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# Ricciocarpin A

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**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.007 Å
*R* factor = 0.061
*wR* factor = 0.146
Data-to-parameter ratio = 7.8

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

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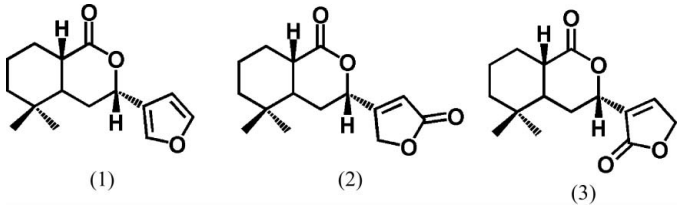
In the crystal structure of the title compound, $C_{15}H_{20}O_3$, the ring junction is *trans* and the cyclohexane ring is in a chair conformation, while the δ-lactone adopts a boat conformation.

## Comment

The ricciocarpins are characteristic sesquiterpenoids first isolated from axenic cultures of the liverwort *Ricciocarpus natans* (Wurzel & Becker, 1990) and were later synthesized (Eicher *et al.*, 1991; Ihara *et al.*, 1993*a*,*b*; Held *et al.*, 2001, 2002; Agapiou & Krische, 2003). Ricciocarpin A, (1), showed significant molluscicidal activity (Wurzel *et al.*, 1990). We have now determined its crystal structure. The results show that the ring junction is *trans*, the cyclohexane ring is in a chair conformation, and the δ-lactone adopts a boat conformation. The conformations of the two six-membered rings are similar to those of ricciocarpin B, (2), and isoricciocarpin B, (3) (Held *et al.*, 2001, 2002).




## Experimental

Fresh *Ricciocarpus natans* (from an axenic culture in Gamborg's B5 medium supplemented with 2% sucrose) was freeze-dried and extracted with methanol. Liquid chromatography afforded ricciocarpin A, which was identified by comparison of the physical data with literature values (Wurzel & Becker, 1990). Colourless single

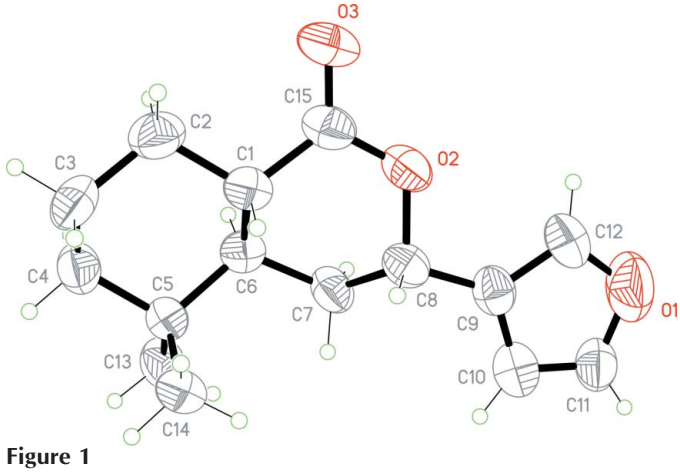



**Figure 1**
A displacement ellipsoid (50% probability) drawing of ricciocarpin A.

crystals were obtained by slow dispersion and evaporation of a methanol solution methanol at 277 K.

*Crystal data*

| | |
|---|---|
| $C_{15}H_{20}O_3$ | $D_x$ = 1.240 Mg m$^{-3}$ |
| $M_r$ = 248.31 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1$ | Cell parameters from 1191 reflections |
| $a$ = 8.5060 (7) Å | $\theta$ = 2.5–26.5° |
| $b$ = 7.4661 (7) Å | $\mu$ = 0.09 mm$^{-1}$ |
| $c$ = 10.733 (1) Å | $T$ = 295 (2) K |
| $\beta$ = 102.626 (2)° | Plate, colourless |
| $V$ = 665.13 (10) Å$^3$ | 0.20 × 0.10 × 0.04 mm |
| $Z$ = 2 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1269 independent reflections |
| | 1188 reflections with $I > 2\sigma(I)$ |
| $\varphi$ and $\omega$ scans | $R_{int}$ = 0.021 |
| Absorption correction: multi-scan (*SADABS*, Sheldrick, 2001) | $\theta_{max}$ = 25.0° |
| | $h = -10 \rightarrow 9$ |
| $T_{min}$ = 0.983, $T_{max}$ = 0.997 | $k = -8 \rightarrow 8$ |
| 3961 measured reflections | $l = -10 \rightarrow 12$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.1173P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.061 | |
| $wR(F^2)$ = 0.146 | |
| $S$ = 1.19 | $(\Delta/\sigma)_{max} < 0.001$ |
| 1269 reflections | $\Delta\rho_{max}$ = 0.20 e Å$^{-3}$ |
| 162 parameters | $\Delta\rho_{min}$ = −0.18 e Å$^{-3}$ |
| H-atom parameters constrained | |

The absolute configuration cannot be determined from X-ray data without the presence of heavy atoms; the Freidel pairs were thus merged before the final refinement. H atoms were placed at calculated positions, with tertiary, secondary, primary and aromatic C—H distances of 0.98, 0.97, 0.96 and 0.93 Å, respectively. The $U_{iso}$(H) values were assigned as 1.20 times $U_{eq}$ of the parent C atom, except in the case of methyl groups where the value were 1.5$U_{eq}$(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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