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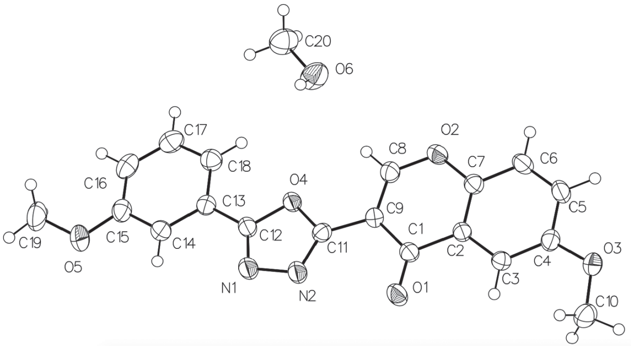
Crystal structure of 6-methoxy-3-(5-(3-methoxyphenyl)-1,3,4-oxadiazol-2-yl)-4*H*-chromen-4-one-methanol (1/1), $C_{20}H_{18}N_2O_6$



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Abstract

$C_{19}H_{14}N_2O_5 \cdot CH_3OH$, monoclinic, $P2_1/c$ (no. 14), $a = 7.9074(3)$ Å, $b = 20.4341(7)$ Å, $c = 11.0406(4)$ Å, $\beta = 92.6004(16)°$, $V = 1783.85(11)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0467$, $wR_{ref}(F^2) = 0.1260$, $T = 223(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The oxadiazole compound was obtained by hypervalent iodine catalyzed oxidative cyclization reaction of corresponding chromenonyl-benzohydrazide (**I**), which was described in the literature [6]. 6-Methoxy–3–formylchromone (100 mg, 0.5 mmol), prepared by previously reported

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.22 \times 0.12 \times 0.08$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	PHOTON 100 CMOS, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	86382, 4456, 0.080
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3129
$N(param)_{refined}$:	257
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4], PublCIF [5]

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

Atom	x	y	z	$U_{iso}*/U_{eq}$
O1	0.00874(17)	0.49997(6)	0.81048(11)	0.0448(3)
O2	0.09386(15)	0.66757(5)	0.61913(10)	0.0349(3)
O3	−0.24451(16)	0.69350(6)	1.04774(10)	0.0403(3)
O4	0.27408(13)	0.49629(5)	0.47887(9)	0.0289(3)
N1	0.2503(2)	0.39338(7)	0.53418(13)	0.0397(4)
N2	0.1644(2)	0.43265(7)	0.61833(13)	0.0383(4)
C1	0.0344(2)	0.55012(7)	0.75304(14)	0.0291(3)
C2	−0.01964(19)	0.61465(7)	0.79644(14)	0.0265(3)
C3	−0.10580(19)	0.62065(8)	0.90656(14)	0.0291(3)
H3	−0.1281	0.5835	0.9539	0.035*
C4	−0.1571(2)	0.68166(8)	0.94426(14)	0.0314(3)
C5	−0.1210(2)	0.73722(8)	0.87532(16)	0.0360(4)
H5	−0.1547	0.7787	0.9030	0.043*
C6	−0.0370(2)	0.73192(8)	0.76803(16)	0.0348(4)
H6	−0.0129	0.7693	0.7217	0.042*
C7	0.0118(2)	0.67026(8)	0.72887(14)	0.0288(3)
C8	0.1427(2)	0.60923(8)	0.57777(15)	0.0323(4)
H8	0.1970	0.6078	0.5025	0.039*
C9	0.11949(19)	0.55233(7)	0.63650(13)	0.0268(3)
C10	−0.2790(2)	0.63973(10)	1.12483(16)	0.0434(4)
H10A	−0.3506	0.6086	1.0823	0.065*
H10B	−0.3363	0.6552	1.1966	0.065*
H10C	−0.1737	0.6187	1.1482	0.065*
C11	0.18235(19)	0.49228(7)	0.58259(13)	0.0271(3)
C12	0.31094(19)	0.43227(7)	0.45487(14)	0.0284(3)
C13	0.40607(19)	0.41612(8)	0.34670(14)	0.0275(3)
C14	0.4379(2)	0.35047(8)	0.32287(14)	0.0316(3)
H14	0.4000	0.3180	0.3764	0.038*

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Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C15	0.5258(2)	0.33311(8)	0.21997(15)	0.0343(4)
C16	0.5819(2)	0.38110(9)	0.14121(15)	0.0386(4)
H16	0.6422	0.3695	0.0716	0.046*
C17	0.5484(2)	0.44623(9)	0.16571(16)	0.0400(4)
H17	0.5864	0.4786	0.1120	0.048*
C18	0.4604(2)	0.46445(8)	0.26759(15)	0.0345(4)
H18	0.4375	0.5088	0.2832	0.041*
O5	0.54987(18)	0.26761(6)	0.20448(12)	0.0499(4)
C19	0.6158(3)	0.24590(11)	0.09180(18)	0.0574(6)
H19A	0.5479	0.2637	0.0259	0.086*
H19B	0.6126	0.1985	0.0886	0.086*
H19C	0.7317	0.2607	0.0841	0.086*
C20	0.2343(3)	0.62376(11)	0.16812(19)	0.0535(5)
H20A	0.1518	0.6434	0.1136	0.080*
H20B	0.2647	0.5807	0.1384	0.080*
H20C	0.3344	0.6511	0.1722	0.080*
O6	0.1652(2)	0.61801(7)	0.28324(12)	0.0571(4)
H6A	0.0891	0.5900	0.2819	0.086*

methods [7], was treated with 3-methoxybenzohydrazide (80 mg, 0.5 mmol) in ethanol (20 mL) and the mixture was refluxed for 5 h in the presence of catalytic amounts of glacial acetic acid. After cooling, the solvent was removed under vacuum and the product 3-methoxy-N′-((6-methoxy-4-oxo-4*H*-chromen-3-yl)methylene) benzohydrazide (**I**) was used for next step. To a solution of **I**, (70 mg, 0.2 mmol) in methylene chloride (20 mL), 1.5 equivalents of diacetoxyiodobenzene (97 mg, 0.3 mmol) [8] were added portion–wise over 30 min. The mixture was stirred at room temperature for 18 h and poured into water (40 mL). After extraction with methylene chloride (25 mL × 2), the combined organic layers were dried over $MgSO_4$. Filtration and evaporation of the organic solvent gave a solid, which was recrystallized in methanol to afford crystals used in this X-ray diffraction study.

Experimental details

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

Comment

Flavonoids are well known secondary metabolites in plants and play important role in various biological processes [9, 10]. The characteristic skeletal structure of flavonoids are chromenons. Oxadiazole derivatives have attracted considerable interests due to their wide application as medicinal pharmacophore [11]. According to recent research, the 1,3,4-oxadiazole units were introduced as potential linkers to meet 'the 5-atoms role' to generate potent pharmacophores [12]. As a continuation of our research program to expand the use of novel synthetic flavones [13, 14], we designed a new flavone which has oxadiazole linker between chromenon and benzene ring.

The title compound crystallized as a methanol solvate (see the Figure). In the title molecule, the whole molecule is approximately planar and dihedral angles formed by central oxadiazole ring with substituated phenyl ring and chromenon moiety are 2.29(2)°, 4.75(1)° respectively. The methoxy group at the phenyl ring is coplanar with the ring [C3—C4—O3—C10 torsion angle = 2.8(2)°], whereas the methoxy group at the chromenonyl moiety is slightly twisted from the corresponding ring [C16—C15—O5—C19 torsion angle = −9.3(3)°]. In the crystal methanol acts as a hydrogen bond donor and an acceptor to form tetramers which are composed of two title molecules and two molecules of methanol. The hydrogen atom of methanol solvent interacts with carbonyl oxygen (O1) and one of nitrogen atom (N2) in the oxadiazole ring through a bifurcated O6—H6A—O1 and O6—H6A—N2 hydrogen bonds to form a six-membered ring. Additional weak intermolecular hydrogen bonds, C8—H8—O6, formed between solvated methanol and the title molecule generates inversion dimers of title compounds, which are intercalated by two molecules of methanol. Another nitrogen atoms (N1) in the oxadiazole ring forms intermolecular hydrogen bond interactions C5—H5—N1 which link the tetramers into chains propagating along the *b*-axis.

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