Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Regiolone from the pericarps of *Juglans regia* L.

Jun-Xi Liu, Duo-Long Di,* Chen Li and Xin-Yi Huang

Key Laboratory for Natural Medicine of Gansu Province, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China

Correspondence e-mail: didl@lzb.ac.cn

Received 13 March 2007
Accepted 21 April 2007

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.063
Data-to-parameter ratio = 13.4

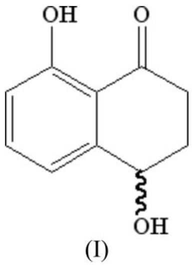
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Regiolone [(4*RS*)-4,8-dihydroxy-3,4-dihydronaphthalen-1(2*H*)-one], $C_{10}H_{10}O_3$, isolated from the pericarps of *Juglans regia* L., is confirmed as a racemate.

Comment

Juglans regia L. (Juglandaceae), a walnut tree, is widely planted in China, Korea and Japan. The pericarps of *J. regia* L. (QingLongYi) have been used as a folk medicine for the treatment of cancer, chilblains and athlete's foot.

Regiolone was isolated from *Juglans* plant materials, and the structure was proposed as (4*S*)-4,8-dihydroxy-α-tetralone [isosclerone (Morita & Aoki, 1974; Fujimoto *et al.*, 1998; Ayer *et al.*, 2000); regiolone (Talapapatra *et al.*, 1988; Li *et al.*, 2003)] by comparing the spectroscopic data and the optical rotation with those reported in the literature. Additionally, its enantiomers in a ratio of *ca* 5:1 were separated by chiral HPLC (Koichi *et al.*, 2005). In a continuation of our investigation to find cytotoxic compounds, we studied the pericarps of *J. regia* L. and a regiolone crystal was obtained. To the best of our knowledge, the crystal structure of the title compound, (I), was previously unreported.



(I)

The conformation of the cyclohexenone ring in regiolone can adopt either of the two nearly half-chair forms having similar free energy, as was deduced from its high-resolution NMR spectroscopic data (Talapapatra *et al.*, 1988). Using CD experiments, Koichi *et al.* (2005) reported that one of the methylene protons at C-2 had β- and α-quasi-axial orientations with respect to the cyclohexenone ring. On the other hand, the sign of the weakly negative and positive Cotton effects could be interpreted under the assumption that the half-chair form of the cyclohexenone ring is a more stable conformation than the sofa form in the molecular structure. The half-chair form of the cyclohexenone ring is confirmed by our results. The asymmetric unit is shown in Fig. 1. Regiolone exists as a racemate and the absolute configurations at C-4 in the two molecules of this arbitrarily chosen asymmetric unit are *R* and S for unprimed and primed molecules, respectively.

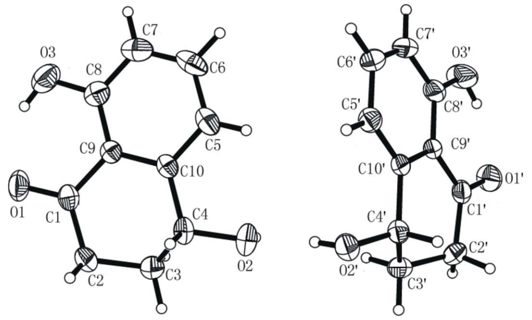


Figure 1
The asymmetric unit of the title compound, with 30% probability displacement ellipsoids.

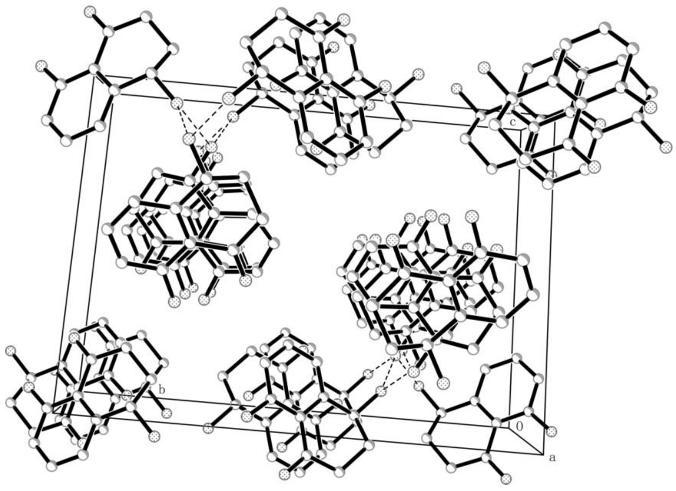


Figure 2
A packing diagram. Hydrogen bonds are shown as dashed lines, and H atoms have been omitted.

Intra- and intermolecular hydrogen bonds are found (Table 1 and Fig. 2).

Experimental

The dried pericarps of *J. regia* L. (10 kg) were powered and extracted with 95% EtOH at room temperature. The combined extracts were evaporated under reduce pressure and extracted three times with petroleum ether (353–383 K). After evaporation of the solvent, the extract (150 g) was chromatographed on a silica gel column, eluted with petroleum ether–EtOAc (6:1) and collected in three fractions: Fr1 (1.0 l), Fr2 (1.5 l) and Fr3 (1.0 l). The residue obtained from fraction Fr2 was recrystallized from EtOAc to yield regiolone (100 mg) as colourless block crystals, $[\alpha]^{20}_{D} = \pm 0°$ (*c* 1.3, CH_2Cl_2), m.p. 366–364 K; ^{1}H NMR (400 MHz, $CDCl_3$, $\delta_{p.p.m.}$): 12.4 (1H, *s*, OH-8), 7.46 (1H, *t*, 8.4, 7.6, H-6), 6.99 (1H, *d*, 7.6, H-5), 6.89 (1H, *d*, 8.4, H-7), 4.88 (1H, *dd*, 7.2, 4.0), 2.97 (1H, *ddd*, 18.0, 8.4, 4.8, H-2a), 2.62 (1H, *ddd*, 18.0, 8.4, 4.8, H-2b), 2.31 (1H, *m*, H-3a), 2.16 (1H, *m*, H-3b); ^{13}C NMR (100 MHz, $\delta_{p.p.m.}$): 204.4 (C-1), 162.4 (C-8), 145.8 (C-9), 136.9 (C-6), 117.5 (C-5), 117.4 (C-7), 115.1 (C-10), 67.5 (C-4), 34.5 (C-2), 31.1 (C-3). These data agree with those reported previously (Talapapatra *et al.*, 1988).

Crystal data

$C_{10}H_{10}O_3$
$M_r = 178.18$
Monoclinic, $P2_1/c$
$a = 12.4859$ (3) Å
$b = 16.6010$ (5) Å
$c = 8.7947$ (2) Å
$\beta = 109.427$ (1)°

$V = 1719.16$ (8) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 294$ (2) K
0.25 × 0.20 × 0.19 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.975$, $T_{max} = 0.981$

9127 measured reflections
3207 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.063$
$S = 1.12$
3207 reflections

240 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3′···O1	0.82	1.86	2.581 (2)	146
O2′—H2E···O2	0.82	1.89	2.713 (2)	178
O3′—H3E···O1′	0.82	1.84	2.564 (2)	147

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

All H atoms were positioned geometrically (C—H = 0.93–0.98 Å, O—H = 0.82 Å) and were refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Great Research Project of CAS (KGCX2-SW-213-08) and the Hundreds of Talents Programme of CAS.

References

Ayer, W. A., Trifonov, L. S. & Hutchison, L. J. (2000). *Nat. Prod. Lett.* **14**, 405–410.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Fujimoto, H., Nagano, J. & Yamaguchi, K. (1998). *Chem. Pharm. Bull.* **46**, 423–429.
Koichi, M., Erika, M. & Takayuki, K. (2005). *Chem. Pharm. Bull.* **53**, 934–937.
Li, G., Xu, M. L. & Choi, H. G. (2003). *Chem. Pharm. Bull.* **51**, 262–264.
Morita, T. & Aoki, H. (1974). *Agric. Biol. Chem.* **38**, 1501–1505.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Talapapatra, S. K., Karmacharya, B. & De, S. C. (1988). *Phytochemistry*, **27**, 3929–3932.

o2714 Liu *et al.* • $C_{10}H_{10}O_3$ *Acta Cryst.* (2007). E**63**, o2713–o2714