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Crystal structure of 5,5'-dimethoxy-2,2'-[1,1'-(ethylenedioxydinitrilo)diethyldiyne]diphenol, $C_{20}H_{24}N_2O_6$

Table 1: Crystal collection and handling.

Crystal:	Needle, colorless
Size:	$0.26 \times 0.23 \times 0.19$ mm
Wavelength:	Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å)
μ :	0.103 mm $^{-1}$
Diffractometer, scan mode:	Bruker APEX-II, Φ and ω -scans
$\theta_{\max}$, completeness:	25.0° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6634, 3266, 0.0627
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2482
$N(\text{param})_{\text{refined}}$:	259
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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Abstract

$C_{20}H_{24}N_2O_6$, triclinic, $P\bar{1}$, $a = 7.1389(5)$ Å, $b = 9.4977(7)$ Å, $c = 14.6456(10)$ Å, $\alpha = 105.118(3)^\circ$, $\beta = 101.188(2)^\circ$, $\gamma = 94.739(3)^\circ$, $Z = 2$, $V = 931.08(11)$ Å 3 , $R_{\text{gt}}(F) = 0.0564$, $wR_{\text{ref}}(F^2) = 0.1646$, $T = 153(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A solution of 1,2-bis(aminoxy)ethane (92.1 mg, 1.00 mmol) in ethanol solution (5 mL) was added gradually to a solution of 2-hydroxy-4-methoxy-1-acetophenone (332.34 mg, 2.00 mmol) in ethanol (15 mL), and the mixture was stirred for 24 h at 333 K. After cooling to room temperature, the mixture was filtered and the precipitate was washed successively

with absolute ethanol and a mixture of *n*-hexane and absolute ethanol in the ratio 4:1, respectively. The crude product was purified by recrystallization with ethanol and the title compound was obtained (260.07 mg, 67%). A solution of the title compound in acetone and absolute ethanol was allowed to stand at room temperature for several days, resulting in clear light colorless crystals; M.p. 413–414 K. Elemental analysis – Anal. Calcd. for $C_{20}H_{24}N_2O_6$: C, 61.85%; H, 6.23%; N, 7.21%. Found: C, 62.05%; H, 6.21%; N, 7.19%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

It is well known that Salen-like compounds have been widely investigated in modern coordination chemistry and organometallic chemistry [4–6]. They are able to form different types of metal complexes with interesting properties [7–9]. These compounds can be applied to luminescent [10] and magnetic [11] materials, catalytic activities [12] and supramolecular building units [13].

The title compound is only built up by the $C_{20}H_{24}N_2O_6$ molecules, in which all bond lengths are within the normal ranges (*cf.* the figure). The molecule adopts an E configuration with respect to each azomethine C=N bond. The imino group is coplanar with the aromatic ring. Within the molecule, the planar units aren't parallel, and extend in opposite directions

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} ^a /U _{eq}
O1	0.8863(2)	−0.1158(2)	0.77513(11)	0.0276(4)
O2	0.9624(2)	0.23875(18)	0.60326(11)	0.0231(4)
H2	0.930530	0.250192	0.547636	0.035
O3	0.7360(2)	0.19814(19)	0.33417(10)	0.0236(4)
O4	0.5344(2)	0.46846(19)	0.34970(11)	0.0275(4)
O5	0.7408(2)	0.63313(19)	0.16622(11)	0.0255(4)
H5	0.727886	0.597299	0.211728	0.038
O6	0.3785(2)	0.7898(2)	−0.08836(11)	0.0330(5)
N1	0.7830(2)	0.1708(2)	0.42553(12)	0.0198(5)
N2	0.5514(3)	0.5304(2)	0.27345(13)	0.0241(5)
C1	1.0205(4)	−0.0199(3)	0.85784(17)	0.0340(7)
H1A	1.144195	−0.000637	0.840587	0.051
H1B	1.039244	−0.066492	0.910648	0.051
H1C	0.970153	0.073248	0.878946	0.051
C2	0.8514(3)	−0.0694(3)	0.69363(16)	0.0210(5)
C3	0.9261(3)	0.0668(3)	0.68938(15)	0.0195(5)
H3	1.006282	0.135260	0.745458	0.023
C4	0.8829(3)	0.1033(3)	0.60217(15)	0.0180(5)
C5	0.7650(3)	0.0039(3)	0.51769(15)	0.0177(5)
C6	0.6914(3)	−0.1326(3)	0.52569(16)	0.0197(5)
H6	0.610902	−0.201760	0.470058	0.024
C7	0.7318(3)	−0.1700(3)	0.61134(16)	0.0222(5)
H7	0.679006	−0.263206	0.614623	0.027
C8	0.7189(3)	0.0390(3)	0.42403(15)	0.0171(5)
C9	0.6063(3)	−0.0749(3)	0.33521(15)	0.0221(5)
H9A	0.605523	−0.038592	0.278378	0.033
H9B	0.473511	−0.095557	0.341795	0.033
H9C	0.665763	−0.165444	0.327116	0.033
C10	0.8222(3)	0.3443(3)	0.34477(17)	0.0261(6)
H10A	0.817583	0.357469	0.279538	0.031
H10B	0.959479	0.357126	0.378419	0.031
C11	0.7255(3)	0.4612(3)	0.40045(16)	0.0265(6)
H11A	0.718276	0.442112	0.463063	0.032
H11B	0.805482	0.557974	0.414943	0.032
C12	0.3873(3)	0.5424(3)	0.22381(16)	0.0216(5)
C13	0.1995(3)	0.4949(3)	0.24579(18)	0.0302(6)
H13A	0.190729	0.390828	0.244345	0.045
H13B	0.092571	0.508388	0.197081	0.045
H13C	0.192469	0.554590	0.310313	0.045
C14	0.3923(3)	0.6079(3)	0.14311(15)	0.0210(5)
C15	0.5656(3)	0.6495(3)	0.11834(16)	0.0208(5)
C16	0.5653(3)	0.7108(3)	0.04174(16)	0.0233(5)
H16	0.683646	0.738811	0.026396	0.028
C17	0.3938(3)	0.7310(3)	−0.01182(16)	0.0250(6)
C18	0.2203(3)	0.6902(3)	0.01058(17)	0.0288(6)
H18	0.101778	0.703305	−0.026671	0.035
C19	0.2209(3)	0.6309(3)	0.08677(17)	0.0255(6)
H19	0.101628	0.604596	0.101796	0.031
C20	0.5523(4)	0.8415(3)	−0.11097(18)	0.0334(7)
H20A	0.626886	0.921359	−0.055630	0.050
H20B	0.520890	0.878210	−0.167961	0.050
H20C	0.628661	0.760549	−0.124846	0.050

from the dimethylene bridge. In the crystal structure, there are intramolecular hydrogen bond interactions (O2—H2···N1, O5—H5···N2) and weak non-classical intermolecular hydrogen bond interactions [15, 16].

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