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Crystal structure of (*E*)-2-(4-((3,4-difluorobenzyl)oxy)styryl)-4,6-dimethoxybenzaldehyde, C₂₄H₂₀F₂O₄

Figure 1: X-ray crystal structure of the title molecule.

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Abstract

C₂₄H₂₀F₂O₄, triclinic, $P\bar{1}$ (no. 2), $a = 8.3962(8)$ Å, $b = 11.1939(19)$ Å, $c = 11.596(2)$ Å, $\alpha = 64.877(17)^\circ$, $\beta = 85.781(11)^\circ$, $\gamma = 86.894(10)^\circ$, $V = 983.8(3)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0658$, $wR_{\text{ref}}(F^2) = 0.1750$, $T = 293(2)$ K.

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The crystal structure is shown in the Figure 1. 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

(*E*)-4-((4-(3,5-Dimethoxystyryl)phenoxy)methyl)-1,2-difluorobenzene (1.91 g, 5 mmol, prepared by a literature method [3]) was dissolved in 10 mL acetonitrile and 0.765 mL DMF. Then, 3 mL of POCl₃ was added dropwise in ice bath. After completion, the mixture was poured into ice water and

Table 1: Data collection and handling.

Crystal:	Clear light yellow block
Size:	0.25 × 0.24 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
θ_{max} , completeness:	26°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6355, 3868, 0.048
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1967
$N(\text{param})_{\text{refined}}$:	273
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	1.5387(2)	0.4013(2)	0.2470(2)	0.0999(8)
F2	1.7266(2)	0.5651(2)	0.05540(19)	0.0947(8)
O1	1.0300(2)	0.6458(2)	0.24903(19)	0.0567(6)
O2	0.1744(3)	1.0323(2)	0.1209(2)	0.0870(9)
O3	−0.2237(2)	1.0504(2)	0.30026(18)	0.0573(6)
O4	−0.0986(2)	0.7298(2)	0.70829(19)	0.0606(6)
C1	1.3228(3)	0.5544(3)	0.1888(3)	0.0567(9)
H1	1.258483	0.495459	0.255090	0.068*
C2	1.4783(4)	0.5205(3)	0.1683(3)	0.0624(9)
C3	1.5743(4)	0.6034(4)	0.0711(3)	0.0639(10)
C4	1.5189(4)	0.7253(4)	−0.0085(3)	0.0756(11)
H4	1.584826	0.782817	−0.074564	0.091*
C5	1.3612(3)	0.7625(4)	0.0110(3)	0.0672(10)
H5	1.321629	0.845281	−0.042757	0.081*
C6	1.2631(3)	0.6769(3)	0.1098(3)	0.0509(8)
C7	1.0934(3)	0.7212(3)	0.1226(3)	0.0574(9)
H7A	1.089524	0.814227	0.104678	0.069*
H7B	1.030071	0.709257	0.061869	0.069*
C8	0.8750(3)	0.6751(3)	0.2795(3)	0.0470(7)
C9	0.7814(3)	0.7768(3)	0.1989(3)	0.0520(8)
H9	0.820358	0.830648	0.116804	0.062*
C10	0.6288(3)	0.7981(3)	0.2412(3)	0.0494(8)
H10	0.565862	0.866685	0.186220	0.059*
C11	0.5669(3)	0.7204(3)	0.3627(3)	0.0462(7)
C12	0.6641(3)	0.6171(3)	0.4415(3)	0.0549(8)
H12	0.624999	0.562029	0.523185	0.066*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C13	0.8163(3)	0.5950(3)	0.4012(3)	0.0540(8)
H13	0.879513	0.526222	0.455597	0.065*
C14	0.4073(3)	0.7406(3)	0.4112(3)	0.0494(8)
H14	0.380697	0.685069	0.495916	0.059*
C15	0.2964(3)	0.8274(3)	0.3501(3)	0.0488(8)
H15	0.321210	0.881880	0.264836	0.059*
C16	0.1377(3)	0.8477(3)	0.4018(3)	0.0435(7)
C17	0.0283(3)	0.9422(3)	0.3245(3)	0.0427(7)
C18	−0.1231(3)	0.9569(3)	0.3806(3)	0.0428(7)
C19	−0.1625(3)	0.8836(3)	0.5079(3)	0.0477(8)
H19	−0.262367	0.894286	0.543087	0.057*
C20	−0.0511(3)	0.7936(3)	0.5829(3)	0.0466(7)
C21	0.0964(3)	0.7747(3)	0.5306(3)	0.0470(7)
H21	0.168681	0.712493	0.582129	0.056*
C22	0.0581(4)	1.0274(3)	0.1902(3)	0.0611(9)
H22	−0.024456	1.086906	0.151969	0.073*
C23	−0.3784(3)	1.0696(3)	0.3514(3)	0.0637(10)
H23A	−0.436812	1.137105	0.285327	0.096*
H23B	−0.366971	1.095905	0.419025	0.096*
H23C	−0.435291	0.988626	0.384072	0.096*
C24	0.0150(4)	0.6442(3)	0.7941(3)	0.0660(10)
H24A	0.045406	0.572982	0.770879	0.099*
H24B	−0.031863	0.609112	0.879582	0.099*
H24C	0.107806	0.692942	0.789196	0.099*

neutralized by aq. NaOH. The mixture was stirred overnight and filtered to afford the crude product as yellow solid. After drying, the product was purified by column chromatography on silica gel to afford the title compound, using petroleum ether/ethylacetate (3:1, v/v, R_f = 0.3) as eluent. Crystals were obtained by slow evaporation of dichloromethane/methanol within 2 days.

Experimental details

The hydrogen atoms were added geometrically and riding on the concerned atoms.

Comment

Stilbene-based compounds are widely represented in nature and have become of particular interest because of their wide range of biological activities [4]. Among them, resveratrol (*trans*-3,4',5-trihydroxystilbene), is widely present in grape skin, peanuts and many medicinal plants [5]. Resveratrol has been found to exhibit many different kinds of biological activities, such as anti-tumor, anti-inflammation, and antioxidant [6–8]. Recently, many derivatives of resveratrol had been made by total synthesis [9]. However, there are only some reports about partially synthesized derivatives starting from resveratrol.

There is one molecule in the asymmetric unit (see the figure). The C14–C15 bond length [1.314(3) Å] is consistent with the expected value for a C=C double bond. Similarly, the C22–O2 bond length [1.206(3)] is consistent with the expected value for a C=O double bond. The angles of C16–C15–C14 and C15–C14–C11 are 127.08(2)° and 128.08(2)° respectively, indicating that it has the E configuration (Figure 1). All bond lengths and angles are within normal ranges to previously reported structures [10–12].

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