IUCrData

ISSN 2414-3146

2-Oxo-2*H*-chromen-7-yl 2,2-dimethylpropionate

Hypolite Bazié,[a] Eric Ziki,[b] Sorgho Brahima,[a] Abdoulaye Djandé[a] and Rita Kakou-Yao[b]*

[a]Laboratory of Molecular Chemistry and Materials (LC2M), University Joseph KI-ZERBO, 03 BP 7021 Ouagadougou 03, Burkina Faso, and [b]Laboratory of Matter, Environmental and Solar Energy Sciences, Research Team: Crystallography and Molecular Physics, University Félix Houphouët-Boigny, 08 BP 582 Abidjan 08, Ivory Coast. *Correspondence e-mail: hypobazie93@gmail.com

Received 28 August 2025
Accepted 29 October 2025

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

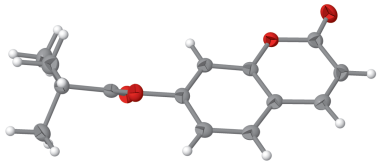
Keywords: crystal structure; coumarin; Hirshfeld surface analysis.

CCDC reference: 2498840

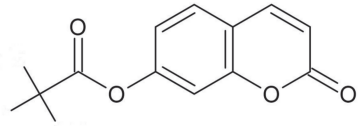
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{14}O_4$, the dihedral angle between the 2*H*-chromen-2-one moiety and the C—CO_2 ester grouping is 54.30 (5)°. In the crystal, the molecules are linked by C—H···O hydrogen bonds forming *C*(6) [100] chains. The contributions to the Hirshfeld surface for the H···H, H···O/ O···H, H···C/ C···H and C···C contacts are 49.5, 29.91, 8.6 and 7.7%, respectively.

3D view



Chemical scheme



Structure description

Herein we describe the synthesis, crystal structure and Hirshfeld surface analysis of the title coumarin derivative, $C_{14}H_{14}O_4$. As reported by several authors, coumarin-derived compounds exhibit various biological activities, such as anticancer (Yadav *et al.*, 2024; Rawat *et al.*, 2022), anti-inflammatory (Todeschini *et al.*, 1998) and anti-glaucoma (Ziki *et al.*, 2023) properties.

As expected, the fused ring system formed by atoms C1–C9/O1/O2 is almost planar with an r.m.s deviation of 0.009 Å and the dihedral angle between this ring system and the plane formed by atoms C11/C11/O3/O4 in the ester grouping is 54.30 (5)° (Fig. 1).

In the crystal, molecules are linked by weak C5—H5···O4($x+1, y, z$) hydrogen bonds (Table 1), thereby generating [100] C(6) chains (Fig. 2). The Hirshfeld surface and two-dimensional fingerprint plot of the title compound generated by *CrystalExplorer21.5* (Spackman *et al.*, 2021) confirmed the above interaction: the C5—H5···O4 bond is indicated by the red spots on Fig. 3*a*. The fingerprint plots show that the most important contributions to the surface are H···H and H···O/O···H contacts with 49.5 and 29.1%, respectively (Fig. 3*c* and 3*e*). The H···C/C···H and C···C contacts contribute 8.6 and 7.7%, respectively. These values are close to those of 2-oxo-2*H*-chromen-7-yl *tert*-butylacetate (Bazié *et al.*, 2025).

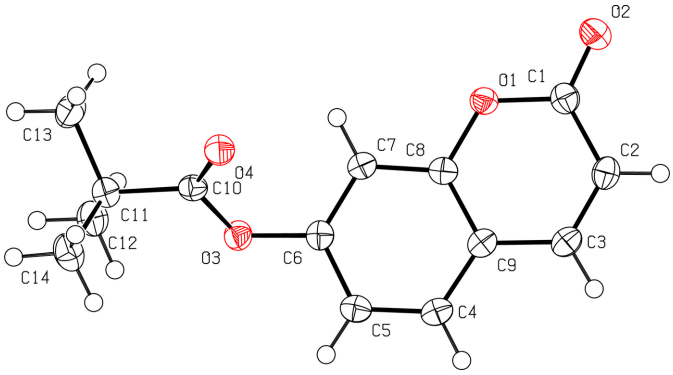


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

In a 100 ml round-bottom flask equipped with a condenser, pivaloyl chloride (0.76 ml, 6.17 mmol, 1 equiv.) was dissolved in 16 ml of dried diethyl ether and then dried pyridine (2.31 ml, 4.7 equiv.) and 7-hydroxycoumarin (1 g, 6.17 mmol, 1 equiv.) were added by small portions over 30 min, with vigorous stirring. The reaction mixture was left stirring at room temperature for 3 h.

The resulting mixture was next poured in a separating funnel containing 40 ml of chloroform and washed with 5% hydrochloric acid until the pH was 2–3. The organic phase was extracted, washed with water to neutrality, dried with magnesium sulfate and the solvent removed *in vacuo* until a cloudy solution was obtained. The occurred precipitate while cooling in an ice bath was filtered off with suction, washed with petroleum ether and recrystallized from a chloroform/*n*-hexane solvent mixture (1:3) giving the title compound as a

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O4^{i}	0.93	2.49	3.343 (4)	153

Symmetry code: (i) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{14}O_4$
M_r	246.25
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	6.242 (7), 7.191 (8), 13.652 (16)
α, β, γ (°)	99.05 (6), 92.85 (5), 91.99 (3)
V (Å^3)	603.9 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.41 × 0.12 × 0.04
Data collection	
Diffractometer	Bruker D8 Venture
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	43182, 3718, 2693
R_{int}	0.056
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.719
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.160, 1.09
No. of reflections	3718
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.27

Computer programs: *APEX4* and *SAINT* (Bruker, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

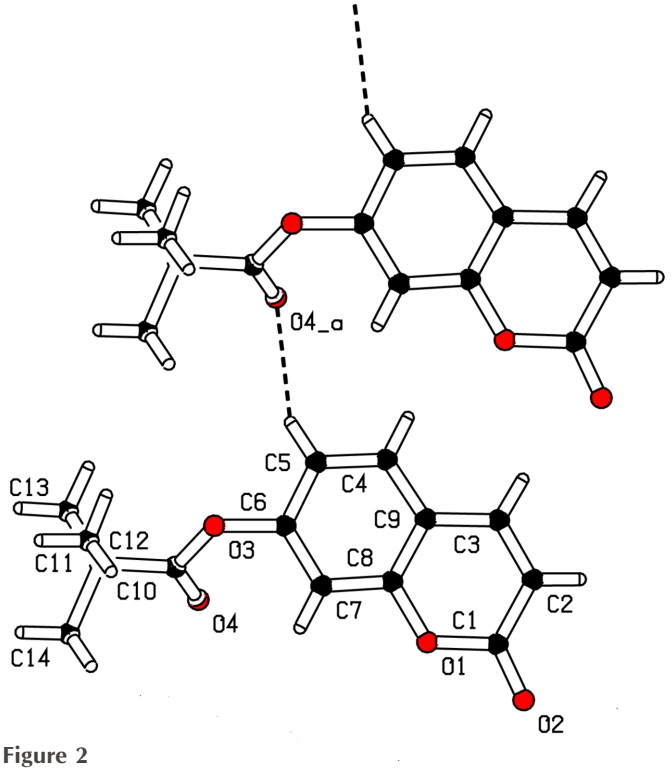


Figure 2
Part of the extended structure of the title compound showing the formation of [100] hydrogen bonded chains. Symmetry code: (*a*) $x+1, y, z$.

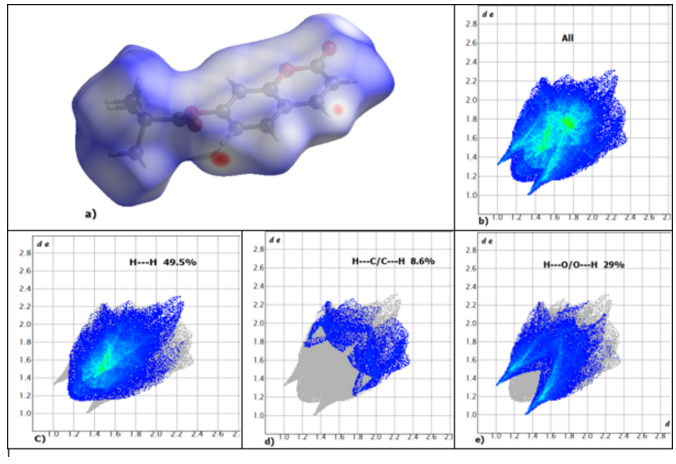


Figure 3
(*a*) Hirshfeld surface of the title compound mapped over d_{norm}, (*b*) the overall two-dimensional fingerprint plots and (*c*)–(*e*) delineated into contributions from different contacts: H···H, H···C/C···H and H···O/O···H.

white powder (0.96 g, yield 63%). Colourless prisms suitable for single-crystal X-ray diffraction analysis were then formed from an acetone solution, after the solvent was left to evaporate slowly at room temperature, m.p. 403–405 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the PMD2X X-ray diffraction facility (https://crm2.univ-lorraine.fr/lab/fr/services/pmd2x) of the Université de Lorraine, for X-ray diffraction measurements and the AFRAMED project. CCDC is also thanked for providing access to the Cambridge Structural Database through the FAIRE program. The authors thank UNESCO, CNRS and the IUCr for their support of the AFRAMED project.

References

Bazié, H., Ziki, E., Brahima, S., Ratsimbazafy, V., Roge, P., Wenger, E., Djandé, A. & Lecomte, C. (2025). *IUCrData* **10**, x250189.

Bruker (2019). *APEX4* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Rawat, A. A. & Reddy, V. B. (2022). *Eur J. Med. Chem. Rep.* **5**, 100038.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Todeschini, A. R., de Miranda, A. L. P., da Silva, K. C. M., Parrini, S. C. & Barreiro, E. J. (1998). *Eur. J. Med. Chem.* **33**, 189–199.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Yadav, A. K., Maharjan Shrestha, R. & Yadav, P. N. (2024). *Eur. J. Med. Chem.* **267**, 116179.

Ziki, E., Akonan, L., Kouman, K. C., Dali, D., Megnassan, E., Kakou-Yao, R., Tenon, A. J., Frecer, V. & Miertus, S. J. (2023). *J. Pharm. Res. Int.* **35**, 10–33.

full crystallographic data

IUCrData (2025). **10**, x250949 [https://doi.org/10.1107/S2414314625009496]

2-Oxo-2*H*-chromen-7-yl 2,2-dimethylpropionate

Hypolite Bazié, Eric Ziki, Sorgho Brahima, Abdoulaye Djandé and Rita Kakou-Yao

2-Oxo-2*H*-chromen-7-yl 2,2-dimethylpropionate

Crystal data

$C_{14}H_{14}O_4$
$M_r = 246.25$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.242$ (7) Å
$b = 7.191$ (8) Å
$c = 13.652$ (16) Å
$\alpha = 99.05$ (6)°
$\beta = 92.85$ (5)°
$\gamma = 91.99$ (3)°
$V = 603.9$ (12) Å^3
$Z = 2$
$F(000) = 260$
$D_x = 1.354$ Mg m^{-3}
Melting point: 403 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3418 reflections
$\theta = 2.9$–$30.7°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Prism, colourless
0.41 × 0.12 × 0.04 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: fine-focus sealed tube
Mirror monochromator
φ and ω scans
43182 measured reflections
3718 independent reflections
2693 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 30.7°$, $\theta_{min} = 2.9°$
$h = -8\rightarrow8$
$k = -10\rightarrow10$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.160$
$S = 1.09$
3718 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.0957P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.033 (8)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.13581 (14)	0.84613 (12)	0.39385 (7)	0.0237 (2)
O4	0.06432 (15)	0.57567 (13)	0.69115 (7)	0.0269 (2)
O3	0.36060 (14)	0.76954 (13)	0.72407 (7)	0.0254 (2)
O2	0.01958 (17)	0.88911 (16)	0.24380 (8)	0.0345 (3)
C8	0.2943 (2)	0.80064 (17)	0.45874 (9)	0.0217 (3)
C9	0.4981 (2)	0.75335 (17)	0.42670 (9)	0.0223 (3)
C7	0.2418 (2)	0.80648 (17)	0.55681 (9)	0.0223 (3)
H7	0.106957	0.841094	0.577328	0.027*
C6	0.3981 (2)	0.75876 (17)	0.62275 (9)	0.0227 (3)
C4	0.6515 (2)	0.70870 (17)	0.49681 (10)	0.0240 (3)
H4	0.787880	0.677217	0.477166	0.029*
C10	0.1826 (2)	0.67743 (17)	0.75051 (9)	0.0223 (3)
C5	0.6034 (2)	0.71067 (18)	0.59473 (10)	0.0242 (3)
H5	0.705698	0.680589	0.640927	0.029*
C3	0.5370 (2)	0.75548 (18)	0.32394 (10)	0.0252 (3)
H3	0.671750	0.727749	0.300907	0.030*
C1	0.1690 (2)	0.84583 (18)	0.29448 (10)	0.0260 (3)
C11	0.1609 (2)	0.71001 (18)	0.86250 (10)	0.0257 (3)
C2	0.3801 (2)	0.79724 (19)	0.26079 (10)	0.0269 (3)
H2	0.407245	0.794946	0.194259	0.032*
C13	−0.0793 (2)	0.7139 (2)	0.88096 (11)	0.0338 (3)
H13A	−0.097988	0.734343	0.951163	0.051*
H13B	−0.141709	0.814129	0.851933	0.051*
H13C	−0.148727	0.595837	0.851352	0.051*
C12	0.2761 (2)	0.8930 (2)	0.91450 (11)	0.0325 (3)
H12A	0.257507	0.906684	0.984621	0.049*
H12B	0.426308	0.889022	0.902742	0.049*
H12C	0.216609	0.998011	0.888773	0.049*
C14	0.2597 (3)	0.5410 (2)	0.90167 (11)	0.0360 (3)
H14A	0.249513	0.555599	0.972376	0.054*
H14B	0.183333	0.426808	0.870998	0.054*
H14C	0.407801	0.535092	0.886066	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0223 (4)	0.0243 (5)	0.0251 (5)	0.0022 (3)	0.0013 (3)	0.0059 (3)
O4	0.0259 (5)	0.0272 (5)	0.0268 (5)	−0.0036 (4)	0.0002 (4)	0.0035 (4)
O3	0.0244 (5)	0.0275 (5)	0.0240 (4)	−0.0028 (4)	0.0014 (3)	0.0045 (4)

O2	0.0309 (5)	0.0431 (6)	0.0320 (5)	0.0033 (4)	−0.0010 (4)	0.0140 (4)
C8	0.0221 (6)	0.0171 (5)	0.0260 (6)	−0.0005 (4)	0.0002 (5)	0.0038 (4)
C9	0.0226 (6)	0.0169 (5)	0.0268 (6)	−0.0018 (4)	0.0032 (5)	0.0015 (4)
C7	0.0212 (6)	0.0195 (5)	0.0264 (6)	0.0006 (4)	0.0027 (5)	0.0035 (4)
C6	0.0249 (6)	0.0192 (5)	0.0236 (6)	−0.0023 (4)	0.0016 (5)	0.0032 (4)
C4	0.0209 (6)	0.0187 (6)	0.0318 (7)	−0.0006 (4)	0.0030 (5)	0.0025 (5)
C10	0.0211 (6)	0.0200 (5)	0.0263 (6)	0.0016 (4)	0.0008 (5)	0.0048 (4)
C5	0.0224 (6)	0.0205 (6)	0.0294 (6)	−0.0008 (4)	−0.0015 (5)	0.0043 (5)
C3	0.0241 (6)	0.0210 (6)	0.0303 (6)	−0.0008 (5)	0.0056 (5)	0.0025 (5)
C1	0.0285 (7)	0.0231 (6)	0.0268 (6)	−0.0008 (5)	0.0015 (5)	0.0062 (5)
C11	0.0278 (6)	0.0261 (6)	0.0232 (6)	0.0011 (5)	0.0009 (5)	0.0042 (5)
C2	0.0302 (7)	0.0250 (6)	0.0256 (6)	−0.0019 (5)	0.0053 (5)	0.0040 (5)
C13	0.0315 (7)	0.0394 (8)	0.0305 (7)	−0.0006 (6)	0.0085 (6)	0.0035 (6)
C12	0.0377 (8)	0.0313 (7)	0.0269 (7)	−0.0007 (6)	−0.0001 (6)	0.0011 (5)
C14	0.0481 (9)	0.0333 (8)	0.0282 (7)	0.0070 (6)	0.0009 (6)	0.0085 (6)

Geometric parameters (Å, º)

O1—C8	1.3772 (19)	C3—C2	1.346 (2)
O1—C1	1.382 (2)	C3—H3	0.9300
O4—C10	1.2058 (19)	C1—C2	1.453 (2)
O3—C10	1.3668 (19)	C11—C12	1.530 (2)
O3—C6	1.405 (2)	C11—C13	1.534 (3)
O2—C1	1.212 (2)	C11—C14	1.539 (2)
C8—C7	1.389 (2)	C2—H2	0.9300
C8—C9	1.402 (2)	C13—H13A	0.9600
C9—C4	1.401 (2)	C13—H13B	0.9600
C9—C3	1.438 (2)	C13—H13C	0.9600
C7—C6	1.383 (2)	C12—H12A	0.9600
C7—H7	0.9300	C12—H12B	0.9600
C6—C5	1.395 (2)	C12—H12C	0.9600
C4—C5	1.383 (2)	C14—H14A	0.9600
C4—H4	0.9300	C14—H14B	0.9600
C10—C11	1.523 (3)	C14—H14C	0.9600
C5—H5	0.9300		
C8—O1—C1	121.89 (12)	O1—C1—C2	117.24 (12)
C10—O3—C6	118.83 (11)	C10—C11—C12	112.83 (13)
O1—C8—C7	116.55 (13)	C10—C11—C13	107.60 (12)
O1—C8—C9	121.17 (13)	C12—C11—C13	110.08 (13)
C7—C8—C9	122.28 (12)	C10—C11—C14	106.56 (12)
C4—C9—C8	118.06 (14)	C12—C11—C14	109.61 (14)
C4—C9—C3	124.18 (13)	C13—C11—C14	110.08 (13)
C8—C9—C3	117.76 (12)	C3—C2—C1	121.31 (14)
C6—C7—C8	117.34 (13)	C3—C2—H2	119.3
C6—C7—H7	121.3	C1—C2—H2	119.3
C8—C7—H7	121.3	C11—C13—H13A	109.5
C7—C6—C5	122.65 (14)	C11—C13—H13B	109.5

C7—C6—O3	120.70 (13)	H13A—C13—H13B	109.5
C5—C6—O3	116.50 (12)	C11—C13—H13C	109.5
C5—C4—C9	121.07 (14)	H13A—C13—H13C	109.5
C5—C4—H4	119.5	H13B—C13—H13C	109.5
C9—C4—H4	119.5	C11—C12—H12A	109.5
O4—C10—O3	122.82 (13)	C11—C12—H12B	109.5
O4—C10—C11	124.93 (13)	H12A—C12—H12B	109.5
O3—C10—C11	112.17 (12)	C11—C12—H12C	109.5
C4—C5—C6	118.57 (12)	H12A—C12—H12C	109.5
C4—C5—H5	120.7	H12B—C12—H12C	109.5
C6—C5—H5	120.7	C11—C14—H14A	109.5
C2—C3—C9	120.62 (14)	C11—C14—H14B	109.5
C2—C3—H3	119.7	H14A—C14—H14B	109.5
C9—C3—H3	119.7	C11—C14—H14C	109.5
O2—C1—O1	116.69 (14)	H14A—C14—H14C	109.5
O2—C1—C2	126.05 (15)	H14B—C14—H14C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O4^{i}	0.93	2.49	3.343 (4)	153

Symmetry code: (i) *x*+1, *y*, *z*.