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Synthesis and crystal structure of methyl 2-(2-((*tert*-butoxycarbonyl)amino)phenyl)-2-(4-oxo-4*H*-chromen-3-yl)acetate, C₂₃H₂₃NO₆

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12760, 3510, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2963
$N(\text{param})_{\text{refined}}$:	279
Programs:	SHELX [1], Bruker [2]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.71916(17)	0.66626(8)	0.54853(6)	0.0256(3)
O2	0.58388(16)	0.50290(7)	0.36722(6)	0.0221(3)
O3	0.85285(15)	0.66269(7)	0.23708(6)	0.0217(3)
O4	0.57052(17)	0.66172(8)	0.17288(6)	0.0293(3)
O5	0.83680(15)	0.79430(7)	0.42312(6)	0.0210(3)
O6	0.66384(16)	0.87639(7)	0.48784(6)	0.0227(3)
N1	0.65063(19)	0.75055(9)	0.27585(7)	0.0179(3)
H1	0.734(3)	0.7555(12)	0.3139(10)	0.026(5)*
C1	0.5813(2)	0.64917(10)	0.42435(8)	0.0172(3)
C2	0.6808(2)	0.61601(11)	0.49421(8)	0.0186(4)
C3	0.7328(2)	0.51983(10)	0.49408(9)	0.0190(4)
C4	0.8356(2)	0.47897(11)	0.55594(9)	0.0225(4)
H4	0.8662(15)	0.5119(6)	0.5997(5)	0.027*
C5	0.8915(2)	0.39057(11)	0.55227(10)	0.0252(4)
H5	0.9588(15)	0.3636(7)	0.5936(8)	0.030*
C6	0.8474(2)	0.34096(11)	0.48651(10)	0.0270(4)
H6	0.8884(15)	0.2815(9)	0.4840(2)	0.032*
C7	0.7443(2)	0.37865(11)	0.42545(10)	0.0246(4)
H7	0.7135(6)	0.3451(2)	0.3819(1)	0.030*
C8	0.6864(2)	0.46806(11)	0.42995(9)	0.0203(4)
C9	0.5358(2)	0.59178(10)	0.36736(9)	0.0192(4)
H9	0.4660(2)	0.6144(9)	0.3247(3)	0.023*
C10	0.5288(2)	0.74868(10)	0.42356(8)	0.0169(3)
H10	0.4559(1)	0.7566(7)	0.4633(0)	0.020*
C11	0.4180(2)	0.78384(10)	0.35211(8)	0.0168(3)
C12	0.2487(2)	0.81868(10)	0.35582(9)	0.0198(4)
H12	0.2082(3)	0.8209(0)	0.4016(6)	0.024*
C13	0.1397(2)	0.84994(11)	0.29313(9)	0.0232(4)

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Abstract

C₂₃H₂₃NO₆, monoclinic, $P2_1/n$ (no. 14), $a = 7.5845(4)$ Å, $b = 14.7024(5)$ Å, $c = 18.1433(6)$ Å, $\beta = 98.753(4)^\circ$, $V = 1999.62(14)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0389$, $wR_{\text{ref}}(F^2) = 0.0965$, $T = 293$ 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.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H13	0.026853	0.872607	0.296762	0.028*
C14	0.1994(2)	0.84735(11)	0.22476(9)	0.0231(4)
H14	0.125814	0.867670	0.182244	0.028*
C15	0.3676(2)	0.81477(10)	0.21935(9)	0.0203(4)
H15	0.407425	0.814028	0.173337	0.024*
C16	0.4780(2)	0.78301(10)	0.28248(8)	0.0169(3)
C17	0.6812(2)	0.68863(10)	0.22313(8)	0.0191(4)
C18	0.9195(2)	0.59164(11)	0.19078(9)	0.0221(4)
C19	0.8202(3)	0.50357(12)	0.19828(10)	0.0320(4)
H19A	0.825584	0.488844	0.250127	0.048*
H19B	0.874252	0.455578	0.173631	0.048*
H19C	0.697907	0.510445	0.175849	0.048*
C20	1.1134(2)	0.58349(12)	0.22529(10)	0.0297(4)
H20A	1.171759	0.640985	0.222080	0.044*
H20B	1.170553	0.538205	0.199009	0.044*
H20C	1.120931	0.566125	0.276683	0.044*
C21	0.9039(3)	0.62367(13)	0.11039(9)	0.0324(5)
H21A	0.780515	0.633111	0.090568	0.049*
H21B	0.953218	0.578433	0.081280	0.049*
H21C	0.968047	0.679706	0.108556	0.049*
C22	0.6954(2)	0.80687(10)	0.44475(8)	0.0174(4)
C23	0.8152(3)	0.93334(12)	0.51410(10)	0.0305(4)
H23A	0.868786	0.953831	0.472420	0.046*
H23B	0.900902	0.899176	0.547457	0.046*
H23C	0.776575	0.984913	0.539903	0.046*

Source of material

The mixture of *tert*-butyl 3-(methylsulfonyloxy)-2-oxo-3-(4-oxo-3,4-dihydro-2*H*-chromen-3-yl) indoline-1-carboxylate (0.1 mmol), Et₃N (0.12 mmol) and MeOH (methanol; 3 mL) was stirred at room temperature for about 2 h. The reaction was monitored by thin-layer chromatography, which showed the disappearance of *tert*-butyl 3-(methylsulfonyloxy)-2-oxo-3-(4-oxo-3,4-dihydro-2*H*-chromen-3-yl) indoline-1-carboxylate. After MeOH was removed under reduced pressure, the residue was purified by column chromatography on silica gel column using petroleum ether/ethyl acetate (8:1, v/v) to give the pure title compound. Crystals were obtained at room temperature from the mother liquor.

Experimental details

All hydrogen atoms placed in geometrically idealized positions. The *U*_{iso} values of the hydrogen atoms of methyl groups were set to 1.5*U*_{eq}(C) and the *U*_{iso} values of all other hydrogen atoms were set to 1.2*U*_{eq}(C).

Comment

Synthesis of heterocyclic compounds has emerged as a powerful technique useful for drug discovery [3]. In particular, synthesis of heterocycles bearing oxygen atom(s) is an important synthetic target, as they are integral parts of many

medicinally interesting compounds. The chromen-4-one is an important oxygen-containing heterocycle which may possess variety of biological activities, such as antiHIV [4, 5], anticancer [6, 7] and antioxidant [8, 9].

As shown in Figure, the title compound is composed of two molecular moieties: 4*H*-chromen-4-one and methyl 2-(2-(*tert*-butoxycarbonyl)phenyl)acetate, the two moieties are linked by C10. The 4*H*-chromen-4-one moiety is essentially planar, with mean deviation from this plane of 0.0312(3) Å, and the chemical bond parameters of it are consistent with previous reports [10, 11]. The dihedral angle between the two ring moieties is 57.9°. The molecular skeleton is stabilized by an intra-molecular hydrogen bond N1–H1···O5.

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