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5,7-Dihydroxy-6,2′-dimethoxyisoflavone

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Key indicators

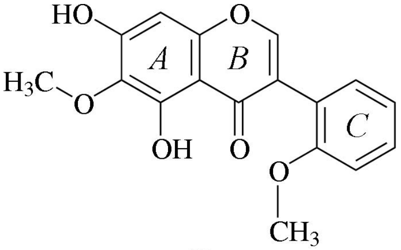
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 12.1

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

The title compound, $C_{17}H_{14}O_6$, has been isolated for the first time from *Rhazya stricta*. The dihedral angle between the mean planes through the benzopyran ring system and *o*-anisole group is 59.78 (5)°. The molecular packing is stabilized by O—H···O and C—H···O hydrogen bonds.

Comment

Rhazya stricta (Apocyanaceae) is a gladiolus, an erect shrub found abundantly in Pakistan, especially in Sindh (Glotter, 1991). The plant is reputed to have antitumour activity. In the indigenous system of medicine, it is used for the treatment of chronic rheumatism, sore throat and debility in general (Chopra *et al.*, 1956; Dymock *et al.*, 1893). It contains several medicinally important indole alkaloids (Rahman *et al.*, 1982). As such, the plant extract was studied and a number of compounds have been isolated. The title compound, (I), is one among the series of compounds isolated and here we report its crystal structure. Previously, this compound was isolated from *Iris missouriensis* (Wong *et al.*, 1987) and *Iris spuria* (Shawl *et al.*, 1984).



(I)

The bond lengths in compound (I) show normal values (Allen *et al.*, 1987). The benzopyran ring system is planar and the methoxyphenyl group (O6/C10–C16) attached at the C2 position is oriented at an angle of 59.78 (5)° (Fig. 1). The methoxy group at the C5 position is twisted away from the plane of the C4–C9 benzene ring, the C17—O4—C5—C4 torsion angle being 75.9 (2)°.

Intramolecular O—H···O hydrogen bonds link symmetry-related molecules into chains that run along the b axis (Table 1). Adjacent chains are interlinked into a three-dimensional network by C—H···O hydrogen bonds (Fig. 2).

Experimental

Air-dried aerial parts (40 kg) of *Rhazya stricta* were extracted with ethanol (200 l). The ethanolic extract of the whole plant was concentrated to a gum (950 g), dissolved in distilled water and extracted thoroughly with petroleum ether. The petroleum ether-soluble portion was evaporated under reduced pressure to a gum

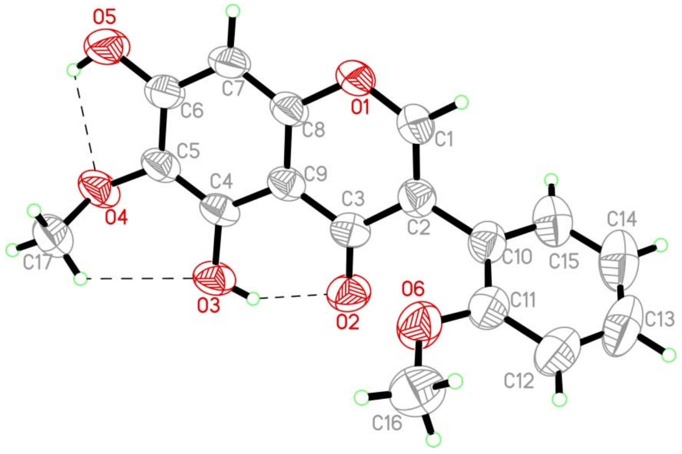


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

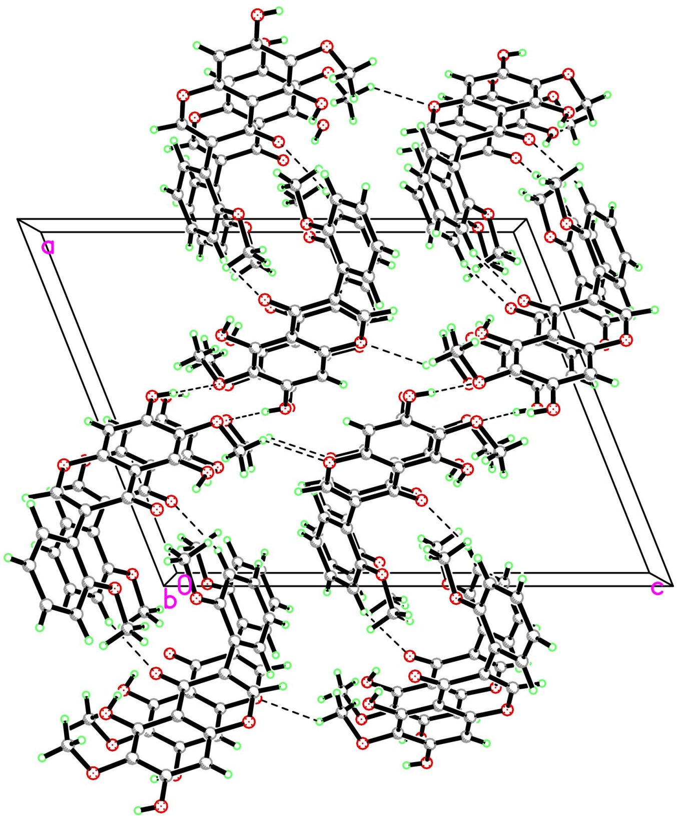


Figure 2
A view, down the *b* axis, of the molecular packing in (I). Dashed lines indicate hydrogen bonds.

(134 g). The remaining aqueous layer was extracted with $CHCl_3$ at neutral pH. The chloroform-soluble portion was evaporated under reduced pressure to a gum (40.0 g) and chromatographed on a silica-gel column (Merck, 70–230 mesh, 800 g) eluted with gradients of mixtures of petroleum ether–chloroform and chloroform–methanol. Compound (I) was obtained as a colourless crystalline solid on elution with chloroform–methanol (9:1) (23.0 mg, 5.6×10^{-5}% yield with $Rf = 0.46$). The compound was recrystallized from a solution in acetone–hexane (1:1) (m.p. 463–464 K).

Crystal data

$C_{17}H_{14}O_6$
$M_r = 314.28$
Monoclinic, $P2_1/c$
$a = 13.6600$ (7) Å
$b = 6.5247$ (3) Å
$c = 17.5754$ (9) Å
$\beta = 111.726$ (1)°
$V = 1455.18$ (12) Å^3
$Z = 4$

$D_x = 1.435$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4463 reflections
$\theta = 1.6$–25.0°
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
$0.45 \times 0.39 \times 0.09$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.952$, $T_{\max} = 0.990$
6924 measured reflections

2560 independent reflections
2004 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 25.0°$
$h = -16 \rightarrow 16$
$k = -7 \rightarrow 7$
$l = -17 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.04$
2560 reflections
212 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 0.2123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O3—H3$A$$\cdots$O2$^{\rm i}$	0.82	1.84	2.566 (2)	147
O5—H5$A$$\cdots$O4$^{\rm i}$	0.82	2.29	2.745 (2)	115
O5—H5$A$$\cdots$O3$^{\rm ii}$	0.82	2.57	3.197 (2)	134
O5—H5$A$$\cdots$O4$^{\rm ii}$	0.82	2.35	3.026 (2)	140
C12—H12$A$$\cdots$O2$^{\rm iii}$	0.93	2.51	3.347 (3)	149
C17—H17$A$$\cdots$O1$^{\rm iv}$	0.96	2.41	3.194 (2)	138
C17—H17$C$$\cdots$O3$^{\rm i}$	0.96	2.55	3.099 (2)	117

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

All H atoms were positioned geometrically and allowed to ride on the parent atoms, with O—H = 0.82 Å, aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å, and with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C/O) for methyl and hydroxyl H atoms and $1.2U_{\rm eq}$(C) for the other H atoms. A rotating group model was used for the methyl and hydroxy groups.

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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Chopra, R. N., Nayar, S. L. & Chopra, I. C. (1956). *A Glossary of Indian Medicinal Plants*, pp. 212. New Delhi: CSIR.
Dymock, W. C., Warden, J. H. & Hooper, H. (1893). *Pharmacographia India*, p. 391. London: Kegan Paul, Trench, Trubner & Co.
Glotter, E. (1991). *Natural Prod. Rep.* **8**, 415–417.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Rahman, A.-ur. & Fatima, K. (1982). *J. Chem. Soc. Pak.* **4**, 121–123.

Acta Cryst. (2005). E**61**, o1812–o1814 Sultana *et al.* • $C_{17}H_{14}O_6$ **o1813**

Shawl, A. S., Zaman, V. A. & Kalla, A. K. (1984). *Phytochemistry*, **23**, 2405–2406.

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.

Wong, S. M., Konno, C., Oshima, Y., Pezzuto, M. J., Fong, H. H. S. & Farnsworth, N. R. (1987). *J. Nat. Prod.* **50**, 178–180.

o1814 Sultana *et al.* • $C_{17}H_{14}O_6$ *Acta Cryst.* (2005). E**61**, o1812–o1814