Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Gerontoxanthone I methanol solvate

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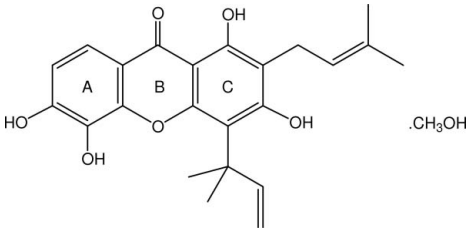
Received 23 August 2007; accepted 26 August 2007

Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.141; data-to-parameter ratio = 13.0.

A methanol solvate of gerontoxanthone I [systematic name: 4-(1,1-dimethylprop-2-enyl)-1,3,5,6-tetrahydroxy-2-(3-methylbut-2-enyl)-9*H*-xanthen-9-one methanol solvate], $C_{23}H_{24}O_6\cdot CH_3OH$, is reported. Gerontoxanthone I was isolated from the roots of *Cratoxylum formosum* ssp. *pruniflorum*. The three rings in the structure are essentially coplanar. The 3-methylbut-2-enyl side chain is equatorially attached to the benzene ring, whereas the 1-methylbut-2-enyl substituent is bisectionally attached to the benzene ring. Intramolecular O—H···O hydrogen bonds generate S(5) and S(6) ring motifs. In the crystal structure, intermolecular O—H···O hydrogen bonds and C—H···O interactions connect the molecules of gerontoxanthone I into chains along the [100] direction. The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds, weak C—H···O intra- and intermolecular interactions, and C—H···π interactions.

Related literature

For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related literature on values of bond lengths, see: Allen *et al.* (1987). For related structures, see, for example: Boonnak *et al.* (2005); Boonnak, Chantrapromma & Fun (2006); Boonnak, Karalai *et al.* (2006); Chantrapromma *et al.* (2005, 2006); Fun *et al.* (2006). For related literature on bioactivities of xanthones, see, for example: Aderson (1986); Boonnak, Karalai *et al.* (2006); Kitanov *et al.* (1988).



Experimental

Crystal data

$C_{23}H_{24}O_6\cdot CH_4O$
M_r = 428.46
Monoclinic, $P2_1/c$
a = 10.0411 (8) Å
b = 20.1500 (16) Å
c = 12.1807 (7) Å
β = 117.534 (5)°
V = 2185.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 297 (2) K
0.55 × 0.29 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.982
11274 measured reflections
3834 independent reflections
3429 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.141
S = 1.05
3834 reflections
296 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O1···O7^{i}	0.85 (3)	1.79 (3)	2.632 (2)	170 (3)
O2—H1O2···O1	0.82	2.29	2.7117 (19)	113
O2—H1O2···O4^{ii}	0.82	2.03	2.7995 (19)	155
O4—H1O4···O3	0.82	1.81	2.5505 (18)	149
O7—H1O7···O3	0.83 (2)	1.954 (19)	2.755 (2)	161 (4)
C18—H18*B*···O6	0.96	2.25	2.638 (2)	103
C19—H19*A*···O2^{iii}	0.97	2.54	3.378 (2)	145
C19—H19*A*···O4	0.97	2.50	2.8451 (19)	101
C22—H22*B*···*Cg*1^{iv}	0.96	3.10	3.705 (2)	123

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

This work was supported by the Thailand Research Fund (Direct Basic Research in Medicinal Chemistry, grant No. DBG4880019). The authors also thank Prince of Songkla University, the Malaysian Government and Universiti Sains Malaysia for a Scientific Advancement Grant Allocation (SAGA) (grant No. 304/PFIZIK/653003/A118).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2198).

  doi:10.1107/S1600536807041906

References

Aderson, E. F. (1986). *Econ. Bot.* **40**, 442–450.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

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

Boonnak, N., Chantrapromma, S., Fun, H.-K. & Karalai, C. (2005). *Acta Cryst.* E**61**, o4376–o4378.

Boonnak, N., Chantrapromma, S. & Fun, H.-K. (2006). *Acta Cryst.* E**62**, o2034–o2036.

Boonnak, N., Karalai, C., Chantrapromma, S., Ponglimanont, C., Fun, H.-K., Kanjana-Opas, A. & Laphookhieo, S. (2006). *Tetrahedron*, **62**, 8850–8859.

Chantrapromma, S., Boonnak, N., Fun, H.-K., Anjum, S. & Atta-ur-Rahman (2005). *Acta Cryst.* E**61**, o2136–o2138.

Chantrapromma, S., Boonnak, N., Fun, H.-K. & Karalai, C. (2006). *Acta Cryst.* E**62**, o360–o362.

Fun, H.-K., Ng, S.-L., Razak, I. A., Boonnak, N. & Chantrapromma, S. (2006). *Acta Cryst.* E**62**, o130–o132.

Kitanov, G. M., Assenov, I. & The Van, D. (1988). *Pharmazie*, **43**, H12–H13.

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

Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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

supplementary materials

Acta Cryst. (2007). E**63**, o3958-o3959 [**doi:10.1107/S1600536807041906**]

Gerontoxanthone I methanol solvate

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Comment

Some species of plants in the genus *Cratoxylum* have been used for the treatment of diuretic, stomachic, and tonic effects (Kitanov *et al.*, 1988), as well as for diarrhea and flatulence (Aderson, 1986). In our ongoing research of bioactive compounds from medicinal plants, the title compound, gerontoxanthone I, was isolated from a dichloromethane extract of the roots of *Cratoxylum formosum* ssp. *pruniflorum*, collected from Nhongkai Province in the northeasthern part of Thailand. As the title compound showed strong antibacterial and cytotoxic activities (Boonnak, Karalai, *et al.*, 2006), its X-ray crystal structure was determined in order to gain more information for further SAR (Structure and Activity Relationship) analysis. In our previous studies, we have reported the crystal structures of xanthone and anthraquinone compounds from the roots and barks of this plant (Boonnak *et al.*, 2005; Boonnak, Chantrapromma & Fun, 2006; Boonnak, Karalai *et al.*, 2006; Chantrapromma *et al.*, 2005; 2006; Fun *et al.*, 2006). We report here the crystal structure of the methanol solvate of gerontoxanthone I.

In the title compound (Fig. 1), the xanthone skeleton (rings A, B and C) is essentially planar, the maximum deviation from planarity being 0.043 (2) Å for atom C3. The O2—H2A···O1 and O4—H4A···O3 hydrogen bonds generate S(5) and S(6) ring motifs, respectively (Bernstein *et al.*, 1995) and help to stabilize the planarity of the structure. There are also weak intramolecular C—H···O interactions; C18—H18B···O6 and C19—H19A···O4 generate S(6) and S(5) ring motifs respectively (Table 1).

The orientation of the 3-methylbut-2-enyl [C19–C23] side chain with respect to the benzene ring *C* is indicated by the torsion angle of C13—C12—C19—C20 = −93.25 (17)°, [90.6 (2)° in the monohydrate compound (Boonnak, Chantrapromma & Fun, 2006)], indicating a (-)-synclinal conformation (Fig. 1). The 1,1-dimethylprop-2-enyl [C14–C18] substituent is attached to the benzene ring at C10 with the torsion angle C9—C10—C14—C15 of −137.06 (17)° [−52.6 (3)° in Boonnak, Chantrapromma & Fun, 2006], indicating a (-)-anticlinal conformation. Bond distances and angles in the title compound are in normal ranges (Allen *et al.*, 1987) and comparable to those reported in the gerontoxanthone I monohydrate (Boonnak, Chantrapromma & Fun, 2006) and other closely related structures (Boonnak *et al.*, 2005; Boonnak, Karalai *et al.*, 2006; Chantrapromma *et al.*, 2005; 2006; Fun *et al.*, 2006). The methanol solvent molecule is also involved in hydrogen bonds (Table 1).

In the crystal packing (Fig. 2), the gerontoxanthone I molecules are linked together into chains along the *a* axis by the intermolecular O2—H1O2···O4 hydrogen bond (symmetry code: −1 + *x*, *y*, *z*) and weak C19—H19A···O2 interaction (symmetry code: 1 + *x*, *y*, *z*) (Table 1) and are further linked to the methanol molecules by O1—H1O1···O7 (symmetry code: −1 + *x*, *y*, −1 + *z*) and O7—H1O7···O3 (symmetry code: 1 − *x*, 1 − *y*, 1 − *z*) hydrogen bonds (Table 1). This packing is different from the three dimensional crystal packing of the monohydrate compound (Boonnak, Chantrapromma & Fun, 2006). The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds, weak C—H···O intra- and intramolecular interactions (Table 1). In addition, the molecular packing is further stabilized by a C—H···π interaction between one of the methyl groups of the 3-methylbut-2-enyl side chain and the centroid of the C1–C6 benzene ring (Cg_1) (Table 1).

Experimental

Air-dried roots of *Cratoxylum formosum* ssp. *pruniflorum* (5.30 kg) were ground and extracted with CH_2Cl_2 (2x20 l for 2x5 days) at room temperature. The residue obtained after evaporation of the solvent was subjected to quick column chromatography (QCC) on silica gel, using hexane as first eluent and then increasing polarity with EtOAc and acetone, to afford 8 fractions (F1–F8). Fraction F3 was separated by CC with 10% acetone–hexane to give the title compound. Yellow needle-shaped single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvents from a $CHCl_3/CH_3OH$ (7:3 *v*/*v*) solution after several days (*M*.p. 452–453 K).

Refinement

H atoms of the methanol molecule and the H atoms attached to O5 and O7 were located in a difference map. The remaining H atoms were placed in calculated positions with O—H distance of 0.82 Å and C—H distances in the range 0.93–0.97 Å. The U_{iso}(H) values were constrained to be $1.5U_{eq}$(carrier atom) for hydroxyl and methyl H atoms and $1.2U_{eq}$(carrier atom) for the remaining H atoms. Owing to a large fraction of weak data at higher angles, the 2θ maximum was limited to 50°. A rotating group model was used for the methyl groups.

Figures

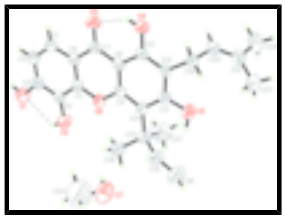

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen bonds are drawn as dashed lines.

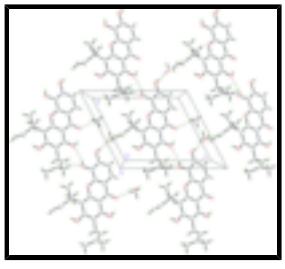

Fig. 2. The crystal packing of the title compound viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines.

4-(1,1-dimethylprop-2-enyl)-1,3,5,6-tetrahydroxy-2-(3-methylbut-2-enyl)- 9*H*-xanthen-9-one methanol solvate

Crystal data

$C_{23}H_{24}O_6{\cdot}CH_4O$	F_{000} = 912
M_r = 428.46	D_x = 1.302 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 452-453 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.0411 (8) Å	Cell parameters from 3834 reflections
b = 20.1500 (16) Å	θ = 2.3–25.0º

c = 12.1807 (7) Å	μ = 0.10 mm^{-1}
β = 117.534 (5)º	T = 297 (2) K
V = 2185.4 (3) Å^3	Needle, yellow
Z = 4	0.55 × 0.29 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer	3834 independent reflections
Radiation source: fine-focus sealed tube	3429 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 25.0º
T = 297(2) K	θ_{min} = 2.3º
ω scans	h = −11→11
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −23→23
T_{min} = 0.950, T_{max} = 0.982	l = −7→14
11274 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.141	$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2 + 0.4621P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3834 reflections	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
296 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	−0.16926 (13)	0.49056 (7)	0.21463 (12)	0.0639 (4)
H1O1	−0.222 (3)	0.5096 (14)	0.146 (3)	0.096*
O2	0.02703 (12)	0.42552 (7)	0.42133 (11)	0.0636 (4)
H1O2	−0.0623	0.4333	0.3972	0.095*
O3	0.48730 (12)	0.48078 (7)	0.25536 (11)	0.0570 (3)
O4	0.73927 (12)	0.42967 (6)	0.40403 (11)	0.0536 (3)
H1O4	0.6776	0.4502	0.3439	0.080*
O5	0.77233 (13)	0.30854 (7)	0.74175 (13)	0.0638 (4)
H5A	0.727 (3)	0.3031 (13)	0.789 (3)	0.100 (8)*
O6	0.30754 (11)	0.40989 (5)	0.47182 (9)	0.0449 (3)
O7	0.6422 (2)	0.53884 (12)	0.99678 (17)	0.0999 (6)
H1O7	0.595 (5)	0.525 (2)	0.9244 (17)	0.176 (18)*
C1	0.17612 (17)	0.50642 (8)	0.18518 (14)	0.0453 (4)
H1B	0.2105	0.5248	0.1329	0.054*
C2	0.02873 (17)	0.51419 (8)	0.15877 (14)	0.0472 (4)
H2A	−0.0361	0.5380	0.0889	0.057*
C3	−0.02461 (16)	0.48670 (8)	0.23586 (14)	0.0459 (4)
C4	0.07148 (16)	0.45290 (8)	0.34219 (14)	0.0434 (3)
C5	0.22065 (15)	0.44518 (7)	0.36748 (13)	0.0387 (3)
C6	0.27565 (16)	0.47090 (7)	0.29057 (13)	0.0395 (3)
C7	0.43129 (16)	0.45928 (7)	0.32226 (13)	0.0408 (3)
C8	0.51977 (15)	0.42168 (7)	0.43328 (13)	0.0385 (3)
C9	0.45495 (15)	0.39753 (7)	0.50637 (13)	0.0381 (3)
C10	0.53244 (16)	0.35998 (7)	0.61333 (14)	0.0419 (3)
C11	0.68362 (16)	0.34693 (7)	0.64363 (14)	0.0437 (4)
C12	0.75637 (15)	0.37038 (7)	0.57675 (14)	0.0407 (3)
C13	0.67322 (15)	0.40703 (7)	0.47158 (13)	0.0397 (3)
C14	0.46210 (18)	0.32904 (9)	0.69122 (16)	0.0530 (4)
C15	0.5654 (2)	0.33895 (13)	0.82710 (18)	0.0704 (6)
H15A	0.6029	0.3816	0.8514	0.084*
C16	0.6087 (3)	0.29370 (19)	0.9158 (3)	0.1141 (12)
H16A	0.5746	0.2502	0.8967	0.137*
H16B	0.6733	0.3054	0.9972	0.137*
C17	0.4329 (4)	0.25566 (12)	0.6555 (3)	0.1012 (9)
H17A	0.3985	0.2339	0.7078	0.152*
H17B	0.5242	0.2352	0.6656	0.152*
H17C	0.3577	0.2519	0.5706	0.152*
C18	0.3143 (2)	0.36078 (13)	0.67513 (19)	0.0753 (6)
H18A	0.2879	0.3426	0.7353	0.113*
H18B	0.2357	0.3516	0.5934	0.113*
H18C	0.3273	0.4079	0.6866	0.113*
C19	0.92107 (15)	0.35478 (7)	0.62011 (14)	0.0430 (4)
H19A	0.9650	0.3897	0.5924	0.052*
H19B	0.9730	0.3543	0.7099	0.052*
C20	0.94472 (16)	0.28944 (8)	0.57296 (15)	0.0456 (4)

H20A	0.9067	0.2860	0.4876	0.055*
C21	1.01301 (18)	0.23594 (8)	0.63764 (17)	0.0533 (4)
C22	1.0272 (3)	0.17404 (10)	0.5744 (2)	0.0791 (6)
H22A	0.9881	0.1823	0.4873	0.119*
H22B	0.9715	0.1387	0.5869	0.119*
H22C	1.1310	0.1616	0.6087	0.119*
C23	1.0826 (3)	0.23100 (11)	0.7756 (2)	0.0756 (6)
H23A	1.0799	0.2737	0.8096	0.113*
H23B	1.1850	0.2166	0.8077	0.113*
H23C	1.0277	0.1995	0.7981	0.113*
C24	0.6202 (7)	0.6016 (2)	0.9882 (4)	0.186 (2)
H24A	0.6362	0.6187	0.9217	0.223*
H24B	0.6890	0.6224	1.0644	0.223*
H24C	0.5190	0.6107	0.9722	0.223*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0327 (6)	0.0978 (10)	0.0607 (7)	0.0177 (6)	0.0213 (5)	0.0222 (7)
O2	0.0348 (6)	0.0987 (10)	0.0647 (8)	0.0163 (6)	0.0292 (6)	0.0302 (7)
O3	0.0383 (6)	0.0864 (9)	0.0505 (6)	0.0073 (5)	0.0240 (5)	0.0220 (6)
O4	0.0333 (5)	0.0794 (8)	0.0545 (7)	0.0092 (5)	0.0256 (5)	0.0206 (6)
O5	0.0421 (6)	0.0850 (9)	0.0676 (8)	0.0232 (6)	0.0282 (6)	0.0380 (7)
O6	0.0290 (5)	0.0620 (6)	0.0457 (6)	0.0085 (4)	0.0189 (5)	0.0151 (5)
O7	0.0808 (11)	0.1318 (17)	0.0661 (10)	0.0343 (11)	0.0160 (9)	0.0218 (10)
C1	0.0373 (8)	0.0569 (9)	0.0414 (8)	0.0024 (6)	0.0178 (6)	0.0067 (7)
C2	0.0375 (8)	0.0566 (9)	0.0416 (8)	0.0087 (6)	0.0133 (6)	0.0075 (7)
C3	0.0311 (7)	0.0554 (9)	0.0480 (8)	0.0067 (6)	0.0155 (6)	0.0009 (7)
C4	0.0327 (7)	0.0547 (9)	0.0456 (8)	0.0049 (6)	0.0204 (6)	0.0047 (6)
C5	0.0310 (7)	0.0444 (7)	0.0389 (7)	0.0032 (5)	0.0147 (6)	0.0023 (6)
C6	0.0323 (7)	0.0448 (7)	0.0401 (7)	0.0011 (6)	0.0156 (6)	0.0012 (6)
C7	0.0334 (7)	0.0508 (8)	0.0398 (7)	−0.0001 (6)	0.0183 (6)	0.0025 (6)
C8	0.0304 (7)	0.0445 (7)	0.0412 (8)	0.0006 (5)	0.0170 (6)	0.0015 (6)
C9	0.0271 (7)	0.0451 (8)	0.0427 (7)	0.0015 (5)	0.0166 (6)	0.0017 (6)
C10	0.0336 (7)	0.0479 (8)	0.0460 (8)	0.0021 (6)	0.0199 (6)	0.0076 (6)
C11	0.0353 (7)	0.0481 (8)	0.0469 (8)	0.0064 (6)	0.0184 (6)	0.0095 (6)
C12	0.0307 (7)	0.0447 (8)	0.0466 (8)	0.0029 (6)	0.0177 (6)	0.0024 (6)
C13	0.0311 (7)	0.0470 (8)	0.0442 (8)	−0.0004 (6)	0.0203 (6)	0.0012 (6)
C14	0.0413 (8)	0.0624 (10)	0.0590 (10)	0.0025 (7)	0.0262 (7)	0.0196 (8)
C15	0.0429 (9)	0.1156 (16)	0.0581 (10)	0.0101 (10)	0.0279 (8)	0.0288 (11)
C16	0.0689 (14)	0.195 (3)	0.0909 (17)	0.0497 (17)	0.0475 (13)	0.078 (2)
C17	0.125 (2)	0.0777 (15)	0.129 (2)	−0.0289 (14)	0.083 (2)	0.0060 (14)
C18	0.0423 (9)	0.1250 (18)	0.0686 (12)	0.0133 (10)	0.0340 (9)	0.0399 (12)
C19	0.0299 (7)	0.0508 (8)	0.0478 (8)	0.0040 (6)	0.0176 (6)	0.0065 (6)
C20	0.0319 (7)	0.0571 (9)	0.0484 (8)	0.0031 (6)	0.0191 (6)	0.0033 (7)
C21	0.0432 (8)	0.0526 (9)	0.0642 (10)	0.0049 (7)	0.0251 (8)	0.0062 (8)
C22	0.0806 (14)	0.0597 (11)	0.0947 (15)	0.0130 (10)	0.0385 (12)	−0.0020 (11)
C23	0.0811 (14)	0.0732 (13)	0.0703 (12)	0.0236 (11)	0.0330 (11)	0.0244 (10)

C24	0.280 (6)	0.137 (3)	0.115 (3)	0.066 (4)	0.070 (4)	−0.008 (3)

Geometric parameters (Å, °)

O1—C3	1.3547 (18)	C12—C19	1.5183 (19)
O1—H1O1	0.85 (3)	C14—C15	1.506 (3)
O2—C4	1.3529 (19)	C14—C17	1.531 (3)
O2—H1O2	0.8200	C14—C18	1.543 (2)
O3—C7	1.2617 (18)	C15—C16	1.324 (3)
O4—C13	1.3526 (17)	C15—H15A	0.9300
O4—H1O4	0.8200	C16—H16A	0.9300
O5—C11	1.3550 (18)	C16—H16B	0.9300
O5—H5A	0.89 (3)	C17—H17A	0.9600
O6—C9	1.3616 (17)	C17—H17B	0.9600
O6—C5	1.3627 (17)	C17—H17C	0.9600
O7—C24	1.279 (5)	C18—H18A	0.9600
O7—H1O7	0.831 (10)	C18—H18B	0.9600
C1—C2	1.370 (2)	C18—H18C	0.9600
C1—C6	1.405 (2)	C19—C20	1.499 (2)
C1—H1B	0.9300	C19—H19A	0.9700
C2—C3	1.393 (2)	C19—H19B	0.9700
C2—H2A	0.9300	C20—C21	1.324 (2)
C3—C4	1.385 (2)	C20—H20A	0.9300
C4—C5	1.391 (2)	C21—C23	1.496 (3)
C5—C6	1.389 (2)	C21—C22	1.507 (3)
C6—C7	1.445 (2)	C22—H22A	0.9600
C7—C8	1.443 (2)	C22—H22B	0.9600
C8—C9	1.410 (2)	C22—H22C	0.9600
C8—C13	1.4193 (19)	C23—H23A	0.9600
C9—C10	1.392 (2)	C23—H23B	0.9600
C10—C11	1.411 (2)	C23—H23C	0.9600
C10—C14	1.551 (2)	C24—H24A	0.9600
C11—C12	1.404 (2)	C24—H24B	0.9600
C12—C13	1.376 (2)	C24—H24C	0.9600
C3—O1—H1O1	109.7 (18)	C18—C14—C10	116.03 (13)
C4—O2—H1O2	109.5	C16—C15—C14	127.2 (3)
C13—O4—H1O4	109.5	C16—C15—H15A	116.4
C11—O5—H5A	108.6 (17)	C14—C15—H15A	116.4
C9—O6—C5	121.30 (11)	C15—C16—H16A	120.0
C24—O7—H1O7	104 (3)	C15—C16—H16B	120.0
C2—C1—C6	120.51 (14)	H16A—C16—H16B	120.0
C2—C1—H1B	119.7	C14—C17—H17A	109.5
C6—C1—H1B	119.7	C14—C17—H17B	109.5
C1—C2—C3	120.42 (14)	H17A—C17—H17B	109.5
C1—C2—H2A	119.8	C14—C17—H17C	109.5
C3—C2—H2A	119.8	H17A—C17—H17C	109.5
O1—C3—C4	115.40 (14)	H17B—C17—H17C	109.5
O1—C3—C2	124.11 (14)	C14—C18—H18A	109.5
C4—C3—C2	120.49 (14)	C14—C18—H18B	109.5

O2—C4—C3	123.39 (13)	H18A—C18—H18B	109.5
O2—C4—C5	118.20 (13)	C14—C18—H18C	109.5
C3—C4—C5	118.39 (14)	H18A—C18—H18C	109.5
O6—C5—C6	122.75 (12)	H18B—C18—H18C	109.5
O6—C5—C4	115.11 (12)	C20—C19—C12	112.88 (12)
C6—C5—C4	122.13 (13)	C20—C19—H19A	109.0
C5—C6—C1	118.02 (13)	C12—C19—H19A	109.0
C5—C6—C7	118.51 (13)	C20—C19—H19B	109.0
C1—C6—C7	123.47 (13)	C12—C19—H19B	109.0
O3—C7—C8	121.37 (13)	H19A—C19—H19B	107.8
O3—C7—C6	121.45 (13)	C21—C20—C19	128.13 (15)
C8—C7—C6	117.18 (12)	C21—C20—H20A	115.9
C9—C8—C13	117.94 (13)	C19—C20—H20A	115.9
C9—C8—C7	120.60 (13)	C20—C21—C23	124.55 (17)
C13—C8—C7	121.46 (13)	C20—C21—C22	121.00 (17)
O6—C9—C10	116.49 (12)	C23—C21—C22	114.45 (16)
O6—C9—C8	119.66 (12)	C21—C22—H22A	109.5
C10—C9—C8	123.84 (13)	C21—C22—H22B	109.5
C9—C10—C11	114.48 (13)	H22A—C22—H22B	109.5
C9—C10—C14	125.11 (13)	C21—C22—H22C	109.5
C11—C10—C14	120.26 (13)	H22A—C22—H22C	109.5
O5—C11—C12	113.44 (13)	H22B—C22—H22C	109.5
O5—C11—C10	121.74 (13)	C21—C23—H23A	109.5
C12—C11—C10	124.81 (13)	C21—C23—H23B	109.5
C13—C12—C11	117.79 (12)	H23A—C23—H23B	109.5
C13—C12—C19	121.96 (13)	C21—C23—H23C	109.5
C11—C12—C19	120.25 (13)	H23A—C23—H23C	109.5
O4—C13—C12	119.35 (12)	H23B—C23—H23C	109.5
O4—C13—C8	119.54 (13)	O7—C24—H24A	109.5
C12—C13—C8	121.11 (13)	O7—C24—H24B	109.5
C15—C14—C17	112.63 (19)	H24A—C24—H24B	109.5
C15—C14—C18	102.58 (16)	O7—C24—H24C	109.5
C17—C14—C18	108.52 (18)	H24A—C24—H24C	109.5
C15—C14—C10	110.00 (14)	H24B—C24—H24C	109.5
C17—C14—C10	107.19 (16)		
C6—C1—C2—C3	−0.3 (2)	C8—C9—C10—C11	−0.6 (2)
C1—C2—C3—O1	−178.16 (15)	O6—C9—C10—C14	2.9 (2)
C1—C2—C3—C4	2.0 (2)	C8—C9—C10—C14	−176.02 (14)
O1—C3—C4—O2	−0.4 (2)	C9—C10—C11—O5	−176.97 (14)
C2—C3—C4—O2	179.48 (15)	C14—C10—C11—O5	−1.3 (2)
O1—C3—C4—C5	177.91 (14)	C9—C10—C11—C12	1.9 (2)
C2—C3—C4—C5	−2.2 (2)	C14—C10—C11—C12	177.57 (15)
C9—O6—C5—C6	−0.3 (2)	O5—C11—C12—C13	176.72 (14)
C9—O6—C5—C4	178.79 (13)	C10—C11—C12—C13	−2.2 (2)
O2—C4—C5—O6	0.1 (2)	O5—C11—C12—C19	−2.8 (2)
C3—C4—C5—O6	−178.31 (13)	C10—C11—C12—C19	178.28 (14)
O2—C4—C5—C6	179.21 (14)	C11—C12—C13—O4	−179.14 (13)
C3—C4—C5—C6	0.8 (2)	C19—C12—C13—O4	0.4 (2)
O6—C5—C6—C1	179.88 (13)	C11—C12—C13—C8	1.2 (2)

C4—C5—C6—C1	0.8 (2)	C19—C12—C13—C8	−179.33 (13)
O6—C5—C6—C7	0.5 (2)	C9—C8—C13—O4	−179.69 (13)
C4—C5—C6—C7	−178.59 (13)	C7—C8—C13—O4	1.2 (2)
C2—C1—C6—C5	−1.1 (2)	C9—C8—C13—C12	0.0 (2)
C2—C1—C6—C7	178.30 (14)	C7—C8—C13—C12	−179.13 (14)
C5—C6—C7—O3	179.25 (14)	C9—C10—C14—C15	−137.06 (17)
C1—C6—C7—O3	−0.1 (2)	C11—C10—C14—C15	47.7 (2)
C5—C6—C7—C8	−0.4 (2)	C9—C10—C14—C17	100.2 (2)
C1—C6—C7—C8	−179.77 (14)	C11—C10—C14—C17	−75.0 (2)
O3—C7—C8—C9	−179.44 (14)	C9—C10—C14—C18	−21.2 (3)
C6—C7—C8—C9	0.2 (2)	C11—C10—C14—C18	163.56 (17)
O3—C7—C8—C13	−0.3 (2)	C17—C14—C15—C16	−12.2 (3)
C6—C7—C8—C13	179.32 (13)	C18—C14—C15—C16	104.3 (2)
C5—O6—C9—C10	−178.87 (13)	C10—C14—C15—C16	−131.7 (2)
C5—O6—C9—C8	0.1 (2)	C13—C12—C19—C20	−93.25 (17)
C13—C8—C9—O6	−179.22 (12)	C11—C12—C19—C20	86.25 (17)
C7—C8—C9—O6	−0.1 (2)	C12—C19—C20—C21	−115.91 (17)
C13—C8—C9—C10	−0.3 (2)	C19—C20—C21—C23	−0.3 (3)
C7—C8—C9—C10	178.84 (14)	C19—C20—C21—C22	−179.77 (16)
O6—C9—C10—C11	178.37 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···O7^{i}	0.85 (3)	1.79 (3)	2.632 (2)	170 (3)
O2—H1O2···O1	0.82	2.29	2.7117 (19)	113
O2—H1O2···O4^{ii}	0.82	2.03	2.7995 (19)	155
O4—H1O4···O3	0.82	1.81	2.5505 (18)	149
O7—H1O7···O3i	0.83 (2)	1.954 (19)	2.755 (2)	161 (4)
C18—H18B···O6	0.96	2.25	2.638 (2)	103
C19—H19A···O2^{iii}	0.97	2.54	3.378 (2)	145
C19—H19A···O4	0.97	2.50	2.8451 (19)	101
C22—H22B···Cg1^{iv}	0.96	3.10	3.705 (2)	123

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

Fig. 1

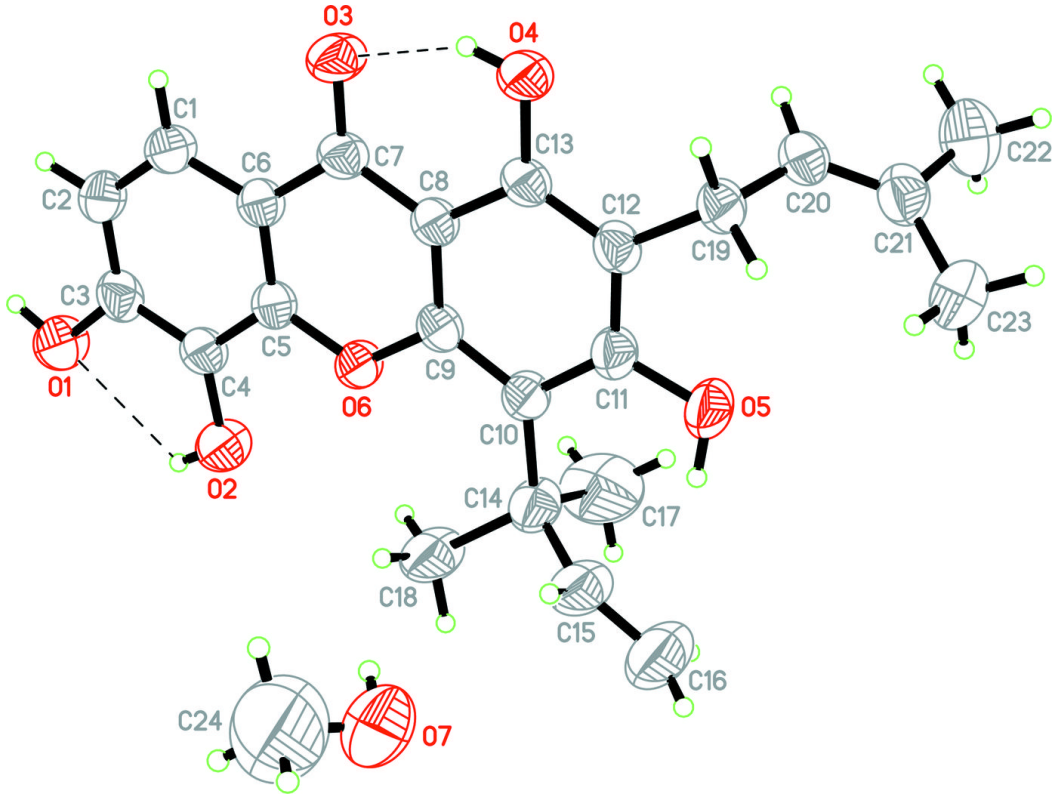
O3
O4
C1
C6
C7
C13
C22
C2
C8
C12
C20
C19
C21
C3
C23
C5
O1
C4
O6
C9
C11
O5
C10
O2
C14
C17
C18
C15
C16
C24
O7

Fig. 2