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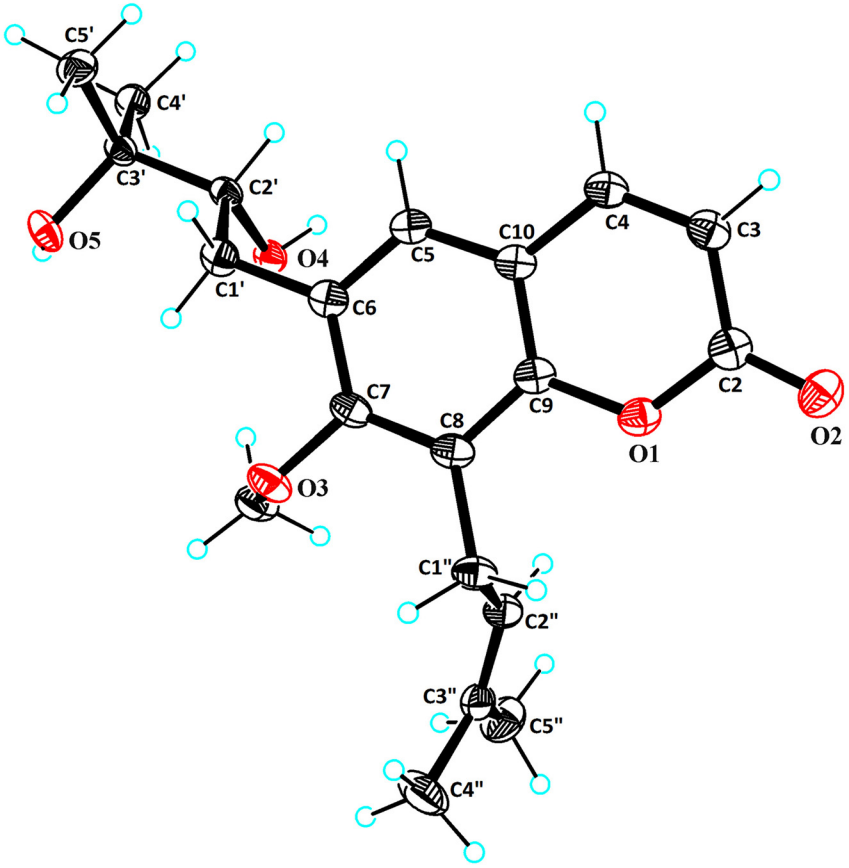
6-(2′,3′-Dihydroxy-3′-methylbutyl)-7-methoxy-8-(3″-methylbut-2″-en-1″-yl)-2*H*-chromen-2-one, $C_{20}H_{26}O_5$



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Abstract

$C_{20}H_{26}O_5$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.2825(10) Å, b = 13.0652(2) Å, c = 26.4176(4) Å, V = 1823.26(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0338, $wR_{ref}(F^2)$ = 0.0821, T = 150 K. CCDC No.: 2377464.

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

The fruits of *Rosa roxburghii* Tratt were extracted with 95 % ethanol under reflux. The crude extract (914.5 g) was loaded onto a silica gel column and eluted with a solvent system of dichloromethane/methanol (1:0 to 1:1, v/v) to afford eight fractions (Fr. A–H). The colourless block crystals were isolated from

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size	0.10 × 0.09 × 0.07 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.73 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12 (RINC), ω
θ_{max}, completeness:	73.9°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17,041, 3,653, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,364
$N(param)_{refined}$:	233
Programs:	Olex2,[1] SHELX[2,3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1′	0.2310 (4)	0.27497 (15)	0.36932 (7)	0.0361 (4)
H1′A	0.129627	0.234968	0.344785	0.043*
H1′B	0.113050	0.318512	0.388927	0.043*
C1″	0.5611 (4)	0.62884 (15)	0.32281 (7)	0.0390 (5)
H1″A	0.625850	0.660446	0.291301	0.047*
H1″B	0.382839	0.650401	0.327229	0.047*
C2′	0.3644 (3)	0.20121 (14)	0.40547 (7)	0.0280 (4)
H2′A	0.482511	0.157884	0.385065	0.034*
C2	1.1047 (4)	0.49086 (16)	0.23038 (7)	0.0382 (5)
C2″	0.7152 (4)	0.66586 (14)	0.36701 (8)	0.0370 (4)
H2″	0.866809	0.629289	0.373890	0.044*
C3′	0.1810 (3)	0.12958 (14)	0.43342 (7)	0.0301 (4)
C3″	0.6636 (4)	0.74378 (16)	0.39750 (7)	0.0384 (4)
C3	1.1042 (4)	0.38152 (16)	0.22168 (7)	0.0394 (5)
H3	1.221991	0.353033	0.198446	0.047*
C4	0.9412 (4)	0.31937 (15)	0.24569 (7)	0.0361 (5)
H4	0.947316	0.247732	0.239649	0.043*
C4′	0.3214 (4)	0.06704 (15)	0.47310 (8)	0.0363 (4)
H4′A	0.205009	0.017505	0.488420	0.054*
H4′B	0.387279	0.112804	0.499375	0.054*
H4′C	0.462208	0.030542	0.457065	0.054*
C4″	0.4483 (6)	0.8158 (2)	0.38997 (12)	0.0685 (8)
H4″A	0.332936	0.788024	0.364376	0.103*
H4″B	0.513166	0.882187	0.378596	0.103*
H4″C	0.357217	0.824518	0.421999	0.103*
C5	0.5869 (4)	0.29991 (15)	0.30770 (7)	0.0326 (4)
H5	0.590302	0.227703	0.303537	0.039*
C5′	0.0477 (4)	0.05945 (16)	0.39589 (8)	0.0419 (5)
H5′A	−0.064601	0.100107	0.374272	0.063*
H5′B	−0.052000	0.008435	0.414371	0.063*
H5′C	0.174085	0.024683	0.374856	0.063*
C5″	0.8246 (6)	0.7657 (2)	0.44297 (9)	0.0576 (7)
H5″A	0.973951	0.721209	0.442509	0.086*
H5″B	0.726621	0.752800	0.473809	0.086*
H5″C	0.878578	0.837468	0.442296	0.086*
C6	0.4125 (4)	0.34266 (15)	0.34071 (7)	0.0336 (4)
C7	0.4091 (4)	0.44986 (15)	0.34505 (7)	0.0346 (4)
C8	0.5728 (4)	0.51340 (14)	0.31806 (7)	0.0344 (4)
C9	0.7493 (4)	0.46614 (14)	0.28684 (7)	0.0331 (4)
C10	0.7577 (4)	0.35997 (14)	0.28039 (6)	0.0320 (4)
C11	0.2921 (5)	0.50260 (17)	0.42739 (8)	0.0453 (5)
H11A	0.149495	0.531230	0.446417	0.068*
H11B	0.440317	0.547013	0.431351	0.068*
H11C	0.331613	0.434126	0.440312	0.068*
O1′	0.5132 (2)	0.25973 (10)	0.44047 (5)	0.0309 (3)
H1′	0.661104	0.236204	0.441405	0.046*
O1	0.9186 (3)	0.52903 (10)	0.26183 (5)	0.0378 (3)
O2′	−0.0158 (3)	0.18863 (12)	0.45742 (5)	0.0375 (3)
H2′	0.020309	0.197435	0.488078	0.056*
O2	1.2502 (3)	0.55211 (12)	0.21228 (5)	0.0474 (4)
O3	0.2264 (3)	0.49593 (11)	0.37495 (5)	0.0439 (4)

fraction B and recrystallized with ethyl acetate/methanol (1:5, v/v). The title compound (23 mg) was obtained after 3 days.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with *d* (C–H) = 0.95–0.99 Å, U_{iso} (H) = 1.5 times U_{eq} (C) and 1.2 times U_{eq} (O).

3 Comment

Recent studies have shown that a series of coumarins with various structures have been found in *R. roxburghii* fruit, and exhibit multifarious biological activities, including antioxidant,[4] antibacterial,[5] anti-inflammatory,[6] anticancer,[7] neuroprotective[8] effects. The title compound contains two hydroxyl groups, one double bond, a methoxy and four methyl groups. The hydroxyl was confirmed by the distances $d(C_{2'}–O_4)$ = 1.434(2) Å and $d(C_{3'}–O_5)$ = 1.442(2) Å, the olefinic bond was identified by the distance $d(C_{2''}–C_{3''})$ = 1.326(3) Å, the methoxy was confirmed by the distances $d(C_7–O_3)$ = 1.385(2) Å, respectively. And the structural characteristics of the title compound are similar to those of 7-methoxy-8-(3-methyl-2-butenyl)coumarin[9] and Buntansin C.[10] and related compounds.[11]

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

References

1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.
2. Sheldrick, G. M. *SHELXTL* – Integrated Space-Group and Crystal-Structure Determination. *Acta Crystallogr.* **2015**, *A71*, 3–8.
3. Sheldrick, G. M. Crystal Structure Refinement with SHELXL. *Acta Crystallogr.* **2015**, *C71*, 3–8.
4. Wu, C. R.; Huang, M. Y.; Lin, Y. T.; Ju, H. J.; Ching, H. Antioxidant Properties of Cortex Fraxini and its Simple Coumarins. *Food Chem.* **2007**, *104*, 1464–1471.
5. Yang, L.; Ding, W.; Xu, Y. Q.; Wu, D. S.; Li, S. L.; Chen, J. N.; Guo, B. New Insights into the Antibacterial Activity of Hydroxycoumarins against Ralstonia Solanacearum. *Molecules* **2016**, *21*, 468.
6. Cho, J. Y.; Hwang, T. L.; Chang, T. H.; Lim, Y. P.; Sung, P. J.; Lee, T. H.; Chen, J. J. New Coumarins and Anti-inflammatory Constituents from Zanthoxylum Avicennae. *Food Chem.* **2012**, *135*, 17–23.
7. Jantamat, P.; Weerapreeyakul, N.; Puthongking, P. Cytotoxicity and Apoptosis Induction of Coumarins and Carbazole Alkaloids from Clausena Harmandiana. *Molecules* **2019**, *24*, 3385.
8. Tran, N. K. S.; Trinh, T. A.; Pyo, J.; Kim, C. G.; Park, J. G.; Kang, K. S. Neuroprotective Potential of Pyranocoumarins from Angelica gigas Nakai on Glutamate-Induced Hippocampal Cell Death. *Antioxidants* **2023**, *12*, 1651.
9. Borowiak, T.; Wolska, I. Structure of 7-Methoxy-8-(3-Methyl-2- Butenyl) Coumarin. *Acta Crystallogr.* **1989**, *45*, 620–622.
10. Wu, T. S.; Huang, S. C.; Lai, J. S. Stem Bark Coumarins of Citrus Grandis. *Phytochemistry* **1994**, *36*, 217–219.
11. Gridunova, G. V.; Yufit, D. S.; Struchkov, Yu. T.; Reznichenko, A. I.; Khrolova, O. R.; Komlev, I. V. *Kristallografiya* **1992**, *37*, 359.