Crystal structure of bis(2-hydroxy-3-ethoxybenzaldehyde) *O*,*O'*-(butane-1,4-diyl)dioxime, $C_{22}H_{28}N_2O_6$

Wen-Kui Dong[*,I], Sen-Hua Liu[I], Shou-Ting Zhang[I], Li Wang[I], Yu-Jie Zhang[I] and Xiong-Hou Gao[II]

[I] Lanzhou Jiaotong University, School of Chemical and Biological Engineering, Lanzhou 730070, P. R. China
[II] Lanzhou Petrochemical Research Center, PetroChina, Lanzhou 730060, P. R. China

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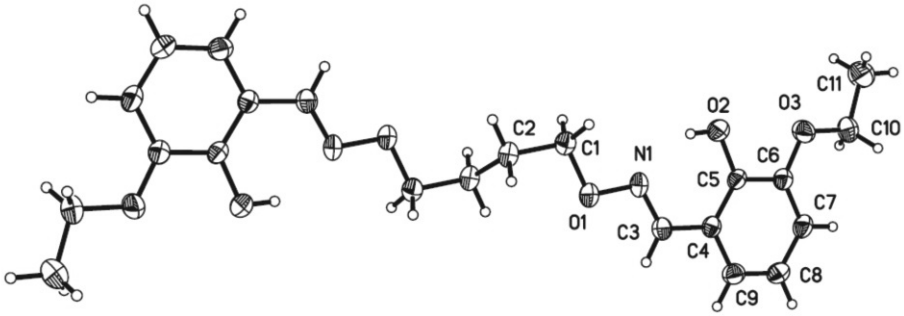

Abstract

$C_{22}H_{28}N_2O_6$, monoclinic, $P2_1/n$ (no. 14), a = 4.6719(5) Å, b = 18.092(2) Å, c = 12.408(1) Å, β = 93.914(1)°, V = 1046.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.086, T = 298 K.

Source of material

To a hot ethanol solution (10 ml) of 3-ethoxysalicylaldehyde (362.2 mg, 2.18 mmol) was added an ethanol solution (5 ml) of 1,4-bis(aminooxy)butane (122.8 mg, 1.022 mmol). After the solution had been stirred at 328 K for 8 h, the reaction mixture was filtered, washed successively with ethanol and ethanol/hexane (1:4), respectively. The isolated compound was dried under reduced pressure and purified with recrystallization from ethanol to yield 365.3 mg of colorless crystalline solid (yield 85.8 %, m.p. 413-414 K).

Experimental details

H atoms were treated as riding atoms with d(C—H) = 0.96 Å (CH_3), d(C—H) = 0.97 Å (CH_2), d(C—H) = 0.93 Å (CH), d(O—H) = 0.82 Å (OH) and with U_{iso}(H) = 1.2 U_{eq}(C)(aromatic), U_{iso}(H) = 1.2 U_{eq}(C) (methylene) and U_{iso}(H) = 1.5 U_{eq}(C) (methyl).

Discussion

Particular attention has recently been paid to the synthesis and crystal structure of dioxime compounds and their analogues [1,2]. These compounds can easily form metallosalen complexes with transition metal ions as versatile chelating ligands [3,4]. Some of them or their metal complexes are used in various organic reaction processes as catalysts, models of reaction centers of metalloenzymes, have fascinating magnetic properties, and are nonlinear optical materials. They can also be used as biological models in understanding the structure of biomolecules and biological processes [5,6]. Because the oxime-type ligands are able to resist the metathesis of C=N bonds, we can use an *O*-alkyloxime unit (-CH=N-O-) instead of -CH=N- group in order to improve the stability of the ligands. Meanwhile, the large electronegativity of oxygen atoms strongly affects the electronic properties of N_2O_2 coordination sphere, which can lead to different and new properties and structures of the resulting complexes.

The title crystal structure is only built up by the $C_{22}H_{28}N_2O_6$ molecules, in which all bond lengths are in normal ranges. The molecule of the title compound has a crystallographic inversion centre and adopts an *E* configuration with respect to the C=N bond. This is similar to what was observed in our previously reported dioxime compounds [7-10]. In the molecule, the two oxime groups are roughly coplanar with the adjacent benzene rings, forming dihedral angles of 2.13° and 2.21°, respectively. Within the molecule, the planar units are parallel but extend in opposite directions from the methylene bridge. The two benzene rings are parallel to each other and with the distance of 1.786 Å. Intramolecular O–H···N hydrogen bonds are formed between the hydroxyl groups and the oxime nitrogens. The distances between the hydroxyl groups and the oxime nitrogen atoms d(O—N) = 2.613 Å indicate strong hydrogen bonding interactions [11,12].

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.10 × 0.13 × 0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.96 cm^{-1}
Diffractometer, scan mode:	Smart 1000 CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5249, 1847
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1019
$N(param)_{refined}$:	137
Programs:	SHELXS-97, SHELXL-97, SHELXTL [13]

* Correspondence author (e-mail: dongwk@126.com)

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.3996	0.2563	0.4271	0.090
H(1A)	4*e*	0.7396	0.4144	0.4037	0.063
H(1B)	4*e*	1.0047	0.3616	0.4269	0.063
H(2A)	4*e*	1.1790	0.4799	0.4180	0.059
H(2B)	4*e*	1.2263	0.4534	0.5381	0.059
H(3)	4*e*	0.5395	0.3160	0.6832	0.057
H(7)	4*e*	–0.2704	0.0877	0.6032	0.064
H(8)	4*e*	–0.1252	0.1439	0.7639	0.069
H(9)	4*e*	0.2105	0.2356	0.7694	0.064
H(10A)	4*e*	–0.4834	0.0807	0.4273	0.076
H(10B)	4*e*	–0.2436	0.0199	0.4451	0.076
H(11A)	4*e*	–0.4264	0.0828	0.2460	0.142
H(11B)	4*e*	–0.4691	0.0000	0.2785	0.142
H(11C)	4*e*	–0.1611	0.0298	0.2602	0.142

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.5856(4)	0.32014(8)	0.5314(1)	0.046(1)	0.036(1)	0.062(1)	–0.0065(9)	0.002(1)	0.0017(8)
O(1)	4*e*	0.7795(3)	0.37791(7)	0.5548(1)	0.064(1)	0.0441(8)	0.059(1)	–0.0162(8)	0.0044(8)	0.0009(7)
O(2)	4*e*	0.2814(3)	0.22309(7)	0.4174(1)	0.070(1)	0.0590(9)	0.052(1)	–0.0169(8)	0.0157(8)	0.0009(7)
O(3)	4*e*	–0.0917(3)	0.11847(8)	0.4109(1)	0.065(1)	0.064(1)	0.058(1)	–0.0231(9)	0.0100(8)	–0.0106(8)
C(1)	4*e*	0.8939(5)	0.4012(1)	0.4565(2)	0.057(2)	0.046(1)	0.055(1)	–0.006(1)	0.007(1)	0.001(1)
C(2)	4*e*	1.0813(4)	0.46675(9)	0.4818(2)	0.045(2)	0.040(1)	0.062(1)	–0.002(1)	0.005(1)	0.004(1)
C(3)	4*e*	0.4806(5)	0.2958(1)	0.6164(2)	0.050(2)	0.040(1)	0.054(1)	0.003(1)	0.004(1)	–0.001(1)
C(4)	4*e*	0.2708(4)	0.2371(1)	0.6102(2)	0.039(1)	0.038(1)	0.048(1)	0.004(1)	0.004(1)	0.003(1)
C(5)	4*e*	0.1798(4)	0.2037(1)	0.5135(2)	0.043(2)	0.039(1)	0.045(1)	0.000(1)	0.010(1)	0.006(1)
C(6)	4*e*	–0.0232(5)	0.1471(1)	0.5099(2)	0.045(2)	0.042(1)	0.051(1)	–0.001(1)	0.006(1)	0.001(1)
C(7)	4*e*	–0.1347(5)	0.1253(1)	0.6042(2)	0.053(2)	0.046(1)	0.062(2)	–0.007(1)	0.013(1)	0.007(1)
C(8)	4*e*	–0.0471(5)	0.1589(1)	0.7004(2)	0.064(2)	0.061(1)	0.049(2)	–0.007(1)	0.014(1)	0.007(1)
C(9)	4*e*	0.1526(5)	0.2137(1)	0.7037(2)	0.058(2)	0.056(1)	0.047(1)	0.002(1)	0.008(1)	0.002(1)
C(10)	4*e*	–0.3046(5)	0.0625(1)	0.4020(2)	0.062(2)	0.053(1)	0.074(2)	–0.014(1)	0.002(1)	–0.006(1)
C(11)	4*e*	–0.3437(6)	0.0420(1)	0.2866(2)	0.118(3)	0.091(2)	0.073(2)	–0.039(2)	–0.010(2)	–0.011(2)

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