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# Desmethylbergenin hemihydrate

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**Key indicators**

Single-crystal X-ray study
$T$ = 293 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.031
$wR$ factor = 0.079
Data-to-parameter ratio = 6.4

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



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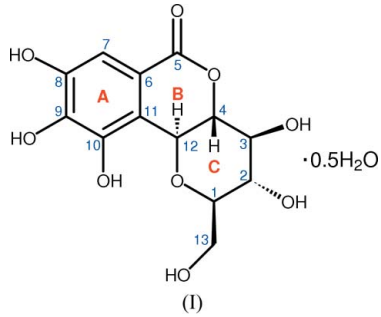
The title compound, 3,4,8,9,10-pentahydroxy-2-hydroxymethyl-2,3,4,4a,6,10b-hexahydropyrano[3,2-*c*]isochromen-6-one hemihydrate, $C_{13}H_{14}O_9\cdot0.5H_2O$, is a naturally occurring isocoumarin isolated from *Rivea hypocrateriformis*. The molecule contains three six-membered rings, *viz*. an aromatic ring, a glucopyranose ring and a $\delta$-lactone ring. The water molecule lies on a twofold axis. The crystal structure is stabilized by an extensive network of O—H···O hydrogen bonds and C—H···O and C—H···$\pi$ interactions.

## Comment

Desmethylbergenin (Taneyama *et al.*, 1983) was isolated from *Rivea hypocrateriformis* (Desr.) Choisy. *Rivea hypocrateriformis*, a robust woody climbing shrub belonging to the family Convolvulaceae is found in dry subtropical forests of India and Pakistan (Austin *et al.*, 1979). The plant is medicinally used by the indigenous population of Tharparkar to cure various type of diseases, such as malaria, and to relieve pain. It is also used as a psychoactive drug plant like the other species of the family Convolvulaceae. For example, *Rivea corymbosa* (Hall.) and *Ipomea violacea* (L.) are psychedelic drug plants (Evans, 1990) found in Mexico where the Indians use these plants for hallucination, anaesthesia, and mood elevation during religious ceremonies. A phytochemical survey revealed that *R. hypocrateriformis* contains alkaloids, glycosides, saponin, tannins and phenolic compounds. The title compound, (I), which is a derivative of bergenin, has been found to show antioxidant activities as well as neuroprotective activity in rats (Takahashi *et al.*, 2003). We report here the X-ray crystal structure of (I).




(I)

The bond lengths and angles in compound (I) show normal values (Allen *et al.*, 1987). The molecule contains three six-membered rings, *viz*. an aromatic ring *A* (atoms C6–C11), a $\delta$-lactone ring *B* (O3/C5/C6/C11/C12/C4) and a glucopyranose ring *C* (C1–C4/C12/O8). Ring *B* is in a half-chair conformation, with puckering parameters $Q$ = 0.477 (2)(Å, $\theta$ = 120.4 (2)° and $\varphi$ = 261.1 (3)°, while ring *C* adopts a chair

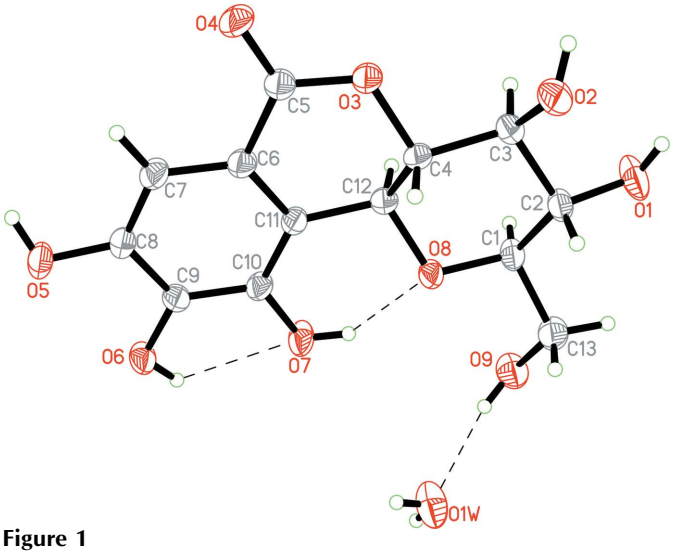



**Figure 1**
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

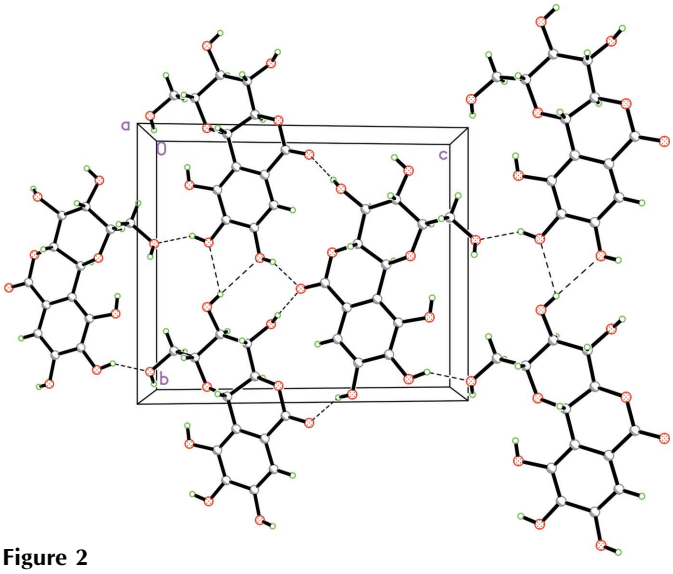



**Figure 2**
A view of the O—H···O hydrogen-bonded (dashed lines) layer. Water molecules have been omitted for clarity.

conformation, with $Q$ = 0.581 (2)(Å, $\theta$ = 166.8 (2)° and $\varphi$ = 134.4 (10)° (Cremer & Pople, 1975). The *B*/*C* junction is *trans* fused. The hydroxyl substituents at C8, C9 and C10 are nearly coplanar with the attached aromatic ring. The conformations of rings *B* and *C* are in agreement with those reported for bergenin monohydrate (Ye *et al.*, 2004).

All hydroxyl groups, except for C3—OH, serve as simultaneous hydrogen-bond donors and acceptors (Table 1). As shown in Fig. 1, the O6—H1*O*6···O7 and O7—H1*O*7···O8 interactions generate rings of graph-set motifs $S(5)$ and $S(6)$, respectively (Bernstein *et al.*, 1995). As seen in Fig. 2, the desmethylbergenin molecules are linked by O—H···O hydrogen bonds, forming a sheet approximately parallel to the (101) plane. Adjacent sheets are cross-linked by O—H···O hydrogen bonds involving the water molecules, and by C—H···O and C—H···$\pi$ interactions (Table 1).

## Experimental

Air-dried vascular parts of the plant *Rivea hypocrateriformis* (Desr.) Choisy (1.0 kg) were chopped and soaked in methanol (6 l) for a period of 30 d at room temperature. The combined methanol extract was concentrated to yield a crude methanol extract (150 g). The methanol extract was suspended in water (700 ml) and the suspension was further extracted with hexane (3 × 1 l, 5.8 g), chloroform (3 × 1 l, 21.3 g), ethyl acetate (3 × 1 l, 13.7 g), butanol (3 × 1 l, 26.5 g) and the residue was soluble in water. The ethyl acetate-soluble fraction was concentrated under reduced pressure and was chromatographed on a silica-gel column using hexane–chloroform (9:1) and the polarity was increased gradually with $CHCl_3$–MeOH (1:24). Various sub-fractions with the same constituents were combined and further purified using flash column chromatography (silica gel) and eluted with $CHCl_3$ to obtain compound (I) in 56.8 mg quantity. An $R_F$ value of 0.35 was noted on thin layer chromatography (30% ethyl acetate–70% petroleum ether) and the compound was recrystallized from chloroform (m.p. 372–374 K).

### *Crystal data*

$C_{13}H_{14}O_9{\cdot}0.5H_2O$
$M_r$ = 323.25
Monoclinic, *C*2
*a* = 8.2820 (19) Å
*b* = 11.566 (3) Å
*c* = 13.959 (3) Å
$\beta$ = 97.545 (5)°
*V* = 1325.5 (5) Å$^3$
*Z* = 4
$D_x$ = 1.620 Mg m$^{-3}$
Mo *K*$\alpha$ radiation
$\mu$ = 0.14 mm$^{-1}$
*T* = 293 (2) K
Needle, colourless
0.33 × 0.13 × 0.08 mm

### *Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.955, $T_{max}$ = 0.989
4048 measured reflections
1373 independent reflections
1320 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019
$\theta_{max}$ = 26.0°

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.079
*S* = 1.22
1373 reflections
214 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.1092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0035 (8)

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

*Cg*3 is the centroid of the ring *A* (atoms C6–C11).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1*O*1···O5$^{i}$ | 0.82 | 2.40 | 3.216 (3) | 179 |
| O1—H1*O*1···O6$^{i}$ | 0.82 | 2.47 | 2.934 (3) | 117 |
| O1*W*—H1*W*1···O1$^{ii}$ | 0.82 (4) | 2.06 (4) | 2.841 (2) | 160 (4) |
| O2—H1*O*2···O4$^{iii}$ | 0.82 | 2.01 | 2.820 (3) | 168 |
| O5—H1*O*5···O4$^{iv}$ | 0.82 | 1.98 | 2.748 (3) | 156 |
| O6—H1*O*6···O7 | 0.82 | 2.28 | 2.709 (3) | 113 |
| O6—H1*O*6···O9$^{ii}$ | 0.82 | 1.90 | 2.632 (3) | 148 |
| O7—H1*O*7···O8 | 0.82 | 1.96 | 2.664 (2) | 144 |
| O9—H1*O*9···O1*W* | 0.84 (4) | 1.95 (4) | 2.776 (3) | 169 (3) |
| C12—H12*A*···O6$^{v}$ | 0.98 | 2.46 | 3.421 (3) | 166 |
| C2—H2*A*···*Cg*3$^{vi}$ | 0.98 | 2.62 | 3.597 (3) | 176 |

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

Atoms H1*W*1 and H1*O*9 were located in a difference map and refined freely. The remaining H atoms were placed in calculated positions (O—H = 0.82 Å and C—H = 0.93–0.98 Å) and allowed to ride on the parent atoms, with $U_{iso}$(H) = 1.5$U_{eq}$(O) for hydroxyl H atoms and 1.2$U_{eq}$(C) for the remaining H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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