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Synthesis and crystal structure of 5,10-dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one

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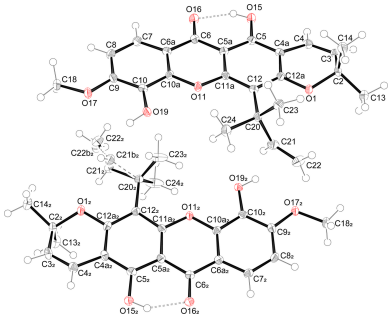
5,10-Dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one, $C_{24}H_{24}O_6$ (**2**), is a prenylated xanthone that was synthesized from 5,9,10-trihydroxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one or macluraxanthone (**1**), a known compound isolated from *Garcinia schomburgkiana* Pierre. The present study describes the synthesis of compound **2** by methylation reaction of **1**, and its crystallographic characterization. Compound 2 features a planar xanthone core and a bent pyrano ring adopting a half-boat conformation. An intermolecular O—H···O hydrogen bond between the hydroxyl hydrogen donor and the ketone acceptor organizes the molecules into a one-dimensional network along the *b*-axis direction. Perpendicular to this network, π–π stacking interactions form the three-dimensional supramolecular architecture. These two key intermolecular interactions are distinctly revealed in the Hirshfeld surface analysis.

1. Chemical context

Pyranoxanthones have long been known for their natural occurrence, showing a broad spectrum of pharmacological and biological activities (Kondedeshmukh & Paradkar, 1994). In the past decade, this scaffold has been subjected to chemical structure identification and synthetic investigations. The pyranoxanthone core also brings a wide range of applications. For example, 1,2-dihydro-2-hydroxy-6-methoxy-3,3-dimethyl-3*H*,7*H*-pyrano[2,3-*c*]xanthen-7-one showed a potent cytotoxicity against leukemia L1210 cell line (Ghirtis *et al.*, 2001). Because various substituents on pyranoxanthones cause different properties, the structure–activity relationships (SAR) play a pivotal role in the discovery of their biological activities.

5,10-Dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one (**2**) is a pyranoxanthone that was isolated from leaves and twigs of *Garcinia speciosa*. This compound showed weak inhibitory activity toward HIV-1 reverse transcriptase (Pailee *et al.*, 2018). In addition, a parent analog 5,9,10-trihydroxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one (**1**), also known as macluraxanthone, was isolated from the same plant (Sangsuwon & Jiratchariyakul, 2015). Compound **1** was first isolated from osage orange (*Maclura pomifera*) in 1964 (Wolfrom *et al.*, 1964). Subsequently, it was also found in different parts of various plants such as *Garcinia bancana* (Rifaldi *et al.*, 2024), and *Cratoxylum soulattri* (Mah *et al.*,



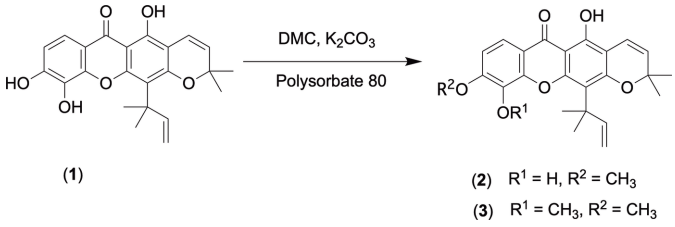
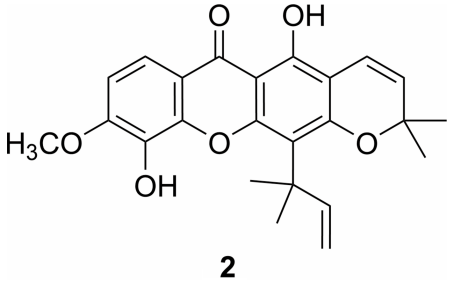


Figure 1
Methylation reaction of macluraxanthone (**1**) to produce xanthones **2** and **3**.

2011). Recently, **1** was also isolated from fruits and twigs of *Garcinia schomburgkiana*, a Thai plant known locally as 'Ma Dun', with a considerable quantity (1.07% from fruits and 5.29% from twigs) (Sukkum *et al.*, 2024). Prompted by the above results, we performed the synthesis of compounds **2** and **3** from macluraxanthone (**1**) *via* methylation reaction using dimethyl carbonate (DMC) as a methylating reagent in the presence of K_2CO_3 as a base, and polysorbate 80 as a phase transfer catalyst (Prakoso *et al.*, 2016) as shown in Fig. 1. The identities of the products were confirmed by ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy and high-resolution electrospray ionization mass spectrometry (HR-ESI-MS). Based on the spectroscopic and spectrometry data and by comparison with the data reported in the literature (Pailee *et al.*, 2018; Wolfrom *et al.*, 1964), compounds **2** and **3** were determined as 5,10-dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one (**2**) and 5-hydroxy-9,10-dimethoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*, 6*H*-pyrano[3,2-*b*]xanthen-6-one (**3**). The structure of **2** was further confirmed by single-crystal X-ray crystallography.



2

2. Structural commentary

Compound **2** crystallizes in the monoclinic $P2_1$ space group with two independent molecules in the asymmetric unit (Z = 4). The structure of **2** shows the expected methylation of the hydroxyl group at O17 position (Fig. 2). The methyl group is coplanar with the core xanthone structure, with C8—C9—O17—C18 torsion angles of 3.8 (3) and −1.4 (3)° for the first and second molecules, respectively. The xanthone core structure (O11, C4*A*–C12*A*) is planar, while the pyrano ring (O1, C2–C4*A*, C12*A*) is bent, adopting a half-boat conformation with the C2 atom deviating out of the plane generated by the remaining 17 atoms (O1, O11, C3–C12*A*) by 0.456 (2) and 0.534 (2) Å, for the first and second molecules,

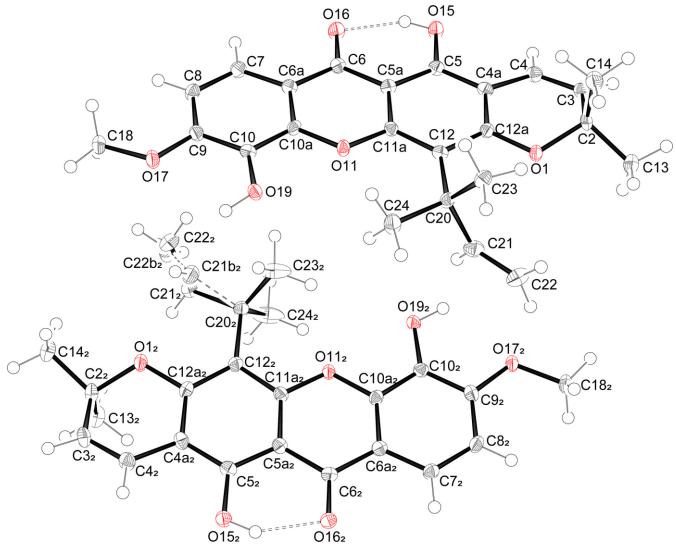


Figure 2
ORTEP view of compound **2** plotted as displacement ellipsoids at the 50% probability level. Two molecules comprise the asymmetric unit.

respectively. This result supports the chemical structure that C2 is not conjugated with the aromatic system. The root-mean-square deviations of the molecular plane formed from these 17 atoms are 0.034 and 0.035 Å for the first and second molecules, respectively.

The structure of **2** features an intramolecular hydrogen bond (Table 1) between its carbonyl and the nearby hydroxyl group, with an O15···O16 distance of 2.530 (2) and 2.547 (2) Å for the first and second molecules in the asymmetric unit, respectively. This distance is considered relatively short for O···O distances involved in hydrogen bonding.

3. Supramolecular features

An intermolecular hydrogen bond is found between O16 and O19 with an O···O distance of 2.719 (2) and 2.704 (2) Å

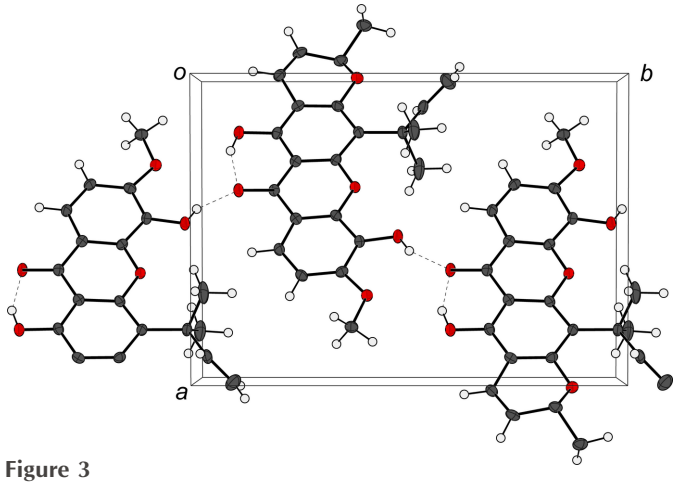


Figure 3
Packing of **2** in the unit cell, consolidated by intermolecular hydrogen bonding. The unit cell is shown as a gray box where the *c* axis is parallel to the reader's view. The *a* and *b* axes and the origin center are labeled in the figure.

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

D—H···A	D—H	H···A	D···A	D—H···A
O15—H15···O16	0.84	1.78	2.530 (2)	147
O15_2—H15_2···O16_2	0.84	1.80	2.547 (2)	147
O19—H19···O16^{i}	0.84	1.92	2.719 (2)	159
O19_2—H19_2···O16_2^{ii}	0.84	1.92	2.704 (2)	156

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

(Table 1) for the first and second molecules in the asymmetric unit, respectively. This hydrogen bonding consolidates the molecular packing, which forms a one-dimensional network of **2** along the *b*-axis direction (Fig. 3). The methoxy group of **2** formed upon the methylation of **1** is not involved in any significant hydrogen-bonding interactions.

The planarity of the molecule facilitates molecular stacking in the structure. The first and second molecules in the asymmetric unit are aligned almost parallel to each other; the angle between the two molecular planes are 11.23 (4)°. The shortest C···C distance between the two molecules is 3.366 (3) Å, found between C4 of the first molecule and C11*A* of the second molecule, indicating possible π–π interactions (Fig. 4*a*). The π–π stackings run along the [10$\bar{1}$] direction perpendicular to the intermolecular hydrogen-bonding network (Fig. 4*b*), altogether forming a three-dimensional supramolecular arrangement.

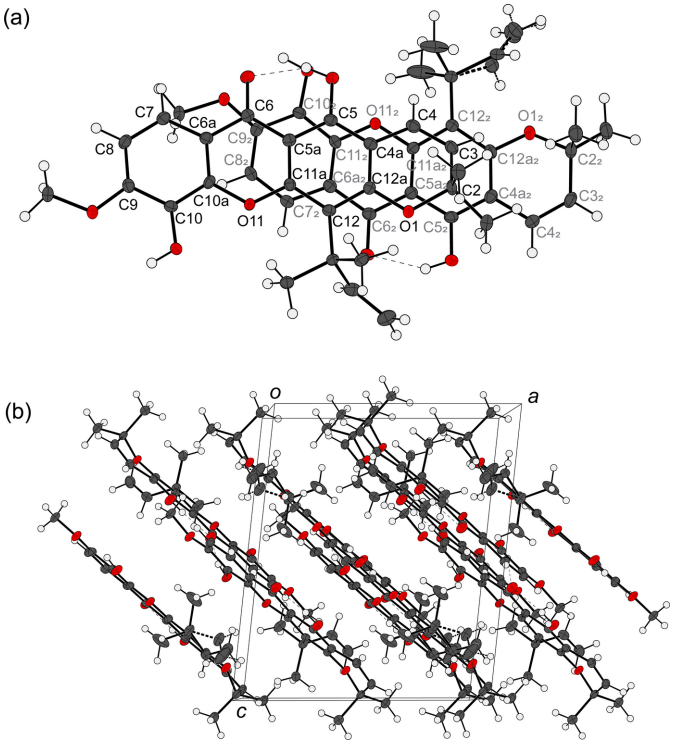


Figure 4
(*a*) Two molecules of **2** in the asymmetric unit viewed perpendicular to the pyranoxanthone rings to highlight π–π stacking. The pyranoxanthone core atoms of the first and second molecules are labeled in black and gray, respectively. (*b*) Stacking of **2** in the unit cell. The unit cell is shown as a gray box where the *b* axis is parallel to the reader's view. The *a* and *c* axes and the origin center are labeled in the figure.

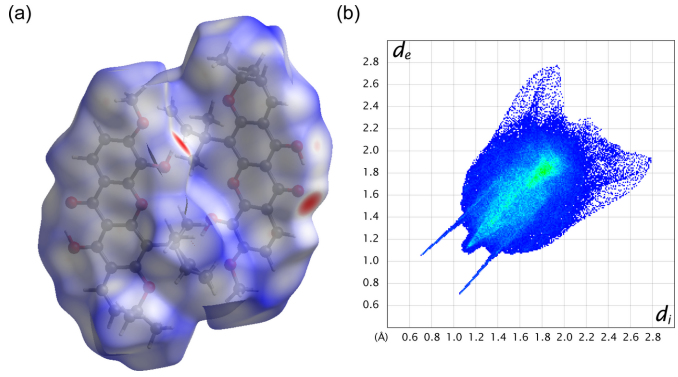


Figure 5
(*a*) Three-dimensional Hirshfeld surface representation of **2** plotted over d_{norm} and (*b*) two-dimensional fingerprint plot of **2** showing all interactions.

4. Hirshfeld surface analysis

Hirshfeld surface analysis was performed to more accurately identify and quantify intermolecular interactions. The analysis was performed using *CrystalExplorer* 21.5 (Spackman *et al.*, 2021). The three-dimensional Hirshfeld surface of **2** is plotted in Fig. 5*a*, including the two molecules of the asymmetric unit, mapped over normalized contact distance (d_{norm}) on a scale from −0.65 to 1.66 a.u. Blue, white, and red regions indicate contacts that are longer, equal, and shorter than the sum of van der Waals radii, respectively. The apparent red regions around O atoms indicate short contacts from intermolecular hydrogen bonding. Fig. 5*b* depicts a two-dimensional fingerprint plot of (d_i, d_e). Two sharp spikes in the fingerprint plot indicate the short intermolecular O—H···O hydrogen bonding interactions, which contribute 15.8% to the overall Hirshfeld surface area. A π–π planar stacking was also identified in the cyan-green region of the plot centered around d_i = d_e = 1.8 Å, contributing 8.7%. The surface corresponding to these C···C interactions spans over two of the four aromatic rings of **2**, indicating that π–π stacking is impeded. The remaining major intermolecular interactions are H···H and C···H interactions, contributing 62.4 and 9.7%, respectively.

5. Database survey

A search for the pyranoxanthone core structure revealed five crystal structures in the Cambridge Structural Database (CSD version 5.45, last update November 2023; Groom *et al.*, 2016). The structure of macluraxanthone (**1**) (QAYTOA; Fun *et al.*, 2006) shows similar structural features including the bent pyrano ring and planar xanthone rings, but differs in the intermolecular packing: **1** engages in hydrogen-bonding interactions involving the O17 position, which is absent in **2** due to the methylation of this oxygen atom. A di-*p*-bromobenzenesulfonylated derivative was also reported (YIZPAZ; Boonnak *et al.*, 2008), featuring a similar bent pyrano ring and planar xanthone rings. In addition, three other pyranoxanthone structures possessing different substituents on C12 were found in the database: CIXSIL (Kosela *et al.*, 1999),

MAPMIA (Chantrapromma *et al.*, 2005), and CABFAP (Sukandar *et al.*, 2016).

6. Synthesis and crystallization

The synthetic reaction was modified from a published procedure according to Prakoso *et al.* (2016). Briefly, a 50 mL round-bottom flask equipped with a magnetic stir bar was charged with **1** (0.13 mmol, 1 equiv.), K_2CO_3 (0.56 mmol, 4.3 equiv.), and polysorbate 80 (0.163 mmol, 1.25 equiv.). Then, dimethyl carbonate (DMC) (1.30 mmol, 10 equiv.) was added to the reaction mixture. After refluxing at 373 K for 5 h, the reaction mixture was quenched with aqueous acetic acid (20 mL) and extracted with dichloromethane (5 × 50 mL). The combined organic layers were washed with a saturated NaCl solution (20 mL) and dried over anhydrous Na_2SO_4. After removal of the solvent, the crude mixture was purified by column chromatography (acetone:hexane, 1:5 *v*/*v*, isocratic system) to afford compound **2** (48% yield), compound **3** (11% yield), and a recovered starting material (**1**) (36% yield).

5,10-Dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one (**2**): yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$), δ 13.48 (*s*, 1H), 7.68 (*d*, *J* = 9.0 Hz,1H), 6.90 (*d*, *J* = 9.0 Hz, 1H), 6.69 (*d*, *J* = 10.0 Hz, 1H), 6.61 (*s*, 1H), 6.59 (*dd*, *J* = 16.0, 10.0 Hz, 1H), 5.54 (*d*, *J* = 10.0 Hz, 1H), 5.12 (*dd*, *J* = 16.0, 1.0 Hz, 1H), 5.09 (*dd*, *J* = 10.0, 1.0 Hz, 1H), 3.95 (*s*, 3H), 1.59 (*s*, 6H), 1.44 (*s*, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$), δ 180.8, 159.1, 156.7, 154.9, 154.4, 151.5, 144.3, 133.5, 127.1, 116.8, 116.0, 114.2, 113.3, 108.4, 105.4, 104.6, 103.6, 78.2, 55.6, 41.3, 28.5, 27.1 ppm. HR-ESI-MS of 407.1482 $[M–H]^-$ (calculated for $C_{24}H_{23}O_6$; 407.1500).

5-Hydroxy-9,10-dimethoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one (**3**): yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$), δ 13.57 (*s*, 1H, OH), 7.90 (*d*, *J* = 8.0 Hz, 1H), 6.92 (*d*, *J* = 8.0 Hz, 1H), 6.68 (*d*, *J* = 9.6 Hz, 1H), 6.30 (*dd*, *J* = 12.1, 8.0 Hz, 1H), 5.52 (*d*, *J* = 9.6 Hz, 1H), 4.86 (*dd*, *J* = 17.0, 1.0 Hz, 1H), 4.78 (*dd*, *J* = 10.0, 1.0 Hz, 1H), 3.94 (*s*, 3H, OCH_3), 3.86 (*s*, 3H, OCH_3), 1.66 (*s*, 6H), 1.40 (*s*, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$), δ 181.0, 159.4, 158.0, 156.5, 155.4, 150.8, 150.0, 136.4, 127.3, 121.4, 116.0, 114.9, 113.7, 108.6, 107.9, 105.2, 103.3, 78.2, 61.5, 55.4, 41.5, 31.9, 27.9 ppm. HR-ESI-MS of 445.1622 $[M + Na]^+$ (calculated for $C_{25}H_{26}O_6Na$; 445.1622).

Single crystals of **2** were obtained as yellow blocks from vapor diffusion of *n*-hexane into an acetone solution.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All non-hydrogen atoms were refined anisotropically. The vinyl group of the second molecule was found to be disordered; refinement was accomplished by modeling over two positions. The occupancy of each disordered component was initially refined freely, and converged to a 0.53:0.47 occupancy ratio. Thus, the occupancy of both components was subsequently constrained to 0.5. The anisotropic displacement refinement of the disordered atoms was stabilized by the application of enhanced rigid bond restraints. Hydrogen atoms bonded to carbon were included in calculated positions and refined using a riding model. Hydrogen atoms bound to O atoms were located in the difference-Fourier map, and refined semi-freely with the help of distance restraints.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{24}O_6$
M_r	408.43
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	101
a, *b*, *c* (Å)	10.6205 (3), 14.8160 (4), 12.7073 (3)
β (°)	96.209 (1)
V (Å^3)	1987.81 (9)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.80
Crystal size (mm)	0.18 × 0.11 × 0.06
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON III C7
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.703, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32650, 7256, 6891
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.604
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.079, 1.06
No. of reflections	7256
No. of parameters	581
No. of restraints	253
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.25
Absolute structure	Flack *x* determined using 3110 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.00 (5)

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT2015* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

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supporting information

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Synthesis and crystal structure of 5,10-dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2*H*,6*H*-pyrano[3,2-*b*]xanthen-6-one

Amporn Saekee, Chutima Kuhakarn, Khetpakorn Chakarawet and Sakchai Hongthong

Computing details

5,10-Dihydroxy-9-methoxy-2,2-dimethyl-12-(2-methylbut-3-en-2-yl)-2H,6H-pyrano[3,2-b]xanthen-6-one

Crystal data

$C_{24}H_{24}O_6$
M_r = 408.43
Monoclinic, $P2_1$
a = 10.6205 (3) Å
b = 14.8160 (4) Å
c = 12.7073 (3) Å
β = 96.209 (1)°
V = 1987.81 (9) Å^3
Z = 4
$F(000)$ = 864
D_x = 1.365 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9895 reflections
θ = 4.6–68.5°
μ = 0.80 mm^{-1}
T = 101 K
Block, clear light yellow
0.18 × 0.11 × 0.06 mm

Data collection

Bruker D8 QUEST PHOTON III C7 diffractometer
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs
ω and φ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.703, T_{max} = 0.753
32650 measured reflections
7256 independent reflections
6891 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 68.6°, θ_{min} = 4.2°
h = −11→12
k = −17→17
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.079
S = 1.06
7256 reflections
581 parameters
253 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.2292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack x determined using 3110 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.00 (5)

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}	Occ. (<1)
O1	0.48934 (14)	0.63254 (10)	0.88990 (12)	0.0157 (3)	
C2	0.56134 (19)	0.68143 (15)	0.97693 (17)	0.0171 (4)	
C3	0.5962 (2)	0.77380 (14)	0.94037 (18)	0.0178 (4)	
H3	0.672019	0.801826	0.970704	0.021*	
C4	0.5213 (2)	0.81629 (15)	0.86586 (17)	0.0172 (4)	
H4	0.539570	0.876651	0.847378	0.021*	
C4A	0.4109 (2)	0.77029 (14)	0.81235 (17)	0.0153 (4)	
C5	0.3218 (2)	0.81468 (14)	0.74379 (16)	0.0151 (4)	
C6	0.13155 (19)	0.81253 (14)	0.61298 (16)	0.0153 (4)	
C6A	0.03222 (19)	0.75829 (14)	0.55740 (17)	0.0150 (4)	
C7	−0.0620 (2)	0.79472 (15)	0.48401 (16)	0.0174 (4)	
H7	−0.063051	0.857719	0.470036	0.021*	
C8	−0.1531 (2)	0.73982 (15)	0.43193 (17)	0.0179 (4)	
H8	−0.216283	0.764913	0.381753	0.021*	
C9	−0.15260 (19)	0.64708 (14)	0.45291 (16)	0.0159 (4)	
C10	−0.0607 (2)	0.60890 (14)	0.52692 (16)	0.0144 (4)	
C10A	0.03125 (19)	0.66544 (14)	0.57792 (16)	0.0141 (4)	
O11	0.11980 (13)	0.62404 (9)	0.64739 (12)	0.0151 (3)	
C11A	0.21661 (18)	0.67169 (14)	0.70155 (16)	0.0134 (4)	
C12	0.30334 (19)	0.62308 (14)	0.77021 (16)	0.0141 (4)	
C12A	0.39954 (19)	0.67621 (14)	0.82519 (16)	0.0137 (4)	
C13	0.6763 (2)	0.62260 (16)	1.00704 (18)	0.0211 (5)	
H13A	0.648844	0.563562	1.030848	0.032*	
H13B	0.731042	0.651523	1.064320	0.032*	
H13C	0.723247	0.614643	0.945423	0.032*	
C14	0.4786 (2)	0.68963 (16)	1.06809 (17)	0.0216 (5)	
H14A	0.399911	0.721312	1.043275	0.032*	
H14B	0.524359	0.723596	1.126368	0.032*	
H14C	0.458469	0.629226	1.092904	0.032*	
O15	0.33355 (14)	0.90468 (10)	0.73218 (12)	0.0197 (3)	
H15	0.275792	0.923517	0.687151	0.029*	
O16	0.14017 (14)	0.89588 (10)	0.59763 (12)	0.0206 (3)	
O17	−0.23789 (14)	0.58673 (10)	0.40592 (12)	0.0196 (3)	
C18	−0.3305 (2)	0.62000 (16)	0.32450 (18)	0.0217 (5)	
H18A	−0.287542	0.645740	0.266961	0.033*	
H18B	−0.381849	0.666699	0.353953	0.033*	
H18C	−0.385346	0.570258	0.296996	0.033*	
O19	−0.05738 (13)	0.51996 (10)	0.55172 (11)	0.0170 (3)	
H19	−0.098952	0.490660	0.503133	0.026*	

C5A	0.2222 (2)	0.76596 (15)	0.68675 (17)	0.0150 (4)
C20	0.30221 (18)	0.51977 (14)	0.79184 (16)	0.0155 (4)
C21	0.4241 (2)	0.47936 (15)	0.76007 (18)	0.0208 (4)
H21	0.445444	0.494286	0.691477	0.025*
C22	0.5035 (2)	0.42587 (17)	0.8174 (2)	0.0270 (5)
H22A	0.577 (3)	0.404 (2)	0.790 (2)	0.036 (8)*
H22B	0.495 (3)	0.413 (2)	0.890 (3)	0.044 (9)*
C23	0.28615 (19)	0.50328 (14)	0.90934 (17)	0.0178 (4)
H23A	0.279587	0.438304	0.922085	0.027*
H23B	0.209051	0.533413	0.927059	0.027*
H23C	0.359603	0.527643	0.953612	0.027*
C24	0.1957 (2)	0.46614 (15)	0.72720 (19)	0.0239 (5)
H24A	0.202985	0.473601	0.651468	0.036*
H24B	0.113204	0.488743	0.743273	0.036*
H24C	0.203356	0.402044	0.745839	0.036*
O1_2	−0.00836 (14)	0.37560 (10)	0.13071 (12)	0.0194 (3)
C2_2	−0.0698 (2)	0.33552 (15)	0.03272 (17)	0.0191 (4)
C3_2	−0.0966 (2)	0.23753 (16)	0.05003 (18)	0.0205 (5)
H3_2	−0.168920	0.209760	0.012961	0.025*
C4_2	−0.0189 (2)	0.18992 (15)	0.11731 (17)	0.0188 (4)
H4_2	−0.031187	0.126751	0.123982	0.023*
C4A_2	0.08530 (19)	0.23455 (15)	0.18108 (17)	0.0162 (4)
C5_2	0.17783 (19)	0.18563 (15)	0.24207 (16)	0.0157 (4)
C5A_2	0.27205 (19)	0.23093 (15)	0.30899 (16)	0.0152 (4)
C6_2	0.36855 (19)	0.18113 (14)	0.37328 (16)	0.0149 (4)
C6A_2	0.46335 (19)	0.23343 (14)	0.43777 (17)	0.0155 (4)
C7_2	0.5642 (2)	0.19459 (15)	0.50217 (17)	0.0171 (4)
H7_2	0.573853	0.130840	0.503783	0.021*
C8_2	0.6496 (2)	0.24795 (15)	0.56324 (17)	0.0179 (4)
H8_2	0.717202	0.220815	0.607165	0.021*
C9_2	0.6371 (2)	0.34219 (15)	0.56086 (17)	0.0166 (4)
C10_2	0.5381 (2)	0.38304 (14)	0.49622 (16)	0.0149 (4)
C10A_2	0.45264 (19)	0.32757 (14)	0.43611 (16)	0.0142 (4)
O11_2	0.35792 (13)	0.37211 (9)	0.37575 (11)	0.0154 (3)
C11A_2	0.26721 (19)	0.32619 (15)	0.31288 (16)	0.0149 (4)
C12_2	0.17537 (19)	0.37960 (15)	0.25460 (17)	0.0174 (5)
C12A_2	0.08667 (19)	0.32959 (14)	0.18777 (17)	0.0162 (4)
C13_2	0.0193 (2)	0.34614 (17)	−0.05354 (18)	0.0243 (5)
H13A_2	0.035750	0.410378	−0.064317	0.037*
H13B_2	−0.020293	0.319796	−0.119763	0.037*
H13C_2	0.099284	0.315045	−0.031762	0.037*
C14_2	−0.1887 (2)	0.39184 (16)	0.00810 (19)	0.0231 (5)
H14A_2	−0.242199	0.385921	0.065903	0.035*
H14B_2	−0.235384	0.370661	−0.058027	0.035*
H14C_2	−0.165275	0.455306	0.000533	0.035*
O15_2	0.17522 (14)	0.09498 (10)	0.23611 (12)	0.0188 (3)
H15_2	0.235733	0.073547	0.276462	0.028*
O16_2	0.36869 (14)	0.09641 (10)	0.37326 (11)	0.0185 (3)

O17_2	0.71448 (13)	0.40131 (10)	0.61783 (12)	0.0185 (3)	
C18_2	0.8165 (2)	0.36517 (16)	0.68843 (17)	0.0202 (5)	
H18A_2	0.873047	0.330205	0.647938	0.030*	
H18B_2	0.782263	0.325871	0.740454	0.030*	
H18C_2	0.864000	0.414731	0.725168	0.030*	
O19_2	0.52116 (14)	0.47341 (10)	0.48892 (12)	0.0197 (3)	
H19_2	0.570615	0.499177	0.535563	0.030*	
C20_2	0.1758 (2)	0.48402 (14)	0.26600 (18)	0.0209 (5)	
C21_2	0.0906 (4)	0.5329 (3)	0.1751 (4)	0.0166 (8)	0.5
H21_2	0.101463	0.518775	0.103741	0.020*	0.5
C21B_2	0.0423 (5)	0.5244 (3)	0.2329 (5)	0.0271 (11)	0.5
H21B_2	−0.028598	0.496895	0.259544	0.032*	0.5
C22_2	0.004 (2)	0.5934 (16)	0.1942 (15)	0.030 (3)	0.5
H22A_2	−0.008289	0.608386	0.265074	0.037*	0.5
H22B_2	−0.046065	0.621701	0.136995	0.037*	0.5
C22B_2	0.022 (2)	0.5936 (15)	0.1710 (15)	0.045 (4)	0.5
H22C_2	0.091559	0.622425	0.143254	0.054*	0.5
H22D_2	−0.061284	0.615457	0.153394	0.054*	0.5
C23_2	0.1694 (3)	0.51099 (16)	0.3812 (2)	0.0372 (6)	
H23A_2	0.095065	0.483015	0.407163	0.056*	
H23B_2	0.162826	0.576792	0.386166	0.056*	
H23C_2	0.246221	0.490474	0.424207	0.056*	
C24_2	0.2965 (3)	0.52333 (17)	0.2285 (2)	0.0374 (7)	
H24A_2	0.370556	0.499658	0.272679	0.056*	
H24B_2	0.294861	0.589264	0.234377	0.056*	
H24C_2	0.301451	0.506234	0.154607	0.056*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0154 (7)	0.0140 (7)	0.0165 (7)	0.0001 (5)	−0.0043 (6)	0.0001 (6)
C2	0.0139 (10)	0.0208 (11)	0.0155 (10)	−0.0014 (8)	−0.0040 (8)	−0.0021 (8)
C3	0.0141 (10)	0.0176 (11)	0.0214 (11)	−0.0031 (8)	0.0008 (8)	−0.0047 (9)
C4	0.0164 (10)	0.0151 (10)	0.0204 (11)	−0.0022 (8)	0.0029 (8)	−0.0013 (8)
C4A	0.0165 (10)	0.0150 (11)	0.0147 (10)	−0.0012 (8)	0.0034 (8)	−0.0013 (8)
C5	0.0171 (11)	0.0126 (10)	0.0157 (10)	−0.0015 (8)	0.0027 (8)	0.0010 (8)
C6	0.0162 (11)	0.0160 (11)	0.0138 (10)	0.0002 (8)	0.0027 (8)	0.0009 (8)
C6A	0.0178 (11)	0.0136 (10)	0.0135 (10)	0.0000 (8)	0.0020 (8)	0.0008 (8)
C7	0.0199 (11)	0.0154 (10)	0.0166 (10)	0.0013 (8)	0.0007 (8)	0.0027 (8)
C8	0.0174 (10)	0.0190 (11)	0.0166 (10)	0.0039 (9)	−0.0012 (8)	0.0022 (9)
C9	0.0146 (10)	0.0190 (11)	0.0137 (10)	−0.0003 (8)	0.0006 (8)	−0.0023 (8)
C10	0.0185 (10)	0.0124 (10)	0.0125 (9)	−0.0001 (8)	0.0031 (8)	0.0003 (8)
C10A	0.0149 (10)	0.0170 (10)	0.0103 (9)	0.0039 (8)	0.0008 (8)	0.0025 (8)
O11	0.0162 (7)	0.0119 (7)	0.0159 (7)	−0.0006 (5)	−0.0044 (6)	0.0023 (6)
C11A	0.0132 (10)	0.0150 (10)	0.0120 (10)	−0.0001 (7)	0.0014 (8)	−0.0006 (8)
C12	0.0176 (10)	0.0123 (11)	0.0127 (10)	−0.0002 (7)	0.0033 (8)	0.0006 (8)
C12A	0.0136 (10)	0.0156 (10)	0.0120 (10)	0.0009 (8)	0.0019 (8)	0.0003 (8)
C13	0.0175 (11)	0.0241 (12)	0.0207 (11)	0.0009 (8)	−0.0031 (8)	0.0003 (9)

C14	0.0208 (11)	0.0247 (12)	0.0189 (11)	0.0000 (9)	0.0003 (8)	−0.0001 (9)
O15	0.0224 (8)	0.0119 (7)	0.0229 (8)	−0.0022 (6)	−0.0056 (6)	0.0038 (6)
O16	0.0248 (8)	0.0127 (8)	0.0229 (8)	−0.0007 (6)	−0.0039 (6)	0.0046 (6)
O17	0.0195 (7)	0.0172 (8)	0.0200 (7)	−0.0002 (6)	−0.0074 (6)	−0.0005 (6)
C18	0.0191 (11)	0.0229 (12)	0.0212 (11)	0.0021 (9)	−0.0071 (9)	−0.0022 (9)
O19	0.0199 (7)	0.0125 (7)	0.0172 (7)	−0.0009 (6)	−0.0048 (6)	−0.0003 (6)
C5A	0.0163 (10)	0.0147 (11)	0.0142 (10)	0.0002 (8)	0.0029 (8)	0.0015 (8)
C20	0.0166 (10)	0.0115 (10)	0.0176 (10)	0.0005 (8)	−0.0019 (8)	0.0023 (8)
C21	0.0263 (11)	0.0154 (10)	0.0214 (11)	−0.0004 (8)	0.0057 (9)	0.0002 (9)
C22	0.0248 (12)	0.0244 (12)	0.0327 (14)	0.0077 (10)	0.0062 (10)	0.0020 (10)
C23	0.0170 (10)	0.0157 (10)	0.0208 (10)	0.0014 (8)	0.0020 (8)	0.0043 (8)
C24	0.0288 (12)	0.0119 (10)	0.0282 (12)	−0.0028 (9)	−0.0093 (9)	0.0043 (9)
O1_2	0.0175 (7)	0.0180 (8)	0.0207 (8)	0.0011 (6)	−0.0062 (6)	0.0006 (6)
C2_2	0.0154 (10)	0.0232 (11)	0.0174 (10)	−0.0022 (8)	−0.0036 (8)	0.0022 (9)
C3_2	0.0146 (10)	0.0245 (11)	0.0215 (11)	−0.0059 (9)	−0.0023 (8)	−0.0008 (9)
C4_2	0.0168 (10)	0.0176 (11)	0.0217 (11)	−0.0039 (8)	0.0010 (8)	0.0006 (9)
C4A_2	0.0149 (10)	0.0192 (11)	0.0145 (10)	−0.0037 (8)	0.0020 (8)	−0.0002 (8)
C5_2	0.0180 (11)	0.0136 (10)	0.0157 (10)	−0.0024 (8)	0.0034 (8)	0.0014 (8)
C5A_2	0.0165 (10)	0.0157 (11)	0.0135 (10)	−0.0008 (8)	0.0019 (8)	0.0006 (8)
C6_2	0.0175 (10)	0.0141 (10)	0.0136 (10)	−0.0001 (8)	0.0039 (8)	0.0006 (8)
C6A_2	0.0167 (10)	0.0161 (11)	0.0139 (10)	0.0011 (8)	0.0027 (8)	0.0007 (8)
C7_2	0.0183 (10)	0.0139 (10)	0.0187 (11)	0.0012 (8)	0.0002 (8)	0.0025 (8)
C8_2	0.0166 (10)	0.0177 (11)	0.0186 (11)	0.0033 (8)	−0.0022 (8)	0.0031 (9)
C9_2	0.0153 (10)	0.0190 (11)	0.0152 (10)	−0.0025 (8)	0.0005 (8)	−0.0016 (8)
C10_2	0.0180 (10)	0.0124 (10)	0.0143 (10)	0.0006 (8)	0.0018 (8)	0.0009 (8)
C10A_2	0.0150 (10)	0.0155 (10)	0.0122 (10)	0.0021 (8)	0.0010 (8)	0.0020 (8)
O11_2	0.0163 (7)	0.0126 (7)	0.0162 (7)	0.0004 (5)	−0.0044 (6)	−0.0002 (6)
C11A_2	0.0157 (11)	0.0139 (10)	0.0149 (10)	−0.0022 (8)	0.0004 (8)	−0.0009 (8)
C12_2	0.0167 (10)	0.0133 (11)	0.0212 (11)	0.0010 (8)	−0.0020 (9)	0.0003 (8)
C12A_2	0.0143 (10)	0.0178 (11)	0.0160 (10)	0.0023 (8)	−0.0002 (8)	0.0028 (8)
C13_2	0.0185 (11)	0.0297 (13)	0.0241 (12)	−0.0011 (9)	−0.0007 (9)	0.0055 (10)
C14_2	0.0175 (10)	0.0281 (13)	0.0227 (11)	0.0018 (9)	−0.0030 (9)	0.0050 (10)
O15_2	0.0218 (8)	0.0120 (7)	0.0212 (8)	−0.0009 (6)	−0.0042 (6)	0.0014 (6)
O16_2	0.0227 (8)	0.0118 (7)	0.0199 (7)	0.0007 (6)	−0.0029 (6)	0.0018 (6)
O17_2	0.0183 (7)	0.0152 (7)	0.0198 (8)	0.0003 (6)	−0.0072 (6)	−0.0008 (6)
C18_2	0.0170 (10)	0.0211 (11)	0.0207 (11)	0.0010 (8)	−0.0067 (8)	0.0006 (9)
O19_2	0.0236 (8)	0.0113 (7)	0.0219 (8)	0.0003 (6)	−0.0083 (6)	−0.0015 (6)
C20_2	0.0211 (11)	0.0114 (10)	0.0281 (12)	0.0010 (8)	−0.0068 (9)	−0.0004 (9)
C21_2	0.021 (2)	0.010 (2)	0.018 (2)	−0.0030 (18)	0.0004 (18)	0.0042 (16)
C21B_2	0.022 (2)	0.019 (2)	0.038 (3)	0.0019 (19)	−0.008 (2)	−0.002 (2)
C22_2	0.028 (4)	0.027 (5)	0.035 (5)	0.012 (3)	−0.003 (4)	−0.001 (4)
C22B_2	0.046 (10)	0.022 (5)	0.059 (10)	0.004 (5)	−0.027 (7)	0.006 (6)
C23_2	0.0562 (16)	0.0155 (11)	0.0456 (16)	0.0025 (11)	0.0319 (13)	−0.0015 (11)
C24_2	0.0649 (18)	0.0137 (11)	0.0394 (15)	−0.0023 (11)	0.0315 (14)	0.0024 (10)

Geometric parameters (Å, º)

O1—C2	1.466 (2)	C2_2—C13_2	1.531 (3)
O1—C12A	1.355 (2)	C2_2—C14_2	1.518 (3)
C2—C3	1.504 (3)	C3_2—H3_2	0.9500
C2—C13	1.515 (3)	C3_2—C4_2	1.325 (3)
C2—C14	1.533 (3)	C4_2—H4_2	0.9500
C3—H3	0.9500	C4_2—C4A_2	1.458 (3)
C3—C4	1.328 (3)	C4A_2—C5_2	1.388 (3)
C4—H4	0.9500	C4A_2—C12A_2	1.411 (3)
C4—C4A	1.459 (3)	C5_2—C5A_2	1.411 (3)
C4A—C5	1.382 (3)	C5_2—O15_2	1.345 (3)
C4A—C12A	1.410 (3)	C5A_2—C6_2	1.443 (3)
C5—O15	1.349 (3)	C5A_2—C11A_2	1.413 (3)
C5—C5A	1.414 (3)	C6_2—C6A_2	1.452 (3)
C6—C6A	1.448 (3)	C6_2—O16_2	1.255 (3)
C6—O16	1.255 (3)	C6A_2—C7_2	1.399 (3)
C6—C5A	1.445 (3)	C6A_2—C10A_2	1.399 (3)
C6A—C7	1.400 (3)	C7_2—H7_2	0.9500
C6A—C10A	1.401 (3)	C7_2—C8_2	1.377 (3)
C7—H7	0.9500	C8_2—H8_2	0.9500
C7—C8	1.378 (3)	C8_2—C9_2	1.403 (3)
C8—H8	0.9500	C9_2—C10_2	1.400 (3)
C8—C9	1.400 (3)	C9_2—O17_2	1.356 (3)
C9—C10	1.399 (3)	C10_2—C10A_2	1.390 (3)
C9—O17	1.364 (3)	C10_2—O19_2	1.353 (3)
C10—C10A	1.393 (3)	C10A_2—O11_2	1.367 (2)
C10—O19	1.354 (3)	O11_2—C11A_2	1.365 (2)
C10A—O11	1.364 (2)	C11A_2—C12_2	1.403 (3)
O11—C11A	1.370 (2)	C12_2—C12A_2	1.409 (3)
C11A—C12	1.398 (3)	C12_2—C20_2	1.554 (3)
C11A—C5A	1.411 (3)	C13_2—H13A_2	0.9800
C12—C12A	1.413 (3)	C13_2—H13B_2	0.9800
C12—C20	1.555 (3)	C13_2—H13C_2	0.9800
C13—H13A	0.9800	C14_2—H14A_2	0.9800
C13—H13B	0.9800	C14_2—H14B_2	0.9800
C13—H13C	0.9800	C14_2—H14C_2	0.9800
C14—H14A	0.9800	O15_2—H15_2	0.8400
C14—H14B	0.9800	O17_2—C18_2	1.434 (2)
C14—H14C	0.9800	C18_2—H18A_2	0.9800
O15—H15	0.8400	C18_2—H18B_2	0.9800
O17—C18	1.435 (3)	C18_2—H18C_2	0.9800
C18—H18A	0.9800	O19_2—H19_2	0.8400
C18—H18B	0.9800	C20_2—C21_2	1.566 (5)
C18—H18C	0.9800	C20_2—C21B_2	1.555 (5)
O19—H19	0.8400	C20_2—C23_2	1.526 (3)
C20—C21	1.520 (3)	C20_2—C24_2	1.530 (3)
C20—C23	1.540 (3)	C21_2—H21_2	0.9500

C20—C24	1.544 (3)	C21_2—C22_2	1.33 (2)
C21—H21	0.9500	C21B_2—H21B_2	0.9500
C21—C22	1.317 (3)	C21B_2—C22B_2	1.29 (2)
C22—H22A	0.95 (3)	C22_2—H22A_2	0.9500
C22—H22B	0.95 (3)	C22_2—H22B_2	0.9500
C23—H23A	0.9800	C22B_2—H22C_2	0.9500
C23—H23B	0.9800	C22B_2—H22D_2	0.9500
C23—H23C	0.9800	C23_2—H23A_2	0.9800
C24—H24A	0.9800	C23_2—H23B_2	0.9800
C24—H24B	0.9800	C23_2—H23C_2	0.9800
C24—H24C	0.9800	C24_2—H24A_2	0.9800
O1_2—C2_2	1.467 (3)	C24_2—H24B_2	0.9800
O1_2—C12A_2	1.360 (2)	C24_2—H24C_2	0.9800
C2_2—C3_2	1.500 (3)		

C12A—O1—C2	119.84 (16)	C3_2—C2_2—C14_2	113.27 (18)
O1—C2—C3	110.01 (17)	C14_2—C2_2—C13_2	111.13 (19)
O1—C2—C13	104.27 (17)	C2_2—C3_2—H3_2	120.2
O1—C2—C14	108.31 (17)	C4_2—C3_2—C2_2	119.6 (2)
C3—C2—C13	112.53 (18)	C4_2—C3_2—H3_2	120.2
C3—C2—C14	109.97 (18)	C3_2—C4_2—H4_2	120.0
C13—C2—C14	111.53 (18)	C3_2—C4_2—C4A_2	120.0 (2)
C2—C3—H3	119.9	C4A_2—C4_2—H4_2	120.0
C4—C3—C2	120.21 (19)	C5_2—C4A_2—C4_2	121.5 (2)
C4—C3—H3	119.9	C5_2—C4A_2—C12A_2	119.04 (19)
C3—C4—H4	120.0	C12A_2—C4A_2—C4_2	119.2 (2)
C3—C4—C4A	120.0 (2)	C4A_2—C5_2—C5A_2	120.08 (19)
C4A—C4—H4	120.0	O15_2—C5_2—C4A_2	118.74 (18)
C5—C4A—C4	122.18 (19)	O15_2—C5_2—C5A_2	121.18 (19)
C5—C4A—C12A	118.84 (19)	C5_2—C5A_2—C6_2	120.81 (19)
C12A—C4A—C4	118.82 (19)	C5_2—C5A_2—C11A_2	118.03 (19)
C4A—C5—C5A	120.23 (19)	C11A_2—C5A_2—C6_2	121.15 (19)
O15—C5—C4A	118.31 (19)	C5A_2—C6_2—C6A_2	116.97 (18)
O15—C5—C5A	121.45 (19)	O16_2—C6_2—C5A_2	120.81 (19)
O16—C6—C6A	122.13 (19)	O16_2—C6_2—C6A_2	122.22 (19)
O16—C6—C5A	121.07 (19)	C7_2—C6A_2—C6_2	123.42 (19)
C5A—C6—C6A	116.79 (18)	C10A_2—C6A_2—C6_2	118.25 (19)
C7—C6A—C6	122.78 (19)	C10A_2—C6A_2—C7_2	118.3 (2)
C7—C6A—C10A	118.93 (19)	C6A_2—C7_2—H7_2	119.7
C10A—C6A—C6	118.30 (18)	C8_2—C7_2—C6A_2	120.6 (2)
C6A—C7—H7	119.8	C8_2—C7_2—H7_2	119.7
C8—C7—C6A	120.4 (2)	C7_2—C8_2—H8_2	119.9
C8—C7—H7	119.8	C7_2—C8_2—C9_2	120.3 (2)
C7—C8—H8	120.0	C9_2—C8_2—H8_2	119.9
C7—C8—C9	120.0 (2)	C10_2—C9_2—C8_2	120.43 (19)
C9—C8—H8	120.0	O17_2—C9_2—C8_2	125.52 (19)
C10—C9—C8	120.86 (19)	O17_2—C9_2—C10_2	114.05 (18)
O17—C9—C8	124.86 (19)	C10A_2—C10_2—C9_2	118.09 (19)

O17—C9—C10	114.27 (18)	O19_2—C10_2—C9_2	123.63 (19)
C10A—C10—C9	118.21 (18)	O19_2—C10_2—C10A_2	118.28 (18)
O19—C10—C9	123.27 (19)	C10_2—C10A_2—C6A_2	122.27 (19)
O19—C10—C10A	118.52 (18)	O11_2—C10A_2—C6A_2	122.90 (19)
C10—C10A—C6A	121.54 (18)	O11_2—C10A_2—C10_2	114.82 (18)
O11—C10A—C6A	122.95 (18)	C11A_2—O11_2—C10A_2	121.19 (17)
O11—C10A—C10	115.51 (18)	O11_2—C11A_2—C5A_2	119.48 (18)
C10A—O11—C11A	121.40 (16)	O11_2—C11A_2—C12_2	115.73 (18)
O11—C11A—C12	117.14 (18)	C12_2—C11A_2—C5A_2	124.78 (19)
O11—C11A—C5A	118.90 (18)	C11A_2—C12_2—C12A_2	113.75 (19)
C12—C11A—C5A	123.95 (19)	C11A_2—C12_2—C20_2	121.16 (18)
C11A—C12—C12A	114.45 (18)	C12A_2—C12_2—C20_2	125.09 (19)
C11A—C12—C20	126.87 (18)	O1_2—C12A_2—C4A_2	117.76 (19)
C12A—C12—C20	118.67 (18)	O1_2—C12A_2—C12_2	117.83 (19)
O1—C12A—C4A	118.63 (18)	C12_2—C12A_2—C4A_2	124.3 (2)
O1—C12A—C12	117.26 (18)	C2_2—C13_2—H13A_2	109.5
C4A—C12A—C12	124.03 (19)	C2_2—C13_2—H13B_2	109.5
C2—C13—H13A	109.5	C2_2—C13_2—H13C_2	109.5
C2—C13—H13B	109.5	H13A_2—C13_2—H13B_2	109.5
C2—C13—H13C	109.5	H13A_2—C13_2—H13C_2	109.5
H13A—C13—H13B	109.5	H13B_2—C13_2—H13C_2	109.5
H13A—C13—H13C	109.5	C2_2—C14_2—H14A_2	109.5
H13B—C13—H13C	109.5	C2_2—C14_2—H14B_2	109.5
C2—C14—H14A	109.5	C2_2—C14_2—H14C_2	109.5
C2—C14—H14B	109.5	H14A_2—C14_2—H14B_2	109.5
C2—C14—H14C	109.5	H14A_2—C14_2—H14C_2	109.5
H14A—C14—H14B	109.5	H14B_2—C14_2—H14C_2	109.5
H14A—C14—H14C	109.5	C5_2—O15_2—H15_2	109.5
H14B—C14—H14C	109.5	C9_2—O17_2—C18_2	117.80 (17)
C5—O15—H15	109.5	O17_2—C18_2—H18A_2	109.5
C9—O17—C18	117.48 (16)	O17_2—C18_2—H18B_2	109.5
O17—C18—H18A	109.5	O17_2—C18_2—H18C_2	109.5
O17—C18—H18B	109.5	H18A_2—C18_2—H18B_2	109.5
O17—C18—H18C	109.5	H18A_2—C18_2—H18C_2	109.5
H18A—C18—H18B	109.5	H18B_2—C18_2—H18C_2	109.5
H18A—C18—H18C	109.5	C10_2—O19_2—H19_2	109.5
H18B—C18—H18C	109.5	C12_2—C20_2—C21_2	113.4 (2)
C10—O19—H19	109.5	C12_2—C20_2—C21B_2	111.4 (2)
C5—C5A—C6	119.86 (19)	C23_2—C20_2—C12_2	110.50 (19)
C11A—C5A—C5	118.49 (19)	C23_2—C20_2—C21_2	120.3 (2)
C11A—C5A—C6	121.62 (19)	C23_2—C20_2—C21B_2	91.4 (3)
C21—C20—C12	108.51 (16)	C23_2—C20_2—C24_2	108.9 (2)
C21—C20—C23	112.43 (17)	C24_2—C20_2—C12_2	110.11 (18)
C21—C20—C24	104.60 (18)	C24_2—C20_2—C21_2	91.8 (2)
C23—C20—C12	109.34 (16)	C24_2—C20_2—C21B_2	122.7 (3)
C23—C20—C24	106.41 (17)	C20_2—C21_2—H21_2	118.8
C24—C20—C12	115.57 (17)	C22_2—C21_2—C20_2	122.4 (9)
C20—C21—H21	116.5	C22_2—C21_2—H21_2	118.8

C22—C21—C20	127.0 (2)	C20_2—C21B_2—H21B_2	118.2
C22—C21—H21	116.5	C22B_2—C21B_2—C20_2	123.6 (12)
C21—C22—H22A	120.5 (18)	C22B_2—C21B_2—H21B_2	118.2
C21—C22—H22B	122.2 (19)	C21_2—C22_2—H22A_2	120.0
H22A—C22—H22B	117 (3)	C21_2—C22_2—H22B_2	120.0
C20—C23—H23A	109.5	H22A_2—C22_2—H22B_2	120.0
C20—C23—H23B	109.5	C21B_2—C22B_2—H22C_2	120.0
C20—C23—H23C	109.5	C21B_2—C22B_2—H22D_2	120.0
H23A—C23—H23B	109.5	H22C_2—C22B_2—H22D_2	120.0
H23A—C23—H23C	109.5	C20_2—C23_2—H23A_2	109.5
H23B—C23—H23C	109.5	C20_2—C23_2—H23B_2	109.5
C20—C24—H24A	109.5	C20_2—C23_2—H23C_2	109.5
C20—C24—H24B	109.5	H23A_2—C23_2—H23B_2	109.5
C20—C24—H24C	109.5	H23A_2—C23_2—H23C_2	109.5
H24A—C24—H24B	109.5	H23B_2—C23_2—H23C_2	109.5
H24A—C24—H24C	109.5	C20_2—C24_2—H24A_2	109.5
H24B—C24—H24C	109.5	C20_2—C24_2—H24B_2	109.5
C12A_2—O1_2—C2_2	119.05 (16)	C20_2—C24_2—H24C_2	109.5
O1_2—C2_2—C3_2	109.96 (18)	H24A_2—C24_2—H24B_2	109.5
O1_2—C2_2—C13_2	108.29 (17)	H24A_2—C24_2—H24C_2	109.5
O1_2—C2_2—C14_2	103.65 (17)	H24B_2—C24_2—H24C_2	109.5
C3_2—C2_2—C13_2	110.26 (19)		

O1—C2—C3—C4	30.5 (3)	C2_2—C3_2—C4_2—C4A_2	5.5 (3)
C2—O1—C12A—C4A	26.1 (3)	C3_2—C4_2—C4A_2—C5_2	−171.6 (2)
C2—O1—C12A—C12	−157.12 (17)	C3_2—C4_2—C4A_2—C12A_2	13.6 (3)
C2—C3—C4—C4A	−5.6 (3)	C4_2—C4A_2—C5_2—C5A_2	−175.24 (18)
C3—C4—C4A—C5	171.99 (19)	C4_2—C4A_2—C5_2—O15_2	4.6 (3)
C3—C4—C4A—C12A	−12.6 (3)	C4_2—C4A_2—C12A_2—O1_2	−2.2 (3)
C4—C4A—C5—O15	−4.6 (3)	C4_2—C4A_2—C12A_2—C12_2	173.6 (2)
C4—C4A—C5—C5A	174.56 (18)	C4A_2—C5_2—C5A_2—C6_2	179.70 (18)
C4—C4A—C12A—O1	2.4 (3)	C4A_2—C5_2—C5A_2—C11A_2	1.1 (3)
C4—C4A—C12A—C12	−174.12 (19)	C5_2—C4A_2—C12A_2—O1_2	−177.14 (18)
C4A—C5—C5A—C6	−178.24 (18)	C5_2—C4A_2—C12A_2—C12_2	−1.3 (3)
C4A—C5—C5A—C11A	−0.1 (3)	C5_2—C5A_2—C6_2—C6A_2	178.57 (18)
C5—C4A—C12A—O1	177.93 (18)	C5_2—C5A_2—C6_2—O16_2	−2.0 (3)
C5—C4A—C12A—C12	1.4 (3)	C5_2—C5A_2—C11A_2—O11_2	−179.13 (17)
C6—C6A—C7—C8	178.87 (18)	C5_2—C5A_2—C11A_2—C12_2	−0.2 (3)
C6—C6A—C10A—C10	−179.52 (18)	C5A_2—C6_2—C6A_2—C7_2	−178.49 (19)
C6—C6A—C10A—O11	−0.3 (3)	C5A_2—C6_2—C6A_2—C10A_2	2.1 (3)
C6A—C6—C5A—C5	180.00 (17)	C5A_2—C11A_2—C12_2—C12A_2	−1.3 (3)
C6A—C6—C5A—C11A	1.9 (3)	C5A_2—C11A_2—C12_2—C20_2	178.5 (2)
C6A—C7—C8—C9	0.6 (3)	C6_2—C5A_2—C11A_2—O11_2	2.3 (3)
C6A—C10A—O11—C11A	−0.3 (3)	C6_2—C5A_2—C11A_2—C12_2	−178.76 (19)
C7—C6A—C10A—C10	0.2 (3)	C6_2—C6A_2—C7_2—C8_2	−178.62 (18)
C7—C6A—C10A—O11	179.41 (18)	C6_2—C6A_2—C10A_2—C10_2	179.21 (18)

C7—C8—C9—C10	0.3 (3)	C6_2—C6A_2—C10A_2—O11_2	−0.7 (3)
C7—C8—C9—O17	−179.88 (18)	C6A_2—C7_2—C8_2—C9_2	−0.6 (3)
C8—C9—C10—C10A	−1.0 (3)	C6A_2—C10A_2—O11_2—C11A_2	0.0 (3)
C8—C9—C10—O19	178.67 (19)	C7_2—C6A_2—C10A_2—C10_2	−0.2 (3)
C8—C9—O17—C18	3.8 (3)	C7_2—C6A_2—C10A_2—O11_2	179.82 (18)
C9—C10—C10A—C6A	0.7 (3)	C7_2—C8_2—C9_2—C10_2	−0.2 (3)
C9—C10—C10A—O11	−178.59 (17)	C7_2—C8_2—C9_2—O17_2	179.52 (19)
C10—C9—O17—C18	−176.38 (17)	C8_2—C9_2—C10_2—C10A_2	0.7 (3)
C10—C10A—O11—C11A	178.99 (16)	C8_2—C9_2—C10_2—O19_2	−179.11 (19)
C10A—C6A—C7—C8	−0.8 (3)	C8_2—C9_2—O17_2—C18_2	−1.4 (3)
C10A—O11—C11A—C12	−179.40 (16)	C9_2—C10_2—C10A_2—C6A_2	−0.5 (3)
C10A—O11—C11A—C5A	1.7 (3)	C9_2—C10_2—C10A_2—O11_2	179.42 (17)
O11—C11A—C12—C12A	−178.88 (16)	C10_2—C9_2—O17_2—C18_2	178.34 (17)
O11—C11A—C12—C20	0.2 (3)	C10_2—C10A_2—O11_2—C11A_2	−179.90 (16)
O11—C11A—C5A—C5	179.38 (17)	C10A_2—C6A_2—C7_2—C8_2	0.8 (3)
O11—C11A—C5A—C6	−2.5 (3)	C10A_2—O11_2—C11A_2—C5A_2	−0.8 (3)
C11A—C12—C12A—O1	−177.53 (16)	C10A_2—O11_2—C11A_2—C12_2	−179.85 (17)
C11A—C12—C12A—C4A	−1.0 (3)	O11_2—C11A_2—C12_2—C12A_2	177.64 (17)
C11A—C12—C20—C21	119.1 (2)	O11_2—C11A_2—C12_2—C20_2	−2.5 (3)
C11A—C12—C20—C23	−117.9 (2)	C11A_2—C5A_2—C6_2—C6A_2	−2.9 (3)
C11A—C12—C20—C24	2.1 (3)	C11A_2—C5A_2—C6_2—O16_2	176.54 (19)
C12—C11A—C5A—C5	0.5 (3)	C11A_2—C12_2—C12A_2—O1_2	177.95 (17)
C12—C11A—C5A—C6	178.64 (18)	C11A_2—C12_2—C12A_2—C4A_2	2.1 (3)
C12—C20—C21—C22	129.5 (2)	C11A_2—C12_2—C20_2—C21_2	164.8 (3)
C12A—O1—C2—C3	−41.4 (2)	C11A_2—C12_2—C20_2—C21B_2	−156.8 (3)
C12A—O1—C2—C13	−162.25 (16)	C11A_2—C12_2—C20_2—C23_2	−56.8 (3)
C12A—O1—C2—C14	78.9 (2)	C11A_2—C12_2—C20_2—C24_2	63.6 (3)
C12A—C4A—C5—O15	179.98 (18)	C12_2—C20_2—C21_2—C22_2	128.5 (13)
C12A—C4A—C5—C5A	−0.8 (3)	C12_2—C20_2—C21B_2—C22B_2	−133.2 (12)
C12A—C12—C20—C21	−61.8 (2)	C12A_2—O1_2—C2_2—C3_2	44.4 (2)
C12A—C12—C20—C23	61.1 (2)	C12A_2—O1_2—C2_2—C13_2	−76.1 (2)
C12A—C12—C20—C24	−178.89 (18)	C12A_2—O1_2—C2_2—C14_2	165.80 (17)
C13—C2—C3—C4	146.3 (2)	C12A_2—C4A_2—C5_2—C5A_2	−0.5 (3)
C14—C2—C3—C4	−88.7 (2)	C12A_2—C4A_2—C5_2—O15_2	179.34 (18)
O15—C5—C5A—C6	0.9 (3)	C12A_2—C12_2—C20_2—C21_2	−15.3 (4)
O15—C5—C5A—C11A	179.09 (18)	C12A_2—C12_2—C20_2—C21B_2	23.0 (4)
O16—C6—C6A—C7	−0.9 (3)	C12A_2—C12_2—C20_2—C23_2	123.1 (2)
O16—C6—C6A—C10A	178.77 (19)	C12A_2—C12_2—C20_2—C24_2	−116.5 (2)
O16—C6—C5A—C5	0.7 (3)	C13_2—C2_2—C3_2—C4_2	86.9 (3)

O16—C6—C5A—C11A	−177.38 (19)	C14_2—C2_2—C3_2—C4_2	−147.9 (2)
O17—C9—C10—C10A	179.24 (17)	O15_2—C5_2—C5A_2—C6_2	−0.1 (3)
O17—C9—C10—O19	−1.1 (3)	O15_2—C5_2—C5A_2—C11A_2	−178.65 (18)
O19—C10—C10A—C6A	−178.94 (18)	O16_2—C6_2—C6A_2—C7_2	2.1 (3)
O19—C10—C10A—O11	1.8 (3)	O16_2—C6_2—C6A_2—C10A_2	−177.35 (19)
C5A—C6—C6A—C7	179.82 (18)	O17_2—C9_2—C10_2—C10A_2	−179.01 (17)
C5A—C6—C6A—C10A	−0.5 (3)	O17_2—C9_2—C10_2—O19_2	1.1 (3)
C5A—C11A—C12—C12A	0.0 (3)	O19_2—C10_2—C10A_2—C6A_2	179.34 (19)
C5A—C11A—C12—C20	179.06 (19)	O19_2—C10_2—C10A_2—O11_2	−0.7 (3)
C20—C12—C12A—O1	3.3 (3)	C20_2—C12_2—C12A_2—O1_2	−1.9 (3)
C20—C12—C12A—C4A	179.87 (18)	C20_2—C12_2—C12A_2—C4A_2	−177.8 (2)
C23—C20—C21—C22	8.5 (3)	C23_2—C20_2—C21_2—C22_2	−5.5 (13)
C24—C20—C21—C22	−106.6 (3)	C23_2—C20_2—C21B_2—C22B_2	114.1 (12)
O1_2—C2_2—C3_2—C4_2	−32.4 (3)	C24_2—C20_2—C21_2—C22_2	−118.7 (13)
C2_2—O1_2—C12A_2—C4A_2	−28.2 (3)	C24_2—C20_2—C21B_2—C22B_2	0.5 (13)
C2_2—O1_2—C12A_2—C12_2	155.71 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O15—H15···O16	0.84	1.78	2.530 (2)	147
O15_2—H15_2···O16_2	0.84	1.80	2.547 (2)	147
O19—H19···O16^{i}	0.84	1.92	2.719 (2)	159
O19_2—H19_2···O16_2^{ii}	0.84	1.92	2.704 (2)	156

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