Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2′-Hydroxy-4,4′,5′,6′-tetramethoxychalcone

Pascal Wafo,[a] Hidayat Hussain,[b] Simeon F. Kouam,[a] Bonaventure T. Ngadjui,[c] Ulrich Flörke[b]* and Karsten Krohn[b]

[a]Department of Chemistry, Teachers Training High School, University of Yaounde 1, BP 46, Yaounde, Cameroon, [b]Department Chemie, Fakultät für Naturwissenschaften, Universität Paderborn, Warburgerstrasse 100, D-33098 Paderborn, Germany, and [c]Department of Organic Chemistry, Faculty of Science, University of Yaounde 1, BP 812, Yaounde, Cameroon

Correspondence e-mail: ulrich.floerke@upb.de

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.088
Data-to-parameter ratio = 8.5

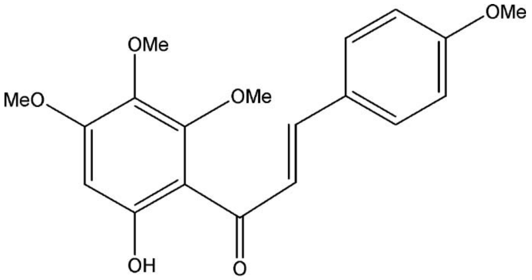
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{19}H_{20}O_6$, the dihedral angle between the two aromatic planes is 31.8 (1)°. Intermolecular C—H···O hydrogen bonds serve to stabilize the structure.

Received 15 August 2005
Accepted 17 August 2005
Online 20 August 2005

Comment

2′-Hydroxy-4,4′,5′,6′-tetramethoxychalcone (or odoratin) is a chalcone derivative that was isolated from the stem bark of *Eupatroium odoratum*, a plant used as a remedy for a variety of ailments, including malaria, fever and infections, and as a haemostatic agent (Shuib *et al.*, 1999). Chalcones represent an important group of natural compounds, showing a variety of biological activities including antifungal (Boeck *et al.*, 2005) and anticoagulant properties (Shuib *et al.*, 1999). The compounds are also used as depigmenting agents (Khatib *et al.*, 2005). Although known for long time (Bose *et al.*, 1973), the title compound, (I), has only now been characterized crystallographically.



(I)

The molecular structure of (I) is closely related to that of the dimethoxychalcone [Cambridge Structural Database (*MOGUL*Version 1.7; Allen, 2002) refcode JEPJAP (Schmalle *et al.*, 1990)] with two methoxy groups at the 5′- and 6′-positions instead of H and OH. Most geometric parameters of the non-H atoms are not significantly different from those

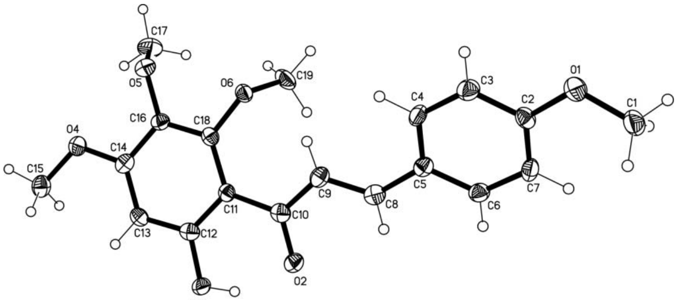


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

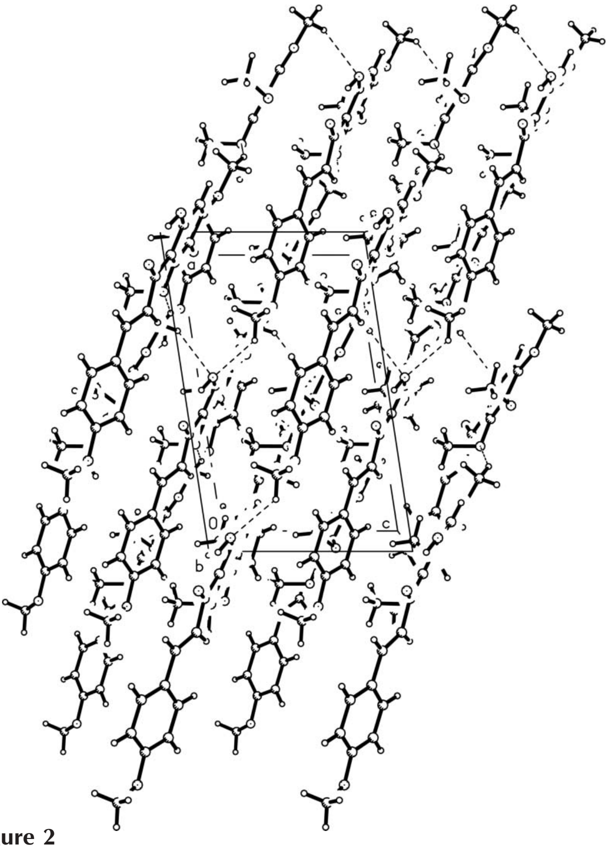


Figure 2
The crystal packing, viewed along [010], with the hydrogen-bonding pattern indicated by dashed lines.

of JEPJAP and need no further discussion. However, the dihedral angle of 31.8 (1)° between the two aromatic planes is considerably increased compared with 13.1 (4)° from the nearly planar JEPJAP molecule. With respect to the C8—C9—C10—O2 enone moiety, both aromatic rings are twisted out of this common plane by 14.7 (2) and 17.2 (3)°, respectively. The methoxy groups O1—C1 and O4—C15 lie in the planes of the attached aromatic rings, whereas both groups O5—C17 and O6—C19 are oriented almost perpendicular, with torsion angles C14—C16—O5—C17 of −78.3 (5)° and C11—C18—O6—C19 of 82.3 (4)°. As in JEPJAP, a strong intramolecular O3—H3···O2 hydrogen bond is observed, with H···O = 1.64 Å and O—H···O = 144°. The intramolecular C9—H9*A*···O6 bridge is less dominant, with H···O = 2.25 Å and C—H···O = 111°. Intermolecular C–H.·O interactions are rather weak, with H···O distances greater than 2.45 Å. All these values are normalized for O—H = 0.938 Å and C—H = 1.080 Å.

Experimental

The leaves of Eupatorium odoratum Linn. were collected in Yaounde, Cameroon. Air-dried and powdered leaves (3 kg) were macerated in a mixture of CH_2Cl_2–MeOH (1:1) for 48 h. The extract was evaporated to yield the residue (200 g) which was then extracted with n-hexane. The extract (20 g) was subjected to column chromatography (CC) over a silica gel column using *n*-hexane with a gradient of acetone. The column eluates were monitored by thin-layer chromatography and similar fractions were combined. Fraction B was subjected to further CC using a gradient solvent *n*-hexane/acetone and yielded odoratin. Recrystallization from *n*-hexane gave yellow crystals (52 mg). The melting point (415–417 K) is identical to that reported in the literature (Bose *et al.*, 1973).

Crystal data

$C_{19}H_{20}O_6$
M_r = 344.35
Monoclinic, *Cc*
a = 13.092 (4) Å
b = 15.041 (5) Å
c = 8.256 (3) Å
β = 98.649 (7)°
V = 1607.3 (9) Å^3
Z = 4

D_x = 1.423 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1038 reflections
θ = 2.7–22.4°
μ = 0.11 mm^{-1}
T = 120 (2) K
Prism, yellow
0.48 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.951, T_{max} = 0.978
7488 measured reflections

1954 independent reflections
1346 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 28.1°
h = −17 → 17
k = −19 → 19
l = −10 → 10

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.088
S = 0.91
1954 reflections
231 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0277P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.357 (4)	O5—C17	1.422 (4)
O1—C1	1.414 (5)	O6—C18	1.379 (4)
O2—C10	1.234 (4)	O6—C19	1.415 (4)
O3—C12	1.349 (4)	C5—C8	1.449 (5)
O4—C14	1.344 (5)	C8—C9	1.336 (5)
O4—C15	1.425 (5)	C9—C10	1.453 (5)
O5—C16	1.364 (4)	C10—C11	1.471 (5)
C2—O1—C1	116.9 (3)	C16—O5—C17	113.3 (3)
C14—O4—C15	116.8 (3)	C18—O6—C19	113.7 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.84	1.72	2.457 (4)	146
C9—H9*A*···O6	0.95	2.31	2.829 (4)	114
C7—H7*A*···O2^{i}	0.95	2.57	3.329 (5)	137
C1—H1*B*···O3^{ii}	0.98	2.54	3.289 (5)	133
C15—H15*B*···O6^{iii}	0.98	2.55	3.410 (5)	147
C15—H15*C*···O3^{iv}	0.98	2.51	3.201 (5)	127

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

H atoms were refined at idealized positions (C—H = 0.95–0.98, O— H = 0.84 Å), riding on the parent C and O atoms with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C and OH). All CH_3 and OH H atoms were allowed to rotate but not to tip. The title compound crystallizes in the non-centrosymmetric space

o3018 Wafo *et al.* • $C_{19}H_{20}O_6$ *Acta Cryst.* (2005). E**61**, o3017–o3019

group *Cc*; however, in the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Accordingly, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Boeck, P., Leal, P. C., Yunes, R. A., Filho, V. C., Lopez, S., Sortino, M., Escalante, A., Furlan, R. L. E. & Zacchino, S. (2005). *Arch. Pharm. Chem. Life Sci.* **338**, 87–95.

Bose, P. K., Chakrabarti, P., Chakravarti, S., Dutta, S. P. & Barua, A. K. (1973). *Phytochemistry,* **12**, 667–668.

Bruker (2002). *SMART* (Version 5.62), *SAINT* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Khatib, S., Nerya, O., Musa, R., Shmuel, M., Tamir, S. & Vaya, J. (2005). *Bioorg. Med. Chem.* **13**, 433–441.

Schmalle, H. W., Adiwidjaja, G., Jarchow, O. H., Hausen, B. & Wollenweber, E. (1990). *Acta Cryst.* C**46**, 1712–1715.

Shuib, N. S., Sam, T. W., Wong, K. C., Chinnakali, K. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 576–578.

Acta Cryst. (2005). E**61**, o3017–o3019 Wafo *et al.* • $C_{19}H_{20}O_6$ **o3019**