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ISSN 2414-3146

2-Oxo-2*H*-chromen-7-yl *tert*-butylacetate

Hypolite Bazié,[a] Eric Ziki,[b]* Sorgho Brahima,[a]* Veroarisinima Ratsimbazafy,[c] Patrick Roge,[c] Emmanuel Wenger,[d] Abdoulaye Djandé[a] and Claude Lecomte[d]

[a]Laboratory of Molecular Chemistry and Materials (LC2M), University Joseph KI-ZERBO, 03 BP 7021 Ouagadougou 03, Burkina Faso, [b]Laboratory of Matter, Environmental and Solar Energy Sciences, Research Team: Crystallography and Molecular Physics, University Félix Houphouët-Boigny, 08 BP 582, Abidjan 22, Côte d'Ivoire, [c]Laboratory of Solid State Physics and Experimental Physics, University of Antananarivo, BP 566, Antananarivo 101, Madagascar, and [d]CRM2, CNRS-Université de Lorraine, Vandoeuvre-lès-Nancy CEDEX BP 70239, France. *Correspondence e-mail: eric.ziki@gmail.com, sorghobrahima3@gmail.com

Received 19 February 2025
Accepted 27 February 2025

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

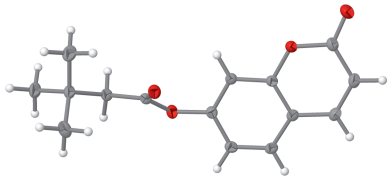
Keywords: crystal structure; Hirshfeld surface; coumarin; hydrogen bond.

CCDC reference: 2427772

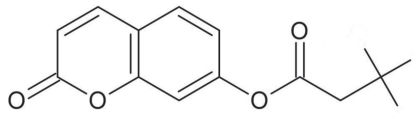
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{16}O_4$, the dihedral angle between the 2*H*-chromen-2-one ring system and the *tert*-butylacetate moiety is 72.72 (9)°. In the crystal, the molecules are connected through C—H···O hydrogen bonds, generating $C(6)$ chains and $R_2^2(20)$ loops that are reinforced by weak aromatic π–π stacking interactions. The H···H, H···O/O···H, H···C/C···H and C···C contacts contribute 50.6, 29.1, 8.5 and 6.8%, respectively, to the Hirshfeld surface.

3D view



Chemical scheme



Structure description

The title coumarin derivative, $C_{15}H_{16}O_4$ (**I**), was synthesized by a research team led by Professor Djandé (LC2M, Ouagadougou, Burkina Faso) as part of the AFRAMED project (Kenfack Tsobnang *et al.*, 2024). Coumarin-derived compounds exhibit various biological activities, such as anticancer (Yadav *et al.*, 2024; Rawat *et al.*, 2022), anti-coagulant (Singh *et al.*, 2019), anti-inflammatory (Todeschini *et al.*, 1998) and anti-glaucoma (Ziki *et al.*, 2023) properties.

As shown in Fig. 1, the 2*H*-chromen-2-one moiety formed by atoms C1–C9/O1/O2 in (**I**) is almost planar with an r.m.s deviation of 0.027 Å and the dihedral angle between this ring system and the plane formed by atoms C10–C12/C14 in the *tert*-butylacetate moiety is 72.72 (9)°. An $S(6)$ ring motif resulting from an intramolecular C13—H13*B*···O4 hydrogen bond is observed (Table 1). The plane passing through atoms C10–C12/C14 of the *tert*-butylacetate moiety contains the ester function atoms (r.m.s = 0.228 Å), but methyl atoms C13 and C15 atoms are on either side of this plane with deviations of 1.275 (1) and −1.244 (1) Å, respectively.

In the crystal of (**I**), molecules are linked by weak hydrogen bonds of the C—H···O type. A pair of C11—H11*B*···O2($-x$ + 2, $-y$, $-z$ + 1) hydrogen bonds generates a

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O4^{i}	0.95	2.50	3.4144 (13)	161
C11—H11B···O2^{ii}	0.99	2.52	3.2523 (13)	131
C13—H13B···O4	0.98	2.43	3.0924 (14)	124

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y, -z+1$.

centrosymmetric $R_2^2(20)$ loop, as shown in Fig. 3. The C5—H5···O4($x-1, y, z$) hydrogen bonds form $C(6)$ chains propagating in the [100] direction (Fig. 2). Aromatic π–π stacking interactions between the pyrone ring (centroid $Cg1$) and benzene ring (centroid $Cg2$) of a symmetry-related ($1-x, -y, 1-z$) molecule reinforce the cohesion of molecules [$Cg1\cdots Cg2$ = 3.5485 (8) with a slippage of 1.042 Å],

The Hirshfeld surface and two-dimensional fingerprint (FP) plot of **(1)** (Fig. 3) generated by *CrystalExplorer21.5* (Spackman *et al.*, 2021) confirmed the above interactions. The fingerprint plots show the different contributions of the atoms in the crystal-to-surface contacts. The most important contributions are H···H and H···O/O···H contacts with 50.6 and 29.1%, respectively (Fig. 3*d* and 3*f*). The H···C/C···H and C···C contacts contribute 8.5 and 6.8%, respectively. These values are close to those of 2-oxo-2*H*-chromen-6-yl 4- *tert*-butylbenzoate (Kenfack Tsobnang *et al.*, 2024).

Synthesis and crystallization

To a solution of *tert*-butylacetyl chloride (6.2 mmol, 0.9 ml) in dried diethyl ether (16 ml) was added dried pyridine (4.7 molar equivalents; 2.31 ml) and 7-hydroxycoumarin (6.17 mmol, 1.00 g) in small portions over 30 min. The mixture was left under agitation at room temperature for 3 h and then poured into 40 ml of chloroform. The solution was acidified with dilute hydrochloric acid (5%) until the pH was 2–3. The organic layer was extracted, washed four times with 25 ml of water to neutrality, dried over $MgSO_4$ and the solvent removed. The resulting crude product was filtered off with suction, washed with petroleum ether and recrystallized from acetone solution as colorless crystals of the title compound. Yield = 79%, m.p. = 368–371 K.

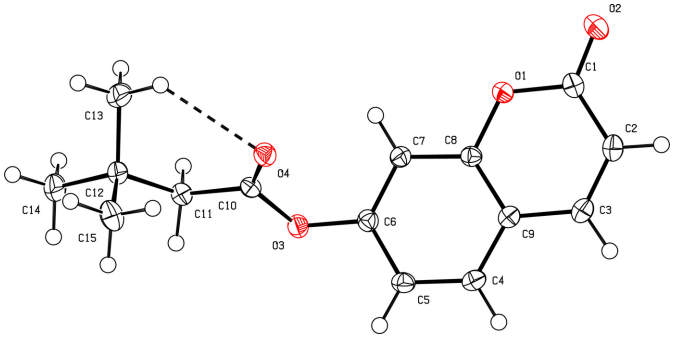


Figure 1
The molecular structure of **(I)** with displacement ellipsoids drawn at the 50% probability level.

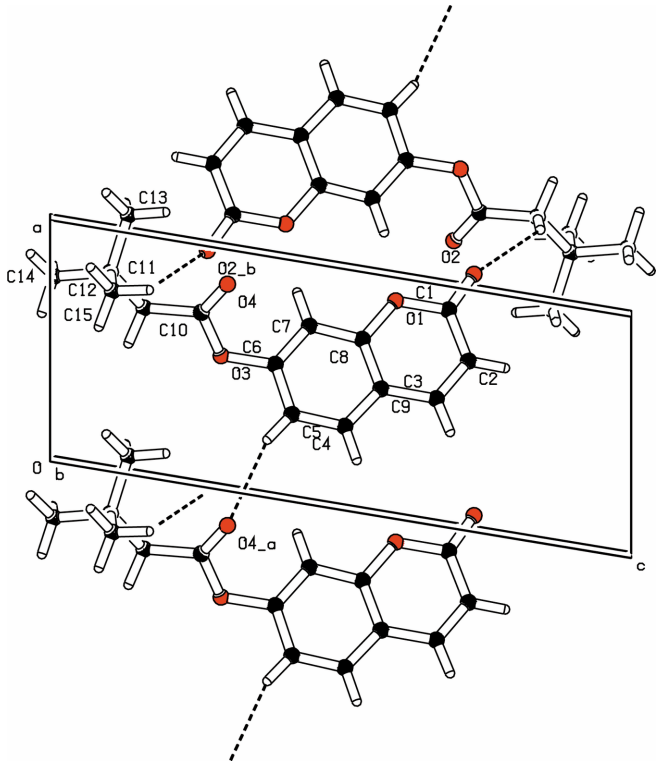


Figure 2
Part of the crystal of **(I)** showing the formation of an undulating network along the *b* axis [$C(6)$ and $R_2^2(20)$ motifs]. Dashed lines indicate hydrogen bonds.

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

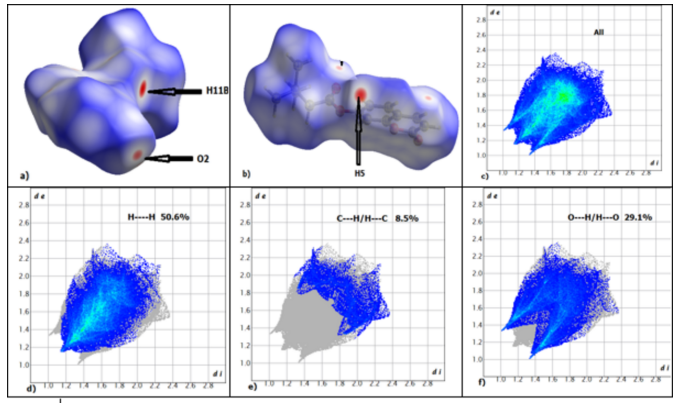


Figure 3
(*a*), (*b*) Hirshfeld surface of **(I)** mapped over d_{norm}, (*c*) overall two-dimensional fingerprint plot of and those delineated into contributions from different contacts: (*d*) H···H, (*d*) H···C/C···H and (*e*) H···O/O···H.

Université de Lorraine for the 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 are very grateful to UNESCO, CNRS and the IUCr for their support of the AFRAMED project.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{16}O_4$
M_r	260.28
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	6.1599 (9), 7.2029 (11), 15.202 (2)
α, β, γ (°)	98.765 (5), 99.335 (5), 91.228 (5)
V (Å^3)	657.05 (17)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.20 × 0.12 × 0.07
Data collection	
Diffractometer	Bruker D8 Venture
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	53961, 4064, 3720
R_{int}	0.050
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.719
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.137, 1.05
No. of reflections	4064
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.28

Computer programs: *APEX4* and *SAINT* (Bruker, 2019), *SHELXS2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

full crystallographic data

IUCrData (2025). **10**, x250189 [https://doi.org/10.1107/S2414314625001890]

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2-Oxo-2*H*-chromen-7-yl *tert*-butylacetate

Crystal data

$C_{15}H_{16}O_4$
M_r = 260.28
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.1599 (9) Å
b = 7.2029 (11) Å
c = 15.202 (2) Å
α = 98.765 (5)°
β = 99.335 (5)°
γ = 91.228 (5)°
V = 657.05 (17) Å^3
Z = 2
$F(000)$ = 276
D_x = 1.316 Mg m^{-3}
Melting point: 368 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4066 reflections
θ = 2.9–30.8°
μ = 0.10 mm^{-1}
T = 100 K
Prism, yellow
0.20 × 0.12 × 0.07 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: fine-focus sealed tube
Mirror monochromator
φ and ω scan
53961 measured reflections
4064 independent reflections
3720 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 30.8°, θ_{min} = 2.9°
h = −8→8
k = −10→10
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.137
S = 1.05
4064 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.2223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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}
O3	0.53573 (12)	0.19418 (10)	0.29426 (5)	0.02062 (16)
O1	0.88247 (11)	0.15470 (10)	0.59464 (5)	0.01913 (16)
O4	0.84148 (12)	0.38219 (10)	0.30532 (5)	0.02129 (16)
O2	1.04542 (14)	0.10787 (12)	0.72944 (5)	0.02755 (18)
C9	0.51037 (15)	0.26016 (12)	0.56924 (6)	0.01750 (18)
C6	0.53813 (16)	0.22422 (13)	0.38729 (6)	0.01796 (18)
C5	0.34391 (15)	0.28555 (13)	0.41609 (7)	0.01937 (18)
H5	0.222778	0.314675	0.374030	0.023*
C4	0.33122 (15)	0.30309 (13)	0.50706 (7)	0.01946 (19)
H4	0.200134	0.344549	0.527611	0.023*
C8	0.70128 (15)	0.20004 (13)	0.53717 (6)	0.01673 (17)
C7	0.71880 (15)	0.18000 (13)	0.44623 (6)	0.01781 (18)
H7	0.849197	0.137745	0.425327	0.021*
C10	0.70385 (15)	0.27069 (13)	0.26024 (6)	0.01775 (18)
C1	0.88143 (17)	0.15948 (14)	0.68557 (6)	0.02053 (19)
C3	0.50766 (17)	0.27137 (14)	0.66442 (7)	0.02070 (19)
H3	0.380241	0.311803	0.688282	0.025*
C2	0.68492 (18)	0.22481 (14)	0.71993 (7)	0.0225 (2)
H2	0.681287	0.234916	0.782707	0.027*
C11	0.68111 (16)	0.19153 (14)	0.16148 (6)	0.01956 (18)
H11A	0.522872	0.186209	0.134991	0.023*
H11B	0.728920	0.060533	0.156411	0.023*
C12	0.80830 (16)	0.29648 (14)	0.10383 (6)	0.02054 (19)
C13	1.05795 (18)	0.29276 (19)	0.13479 (8)	0.0305 (2)
H13A	1.135176	0.360602	0.096951	0.046*
H13B	1.096000	0.353175	0.197906	0.046*
H13C	1.102008	0.162119	0.129266	0.046*
C15	0.7374 (2)	0.50003 (16)	0.10722 (7)	0.0284 (2)
H15A	0.820076	0.565015	0.070105	0.043*
H15B	0.579442	0.500204	0.083962	0.043*
H15C	0.767411	0.564855	0.169777	0.043*
C14	0.74935 (19)	0.19270 (17)	0.00628 (7)	0.0277 (2)
H14A	0.827400	0.255304	−0.033149	0.041*
H14B	0.792917	0.062204	0.003871	0.041*
H14C	0.590176	0.194470	−0.014024	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0191 (3)	0.0257 (3)	0.0169 (3)	−0.0023 (3)	0.0020 (2)	0.0045 (3)
O1	0.0186 (3)	0.0219 (3)	0.0171 (3)	0.0041 (2)	0.0023 (2)	0.0040 (2)
O4	0.0204 (3)	0.0241 (3)	0.0187 (3)	−0.0014 (3)	0.0019 (2)	0.0032 (3)
O2	0.0274 (4)	0.0334 (4)	0.0216 (4)	0.0057 (3)	−0.0004 (3)	0.0074 (3)
C9	0.0181 (4)	0.0150 (4)	0.0201 (4)	0.0005 (3)	0.0050 (3)	0.0028 (3)
C6	0.0184 (4)	0.0182 (4)	0.0177 (4)	−0.0005 (3)	0.0030 (3)	0.0041 (3)
C5	0.0156 (4)	0.0199 (4)	0.0229 (4)	0.0003 (3)	0.0018 (3)	0.0058 (3)
C4	0.0163 (4)	0.0188 (4)	0.0244 (4)	0.0015 (3)	0.0057 (3)	0.0042 (3)
C8	0.0167 (4)	0.0155 (4)	0.0181 (4)	0.0014 (3)	0.0026 (3)	0.0034 (3)
C7	0.0175 (4)	0.0186 (4)	0.0180 (4)	0.0023 (3)	0.0041 (3)	0.0035 (3)
C10	0.0176 (4)	0.0189 (4)	0.0176 (4)	0.0031 (3)	0.0024 (3)	0.0054 (3)
C1	0.0244 (5)	0.0197 (4)	0.0173 (4)	0.0006 (3)	0.0024 (3)	0.0037 (3)
C3	0.0222 (4)	0.0195 (4)	0.0214 (4)	0.0010 (3)	0.0078 (3)	0.0019 (3)
C2	0.0274 (5)	0.0232 (4)	0.0174 (4)	0.0008 (4)	0.0060 (3)	0.0021 (3)
C11	0.0197 (4)	0.0213 (4)	0.0171 (4)	0.0010 (3)	0.0020 (3)	0.0029 (3)
C12	0.0202 (4)	0.0258 (4)	0.0156 (4)	0.0012 (3)	0.0030 (3)	0.0032 (3)
C13	0.0198 (5)	0.0468 (7)	0.0245 (5)	−0.0003 (4)	0.0048 (4)	0.0032 (4)
C15	0.0382 (6)	0.0250 (5)	0.0223 (5)	0.0003 (4)	0.0024 (4)	0.0075 (4)
C14	0.0294 (5)	0.0354 (6)	0.0172 (4)	0.0008 (4)	0.0040 (4)	0.0011 (4)

Geometric parameters (Å, º)

O3—C10	1.3710 (11)	C3—C2	1.3490 (14)
O3—C6	1.3953 (11)	C3—H3	0.9500
O1—C1	1.3785 (11)	C2—H2	0.9500
O1—C8	1.3791 (11)	C11—C12	1.5339 (14)
O4—C10	1.2033 (12)	C11—H11A	0.9900
O2—C1	1.2151 (12)	C11—H11B	0.9900
C9—C8	1.3970 (13)	C12—C15	1.5344 (15)
C9—C4	1.4044 (13)	C12—C13	1.5355 (15)
C9—C3	1.4396 (13)	C12—C14	1.5380 (14)
C6—C7	1.3862 (13)	C13—H13A	0.9800
C6—C5	1.3971 (13)	C13—H13B	0.9800
C5—C4	1.3845 (13)	C13—H13C	0.9800
C5—H5	0.9500	C15—H15A	0.9800
C4—H4	0.9500	C15—H15B	0.9800
C8—C7	1.3900 (13)	C15—H15C	0.9800
C7—H7	0.9500	C14—H14A	0.9800
C10—C11	1.5054 (13)	C14—H14B	0.9800
C1—C2	1.4540 (14)	C14—H14C	0.9800
C10—O3—C6	119.43 (7)	C1—C2—H2	119.3
C1—O1—C8	121.85 (8)	C10—C11—C12	117.18 (8)
C8—C9—C4	118.47 (9)	C10—C11—H11A	108.0
C8—C9—C3	117.80 (9)	C12—C11—H11A	108.0

C4—C9—C3	123.72 (9)	C10—C11—H11B	108.0
C7—C6—O3	120.83 (8)	C12—C11—H11B	108.0
C7—C6—C5	122.58 (9)	H11A—C11—H11B	107.2
O3—C6—C5	116.39 (8)	C11—C12—C15	110.51 (8)
C4—C5—C6	118.78 (9)	C11—C12—C13	111.12 (8)
C4—C5—H5	120.6	C15—C12—C13	110.36 (9)
C6—C5—H5	120.6	C11—C12—C14	106.66 (8)
C5—C4—C9	120.60 (9)	C15—C12—C14	109.16 (8)
C5—C4—H4	119.7	C13—C12—C14	108.94 (8)
C9—C4—H4	119.7	C12—C13—H13A	109.5
O1—C8—C7	116.34 (8)	C12—C13—H13B	109.5
O1—C8—C9	121.32 (8)	H13A—C13—H13B	109.5
C7—C8—C9	122.33 (9)	C12—C13—H13C	109.5
C6—C7—C8	117.24 (8)	H13A—C13—H13C	109.5
C6—C7—H7	121.4	H13B—C13—H13C	109.5
C8—C7—H7	121.4	C12—C15—H15A	109.5
O4—C10—O3	123.07 (9)	C12—C15—H15B	109.5
O4—C10—C11	128.49 (9)	H15A—C15—H15B	109.5
O3—C10—C11	108.45 (8)	C12—C15—H15C	109.5
O2—C1—O1	116.63 (9)	H15A—C15—H15C	109.5
O2—C1—C2	126.14 (9)	H15B—C15—H15C	109.5
O1—C1—C2	117.23 (9)	C12—C14—H14A	109.5
C2—C3—C9	120.42 (9)	C12—C14—H14B	109.5
C2—C3—H3	119.8	H14A—C14—H14B	109.5
C9—C3—H3	119.8	C12—C14—H14C	109.5
C3—C2—C1	121.30 (9)	H14A—C14—H14C	109.5
C3—C2—H2	119.3	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}	0.95	2.50	3.4144 (13)	161
C11—H11*B*···O2^{ii}	0.99	2.52	3.2523 (13)	131
C13—H13*B*···O4	0.98	2.43	3.0924 (14)	124

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+2, −*y*, −*z*+1.