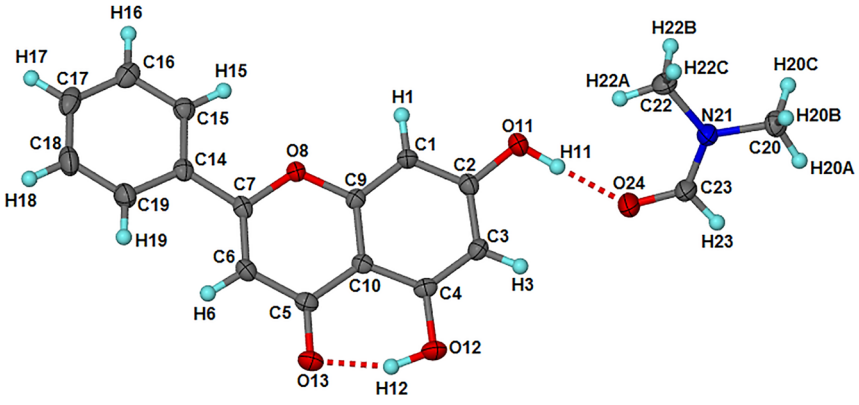
Ayesha Jacobs* and Marie-Louise Tangouna Liambo Bissa

Crystal structure of 5,7-dihydroxy-2-phenyl-4*H*-chromen-4-one–*N*,*N*-dimethylformamide(1/1), $C_{18}H_{17}NO_5$



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Abstract

$C_{18}H_{17}NO_5$, orthorhombic, $P2_12_12_1$ (no. 19), a = 4.6793(9) Å, b = 18.118(4) Å, c = 18.380(4) Å, V = 1,558.2(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0448, w$R_{ref}(F^2)$ = 0.1246, T = 173(2) 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:	Colourless needle
Size:	0.24 × 0.11 × 0.09 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker Kappa Duo Apex II,
θ_{max}, completeness:	27.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21,208, 3,605, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,887
$N(param)_{refined}$:	227
Programs:	Bruker,[1,2] X-Seed,[3] SHELX[4,5]

1 Source of material

5,7-Dihydroxy-2-phenyl-4*H*-chromen-4-one was purchased from Sigma Aldrich and *N*,*N*-dimethylformamide (DMF) was obtained from Merck. 5,7-Dihydroxy-2-phenyl-4*H*-chromen-4-one (0.118 mmol, 0.03 g) was dissolved in 2.0 mL of DMF with heating. The solution was allowed to cool to room temperature. Needle crystals were obtained after 3 weeks of slow evaporation.

2 Experimental details

The hydroxyl hydrogen atoms were located in the difference electron density map and freely refined with isotropic temperature factors. The C–H atoms were geometrically constrained at 0.95 Å (for aromatic and 0.98 Å for methyl H atoms with U_{iso} (H) = 1.2 for aromatic and U_{iso} (H) = 1.5 U_{eq} (C) for methyl H atoms).

3 Comment

5,7-Dihydroxy-2-phenyl-4*H*-chromen-4-one, more commonly known as chrysin is a flavanoid found in plants, honey and

***Corresponding author: Ayesha Jacobs**, Chemistry Department, Faculty of Applied Sciences, Cape Peninsula University of Technology, PO Box 1906, Bellville 7535, South Africa, E-mail: jacobsa@cput.ac.za. https://orcid.org/0000-0002-5508-3291
Marie-Louise Tangouna Liambo Bissa, Chemistry Department, Faculty of Applied Sciences, Cape Peninsula University of Technology, PO Box 1906, Bellville 7535, South Africa

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1975 (6)	0.64458 (15)	0.63362 (14)	0.0259 (6)
H1	−0.118021	0.692736	0.630527	0.031*
C2	−0.4050 (6)	0.62778 (14)	0.68516 (14)	0.0253 (6)
C3	−0.5242 (6)	0.55714 (15)	0.69102 (14)	0.0261 (6)
H3	−0.664881	0.546774	0.726852	0.031*
C4	−0.4332 (6)	0.50291 (14)	0.64375 (14)	0.0247 (6)
C5	−0.1295 (6)	0.46294 (14)	0.53830 (14)	0.0255 (6)
C6	0.0764 (6)	0.48697 (15)	0.48587 (14)	0.0271 (6)
H6	0.142710	0.453092	0.450207	0.032*
C7	0.1795 (6)	0.55703 (15)	0.48586 (14)	0.0239 (6)
O8	0.0938 (4)	0.60773 (10)	0.53615 (9)	0.0248 (4)
C9	−0.1103 (6)	0.58916 (14)	0.58707 (13)	0.0214 (5)
C10	−0.2232 (6)	0.51760 (15)	0.59008 (14)	0.0228 (5)
O11	−0.4904 (5)	0.68329 (11)	0.72941 (11)	0.0350 (5)
H11	−0.618 (9)	0.668 (2)	0.766 (2)	0.053 (11)*
O12	−0.5477 (5)	0.43424 (11)	0.64819 (11)	0.0345 (5)
H12	−0.465 (10)	0.410 (2)	0.610 (2)	0.059 (12)*
O13	−0.2280 (5)	0.39774 (10)	0.53903 (11)	0.0341 (5)
C14	0.3910 (6)	0.58683 (15)	0.43465 (14)	0.0251 (6)
C15	0.5190 (6)	0.65539 (15)	0.44609 (13)	0.0277 (6)
H15	0.466194	0.684041	0.487239	0.033*
C16	0.7226 (6)	0.68217 (18)	0.39799 (16)	0.0337 (7)
H16	0.810140	0.728618	0.406844	0.040*
C17	0.7988 (7)	0.64177 (19)	0.33733 (16)	0.0374 (7)
H17	0.939299	0.660114	0.304707	0.045*
C18	0.6687 (7)	0.57413 (19)	0.32429 (17)	0.0408 (8)
H18	0.718657	0.546591	0.282179	0.049*
C19	0.4663 (7)	0.54646 (17)	0.37234 (14)	0.0345 (7)
H19	0.378661	0.500121	0.363029	0.041*
C20	−1.3104 (7)	0.76011 (18)	0.92577 (16)	0.0346 (7)
H20A	−1.342695	0.722309	0.963015	0.052*
H20B	−1.490483	0.770621	0.900585	0.052*
H20C	−1.239535	0.805292	0.948869	0.052*
N21	−1.1004 (5)	0.73353 (13)	0.87355 (12)	0.0285 (5)
C22	−1.0210 (8)	0.78346 (16)	0.81458 (16)	0.0344 (7)
H22A	−0.879684	0.759399	0.783175	0.052*
H22C	−0.938909	0.828695	0.835018	0.052*
H22B	−1.191106	0.795797	0.785956	0.052*
C23	−1.0009 (7)	0.66530 (16)	0.87747 (15)	0.0354 (7)
H23	−1.073683	0.634978	0.915336	0.042*
O24	−0.8212 (6)	0.63729 (12)	0.83624 (13)	0.0484 (7)

propolis.[6] The anti-oxidant, anti-inflammatory and other biological activities of chrysin and its derivatives have been reported.[7] The Cambridge Structural Database[8] contains eleven structures involving chrysin[9–14] with only one solvate.[15] The solubility of chrysin in several solvents including *N*,*N*-dimethylformamide has been studied.[16]

The asymmetric unit contains one chrysin molecule and one *N*,*N*-dimethylformamide molecule both in general positions. The phenyl ring of chrysin is out of plane with the fused ring system and the torsion angle τ(C15–C14–C7–O8) is 10.1(3)°.

The structure is characterised by intramolecular O12–H12···O13 (H12···O13: 1.73(4) Å; O12···O13: 2.589(3) Å; O12–H12···O13 = 157(4)°) and intermolecular O11–H11···O24 (H11···O24: 1.70(4) Å; O11···O24: 2.636(3) Å; O11–H11···O24 = 175(4)°) hydrogen bonds. Furthermore, there are a few C–H···O contacts between chrysin and DMF. These include C19···O24: 3.462(4) Å, C20···O13: 3.542(4) Å, C22···O11: 3.451(4) Å and C23···O13: 3.425(4) Å. There are also π···π interactions between chrysin molecules with the shortest distance between the ring centroid of the phenyl ring (C14, C15, C16, C17, C18, C19) and that of the pyrone ring (C5, C6, C7, O8, C9, C10) of 3.6056(8) Å. The *N*,*N*-dimethylformamide molecules occupy channels parallel to [100].

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