Crystal structure of {μ_2-6,6'-dimethoxy-2,2'-[propane-1,3-diylbis (nitrilomethylylidene)]diphenolato-1κ^4*N*,*N'*,*O*,*O'*:2κ^4*O*,*O'*,*O''*,*O'''*} trinitratocopper(II)neodymium(III), $[CuNd(C_{19}H_{20}N_2O_4)(NO_3)_3]$, $C_{19}H_{20}CuN_5NdO_{13}$

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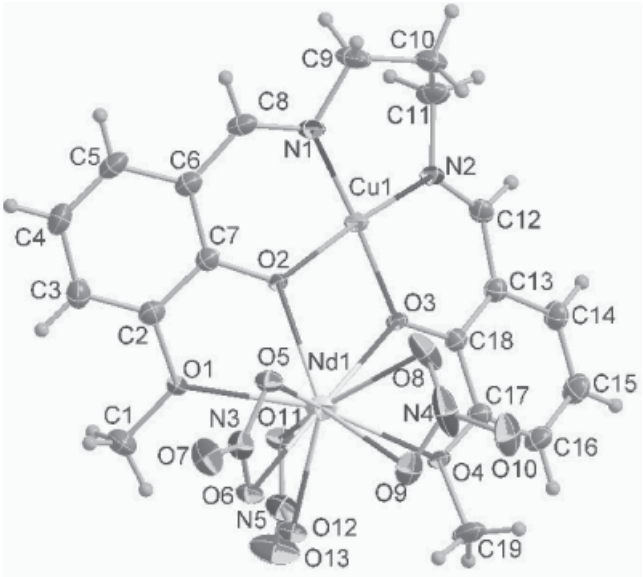

Abstract

$C_{19}H_{20}CuN_5NdO_{13}$, monoclinic, $P2_1/n$ (no. 14), a = 11.679(2) Å, b = 14.708(3) Å, c = 14.910(3) Å, β = 101.78(3)°, V = 2507.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.0284, $wR_{ref}(F^2)$ = 0.0704, T = 295 K.

Table 1. Data collection and handling.

Crystal:	brown prisms, size 0.15×0.18×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	29.72 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	23963, 5702
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4898
$N(param)_{refined}$:	364
Programs:	SHELX [3]

Source of material

The title complex was obtained by the treatment of copper(II) acetate monohydrate (0.05 g, 0.25 mmol) with the 6,6'-((propane-1,3-diylbis(azanylylidene))bis(methanylylidene))bis(2-methoxyphenol) (0.086 g, 0.25 mmol) in mixed methanol/acetone (20/5) at room temperature. Then the mixture was refluxed for 3h after the addition of neodymium (III) nitrate hexahydrate (0.160 g, 0.25 mmol). The reaction mixture was cooled and filtered; diethyl ether was allowed to diffuse slowly into the solution of the filtrate. Single crystals were obtained after several days. Analysis calcd. for $C_{19}H_{20}CuN_5O_{13}Nd$: C, 31.06; H, 2.72; N, 9.53 %; found: C, 31.13; H, 2.73; N, 9.51%.

Experimental details

One of the coordinated nitrato ligands appeared disordered.

Discussion

In the title complex, the Cu(II) ion is four-coordinated in a square-planar geometry by two O atoms and two N atoms of the deprotonated Schiff-base type ligand. The Nd(III) ion is ten-coordinated by three chelating nitrate anions and four chelating O atoms of the deprotonated Schiff-base type ligand. As shown in the figure, the octodentate ligand links Cu and Nd atoms into a dinuclear complex through two phenolate O atoms, which is similar with the bonding characteristic reported for a similar copper-lanthanum complex [1, 2]. The dihedral angle between the plane N1–O1–Cu1 and plane N2–O3–Cu1 is 12.34°. The distance of Cu1–O2 (1.934(19) Å) is almost equal to Cu1–O3 (1.936(20) Å). The Nd(III) ion is 10-coordinated exclusively to oxygen atoms (two from phenolic hydroxyl group, two from methoxy group and six from three bidentate nitrate anions) forming a bicapped square antiprismatic coordination polyhedron. The Nd–O bond lengths are in the range of 2.4141(19)-2.624(2) Å. The dihedral angle between the aromatic two benzene ring planes is 33.81°. The bonds of Nd–O (phenolate oxygen atoms) are the shortest and the Nd–O (methoxy oxygen atoms) are the longest. The Cu–Nd intermolecular distance is 3.571 Å. Notably, there is no intermolecular hydrogen bond and π-π interaction observed in the packing structure of the complex.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.0676	0.9128	0.3606	0.104
H(1B)	4*e*	0.0388	0.8132	0.3860	0.104
H(1C)	4*e*	0.0937	0.8313	0.3001	0.104
H(3A)	4*e*	0.2036	0.9741	0.2953	0.057
H(4A)	4*e*	0.3630	1.0521	0.2637	0.066
H(5A)	4*e*	0.5488	1.0134	0.3362	0.060
H(8A)	4*e*	0.6802	0.9328	0.4491	0.051
H(9A)	4*e*	0.8079	0.8800	0.5923	0.068
H(9B)	4*e*	0.8301	0.8337	0.5029	0.068
H(10A)	4*e*	0.8067	0.7441	0.6641	0.065

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Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(10B)	4*e*	0.9189	0.7458	0.6217	0.065
H(11A)	4*e*	0.7971	0.6649	0.4918	0.063
H(11B)	4*e*	0.8487	0.6084	0.5803	0.063
H(12A)	4*e*	0.7203	0.5119	0.5945	0.042
H(14A)	4*e*	0.6229	0.3892	0.6430	0.050

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(15A)	4*e*	0.4647	0.3262	0.6891	0.058
H(16A)	4*e*	0.2996	0.4132	0.6908	0.053
H(19A)	4*e*	0.1384	0.4992	0.6554	0.093
H(19B)	4*e*	0.1162	0.5988	0.6850	0.093
H(19C)	4*e*	0.2086	0.5376	0.7486	0.093

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}
Nd(1)	4*e*		0.26133(1)	0.747130(9)	0.56908(1)	0.02325(9)	0.03194(9)	0.03570(9)	–0.00114(5)	0.00962(6)	–0.00032(5)
Cu(1)	4*e*		0.55663(3)	0.72778(2)	0.54075(2)	0.0238(2)	0.0332(2)	0.0428(2)	–0.0000(1)	0.0118(1)	0.0049(1)
O(1)	4*e*		0.2073(2)	0.8422(2)	0.4174(1)	0.031(1)	0.051(1)	0.044(1)	–0.0029(9)	0.0030(9)	0.010(1)
O(2)	4*e*		0.4186(2)	0.8015(1)	0.5004(1)	0.028(1)	0.037(1)	0.049(1)	0.0027(8)	0.0129(9)	0.0148(9)
O(3)	4*e*		0.4398(2)	0.6526(1)	0.5797(1)	0.030(1)	0.0317(9)	0.046(1)	0.0001(7)	0.0156(8)	0.0054(8)
O(4)	4*e*		0.2638(2)	0.5849(1)	0.6405(2)	0.034(1)	0.042(1)	0.055(1)	–0.0010(8)	0.021(1)	0.011(1)
O(5)	4*e*		0.2559(2)	0.9131(2)	0.6070(2)	0.041(1)	0.043(1)	0.068(2)	–0.0067(9)	0.013(1)	–0.006(1)
O(6)	4*e*		0.0853(2)	0.8521(2)	0.5721(2)	0.032(1)	0.050(1)	0.070(2)	0.0023(9)	0.013(1)	–0.004(1)
O(7)	4*e*		0.1028(3)	0.9957(2)	0.6042(2)	0.093(2)	0.050(1)	0.104(2)	0.031(2)	0.015(2)	–0.008(2)
O(8)	4*e*		0.3985(3)	0.7694(2)	0.7212(2)	0.066(2)	0.124(3)	0.063(2)	0.041(2)	–0.014(2)	–0.031(2)
O(9)	4*e*		0.2179(3)	0.7554(2)	0.7275(2)	0.093(3)	0.069(2)	0.052(2)	0.023(2)	0.034(2)	0.002(1)
O(10)	4*e*	0.49	0.322(5)	0.745(3)	0.855(1)	0.13(2)	0.13(2)	0.034(3)	0.04(2)	0.014(6)	0.000(5)
O(10')	4*e*	0.51	0.369(4)	0.788(4)	0.849(2)	0.11(1)	0.14(2)	0.035(4)	0.06(1)	–0.006(6)	–0.025(8)
O(11)	4*e*		0.2046(2)	0.6377(2)	0.4368(2)	0.047(1)	0.055(1)	0.057(1)	0.001(1)	0.017(1)	–0.015(1)
O(12)	4*e*		0.0693(2)	0.6693(2)	0.5107(2)	0.032(1)	0.054(1)	0.062(1)	–0.0039(9)	0.011(1)	–0.012(1)
O(13)	4*e*		0.0442(3)	0.5618(2)	0.4093(3)	0.076(2)	0.100(2)	0.159(3)	–0.036(2)	0.018(2)	–0.078(2)
N(1)	4*e*		0.6608(2)	0.8262(2)	0.5149(2)	0.027(1)	0.038(1)	0.058(2)	–0.003(1)	0.015(1)	0.002(1)
N(2)	4*e*		0.6756(2)	0.6338(2)	0.5663(2)	0.027(1)	0.041(1)	0.043(1)	0.003(1)	0.013(1)	0.003(1)
N(3)	4*e*		0.1455(3)	0.9220(2)	0.5946(2)	0.054(2)	0.041(1)	0.049(2)	0.010(1)	0.013(1)	–0.002(1)
N(4)	4*e*		0.3216(5)	0.7639(3)	0.7689(2)	0.123(4)	0.108(3)	0.041(2)	0.082(3)	–0.002(2)	–0.011(2)
N(5)	4*e*		0.1037(3)	0.6212(2)	0.4507(2)	0.047(2)	0.047(2)	0.068(2)	–0.005(1)	0.004(2)	–0.017(1)
C(1)	4*e*		0.0923(3)	0.8506(3)	0.3614(3)	0.034(2)	0.089(3)	0.077(3)	–0.006(2)	–0.005(2)	0.029(2)
C(2)	4*e*		0.2958(3)	0.8919(2)	0.3916(2)	0.038(2)	0.038(1)	0.037(1)	–0.003(1)	0.011(1)	–0.001(1)
C(3)	4*e*		0.2788(3)	0.9595(2)	0.3262(2)	0.052(2)	0.049(2)	0.041(2)	0.003(1)	0.004(1)	0.010(1)
C(4)	4*e*		0.3743(3)	1.0055(2)	0.3066(2)	0.063(2)	0.054(2)	0.048(2)	0.000(2)	0.013(2)	0.022(2)
C(5)	4*e*		0.4854(3)	0.9825(2)	0.3504(2)	0.058(2)	0.047(2)	0.052(2)	–0.009(2)	0.027(2)	0.011(2)
C(6)	4*e*		0.5051(3)	0.9126(2)	0.4166(2)	0.040(2)	0.036(1)	0.043(2)	–0.003(1)	0.017(1)	0.001(1)
C(7)	4*e*		0.4082(2)	0.8675(2)	0.4376(2)	0.034(2)	0.032(1)	0.035(1)	–0.000(1)	0.011(1)	0.002(1)
C(8)	4*e*		0.6236(3)	0.8927(2)	0.4609(2)	0.037(2)	0.039(2)	0.056(2)	–0.007(1)	0.023(1)	0.000(1)
C(9)	4*e*		0.7894(3)	0.8272(2)	0.5531(3)	0.026(2)	0.051(2)	0.094(3)	–0.006(1)	0.012(2)	0.001(2)
C(10)	4*e*		0.8341(3)	0.7439(2)	0.6070(3)	0.027(2)	0.066(2)	0.070(2)	–0.004(1)	0.010(2)	0.004(2)
C(11)	4*e*		0.7955(3)	0.6573(2)	0.5562(3)	0.034(2)	0.048(2)	0.081(2)	0.004(1)	0.027(2)	0.006(2)
C(12)	4*e*		0.6579(2)	0.5521(2)	0.5906(2)	0.031(2)	0.037(1)	0.038(1)	0.008(1)	0.011(1)	0.001(1)
C(13)	4*e*		0.5528(2)	0.5155(2)	0.6124(2)	0.035(2)	0.033(1)	0.029(1)	0.001(1)	0.008(1)	–0.001(1)
C(14)	4*e*		0.5562(3)	0.4241(2)	0.6424(2)	0.047(2)	0.035(1)	0.043(2)	0.006(1)	0.009(1)	0.003(1)
C(15)	4*e*		0.4622(3)	0.3866(2)	0.6703(2)	0.058(2)	0.033(1)	0.055(2)	–0.000(1)	0.014(2)	0.007(1)
C(16)	4*e*		0.3628(3)	0.4386(2)	0.6707(2)	0.046(2)	0.037(1)	0.052(2)	–0.007(1)	0.018(2)	0.006(1)
C(17)	4*e*		0.3578(2)	0.5273(2)	0.6415(2)	0.032(2)	0.036(1)	0.037(1)	–0.001(1)	0.010(1)	0.001(1)
C(18)	4*e*		0.4520(2)	0.5675(2)	0.6098(2)	0.032(1)	0.030(1)	0.026(1)	–0.002(1)	0.006(1)	–0.002(1)
C(19)	4*e*		0.1743(3)	0.5524(3)	0.6862(3)	0.040(2)	0.067(2)	0.087(3)	–0.001(2)	0.035(2)	0.024(2)

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