Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,7-Dimethoxy-2-phenyl-4*H*-chromen-4-one

Angannan Nallasivam,[a] Munirathinam Nethaji,[b] Nagarajan Vembu,[c]* Venkatraman Ragunathan[d] and Nagarajan Sulochana[a]

[a]Department of Chemistry, National Institute of Technology, Tiruchirappalli 620 015, India, [b]Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore 560 012, India, [c]Department of Chemistry, Urumu Dhanalakshmi College, Tiruchirappalli 620 019, India, and [d]Department of Chemistry, Kandasamy Kandar College, Velur 638 182, India
Correspondence e-mail: vembu57@yahoo.com

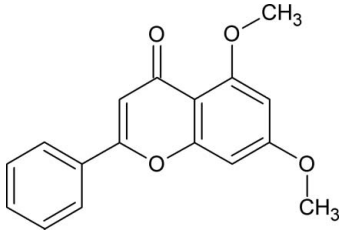
Received 26 January 2009; accepted 2 February 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.126; data-to-parameter ratio = 15.8.

The asymmetric unit of the title compound, $C_{17}H_{14}O_4$, contains two independent molecules which differ in the relative orientations of the phenyl rings with repect to the essentially planar [maximum deviations of 0.029 (2) and 0.050 (2) Å in the two molecules] chromene fused-ring system, forming dihedral angles of 10.3 (5) and 30.86 (5)° in the two molecules. The crystal structure is stabilized by weak C—H···O and C—-H···π interactions, and π–π stacking interactions.

Related literature

For the biological and pharmacological properties of benzopyrans and their derivatives, see Brooks (1998); Hatakeyama *et al.* (1988); Hyana & Saimoto (1987); Tang *et al.* (2007). For the importance of 4*H*-chromenes, see Liu *et al.* (2007); Wang, Fang *et al.* (2003); Wang, Zhang *et al.* (2003). For hydrogen bonding, see: Bernstein *et al.* (1995); Desiraju (1989); Desiraju & Steiner (1999); Etter (1990).



Experimental

Crystal data

$C_{17}H_{14}O_4$
M_r = 282.28
Triclinic, $P\overline{1}$
a = 7.3938 (17) Å
b = 11.430 (3) Å
c = 16.547 (4) Å
α = 92.414 (4)°
β = 102.723 (4)°
γ = 91.916 (4)°
V = 1361.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.35 × 0.32 × 0.29 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.967, T_{max} = 0.977
15401 measured reflections
6096 independent reflections
3897 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.126
S = 1.01
6096 reflections
386 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C17*A*—H17*A*···O1*A*	0.93	2.38	2.713 (2)	101
C13*B*—H13*E*···O11*A*^{i}	0.96	2.37	3.169 (2)	141
C19*A*—H19*A*···O11*B*^{ii}	0.93	2.57	3.256 (2)	131
C19*A*—H19*A*···O12*B*^{ii}	0.93	2.55	3.442 (2)	161
C15*B*—H15*D*···*Cg*5	0.96	2.85	3.786	166

Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $x, y-1, z$. *Cg*5 is the centroids of the C16*A*–C21*A* ring.

Table 2
π–π Stacking interactions (Å, °).

Cg_i	Cg_j	Cg_i···Cg_j	α	perp
*Cg*1	*Cg*2	3.972 (1)	10.9	3.575
*Cg*1	*Cg*4	3.646 (1)	8.1	3.578
*Cg*1	*Cg*4^{i}	3.785 (1)	8.1	3.535
*Cg*2	*Cg*3^{i}	3.792 (1)	9.9	3.599
*Cg*2	*Cg*3	3.883 (1)	9.9	3.743
*Cg*3	*Cg*4^{i}	3.769 (1)	7.1	3.516

Symmetry code: (i) $1+x, y, z$. *Cg*1, *Cg*2, *Cg*3, and *Cg*4 are the centroids of the O1*A*/C2*A*–C4*A*/C9*A*/C10*A*, O1*B*/C2*B*–C4*B*/C9*B*/C10*B*, C5*A*–C10*A* and C5*B*–C10*B* rings, respectively. α is the dihedral angle between ring planes and perp is the perpendicular distance between ring planes.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

AN thanks Dr S. Kannan and Dr B. S. Krishnamurthy, School of Chemistry, Bharathidasan University, Tiruchirappalli, and Organica Aromatics Pvt Ltd Bangalore, India, for providing laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2766).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Brooks, G. T. (1998). *Pestic. Sci.* **22**, 41–50.

Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. (1989). *Crystal Engineering: The Design of Organic Solids*, pp. 125–167. Amsterdam: Elsevier.

Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*, pp. 11–40. New York: Oxford University Press.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Hatakeyama, S., Ochi, N., Numata, H. & Takano, S. (1988). *J. Chem. Soc. Chem. Commun.* pp. 1022–1024.

Hyana, T. & Saimoto, H. (1987). Jpn Patent JP 621 812 768.

Liu, C.-B., Chen, Y.-H., Zhou, X.-Y., Ding, L. & Wen, H.-L. (2007). *Acta Cryst.* E**63**, o90–o91.

Sheldrick, G. M. (1996). *SADABS*. University of Gottingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tang, Q.-G., Wu, W.-Y., He, W., Sun, H.-S. & Guo, C. (2007). *Acta Cryst.* E**63**, o1437–o1438.

Wang, J.-F., Fang, M.-J., Huang, H.-Q., Li, G.-L., Su, W.-J. & Zhao, Y.-F. (2003). *Acta Cryst.* E**59**, o1517–o1518.

Wang, J.-F., Zhang, Y.-J., Fang, M.-J., Huang, Y.-J., Wei, Z.-B., Zheng, Z.-H., Su, W.-J. & Zhao, Y.-F. (2003). *Acta Cryst.* E**59**, o1244–o1245.

supporting information

Acta Cryst. (2009). E**65**, o504–o505 [doi:10.1107/S1600536809003948]

5,7-Dimethoxy-2-phenyl-4*H*-chromen-4-one

Angannan Nallasivam, Munirathinam Nethaji, Nagarajan Vembu, Venkatraman Ragunathan and Nagarajan Sulochana

S1. Comment

Chromenes (benzopyrans) and their derivatives have numerous biological and pharmacological properties (Tang *et al.*, 2007) such as antisterility (Brooks, 1998) and anticancer activity (Hyana & Saimoto, 1987). In addition, polyfunctionalized chromene units are present in numerous natural products (Hatakeyama *et al.*, 1988). 4*H*-chromenes are important synthons for some natural products (Liu *et al.*, 2007). As a part of our structural investigations on 4*H*-chromene derivatives and compounds containing the benzopyran fragment, the single-crystal X-ray diffraction study on the title compound was carried out.

The two molecules (A and B) in the asymmetric unit are shown in Fig. 1. In each molecule, the chromene ring is essentially planar as found in the related chromene derivatives (Wang, Zhang *et al.*, 2003; Wang, Fang *et al.*, 2003). In the title compound, the maximun deviation for the chromene ring in each molecule is 0.029 (2) and 0.050 (2) Å, for atoms C4A and C4B, respectively. The dihedral angle between the chromene ring mean-plane and the phenyl ring is 10.3 (5)° in molecule A and 30.86 (5)° in molecule B. The methoxy substituent at C5 forms dihedral angles of 8.4 (1) and 2.1 (1)° in molecules A and B, respectively. The methoxy substituent at C7 forms dihedral angles of 1.75 (1) & 12.09 (8)° in molecules A and B, respectively.

In the crystal structure (see Fig.2), the intramolecular C17A–H17A···O1A interaction generates a S(5) ring motif (Bernstein *et al.*, 1995; Etter, 1990) and the C19A–H19A···O11B^{ii} and C19A—H19A···O12B^{ii} interactions constitute two bifurcated weak hydrogen bonds bonds that generate a $R^2_1(6)$ ring motif. In addition, the crystal structure contains significant C—H···π interactions (Table 1) and π..π interactions (Table 2) whose distances agree with those described by Desiraju & Steiner (1999) and Desiraju (1989).

S2. Experimental

In to the RBF, a suspension of chrysin (1 g, 3.93 mmol) and potassium carbonate (1.62 g, 11.81 mmol) in dimethyl formamide (10 ml) were added. The reaction mixture was heated to 383 K for 2–3 hrs. The reaction mixture was cooled to 313 K and methyl iodide (10 ml, 15.74 mmol) was slowly added with the help of dropping funnel. The reaction mixture was maintained for 8–9 hr at 313 K and monitored by HPLC. After completion of the reaction, the contents were quenched with water and stirred for 30–45 min at 303 K. The crude solid obtained was filtered and washed with plenty of water followed by methanol and dried under vacuum at 343 K. The compound was purified by column chromatography using ethyl acetate: n-hexane (30:70) as diluent. All the fractions were analyzed by HPLC. The highly pure column fractions were mixed and concentrated in a rotary evaporator. The resulting product was recrystallized from dichloro-methane: n-hexane mixture (10 ml each). The obtained crystals were washed with n-hexane and dried under vacuum at 348 K. Yield: 70%

S3. Refinement

All H atoms were observed in a difference Fourier map. However, they were placed in idealized positions with C–H = 0.93 and 0.96 Å for aryl and methyl H, respectively. Their isotropic displacement parameters were tied to common free variables which were refined in subsequent cycles (for the aryl H-atoms the free variable was set to 0.06 which refined to 0.0716 (16) Å^2 and for the methyl H-atoms the free variable was set to 0.07 and refined to 0.0795 (19) Å^2).

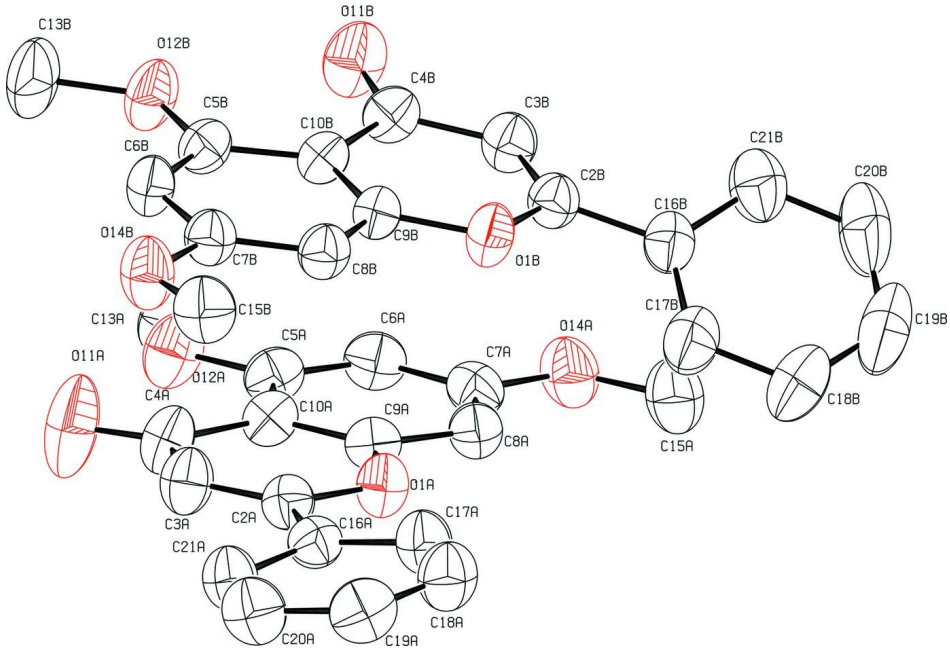


Figure 1

The asymmetric unit of the title compound with displacement ellipsoids shown at the 50% probability level for all non-H atoms. H-atoms are drawn as spheres of arbitrary radius.

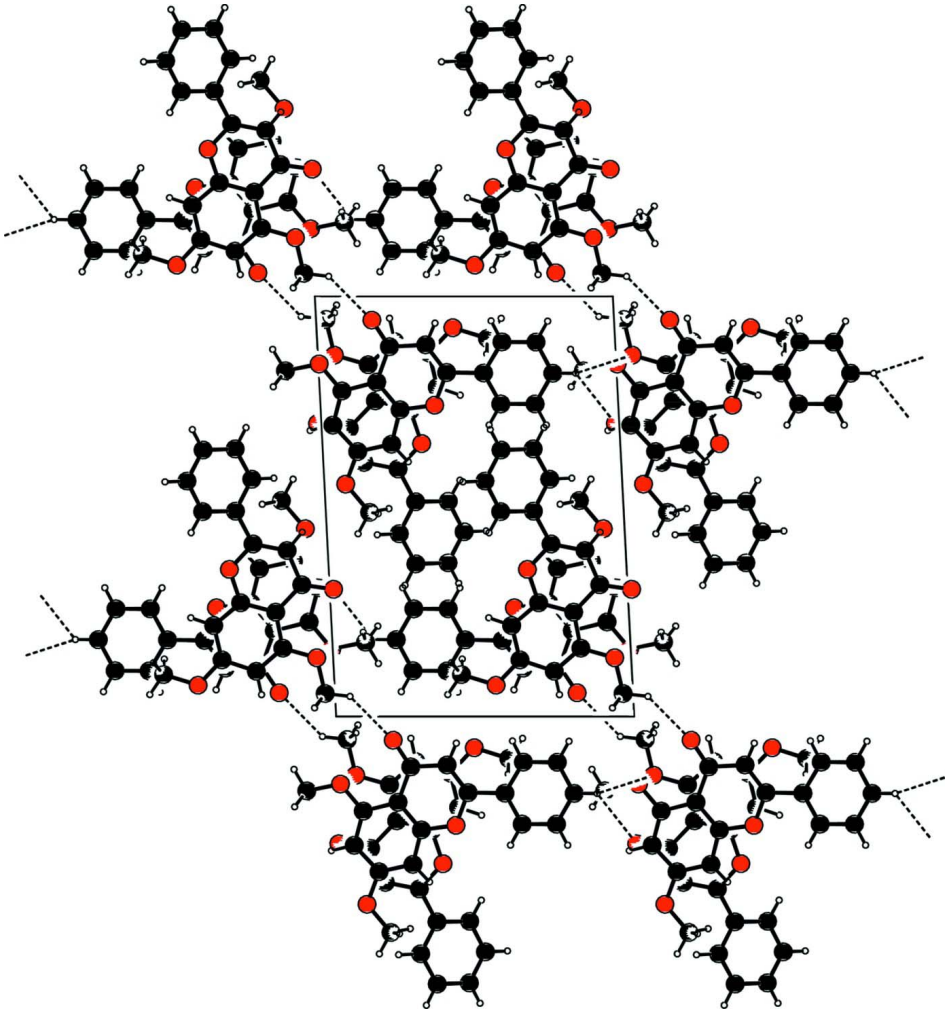


Figure 2

Part of the crystal structure viewed along the *a*-axis. Dashed lines represent weak C–H···O interactions.

5,7-Dimethoxy-2-phenyl-4*H*-chromen-4-one

Crystal data

$C_{17}H_{14}O_4$
$M_r = 282.28$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3938\ (17)$ Å
$b = 11.430\ (3)$ Å
$c = 16.547\ (4)$ Å
$\alpha = 92.414\ (4)°$
$\beta = 102.723\ (4)°$
$\gamma = 91.916\ (4)°$
$V = 1361.5\ (6)$ Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.377$ Mg m^{-3}
Melting point = 413–415 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 547 reflections
$\theta = 2.7–27.0°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Rectangular, colourless
0.35 × 0.32 × 0.29 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.3 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.967, T_{max} = 0.977

15401 measured reflections
6096 independent reflections
3897 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.0°, θ_{min} = 2.1°
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.126
S = 1.01
6096 reflections
386 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
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.201P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.17 e $Å^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.051 (3)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.84670 (17)	0.61660 (10)	0.25891 (7)	0.0474 (3)
C2A	0.7660 (2)	0.57055 (15)	0.18112 (10)	0.0424 (4)
C3A	0.7470 (3)	0.63612 (16)	0.11527 (11)	0.0511 (5)
H3A	0.6915	0.6014	0.0635	0.0716 (16)*
C4A	0.8077 (3)	0.75819 (17)	0.11977 (12)	0.0534 (5)
C5A	0.9527 (3)	0.92519 (15)	0.22512 (12)	0.0482 (4)
C6A	1.0295 (3)	0.96129 (16)	0.30639 (12)	0.0506 (5)
H6A	1.0710	1.0390	0.3193	0.0716 (16)*
C7A	1.0455 (3)	0.88267 (16)	0.36930 (11)	0.0471 (4)
C8A	0.9833 (2)	0.76767 (15)	0.35194 (11)	0.0459 (4)
H8A	0.9931	0.7148	0.3937	0.0716 (16)*
C9A	0.9053 (2)	0.73324 (14)	0.26986 (11)	0.0416 (4)
C10A	0.8873 (2)	0.80697 (15)	0.20411 (11)	0.0437 (4)
O11A	0.7924 (3)	0.81279 (13)	0.05644 (9)	0.0885 (6)

O12A	0.9347 (2)	0.99695 (11)	0.16087 (8)	0.0626 (4)
C13A	1.0217 (3)	1.11127 (17)	0.17698 (14)	0.0670 (6)
H13A	0.9654	1.1550	0.2150	0.0795 (19)*
H13B	1.0068	1.1507	0.1260	0.0795 (19)*
H13C	1.1515	1.1051	0.2008	0.0795 (19)*
O14A	1.1259 (2)	0.92860 (11)	0.44691 (8)	0.0607 (4)
C15A	1.1399 (3)	0.85301 (18)	0.51405 (12)	0.0651 (6)
H15A	1.0180	0.8247	0.5172	0.0795 (19)*
H15B	1.1978	0.8955	0.5650	0.0795 (19)*
H15C	1.2131	0.7879	0.5052	0.0795 (19)*
C16A	0.7055 (2)	0.44676 (15)	0.18240 (10)	0.0419 (4)
C17A	0.7040 (3)	0.39431 (16)	0.25611 (11)	0.0525 (5)
H17A	0.7461	0.4370	0.3062	0.0716 (16)*
C18A	0.6406 (3)	0.27909 (17)	0.25620 (13)	0.0596 (5)
H18A	0.6410	0.2451	0.3063	0.0716 (16)*
C19A	0.5772 (3)	0.21453 (17)	0.18291 (13)	0.0583 (5)
H19A	0.5341	0.1372	0.1832	0.0716 (16)*
C20A	0.5780 (3)	0.26564 (17)	0.10883 (13)	0.0591 (5)
H20A	0.5342	0.2227	0.0589	0.0716 (16)*
C21A	0.6430 (3)	0.37968 (16)	0.10844 (11)	0.0521 (5)
H21A	0.6455	0.4125	0.0582	0.0716 (16)*
O1B	0.47575 (17)	0.66672 (10)	0.35030 (7)	0.0481 (3)
C2B	0.5506 (2)	0.75030 (15)	0.41057 (10)	0.0430 (4)
C3B	0.5514 (3)	0.86351 (16)	0.39514 (11)	0.0482 (4)
H3B	0.5994	0.9175	0.4389	0.0716 (16)*
C4B	0.4812 (2)	0.90719 (15)	0.31364 (11)	0.0458 (4)
C5B	0.3403 (2)	0.83268 (15)	0.16260 (11)	0.0432 (4)
C6B	0.2695 (3)	0.73982 (16)	0.10767 (11)	0.0474 (4)
H6B	0.2246	0.7531	0.0519	0.0716 (16)*
C7B	0.2645 (2)	0.62654 (15)	0.13467 (10)	0.0439 (4)
C8B	0.3323 (2)	0.60447 (15)	0.21647 (10)	0.0425 (4)
H8B	0.3293	0.5289	0.2350	0.0716 (16)*
C9B	0.4054 (2)	0.69884 (15)	0.27057 (10)	0.0397 (4)
C10B	0.4104 (2)	0.81474 (14)	0.24840 (10)	0.0407 (4)
O11B	0.4835 (2)	1.01305 (11)	0.30281 (8)	0.0661 (4)
O12B	0.34727 (19)	0.94462 (10)	0.13977 (8)	0.0578 (4)
C13B	0.2722 (4)	0.96692 (18)	0.05480 (12)	0.0726 (7)
H13D	0.3437	0.9288	0.0204	0.0795 (19)*
H13E	0.2766	1.0498	0.0478	0.0795 (19)*
H13F	0.1457	0.9371	0.0392	0.0795 (19)*
O14B	0.19111 (19)	0.54206 (11)	0.07452 (7)	0.0582 (4)
C15B	0.1540 (3)	0.42788 (16)	0.09996 (12)	0.0571 (5)
H15D	0.2679	0.3959	0.1280	0.0795 (19)*
H15E	0.0984	0.3781	0.0521	0.0795 (19)*
H15F	0.0705	0.4326	0.1369	0.0795 (19)*
C16B	0.6345 (2)	0.69686 (16)	0.48880 (11)	0.0477 (4)
C17B	0.7041 (3)	0.5870 (2)	0.48775 (13)	0.0708 (6)
H17B	0.6916	0.5444	0.4375	0.0716 (16)*

C18B	0.7922 (4)	0.5393 (2)	0.56032 (16)	0.0905 (8)
H18B	0.8401	0.4653	0.5584	0.0716 (16)*
C19B	0.8101 (4)	0.5986 (3)	0.63429 (16)	0.0886 (9)
H19B	0.8713	0.5662	0.6829	0.0716 (16)*
C20B	0.7371 (4)	0.7066 (3)	0.63683 (13)	0.0845 (8)
H20B	0.7460	0.7467	0.6877	0.0716 (16)*
C21B	0.6504 (3)	0.7572 (2)	0.56492 (12)	0.0640 (6)
H21B	0.6029	0.8313	0.5674	0.0716 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0561 (8)	0.0411 (7)	0.0418 (7)	−0.0064 (6)	0.0050 (6)	0.0036 (5)
C2A	0.0397 (10)	0.0445 (10)	0.0415 (9)	−0.0022 (8)	0.0068 (8)	0.0014 (8)
C3A	0.0570 (12)	0.0483 (11)	0.0431 (10)	−0.0088 (9)	0.0020 (9)	0.0040 (8)
C4A	0.0564 (12)	0.0493 (11)	0.0490 (11)	−0.0052 (9)	−0.0011 (9)	0.0123 (9)
C5A	0.0462 (11)	0.0412 (10)	0.0572 (11)	0.0008 (8)	0.0108 (9)	0.0066 (9)
C6A	0.0551 (12)	0.0382 (10)	0.0579 (12)	−0.0019 (8)	0.0131 (9)	−0.0017 (9)
C7A	0.0470 (11)	0.0482 (11)	0.0463 (10)	0.0001 (8)	0.0125 (8)	−0.0051 (8)
C8A	0.0487 (11)	0.0457 (11)	0.0431 (10)	0.0013 (8)	0.0098 (8)	0.0023 (8)
C9A	0.0395 (10)	0.0369 (10)	0.0479 (10)	−0.0021 (7)	0.0094 (8)	0.0004 (8)
C10A	0.0410 (10)	0.0407 (10)	0.0475 (10)	−0.0014 (8)	0.0056 (8)	0.0041 (8)
O11A	0.1298 (15)	0.0642 (10)	0.0536 (9)	−0.0281 (9)	−0.0169 (9)	0.0215 (8)
O12A	0.0768 (10)	0.0429 (8)	0.0616 (8)	−0.0094 (7)	0.0014 (7)	0.0124 (6)
C13A	0.0828 (16)	0.0411 (11)	0.0732 (14)	−0.0029 (11)	0.0086 (12)	0.0073 (10)
O14A	0.0793 (10)	0.0523 (8)	0.0473 (8)	−0.0068 (7)	0.0104 (7)	−0.0076 (6)
C15A	0.0802 (16)	0.0634 (14)	0.0472 (11)	−0.0097 (11)	0.0077 (10)	−0.0046 (10)
C16A	0.0382 (10)	0.0424 (10)	0.0455 (10)	0.0002 (8)	0.0103 (8)	0.0033 (8)
C17A	0.0615 (13)	0.0478 (11)	0.0468 (10)	−0.0043 (9)	0.0101 (9)	0.0012 (9)
C18A	0.0715 (14)	0.0504 (12)	0.0575 (12)	−0.0033 (10)	0.0150 (11)	0.0125 (10)
C19A	0.0613 (13)	0.0415 (11)	0.0712 (14)	−0.0060 (9)	0.0139 (11)	0.0045 (10)
C20A	0.0670 (14)	0.0486 (12)	0.0583 (12)	−0.0090 (10)	0.0103 (10)	−0.0081 (10)
C21A	0.0586 (12)	0.0505 (11)	0.0458 (10)	−0.0063 (9)	0.0098 (9)	0.0015 (9)
O1B	0.0634 (8)	0.0400 (7)	0.0363 (6)	0.0004 (6)	0.0005 (6)	0.0056 (5)
C2B	0.0442 (10)	0.0437 (10)	0.0389 (9)	0.0000 (8)	0.0052 (8)	−0.0002 (8)
C3B	0.0537 (12)	0.0429 (11)	0.0447 (10)	−0.0017 (8)	0.0052 (9)	−0.0014 (8)
C4B	0.0467 (11)	0.0385 (10)	0.0503 (10)	−0.0006 (8)	0.0070 (8)	0.0032 (8)
C5B	0.0458 (10)	0.0379 (10)	0.0458 (10)	0.0026 (8)	0.0086 (8)	0.0094 (8)
C6B	0.0551 (11)	0.0485 (11)	0.0368 (9)	0.0023 (9)	0.0054 (8)	0.0084 (8)
C7B	0.0466 (11)	0.0438 (10)	0.0400 (9)	0.0000 (8)	0.0070 (8)	0.0013 (8)
C8B	0.0485 (11)	0.0359 (9)	0.0427 (10)	0.0003 (8)	0.0091 (8)	0.0054 (7)
C9B	0.0419 (10)	0.0410 (10)	0.0357 (9)	0.0024 (7)	0.0066 (7)	0.0059 (7)
C10B	0.0402 (10)	0.0387 (9)	0.0419 (9)	0.0030 (7)	0.0057 (8)	0.0055 (7)
O11B	0.0908 (11)	0.0367 (8)	0.0641 (9)	−0.0052 (7)	0.0034 (8)	0.0062 (6)
O12B	0.0784 (10)	0.0411 (7)	0.0495 (7)	0.0003 (6)	0.0023 (7)	0.0152 (6)
C13B	0.112 (2)	0.0524 (13)	0.0505 (12)	0.0059 (12)	0.0084 (12)	0.0219 (10)
O14B	0.0795 (10)	0.0478 (8)	0.0414 (7)	−0.0099 (7)	0.0034 (6)	−0.0021 (6)
C15B	0.0674 (14)	0.0449 (11)	0.0562 (12)	−0.0079 (9)	0.0101 (10)	−0.0039 (9)

C16B	0.0450 (11)	0.0562 (12)	0.0401 (10)	−0.0037 (9)	0.0058 (8)	0.0066 (8)
C17B	0.0904 (17)	0.0693 (15)	0.0514 (12)	0.0209 (12)	0.0086 (11)	0.0136 (11)
C18B	0.104 (2)	0.0924 (19)	0.0740 (17)	0.0314 (16)	0.0065 (15)	0.0352 (15)
C19B	0.0694 (17)	0.131 (3)	0.0593 (15)	−0.0098 (16)	−0.0052 (12)	0.0459 (17)
C20B	0.0812 (18)	0.126 (2)	0.0370 (11)	−0.0270 (17)	−0.0028 (11)	0.0029 (13)
C21B	0.0662 (14)	0.0742 (15)	0.0465 (11)	−0.0095 (11)	0.0047 (10)	−0.0034 (10)

Geometric parameters (Å, º)

O1A—C2A	1.369 (2)	O1B—C2B	1.362 (2)
O1A—C9A	1.380 (2)	O1B—C9B	1.3773 (19)
C2A—C3A	1.333 (2)	C2B—C3B	1.329 (2)
C2A—C16A	1.472 (2)	C2B—C16B	1.474 (2)
C3A—C4A	1.445 (3)	C3B—C4B	1.450 (2)
C3A—H3A	0.9300	C3B—H3B	0.9300
C4A—O11A	1.228 (2)	C4B—O11B	1.231 (2)
C4A—C10A	1.468 (3)	C4B—C10B	1.474 (2)
C5A—O12A	1.356 (2)	C5B—O12B	1.3517 (19)
C5A—C6A	1.379 (3)	C5B—C6B	1.378 (2)
C5A—C10A	1.422 (2)	C5B—C10B	1.426 (2)
C6A—C7A	1.392 (2)	C6B—C7B	1.389 (2)
C6A—H6A	0.9300	C6B—H6B	0.9300
C7A—O14A	1.365 (2)	C7B—O14B	1.365 (2)
C7A—C8A	1.375 (2)	C7B—C8B	1.373 (2)
C8A—C9A	1.388 (2)	C8B—C9B	1.387 (2)
C8A—H8A	0.9300	C8B—H8B	0.9300
C9A—C10A	1.390 (2)	C9B—C10B	1.391 (2)
O12A—C13A	1.425 (2)	O12B—C13B	1.430 (2)
C13A—H13A	0.9600	C13B—H13D	0.9600
C13A—H13B	0.9600	C13B—H13E	0.9600
C13A—H13C	0.9600	C13B—H13F	0.9600
O14A—C15A	1.424 (2)	O14B—C15B	1.425 (2)
C15A—H15A	0.9600	C15B—H15D	0.9600
C15A—H15B	0.9600	C15B—H15E	0.9600
C15A—H15C	0.9600	C15B—H15F	0.9600
C16A—C17A	1.384 (2)	C16B—C17B	1.374 (3)
C16A—C21A	1.395 (2)	C16B—C21B	1.390 (3)
C17A—C18A	1.383 (3)	C17B—C18B	1.378 (3)
C17A—H17A	0.9300	C17B—H17B	0.9300
C18A—C19A	1.373 (3)	C18B—C19B	1.352 (4)
C18A—H18A	0.9300	C18B—H18B	0.9300
C19A—C20A	1.381 (3)	C19B—C20B	1.366 (4)
C19A—H19A	0.9300	C19B—H19B	0.9300
C20A—C21A	1.375 (3)	C20B—C21B	1.381 (3)
C20A—H20A	0.9300	C20B—H20B	0.9300
C21A—H21A	0.9300	C21B—H21B	0.9300
C2A—O1A—C9A	119.71 (13)	C2B—O1B—C9B	119.73 (13)

C3A—C2A—O1A	120.94 (16)	C3B—C2B—O1B	121.51 (15)
C3A—C2A—C16A	127.26 (16)	C3B—C2B—C16B	127.33 (16)
O1A—C2A—C16A	111.79 (14)	O1B—C2B—C16B	111.08 (15)
C2A—C3A—C4A	123.68 (17)	C2B—C3B—C4B	123.40 (17)
C2A—C3A—H3A	118.2	C2B—C3B—H3B	118.3
C4A—C3A—H3A	118.2	C4B—C3B—H3B	118.3
O11A—C4A—C3A	120.59 (18)	O11B—C4B—C3B	120.83 (16)
O11A—C4A—C10A	124.93 (17)	O11B—C4B—C10B	125.04 (16)
C3A—C4A—C10A	114.47 (16)	C3B—C4B—C10B	114.12 (15)
O12A—C5A—C6A	123.64 (16)	O12B—C5B—C6B	123.15 (15)
O12A—C5A—C10A	115.79 (16)	O12B—C5B—C10B	116.05 (15)
C6A—C5A—C10A	120.57 (17)	C6B—C5B—C10B	120.80 (15)
C5A—C6A—C7A	120.67 (17)	C5B—C6B—C7B	120.73 (16)
C5A—C6A—H6A	119.7	C5B—C6B—H6B	119.6
C7A—C6A—H6A	119.7	C7B—C6B—H6B	119.6
O14A—C7A—C8A	124.12 (17)	O14B—C7B—C8B	123.99 (16)
O14A—C7A—C6A	115.13 (16)	O14B—C7B—C6B	115.29 (15)
C8A—C7A—C6A	120.76 (17)	C8B—C7B—C6B	120.71 (16)
C7A—C8A—C9A	117.70 (16)	C7B—C8B—C9B	117.70 (16)
C7A—C8A—H8A	121.2	C7B—C8B—H8B	121.1
C9A—C8A—H8A	121.2	C9B—C8B—H8B	121.1
O1A—C9A—C8A	113.28 (15)	O1B—C9B—C8B	113.06 (14)
O1A—C9A—C10A	122.35 (15)	O1B—C9B—C10B	122.23 (15)
C8A—C9A—C10A	124.36 (16)	C8B—C9B—C10B	124.70 (15)
C9A—C10A—C5A	115.92 (16)	C9B—C10B—C5B	115.31 (15)
C9A—C10A—C4A	118.78 (16)	C9B—C10B—C4B	118.81 (15)
C5A—C10A—C4A	125.28 (16)	C5B—C10B—C4B	125.86 (15)
C5A—O12A—C13A	117.71 (15)	C5B—O12B—C13B	117.79 (14)
O12A—C13A—H13A	109.5	O12B—C13B—H13D	109.5
O12A—C13A—H13B	109.5	O12B—C13B—H13E	109.5
H13A—C13A—H13B	109.5	H13D—C13B—H13E	109.5
O12A—C13A—H13C	109.5	O12B—C13B—H13F	109.5
H13A—C13A—H13C	109.5	H13D—C13B—H13F	109.5
H13B—C13A—H13C	109.5	H13E—C13B—H13F	109.5
C7A—O14A—C15A	117.48 (15)	C7B—O14B—C15B	117.91 (13)
O14A—C15A—H15A	109.5	O14B—C15B—H15D	109.5
O14A—C15A—H15B	109.5	O14B—C15B—H15E	109.5
H15A—C15A—H15B	109.5	H15D—C15B—H15E	109.5
O14A—C15A—H15C	109.5	O14B—C15B—H15F	109.5
H15A—C15A—H15C	109.5	H15D—C15B—H15F	109.5
H15B—C15A—H15C	109.5	H15E—C15B—H15F	109.5
C17A—C16A—C21A	118.04 (16)	C17B—C16B—C21B	118.44 (18)
C17A—C16A—C2A	121.46 (16)	C17B—C16B—C2B	120.39 (17)
C21A—C16A—C2A	120.47 (15)	C21B—C16B—C2B	121.14 (18)
C18A—C17A—C16A	120.81 (18)	C16B—C17B—C18B	120.7 (2)
C18A—C17A—H17A	119.6	C16B—C17B—H17B	119.7
C16A—C17A—H17A	119.6	C18B—C17B—H17B	119.7
C19A—C18A—C17A	120.55 (18)	C19B—C18B—C17B	120.9 (3)

C19A—C18A—H18A	119.7	C19B—C18B—H18B	119.5
C17A—C18A—H18A	119.7	C17B—C18B—H18B	119.5
C18A—C19A—C20A	119.32 (18)	C18B—C19B—C20B	119.3 (2)
C18A—C19A—H19A	120.3	C18B—C19B—H19B	120.4
C20A—C19A—H19A	120.3	C20B—C19B—H19B	120.4
C21A—C20A—C19A	120.36 (18)	C19B—C20B—C21B	121.0 (2)
C21A—C20A—H20A	119.8	C19B—C20B—H20B	119.5
C19A—C20A—H20A	119.8	C21B—C20B—H20B	119.5
C20A—C21A—C16A	120.90 (17)	C20B—C21B—C16B	119.7 (2)
C20A—C21A—H21A	119.5	C20B—C21B—H21B	120.2
C16A—C21A—H21A	119.5	C16B—C21B—H21B	120.2
C9A—O1A—C2A—C3A	1.6 (2)	C9B—O1B—C2B—C3B	2.6 (2)
C9A—O1A—C2A—C16A	−177.46 (14)	C9B—O1B—C2B—C16B	−174.21 (14)
O1A—C2A—C3A—C4A	−0.1 (3)	O1B—C2B—C3B—C4B	−2.6 (3)
C16A—C2A—C3A—C4A	178.82 (18)	C16B—C2B—C3B—C4B	173.59 (17)
C2A—C3A—C4A—O11A	177.3 (2)	C2B—C3B—C4B—O11B	179.30 (18)
C2A—C3A—C4A—C10A	−2.1 (3)	C2B—C3B—C4B—C10B	−0.8 (3)
O12A—C5A—C6A—C7A	−179.36 (17)	O12B—C5B—C6B—C7B	−179.50 (16)
C10A—C5A—C6A—C7A	0.4 (3)	C10B—C5B—C6B—C7B	0.1 (3)
C5A—C6A—C7A—O14A	179.18 (16)	C5B—C6B—C7B—O14B	179.95 (16)
C5A—C6A—C7A—C8A	−0.7 (3)	C5B—C6B—C7B—C8B	−0.9 (3)
O14A—C7A—C8A—C9A	−179.62 (16)	O14B—C7B—C8B—C9B	179.06 (16)
C6A—C7A—C8A—C9A	0.3 (3)	C6B—C7B—C8B—C9B	0.0 (3)
C2A—O1A—C9A—C8A	179.66 (14)	C2B—O1B—C9B—C8B	−179.34 (15)
C2A—O1A—C9A—C10A	−0.7 (2)	C2B—O1B—C9B—C10B	1.1 (2)
C7A—C8A—C9A—O1A	−179.81 (15)	C7B—C8B—C9B—O1B	−177.72 (15)
C7A—C8A—C9A—C10A	0.6 (3)	C7B—C8B—C9B—C10B	1.8 (3)
O1A—C9A—C10A—C5A	179.54 (15)	O1B—C9B—C10B—C5B	176.98 (15)
C8A—C9A—C10A—C5A	−0.9 (3)	C8B—C9B—C10B—C5B	−2.5 (3)
O1A—C9A—C10A—C4A	−1.6 (3)	O1B—C9B—C10B—C4B	−4.5 (2)
C8A—C9A—C10A—C4A	177.98 (16)	C8B—C9B—C10B—C4B	176.01 (16)
O12A—C5A—C10A—C9A	−179.87 (15)	O12B—C5B—C10B—C9B	−178.86 (15)
C6A—C5A—C10A—C9A	0.4 (3)	C6B—C5B—C10B—C9B	1.5 (2)
O12A—C5A—C10A—C4A	1.4 (3)	O12B—C5B—C10B—C4B	2.7 (3)
C6A—C5A—C10A—C4A	−178.39 (17)	C6B—C5B—C10B—C4B	−176.92 (17)
O11A—C4A—C10A—C9A	−176.5 (2)	O11B—C4B—C10B—C9B	−175.93 (17)
C3A—C4A—C10A—C9A	2.9 (3)	C3B—C4B—C10B—C9B	4.1 (2)
O11A—C4A—C10A—C5A	2.2 (3)	O11B—C4B—C10B—C5B	2.5 (3)
C3A—C4A—C10A—C5A	−178.40 (17)	C3B—C4B—C10B—C5B	−177.47 (16)
C6A—C5A—O12A—C13A	8.5 (3)	C6B—C5B—O12B—C13B	1.8 (3)
C10A—C5A—O12A—C13A	−171.26 (17)	C10B—C5B—O12B—C13B	−177.77 (17)
C8A—C7A—O14A—C15A	−2.3 (3)	C8B—C7B—O14B—C15B	11.0 (3)
C6A—C7A—O14A—C15A	177.86 (17)	C6B—C7B—O14B—C15B	−169.90 (16)
C3A—C2A—C16A—C17A	−168.56 (19)	C3B—C2B—C16B—C17B	−148.0 (2)
O1A—C2A—C16A—C17A	10.4 (2)	O1B—C2B—C16B—C17B	28.5 (3)
C3A—C2A—C16A—C21A	9.7 (3)	C3B—C2B—C16B—C21B	30.0 (3)
O1A—C2A—C16A—C21A	−171.29 (15)	O1B—C2B—C16B—C21B	−153.46 (17)

C21A—C16A—C17A—C18A	−0.6 (3)	C21B—C16B—C17B—C18B	−1.8 (3)
C2A—C16A—C17A—C18A	177.72 (17)	C2B—C16B—C17B—C18B	176.3 (2)
C16A—C17A—C18A—C19A	−0.3 (3)	C16B—C17B—C18B—C19B	0.9 (4)
C17A—C18A—C19A—C20A	0.3 (3)	C17B—C18B—C19B—C20B	0.9 (4)
C18A—C19A—C20A—C21A	0.5 (3)	C18B—C19B—C20B—C21B	−1.8 (4)
C19A—C20A—C21A—C16A	−1.5 (3)	C19B—C20B—C21B—C16B	0.9 (4)
C17A—C16A—C21A—C20A	1.5 (3)	C17B—C16B—C21B—C20B	0.9 (3)
C2A—C16A—C21A—C20A	−176.89 (17)	C2B—C16B—C21B—C20B	−177.16 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17*A*—H17*A*···O1*A*	0.93	2.38	2.713 (2)	101
C13*B*—H13*E*···O11*A*^{i}	0.96	2.37	3.169 (2)	141
C19*A*—H19*A*···O11*B*^{ii}	0.93	2.57	3.256 (2)	131
C19*A*—H19*A*···O12*B*^{ii}	0.93	2.55	3.442 (2)	161
C13*A*—H13*A*···*Cg*5^{iii}	0.96	3.14	3.838	131
C15*A*—H15*A*···*Cg*6	0.96	3.16	4.059	156
C15*B*—H15*D*···*Cg*5	0.96	2.85	3.786	166

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