118(4)	0.8207(4)	0.5599(5)	0.115(4)	0.075(3)	0.181(8)	0.003(3)	0.072(4)	-0.047(4)
C(20A)	4e	0.5	0.9932(4)	0.8718(3)	0.5415(7)	0.115	0.075	0.181	0.003	0.072	-0.047
C(19B)	4e	0.5	0.7786(3)	0.8189(4)	0.5115(7)	0.115	0.075	0.181	0.003	0.072	-0.047
C(20B)	4e	0.5	0.9823(4)	0.8614(4)	0.5850(6)	0.115	0.075	0.181	0.003	0.072	-0.047
C(23A)	4e	0.5	0.6911(6)	0.4856(4)	0.2309(3)	0.098(4)	0.124(5)	0.057(2)	0.009(3)	0.011(3)	-0.019(3)
C(24A)	4e	0.5	0.7372(6)	0.3041(3)	0.2666(3)	0.098	0.124	0.057	0.009	0.011	-0.019
C(23B)	4e	0.5	0.6479(2)	0.4086(6)	0.2463(4)	0.098	0.124	0.057	0.009	0.011	-0.019
C(24B)	4e	0.5	0.8282(5)	0.3208(5)	0.2626(4)	0.098	0.124	0.057	0.009	0.011	-0.019

Acknowledgments. The project was supported by Jiangxi Provincial Educational foundation (grant no. 2005-146) and Jiangxi University of Science and Technology foundation (grant no. 2003-1).

References

1. Lu, Y.-Q.; Lu, W.-M.; Wu, B.; Wang, L.-N.: Synthesis, characterization and crystal structure of the bis(1,10-phenanthroline) tris(*trans*-2,3-dimethylacrylato)lanthanide(III) dimer. J. Coord. Chem. **53** (2001) 15-23.
2. Lu, W.-M.; Wu, B.; Wang, L.-N.: Synthesis and characterization of bis(1,10-phenanthroline) tris(*trans*-2,3-dimethylacrylato)lanthanide(III) dinuclear Complexes. Chem. J. Chinese Univ. **22** (2001) 535-538.
3. Lu, W.-Q.; Wu, B.; Zheng, X.-M.: Terbium and holmium *trans*-2,3-dimethylacrylic acid complexes with 1,10-phenanthroline. J. Chem. Cryst. **30** (2000) 777-782.
4. Sheldrick, G. M.: Phase Annealing in SHELXL-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.