Crystal structure of [3,3′-iminobis(*N*,*N*-dimethylpropylamine)] (flavonolato)zinc(II) perchlorate, [Zn(fla)(idpaH)]ClO_4

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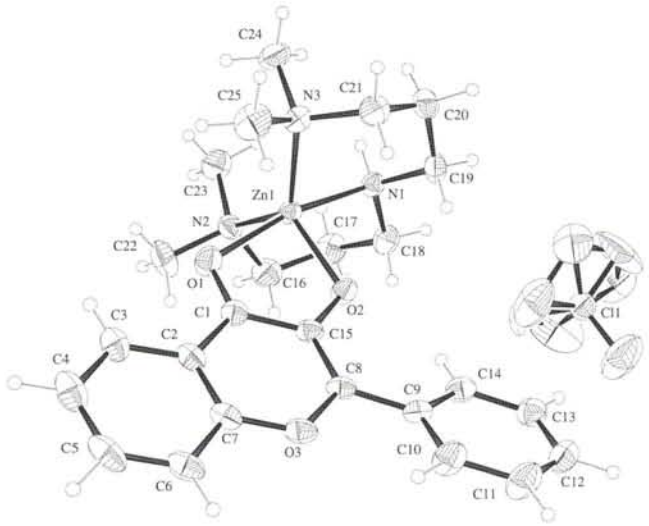

Abstract

$C_{25}H_{34}ClN_3O_7Zn$, triclinic, $P\bar{1}$ (No. 2), a = 9.1974(3) Å, b = 11.7178(5) Å, c = 12.8801(6) Å, α = 95.278(2)°, β = 90.593(2)°, γ = 101.721(2)°, V = 1352.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.048, T = 298 K.

Source of material

The compound was prepared by stirring $Zn(ClO_4)_2 \cdot 6H_2O$ with equimolar amount of 3,3′-iminobis(*N*,*N*-dimethylpropylamine) and flavonol in toluene-ethanol (1:1) at 333 K under N_2 for 3 h. Recrystallization was done from toluene-ethanol (1:1).

Experimental details

Because of some disorder, three oxygen atoms of the perchlorate anion have been splitted in two distinct sites: O4 and O8, O5 and O9, O6 and O10 with a multiplicity of 0.7 for O4, O5 and O6 and multiplicity 0.3 for O8, O9 and O10.

Discussion

In the complex the environment around the zinc(II) centre is approximately trigonal bipyramidal, with τ = 0.71 [1]. Two N atoms of the tridentate idpaH ligand, with Zn—N distances of 2.105(1) Å and 2.129 (1) Å, and the O atom of the 3-hydroxy group of the flavonolate ligand occupy basal positions. The O atom of the 4-carbonyl group of the flavonolate and the N atom of the idpaH ligand, with a Zn—N distance of 2.124(1) Å, are in apical positions. The Zn—O bond distance of the 4-carbonyl O atom is longer [Zn—O1 2.178 (1) Å] and the Zn—N1 bond distance of the tridentate idpaH ligand is shorter [Zn—N1 2.124(1) Å] then those found in the other known zinc(II) flavonolate complex [2].

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Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.3 × 0.3 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.9 cm^{-1}
Diffractometer, scan mode:	Kappa CCD, φ
$2\theta_{max}$:	52.62°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4972, 4972
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 4436
$N(param)_{refined}$:	361
Programs:	SIR92 [3], MAXUS [4], ORTEPII [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.4165	0.6973	0.4459	0.0579
H(3)	2*i*	–0.0528	0.9172	0.0796	0.0768
H(4)	2*i*	–0.2316	0.9359	–0.0431	0.0887
H(5)	2*i*	–0.3916	0.7717	–0.1220	0.0895
H(6)	2*i*	–0.3817	0.5822	–0.0734	0.0756
H(10)	2*i*	–0.3330	0.3418	0.0762	0.0801
H(11)	2*i*	–0.3746	0.1432	0.1060	0.0901
H(12)	2*i*	–0.1898	0.0684	0.1852	0.0798
H(13)	2*i*	0.0346	0.1904	0.2376	0.0723
H(14)	2*i*	0.0775	0.3901	0.2123	0.0635
H(16A)	2*i*	0.5834	0.7637	0.1608	0.0827
H(16B)	2*i*	0.4301	0.6757	0.1495	0.0827
H(17A)	2*i*	0.6111	0.7054	0.3264	0.0865
H(17B)	2*i*	0.6124	0.6045	0.2377	0.0865
H(18A)	2*i*	0.3639	0.5268	0.2782	0.0755
H(18B)	2*i*	0.4755	0.5228	0.3701	0.0755
H(19A)	2*i*	0.3018	0.5186	0.5001	0.0703
H(19B)	2*i*	0.1718	0.5152	0.4194	0.0703
H(20A)	2*i*	0.2470	0.6920	0.5802	0.0748
H(20B)	2*i*	0.1224	0.5814	0.5914	0.0748
H(21A)	2*i*	–0.0098	0.6431	0.4531	0.0736
H(21B)	2*i*	0.0034	0.7276	0.5561	0.0736
H(22A)	2*i*	0.3305	0.8362	0.1038	0.0959
H(22B)	2*i*	0.4874	0.9176	0.1239	0.0959
H(22C)	2*i*	0.3494	0.9500	0.1811	0.0959
H(23A)	2*i*	0.5450	0.8617	0.3763	0.0807
H(23B)	2*i*	0.4763	0.9653	0.3426	0.0807
H(23C)	2*i*	0.6142	0.9330	0.2854	0.0807

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(24A)	2*i*	0.3041	0.8597	0.5300	0.0792
H(24B)	2*i*	0.1684	0.9123	0.5695	0.0792
H(24C)	2*i*	0.2537	0.9579	0.4719	0.0792

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(25A)	2*i*	–0.0752	0.7932	0.3643	0.0954
H(25B)	2*i*	0.0270	0.9182	0.3727	0.0954
H(25C)	2*i*	–0.0583	0.8727	0.4703	0.0954

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}
Zn(1)	2*i*		0.22890(1)	0.7355	0.3094	0.03440(3)	0.03320(3)	0.03780(3)	0.00350(2)	–0.00590(2)	–0.00010(2)
Cl(1)	2*i*		0.35340(2)	0.21340(1)	0.37080(1)	0.05800(9)	0.05370(8)	0.05630(8)	0.01420(7)	–0.00810(6)	0.00900(6)
O(1)	2*i*		0.09660(4)	0.80300(3)	0.19770(3)	0.0551(2)	0.0418(2)	0.0625(2)	0.0048(2)	–0.0185(2)	0.0050(2)
O(2)	2*i*		0.10720(4)	0.58790(3)	0.24170(2)	0.0480(2)	0.0388(2)	0.0542(2)	0.0071(2)	–0.0235(2)	–0.0013(2)
O(3)	2*i*		–0.20210(4)	0.53020(3)	0.05450(2)	0.0402(2)	0.0584(2)	0.0416(2)	0.0113(2)	–0.0139(1)	–0.0058(2)
O(4)	2*i*	0.70	0.2736(1)	0.29700(7)	0.3444(1)	0.1203(8)	0.0709(5)	0.205(1)	0.0496(5)	–0.0370(8)	0.0061(6)
O(5)	2*i*	0.70	0.4805(1)	0.2265(2)	0.3186(1)	0.0639(6)	0.313(2)	0.140(1)	0.053(1)	0.0394(6)	0.041(1)
O(6)	2*i*	0.70	0.3727(2)	0.2071(2)	0.47280(7)	0.178(1)	0.275(2)	0.0531(6)	–0.067(1)	–0.0438(7)	0.0552(9)
O(7)	2*i*		0.25660(8)	0.11220(5)	0.32770(6)	0.1662(6)	0.0731(3)	0.1938(6)	–0.0029(4)	–0.1066(5)	0.0005(4)
O(8)	2*i*	0.30	0.3135(3)	0.2904(2)	0.4533(2)	0.182(2)	0.099(1)	0.123(2)	0.079(2)	0.077(2)	0.001(1)
O(9)	2*i*	0.30	0.4314(5)	0.2963(2)	0.3092(2)	0.277(4)	0.046(1)	0.081(1)	0.002(2)	0.039(2)	0.0320(9)
O(10)	2*i*	0.30	0.4664(2)	0.1702(2)	0.4248(2)	0.078(1)	0.071(1)	0.128(2)	0.031(1)	–0.067(1)	–0.009(1)
N(1)	2*i*		0.35060(4)	0.64250(3)	0.40020(3)	0.0358(2)	0.0346(2)	0.0431(2)	0.0034(2)	–0.0037(2)	0.0029(2)
N(2)	2*i*		0.42840(5)	0.81940(3)	0.24250(3)	0.0455(3)	0.0459(2)	0.0384(2)	0.0038(2)	0.0018(2)	0.0060(2)
N(3)	2*i*		0.12380(4)	0.79930(3)	0.44030(3)	0.0416(3)	0.0397(2)	0.0511(2)	0.0077(2)	0.0023(2)	–0.0017(2)
C(1)	2*i*		0.00480(5)	0.71980(4)	0.15090(3)	0.0411(3)	0.0450(3)	0.0398(3)	0.0085(2)	–0.0018(2)	0.0024(2)
C(2)	2*i*		–0.10520(5)	0.73730(4)	0.07640(3)	0.0403(3)	0.0564(3)	0.0353(3)	0.0165(3)	0.0016(2)	0.0086(2)
C(3)	2*i*		–0.11460(6)	0.84850(5)	0.04830(4)	0.0506(3)	0.0650(3)	0.0546(3)	0.0180(3)	0.0040(2)	0.0176(3)
C(4)	2*i*		–0.21970(7)	0.85970(6)	–0.02430(5)	0.0578(4)	0.0832(4)	0.0651(4)	0.0310(3)	0.0082(3)	0.0336(3)
C(5)	2*i*		–0.31690(7)	0.76110(7)	–0.07010(4)	0.0519(4)	0.1133(5)	0.0432(3)	0.0376(4)	0.0032(3)	0.0234(3)
C(6)	2*i*		–0.31090(6)	0.65130(5)	–0.04380(4)	0.0409(3)	0.0861(4)	0.0398(3)	0.0200(3)	–0.0059(2)	0.0024(3)
C(7)	2*i*		–0.20300(5)	0.64110(5)	0.03020(3)	0.0405(3)	0.0645(3)	0.0323(2)	0.0187(3)	0.0010(2)	0.0037(2)
C(8)	2*i*		–0.09880(5)	0.51060(4)	0.12540(3)	0.0334(3)	0.0491(3)	0.0333(2)	0.0101(2)	–0.0058(2)	–0.0048(2)
C(9)	2*i*		–0.12300(5)	0.38610(4)	0.14200(3)	0.0364(3)	0.0438(3)	0.0358(2)	0.0059(2)	0.0005(2)	–0.0069(2)
C(10)	2*i*		–0.25630(6)	0.31090(5)	0.11020(5)	0.0438(3)	0.0554(3)	0.0811(4)	0.0024(3)	–0.0168(3)	0.0016(3)
C(11)	2*i*		–0.28080(7)	0.19420(5)	0.12710(5)	0.0536(4)	0.0541(3)	0.1026(5)	–0.0091(3)	–0.0160(3)	–0.0004(3)
C(12)	2*i*		–0.17240(7)	0.14990(5)	0.17410(5)	0.0634(4)	0.0439(3)	0.0722(4)	0.0020(3)	0.0053(3)	0.0011(3)
C(13)	2*i*		–0.03960(6)	0.22230(5)	0.20490(4)	0.0543(3)	0.0510(3)	0.0515(3)	0.0160(3)	0.0008(2)	0.0006(2)
C(14)	2*i*		–0.01390(5)	0.34030(4)	0.19000(4)	0.0375(3)	0.0483(3)	0.0447(3)	0.0059(2)	–0.0011(2)	–0.0050(2)
C(15)	2*i*		0.00560(5)	0.60110(4)	0.17310(3)	0.0373(3)	0.0427(3)	0.0351(2)	0.0108(2)	–0.0070(2)	–0.0025(2)
C(16)	2*i*		0.50410(7)	0.72570(5)	0.19790(4)	0.0630(4)	0.0661(4)	0.0591(3)	0.0107(3)	0.0206(3)	0.0039(3)
C(17)	2*i*		0.55470(7)	0.65100(5)	0.27490(5)	0.0569(4)	0.0658(4)	0.0768(4)	0.0266(3)	0.0177(3)	0.0029(3)
C(18)	2*i*		0.43500(6)	0.57330(5)	0.32990(4)	0.0542(3)	0.0465(3)	0.0659(3)	0.0210(3)	0.0023(3)	0.0061(3)
C(19)	2*i*		0.24870(6)	0.56530(4)	0.46490(4)	0.0549(3)	0.0407(3)	0.0551(3)	0.0043(3)	–0.0004(2)	0.0168(2)
C(20)	2*i*		0.17230(6)	0.63500(5)	0.54390(4)	0.0621(4)	0.0534(3)	0.0488(3)	–0.0017(3)	0.0098(3)	0.0133(2)
C(21)	2*i*		0.06110(6)	0.69770(5)	0.50010(4)	0.0479(3)	0.0544(3)	0.0585(3)	0.0030(3)	0.0139(3)	0.0027(3)
C(22)	2*i*		0.40000(7)	0.88670(6)	0.15510(5)	0.0753(5)	0.0862(5)	0.0663(4)	0.0049(4)	0.0067(3)	0.0413(3)
C(23)	2*i*		0.52820(7)	0.90220(5)	0.31830(5)	0.0554(4)	0.0559(3)	0.0707(4)	–0.0119(3)	–0.0004(3)	0.0023(3)
C(24)	2*i*		0.22470(7)	0.89040(5)	0.50890(4)	0.0738(4)	0.0447(3)	0.0591(3)	0.0015(3)	0.0045(3)	–0.0103(3)
C(25)	2*i*		–0.00360(7)	0.85050(6)	0.40910(5)	0.0596(4)	0.0768(4)	0.0899(5)	0.0368(3)	0.0092(3)	0.0008(3)

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