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The crystal structure of 2-(4-fluorophenyl)-1,3,4-oxadiazole, $C_8H_5F_2N_2O$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	APEX2, ϕ and ω scans
$\theta_{\max}$, completeness:	25.1°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4791, 2635, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2048
$N(\text{param})_{\text{refined}}$:	217
Programs:	Bruker [1], SHELX [2, 3]

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Abstract

$C_8H_5N_2O$, monoclinic, $P2_1$ (no. 4) $a = 7.892(8)$ Å, $b = 6.088(6)$ Å, $c = 15.701(16)$ Å, $\beta = 90.08(3)^\circ$, $V = 754.4(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0423$, $wR_{\text{ref}}(F^2) = 0.1032$, $T = 273$ K.

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The asymmetric unit of the title 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 material

All chemicals, reagents and solvents are of analytical grade and are commercially available. Preparation of ethyl 4-fluorobenzoate: Thionyl chloride (17.0 g, 142.8 mmol) was dropped into a stirred solution of parafluorobenzoic acid (10 g, 71.4 mmol) in absolute ethanol (100 mL) under refluxing. The reaction mixture was monitored by TLC and then

evaporated to dryness in vacuum to get the crude product (11.6 g, 97%), which was used in the next reaction without purification. Preparation of 4-fluorobenzohydrazide: ethyl 4-fluorobenzoate (11.6 g, 69.3 mmol) in 30 mL ethanol was dropped into a stirred solution of hydrazine hydrate (28 g, 554.4 mmol) in 100 mL ethanol under refluxing. The mixture was filtrated and refined with ethanol to obtain a white solid (9.5 g, 89%). Preparation of 2-(4-fluorophenyl)-1,3,4-oxadiazole: in a 100 mL round bottom flask, 4-fluorobenzohydrazide (9.5 g, 61.7 mmol) was dissolved in triethyl orthoformate (274 g, 185.1 mmol) and the reaction solution was heated to 150 °C to reflux for 5 h. The mixture was poured into water and extracted by ethyl acetate (3 × 200 mL) to get the crude product. The crude was purified by silica gel column chromatography to afford the title compound. Yield: (5.8 g, 57%). ¹H-NMR (400 MHz, CDCl₃) δ 8.49 (s, 1H), 8.16–8.04 (m, 2H), 7.27–7.15 (m, 2H). Crystals suitable for X-ray analysis were grown in hexane/chloroform at room temperature.

Experimental details

All hydrogen atoms were placed in geometrically calculated positions. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(\text{C})$.

Comment

Oxadiazole and its derivatives are an important class of physiologically active substances widely used in medicine and agriculture as antibiotics [4], and studied in terms of micro wave synthesis [5, 6].

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.1106(4)	0.4691(7)	0.6429(3)	0.0744(10)
C2	0.1149(4)	0.5379(6)	0.7264(2)	0.0739(10)
H2	0.0689	0.6728	0.7419	0.089*
C3	0.1888(4)	0.4029(6)	0.7871(2)	0.0661(9)
H3	0.1925	0.4464	0.8439	0.079*
C4	0.2576(4)	0.2019(5)	0.7628(2)	0.0560(8)
C5	0.2508(4)	0.1385(6)	0.6775(2)	0.0693(9)
H5	0.2972	0.0047	0.6609	0.083*
C6	0.1754(4)	0.2736(7)	0.6170(3)	0.0785(11)
H6	0.1695	0.2312	0.5601	0.094*
C7	0.3388(4)	0.0599(5)	0.8258(2)	0.0566(8)
C8	0.4258(5)	−0.0442(6)	0.9472(2)	0.0783(10)
H8	0.4477	−0.0500	1.0053	0.094*
C9	0.3655(4)	0.8908(7)	0.4014(3)	0.0812(11)
C10	0.2907(4)	0.6886(7)	0.4021(2)	0.0803(11)
H10	0.2766	0.6112	0.4527	0.096*
C11	0.2361(4)	0.6019(6)	0.3247(2)	0.0663(9)
H11	0.1853	0.4642	0.3233	0.080*
C12	0.2571(3)	0.7198(6)	0.2496(2)	0.0561(8)
C13	0.3348(4)	0.9241(5)	0.2519(2)	0.0685(9)
H13	0.3497	1.0035	0.2019	0.082*
C14	0.3901(4)	1.0095(7)	0.3287(3)	0.0804(11)
H14	0.4432	1.1457	0.3308	0.096*
C15	0.1985(4)	0.6311(6)	0.1674(2)	0.0600(8)
C16	0.0946(5)	0.4029(9)	0.0806(3)	0.0905(12)
H16	0.0469	0.2758	0.0581	0.109*
F1	0.0381(3)	0.6011(4)	0.58288(15)	0.1069(8)
F2	0.4186(3)	0.9754(5)	0.47792(16)	0.1269(11)
N1	0.4158(4)	−0.1219(5)	0.81395(18)	0.0702(8)
N2	0.4732(4)	−0.1923(5)	0.8952(2)	0.0768(8)
N3	0.1987(4)	0.7232(6)	0.0931(2)	0.0812(9)
N4	0.1296(4)	0.5694(7)	0.0357(2)	0.0912(10)
O1	0.3413(3)	0.1213(4)	0.90992(14)	0.0765(7)
O2	0.1333(3)	0.4263(4)	0.16505(15)	0.0762(7)

The structure of the title compound was clarified by spectroscopy and X-ray diffraction. There are two crystallographically independent molecules in the asymmetric unit (*cf.* the figure). Both molecules are almost identical concerning their geometric parameters. All atoms in each of the two molecules are on the same plane. Molecules are packed in the crystal structure in a layered manner and stabilized by van der Waals forces.

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