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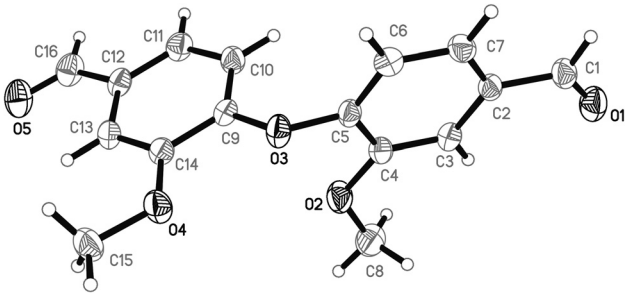
Crystal structure of 3,3′-dimethoxy-4,4′-oxy-di-benzaldehyde, $C_{16}H_{14}O_5$



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Abstract

$C_{16}H_{14}O_5$, monoclinic, $P2_1/c$ (no. 14), a = 11.1420(17) Å, b = 8.4700(12) Å, c = 15.429(2) Å, β = 104.624(4)°, V = 1408.9(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0513, w$R_{ref}(F^2)$ = 0.1243, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Quest, φ and ω
θ_{max}, completeness:	25.1°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21309, 2470, 0.074
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1566
$N(param)_{refined}$:	192
Programs:	Bruker [1], Shelx [2, 3]

1 Source of material

A 150 mL three-neck flask was charged with vanillin (0.01 mol), 4-chloro-3-methoxybenzaldehyde (0.01 mol), potassium carbonate (0.01 mol), and *N*,*N*-dimethylformamide (40 mL). This mixture was stirred at room temperature for 8 h, with the progress of the reaction monitored by thin-layer chromatography (TLC) until completion. Upon completion, the reaction was quenched by the addition of water. The organic phase was extracted three times with dichloromethane (30 mL × 3). After removing the solvent under reduced pressure *via* rotary evaporation, the crude product was purified using silica gel column chromatography, yielding 3,3′-dimethoxy-4,4′-oxy-di-benzaldehyde.

2 Experimental details

The C–H atoms were constrained to an ideal geometry, with C–H distances of 0.93 Å–0.98 Å. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}(C_{methyl})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}(C)$.

3 Comment

Diphenyl ether herbicides are the earliest commercialized class of herbicides in the family of protoporphyrinogen oxidase inhibitors, and there are still a variety of products on the market, which are widely used because of their excellent characteristics, such as high efficiency, low toxicity, and broad spectrum [4–6]. Therefore, 3,3′-dimethoxy-4,4′-oxy-di-benzaldehyde and its derivatives have potential herbicidal potential in the field of pesticides. The crystal structure of the

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

Atom	*x*	*y*	*z*	U_{iso}^*/U_{eq}
C1	1.1650 (2)	0.9047 (3)	0.52117 (19)	0.0539 (7)
H1	1.2046	0.9239	0.5809	0.065*
C2	1.05368 (19)	0.8041 (3)	0.50297 (16)	0.0430 (6)
C3	0.98582 (19)	0.7741 (3)	0.41546 (16)	0.0454 (6)
H3	1.0093	0.8200	0.3675	0.055*
C4	0.8832 (2)	0.6756 (3)	0.40005 (15)	0.0434 (6)
C5	0.84947 (19)	0.6099 (2)	0.47331 (16)	0.0405 (6)
C6	0.9162 (2)	0.6387 (3)	0.55908 (16)	0.0475 (6)
H6	0.8929	0.5926	0.6070	0.057*
C7	1.0188 (2)	0.7370 (3)	0.57429 (16)	0.0477 (6)
H7	1.0642	0.7577	0.6326	0.057*
C8	0.8557 (2)	0.6697 (4)	0.24285 (17)	0.0698 (8)
H8A	0.8023	0.6227	0.1905	0.105*
H8B	0.9383	0.6295	0.2510	0.105*
H8C	0.8562	0.7822	0.2356	0.105*
C9	0.63412 (19)	0.5543 (2)	0.42516 (14)	0.0398 (6)
C10	0.5975 (2)	0.7069 (3)	0.43386 (16)	0.0474 (6)
H10	0.6549	0.7825	0.4616	0.057*
C11	0.4736 (2)	0.7468 (3)	0.40067 (16)	0.0517 (7)
H11	0.4480	0.8498	0.4069	0.062*
C12	0.3879 (2)	0.6358 (3)	0.35856 (15)	0.0462 (6)
C13	0.4258 (2)	0.4810 (3)	0.35023 (15)	0.0451 (6)
H13	0.3684	0.4058	0.3220	0.054*
C14	0.5482 (2)	0.4393 (2)	0.38366 (15)	0.0407 (6)
C15	0.5101 (2)	0.1684 (3)	0.34668 (19)	0.0655 (8)
H15A	0.4669	0.1903	0.2858	0.098*
H15B	0.4516	0.1612	0.3827	0.098*
H15C	0.5539	0.0702	0.3493	0.098*
C16	0.2593 (2)	0.6818 (3)	0.31959 (19)	0.0628 (7)
H16	0.2384	0.7864	0.3269	0.075*
O1	1.20977 (16)	0.9648 (2)	0.46522 (13)	0.0672 (5)
O2	0.81204 (14)	0.6325 (2)	0.31810 (11)	0.0627 (5)
O3	0.75318 (13)	0.50078 (17)	0.45858 (11)	0.0494 (4)
O4	0.59560 (14)	0.29136 (17)	0.37947 (12)	0.0578 (5)
O5	0.17784 (16)	0.5966 (2)	0.27880 (13)	0.0742 (6)

compound was analyzed to provide a precise structure for predicting the binding conformation of the small molecule ligand to the target binding site from the molecular level. The crystalline structure elucidates that the two phenyl rings are not coplanar [7]. The dihedral angle between the best planes of the benzene ring (C2/C3/C4/C5/C6/C7) and benzene ring (C9/C10/C11/C12/C13/C14) is 83.79°. In the crystal, molecular cohesion is maintained through C–H···O hydrogen bond interactions.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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