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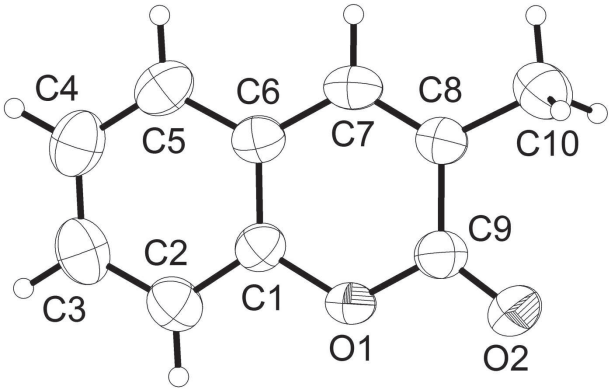
Crystal structure of 3-methyl-2*H*-chromen-2-one, $C_{10}H_8O_2$



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Abstract

$C_{10}H_8O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 6.524(3)$ Å, $b = 17.561(9)$ Å, $c = 6.989(4)$ Å, $\beta = 100.058(7)°$, $V = 788.4(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0418$, $wR_{ref}(F^2) = 0.1069$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless, blocks
	Size 0.22 × 0.18 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	51°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5958, 1469, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 856
$N(param)_{refined}$:	110
Programs:	SHELX [11]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	−0.03429(17)	0.22639(7)	0.28174(17)	0.0547(4)
O2	−0.1471(2)	0.10841(8)	0.2683(2)	0.0758(5)
C10	0.2516(3)	0.04029(11)	0.3311(3)	0.0669(6)
H10A	0.3981	0.0299	0.3469	0.100*
H10B	0.1825	0.0193	0.2101	0.100*
H10C	0.1963	0.0176	0.4362	0.100*
C8	0.2175(3)	0.12382(10)	0.3314(2)	0.0465(5)
C9	0.0031(3)	0.14962(11)	0.2932(2)	0.0512(5)
C7	0.3705(3)	0.17564(11)	0.3627(2)	0.0500(5)
H7	0.5079	0.1590	0.3881	0.060*
C6	0.3300(3)	0.25549(10)	0.3584(2)	0.0452(4)
C1	0.1255(3)	0.27903(10)	0.3142(2)	0.0451(5)
C2	0.0704(3)	0.35431(11)	0.2989(3)	0.0576(5)
H2	−0.0687	0.3685	0.2664	0.069*
C3	0.2248(4)	0.40857(12)	0.3327(3)	0.0642(6)
H3	0.1903	0.4600	0.3221	0.077*
C4	0.4304(4)	0.38692(13)	0.3820(3)	0.0668(6)
H4	0.5340	0.4238	0.4071	0.080*
C5	0.4824(3)	0.31146(12)	0.3943(2)	0.0593(5)
H5	0.6215	0.2974	0.4272	0.071*

Source of material

The synthesis of 3-methylcoumarin is carried out using salicylaldehyde and propionic anhydride for starting material, according to a modified Perkin reaction [9]. All chemicals used were commercially available of AR grade, and were used as received without further purification. The mixture of salicylaldehyde (1 eq), propionic anhydride (3 eq), and K_2CO_3

(0.25 eq) was refluxed in sealed tube until the color of the reaction mixture became brown. The mixture was poured into crushed ice, and the pH was adjusted to 7 using $NaHCO_3$. The precipitate was filtered off, washed with water, dried, and recrystallized from a mixture of hexane/ethyl acetate (4:1) to give 420.5 mg of 3-methyl-2H-chromen-2-one as colourless needles: mp 89–91°C. The colourless crystals of the title compound were obtained by slowly evaporating of an ethanol solution at room temperature. The crystalline product was filtered, washed with ethanol and dried. Analysis calculated for $C_{10}H_8O_2$: C, 74.99; H, 5.03; O, 19.98 %; found: C, 75.16; H, 5.12; O, 19.75 %.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C-H = 0.96–0.97 Å with $U_{iso}(H) = 1.5\,U_{eq}(C)$ for methyl H atoms and $1.2\,U_{eq}(C)$ for all other H atoms.

Discussion

Coumarins, considered as cinnamic acid derivatives, are a kind of aromatic secondary metabolites, which are widely existed in plants [1]. These compounds have been reported to exhibit a variety of pharmacological activities, such as anti-virus, anti-HIV, anti-neoplasm,anti-cancer, anti-coagulant agents [2–4]. The synthesis of coumarin and its derivatives have attracted increasing attention due to their extensive biological applications [5–8]. In this paper, we report the synthesis and crystal structure of 3-methyl-2H-chromen-2-one. In the molecule of the title compound (Figure), bond lengths and angles within coumarin are very similar to those given in the literature for structurally related derivatives [10]. The molecular conformation is characterized by the O2—C9—C8—C7 and O2—C9—O1—C1 torsion angles of 177.9° and 177.8°, respectively. The dihedral angle of benzene ring and pyranose ring is 1.7°.

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