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Crystal structure of 4-(2,2-difluoroethyl)-2,4-dimethyl-6-(trifluoromethyl)isoquinoline-1,3(2*H*,4*H*)-dione, C₁₄H₁₂F₅NO₂

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.22 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.15 mm ⁻¹
Diffractometer, scan mode:	φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7263, 2410, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1846
$N(\text{param})_{\text{refined}}$:	201
Programs:	Bruker [1], SHELX [2]

Source of material

The title compound was synthesized according to the literature method [3]. Colorless single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of its anhydrous tetrahydrofuran solution at room temperature.

Experimental details

All H atoms were placed in calculated positions (C–H = 0.93 Å for aryl, 0.96 Å for methyl, 0.97 Å for methylene and 0.98 Å for methine). The U_{iso} values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for methyl groups and $1.2U_{\text{eq}}$ for the remaining H atoms.

Comment

As a class of important nitrogen containing heterocycles, isoquinoline-1,3-diones are widely found in biologically active natural products, pharmaceuticals and plant alkaloids due to their high pharmaceutical and biological activities [3–6]. In recent years, much efforts has been made to explore the incorporation of difluoromethyl group into the skeleton of isoquinoline-1,3-dione to construct CF₂H-containing isoquinoline-1,3-diones [3, 7–11]. In order to study the relationship between the structural and

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Abstract

C₁₄H₁₂F₅NO₂, monoclinic, $P2_1/c$ (no. 14), $a = 11.8292(10)$ Å, $b = 11.0987(8)$ Å, $c = 11.6480(10)$ Å, $\beta = 115.282(11)^\circ$, $V = 1382.8(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0913$, $wR_{\text{ref}}(F^2) = 0.1609$, $T = 293(2)$ 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.

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.7199 (4)	−0.0363 (4)	0.9590 (4)	0.0473 (11)
H1	0.645125	−0.075047	0.909284	0.057 [*]
C2	0.8152 (4)	−0.0997 (4)	1.0529 (4)	0.0468 (11)
C3	0.9265 (4)	−0.0426 (5)	1.1295 (4)	0.0522 (12)
H3	0.989931	−0.084825	1.193888	0.063 [*]
C4	0.9419 (4)	0.0762 (5)	1.1093 (4)	0.0507 (12)
H4	1.016480	0.114813	1.159842	0.061 [*]
C5	0.8470 (4)	0.1399 (4)	1.0138 (4)	0.0388 (10)
C6	0.7348 (4)	0.0849 (4)	0.9382 (4)	0.0394 (10)
C7	0.6287 (4)	0.1541 (4)	0.8369 (4)	0.0422 (10)
C8	0.6618 (4)	0.2836 (4)	0.8179 (4)	0.0496 (11)
C9	0.8703 (4)	0.2688 (4)	0.9972 (4)	0.0463 (11)
C10	0.8046 (5)	0.4551 (4)	0.8751 (6)	0.0720 (16)
H10A	0.763655	0.473390	0.786061	0.108 [*]
H10B	0.893186	0.465536	0.904768	0.108 [*]
H10C	0.774410	0.508287	0.920767	0.108 [*]
C11	0.5162 (4)	0.1616 (5)	0.8723 (5)	0.0598 (13)
H11A	0.489418	0.081730	0.880624	0.090 [*]
H11B	0.448499	0.203982	0.806907	0.090 [*]
H11C	0.541109	0.203776	0.951388	0.090 [*]
C12	0.7967 (5)	−0.2293 (5)	1.0733 (6)	0.0664 (15)
C13	0.5837 (4)	0.0916 (4)	0.7068 (4)	0.0482 (11)
H13A	0.540940	0.017679	0.708874	0.058 [*]
H13B	0.523669	0.143533	0.642740	0.058 [*]
C14	0.6861 (5)	0.0622 (5)	0.6685 (4)	0.0614 (14)
H14	0.740467	−0.000405	0.724089	0.074 [*]
F1	0.8981 (3)	−0.2943 (3)	1.1072 (4)	0.0901 (11)
F2	0.7691 (4)	−0.2434 (3)	1.1743 (5)	0.1122 (14)
F3	0.7088 (4)	−0.2797 (3)	0.9782 (4)	0.163 (3)
F4	0.6379 (4)	0.0256 (4)	0.5459 (3)	0.1155 (14)
F5	0.7533 (4)	0.1608 (4)	0.6720 (4)	0.1011 (13)
N1	0.7778 (3)	0.3295 (3)	0.8959 (4)	0.0470 (9)
O1	0.5855 (3)	0.3455 (3)	0.7366 (4)	0.0756 (12)
O2	0.9643 (3)	0.3203 (3)	1.0675 (3)	0.0713 (11)

biological properties of CF₂H-containing isoquinoline-1,3-diones, we report the molecular and crystal structures of the title compound.

The asymmetric unit of the title compound contains one molecule (see the Figure). The bond lengths and angles are in the normal ranges [12]. The C=O bond lengths of 1.205(5) Å (C8=O1) and 1.206(5) Å (C9=O2) illustrate typical double-bond character. The six-membered ring (C5–C6–C7–C8–N1–C9) is slightly deviated from planarity, the mean deviation from the plane of 0.0301 Å C9 has the largest deviation of 0.0424 Å. The two rings are almost coplanar. Shortest distance (2.6 Å) is larger than sum of vdW radii.

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