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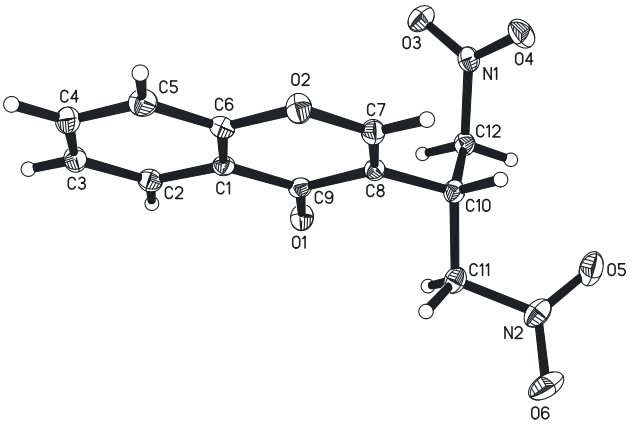
Crystal structure of 3-(1,3-dinitropropan-2-yl)-4*H*-chromen-4-one, $C_{12}H_{10}N_2O_6$



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Abstract

$C_{12}H_{10}N_2O_6$, monoclinic, $P2_1/c$ (no. 14), a = 14.6017(10) Å, b = 6.8095(4) Å, c = 13.0101(8) Å, β = 113.075(7)°, V = 1190.10(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0405, $wR_{ref}(F^2)$ = 0.0976, 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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5022, 2091, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 1740
$N(param)_{refined}$:	181
Programs:	SHELX [1], Bruker [2]

Source of material

The mixture of 4-oxo-4*H*-chromene-3-carbaldehyde (261 mg, 1.5 mmol), di-*tert*-butyl malonate (368 mg, 1.7 mmol) and 4-dimethylaminopyridine (DMAP, 12 mg, 0.1 mmol) was stirred in nitromethane (50 mL) at room temperature for 40 min. The reaction mixture was poured into saturated NaCl solution (100 mL), and a yellow precipitate appeared. The precipitate was filtered and washed with saturated NaCl solution. The yellow precipitate was recrystallized form ethyl acetate to give 3-(1,3-dinitropropan-2-yl)-4*H*-chromen-4-one.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses and placed in geometrically idealized positions. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Comment

Chromone derivatives are naturally occurring compounds spread in the plant kingdom [3], and therefore present in representative amounts in the normal human diet [4]. Some chromone derivatives possess biological activities including antiinflammatory [5], anti—HIV [6], anticancer [7], antibacterial [8], antimalarial [9], and antitumor [10], as well as treatment of Alzheimer's disease [11].

The molecular structure of title compound (*cf.* the figure) consists of a benzene ring A (C1—C6), a pyranone ring B (C1/C6—C9/O2) and a 1, 3-dinitropropyl group. Ring A and ring B constitute the chromone skeleton. The chromone skeleton is essentially planar, with mean deviation from this plane of 0.0054(3) Å. The dihedral angle between rings A and B is 0.72(2)°, similar to the value found in literature [12, 13]. In the crystal structure of title compound, the chromone skeleton is stabilized by three weak intramolecular

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.44312 (13)	0.7713 (3)	0.54597 (14)	0.0166 (4)
C2	0.51132 (13)	0.7369 (3)	0.49654 (15)	0.0202 (4)
H2	0.488382	0.703780	0.421264	0.024*
C3	0.61178 (14)	0.7517 (3)	0.55845 (16)	0.0228 (4)
H3	0.656617	0.727432	0.525213	0.027*
C4	0.64678 (14)	0.8033 (3)	0.67131 (16)	0.0248 (5)
H4	0.714913	0.813757	0.712690	0.030*
C5	0.58145 (14)	0.8387 (3)	0.72175 (15)	0.0235 (5)
H5	0.604702	0.873016	0.796866	0.028*
C6	0.48016 (14)	0.8223 (3)	0.65837 (15)	0.0187 (4)
C7	0.31896 (13)	0.8486 (3)	0.65442 (15)	0.0202 (4)
H7	0.277923	0.874945	0.692265	0.024*
C8	0.27491 (13)	0.8022 (3)	0.54564 (14)	0.0163 (4)
C9	0.33550 (13)	0.7581 (3)	0.48188 (14)	0.0175 (4)
C10	0.16258 (13)	0.7980 (3)	0.48815 (15)	0.0183 (4)
H10	0.134810	0.841982	0.541668	0.022*
C11	0.12853 (13)	0.9405 (3)	0.38950 (15)	0.0213 (4)
H11A	0.174095	1.050908	0.407157	0.026*
H11B	0.131285	0.874663	0.324655	0.026*
C12	0.12402 (14)	0.5901 (3)	0.44964 (14)	0.0198 (4)
H12A	0.052132	0.592338	0.411119	0.024*
H12B	0.152104	0.541700	0.398153	0.024*
N1	0.15299 (11)	0.4580 (2)	0.54878 (12)	0.0208 (4)
N2	0.02524 (12)	1.0153 (3)	0.36135 (13)	0.0260 (4)
O1	0.29803 (9)	0.7139 (2)	0.38197 (10)	0.0224 (3)
O2	0.41817 (9)	0.8598 (2)	0.71304 (10)	0.0229 (3)
O3	0.20439 (10)	0.3135 (2)	0.55230 (11)	0.0290 (4)
O4	0.12560 (10)	0.5022 (2)	0.62346 (11)	0.0295 (4)
O5	−0.03394 (11)	0.9161 (2)	0.38404 (13)	0.0362 (4)
O6	0.00517 (12)	1.1751 (2)	0.31429 (13)	0.0418 (4)

hydrogen bonds, with C11—H11B···O1 to be 2.502(4) Å, C12—H12B···O1 to be 2.512(5) Å and C12—H12A···O5 to be 2.493(3) Å. Additionally, hydrogen bonds C2—H2···O2 and C7—H7···O7 connect neighboring molecules to form chain structure along *c* axis. Neighboring molecules were linked together through C11—H11···O3, C11—H11B···O6 and C12—H12A···O4 to form another chain structure along *b* axis.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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