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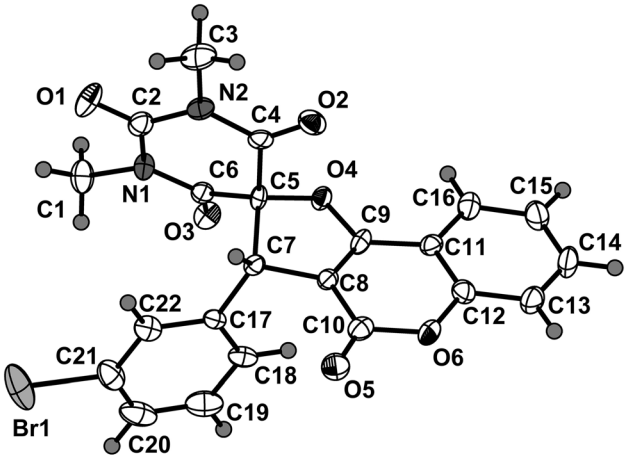
Crystal structure of 3-(3-bromophenyl)-1′,3′-dimethyl-2′*H*,3*H*,4*H*-spiro[furo[3, 2-*c*] chromene-2,5′-pyrimidine]-2′,4,4′,6′(1′*H*,3′*H*) tetraone, $C_{22}H_{15}BrN_2O_6$



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Abstract

$C_{22}H_{15}BrN_2O_6$, monoclinic, $P2_1/c$ (No. 14), a = 5.1760 (5) Å, b = 17.5652(19) Å, c = 21.734(2) Å, β = 94.690(3) °, Z = 4, V = 1969.3(3) Å^3, $R_{gt}(F)$ = 0.0569, $wR_{ref}(F^2)$ = 0.1206, T = 170 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.

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.15 × 0.07 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.13 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω-scans
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18236, 4016, 0.100
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2407
$N(param)_{refined}$:	282
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3, 4]

Source of materials

All reagents and chemicals were purchased from commercial surces and used without further purification. The starting material 5-[(3–bromophenyl)methylene]-1,3-dimethyl-2,4,6(1*H*,3*H*,5*H*)-pyrimidinetrione was synthesized following the literature procedures [5]. A dry 100 ml flask was charged with 5-[(3–bromophenyl)methylene]-1,3-dimethyl-2,4,6(1*H*,3*H*,5*H*)-pyrimidinetrione (0.9 g, 2.8 mmol), 4-hydroxycoumarin (0.55 g, 3.36 mmol), iodine (0.14 g, 0.56 mmol) and 30 mL ethanol. The mixture was stirred at 80 °C for 6.5 h. The precipitate was filtrated out and washed with ethanol for 2–3 times and purified by recrystallization from ethanol to give the target material. Crystals of the title compound were obtained by slow evaporation from a mixture of methanol and tetrahydrofuran.

Experimental details

The C-bound H atoms were geometrically placed and refined as riding with U_{ISO}(H) = 1.2–1.5Ueq(C) using the appropriate *SHELXL* commands.

Discussion

In the past years, coumarin derivatives have drawn more and more attentions of the scientists all over the world, for the reason that these compounds demonstrated promising

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.91013 (11)	0.59854 (4)	0.05991 (2)	0.0593 (2)
O4	0.5038 (5)	0.33479 (16)	0.28634 (11)	0.0213 (7)
O3	0.3133 (5)	0.37862 (17)	0.17096 (12)	0.0267 (7)
O6	0.8550 (6)	0.46066 (17)	0.42791 (12)	0.0274 (7)
O2	0.9228 (5)	0.24456 (17)	0.29274 (13)	0.0301 (7)
O5	1.1097 (6)	0.51329 (19)	0.36196 (13)	0.0331 (8)
O1	0.9740 (6)	0.2605 (2)	0.08577 (14)	0.0397 (9)
N1	0.6628 (6)	0.3301 (2)	0.12743 (14)	0.0235 (8)
N2	0.9486 (6)	0.2495 (2)	0.18909 (15)	0.0250 (8)
C9	0.6137 (7)	0.3746 (3)	0.33611 (17)	0.0213 (10)
C8	0.8085 (7)	0.4206 (2)	0.32313 (17)	0.0197 (9)
C5	0.6807 (7)	0.3431 (2)	0.23910 (17)	0.0202 (9)
C17	0.7447 (7)	0.4880 (2)	0.22013 (17)	0.0192 (9)
C10	0.9391 (8)	0.4679 (3)	0.36914 (18)	0.0246 (10)
C4	0.8580 (8)	0.2746 (2)	0.24357 (19)	0.0219 (10)
C7	0.8452 (7)	0.4176 (2)	0.25530 (16)	0.0186 (9)
H7	1.031728	0.408755	0.248666	0.022*
C2	0.8703 (8)	0.2795 (3)	0.13116 (19)	0.0255 (10)
C6	0.5277 (8)	0.3506 (2)	0.17672 (18)	0.0210 (9)
C12	0.6603 (8)	0.4112 (3)	0.44131 (18)	0.0257 (10)
C11	0.5260 (8)	0.3661 (2)	0.39639 (18)	0.0217 (9)
C16	0.3250 (8)	0.3185 (3)	0.41299 (19)	0.0273 (10)
H16	0.232650	0.287497	0.382877	0.033*
C22	0.8560 (8)	0.5089 (3)	0.16667 (18)	0.0265 (10)
H22	0.999581	0.481366	0.153503	0.032*
C18	0.5309 (8)	0.5282 (3)	0.2385 (2)	0.0272 (10)
H18	0.451822	0.513726	0.274650	0.033*
C21	0.7556 (9)	0.5701 (3)	0.1329 (2)	0.0333 (12)
C13	0.6007 (9)	0.4086 (3)	0.50256 (19)	0.0351 (12)
H13	0.695206	0.438125	0.533336	0.042*
C15	0.2637 (9)	0.3174 (3)	0.4731 (2)	0.0336 (11)
H15	0.126434	0.285854	0.484591	0.040*
C14	0.4008 (10)	0.3619 (3)	0.5173 (2)	0.0381 (12)
H14	0.355698	0.360206	0.558824	0.046*
C1	0.5597 (9)	0.3546 (3)	0.06500 (18)	0.0359 (12)
H1A	0.697071	0.351296	0.036641	0.054*
H1B	0.498530	0.407307	0.066716	0.054*
H1C	0.415283	0.321466	0.050316	0.054*
C19	0.4352 (9)	0.5897 (3)	0.2034 (2)	0.0377 (12)
H19	0.290348	0.617094	0.216046	0.045*
C20	0.5457 (9)	0.6119 (3)	0.1505 (2)	0.0405 (13)
H20	0.480472	0.654320	0.126947	0.049*
C3	1.1454 (9)	0.1890 (3)	0.1919 (2)	0.0405 (13)
H3A	1.229580	0.185786	0.233879	0.061*
H3B	1.275573	0.200544	0.162937	0.061*
H3C	1.062344	0.140199	0.180734	0.061*

activities such as anticancer [6], antiviral [7, 8], anti-inflammatory [9] and antibacterial properties [10]. Thus the synthesis and property studies of novel coumarin derivatives is an active fields in medicinal chemistry.

The title structure is shown in the figure. In the title compound, the C–N bond distances are 1.391(5)Å (N1–C2), 1.374(5)Å (N1–C6), 1.481(5)Å (N1–C1), 1.381(5)Å (N2–C4), 1.394(5)Å (N2–C2), 1.471(6)Å (N2–C3), respectively. The O–C bond distances are 1.372(5)Å (O4–C9), 1.438(4)Å (O4–C5), 1.211(5)Å (O3–C6), 1.389(5)Å (O6–C10), 1.214(5)Å (O2–C4), 1.210(5)Å (O5–C10), 1.208(5)Å (O1–C2), respectively. The bond lengths and angles are in the expected ranges [11, 12].

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