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5-Formyl-2,3-dihydroisocoumarin

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.069
wR factor = 0.143
Data-to-parameter ratio = 13.3

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

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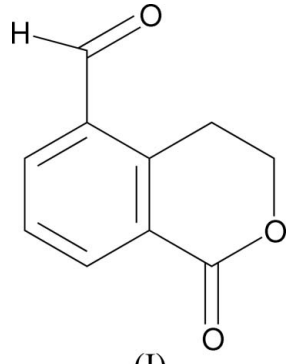
The title compound, erythrocentaurin, $C_{10}H_8O_3$, is a furocoumarin which was isolated from *Enicostema hyssopifolium*. The crystal structure is stabilized by intramolecular C—H···O hydrogen bonds.

Comment

The title compound, erythrocentaurin, (I), was isolated from *Enicostema hyssopifolium* (Willd.) Verdoon of Gentianaceae, which is widely distributed in Southern Pakistan (Omer *et al.*, 1995 and 1996). This plant is considered as medicinally important and used locally by the indigenous people of Tharparkar as a remedy for malaria. In different regions of Pakistan, other species from the same family are used as medicinal plants; they are used as digestive aids, stomachic tonics and also for their depurative, sedative and antipyretic effects (Newall *et al.*, 1996; Sastri, 1952). Chemical surveys reveal that *E. hyssopifolium* contains alkaloids, flavones and their derivatives (Chaudhri *et al.*, 1975; Ghosal *et al.*, 1974; Popov & Marekov, 1959). Erythrocentaurin, (I), has also been found to be an active agent against serine proteases such as chymotrypsin and trypsin; these proteases are involved in the destruction of certain fibrous proteins (Starkey, 1977). We report here the X-ray crystal structure of erythrocentaurin (I).



(I)

The bond lengths in compound (I) show normal values (Allen *et al.*, 1987). The pyrone ring is in a twist-boat conformation, with puckering parameters Q = 0.548 (2) Å, θ = 24.8 (2)° and φ = 272.5 (5)° (Cremer & Pople, 1975). The formyl group is nearly coplanar with the attached benzene ring [O3—C10—C1—C8 = 6.3 (5)°].

The intramolecular C7—H7*B*···O3 hydrogen bond generates a ring of graph-set motif *S*(6) (Bernstein *et al.*, 1995). The stabilization of the structure is supported by this intramolecular C—H···O hydrogen bond. A view of the molecular packing along the *c* axis is shown in Fig. 2.

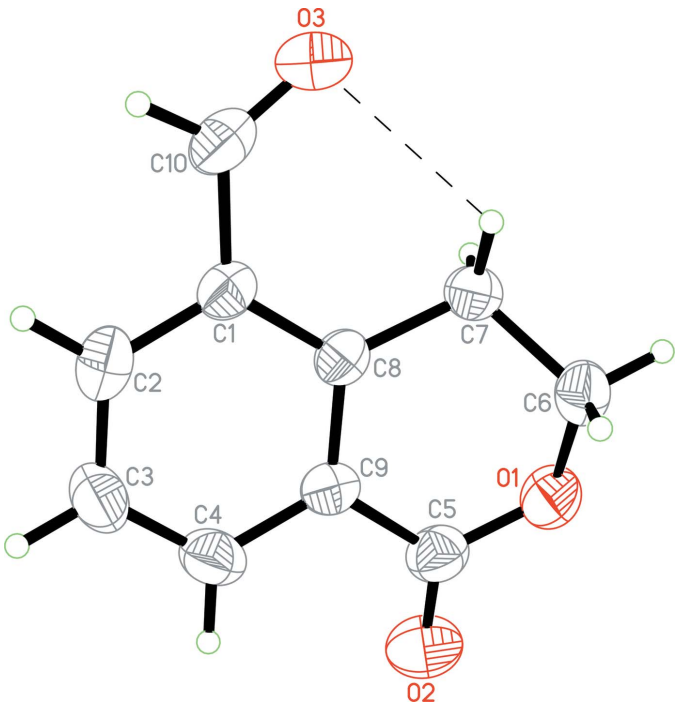


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The hydrogen bond is shown as a dashed line.

Experimental

Air-dried plants of *E. hyssopifolium* (2.5 kg) were chopped and soaked in methanol for a period of 30 d at room temperature. The combined methanol extract was concentrated to yield a crude methanol extract (300 g). This was suspended in water (1 l) and the suspension was further extracted with *n*-hexane (175 g, 3 l), $CHCl_3$ (50 g, 3 l) and *n*-butanol (40 g, 3 l). The $CHCl_3$-soluble fraction was chromatographed on a silica gel column using hexane–$CHCl_3$; the polarity was increased gradually to afford thirteen fractions. Fraction 5 was submitted to repeated FC (230–400 mesh) and eluted with $CHCl_3$:*n*-hexane (30:70) to afford the title compound, (I). An R_f value of 0.50 was noted on thin layer chromatography (same solvent mixture) and the compound was recrystallized from chloroform (m.p. 413–414 K).

Crystal data

$C_{10}H_8O_3$
M_r = 176.16
Monoclinic, $P2_1/c$
a = 7.651 (3) Å
b = 4.0197 (14) Å
c = 26.701 (9) Å
β = 100.337 (9)°
V = 807.9 (5) Å^3
Z = 4
D_x = 1.448 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2249 reflections
θ = 1.5–25.0°
μ = 0.11 mm^{-1}
T = 293 (2) K
Needle, colorless
0.37 × 0.11 × 0.07 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.993
4155 measured reflections
1573 independent reflections
1151 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.0°
$h = -6 \rightarrow 9$
$k = -4 \rightarrow 4$
$l = -32 \rightarrow 32$

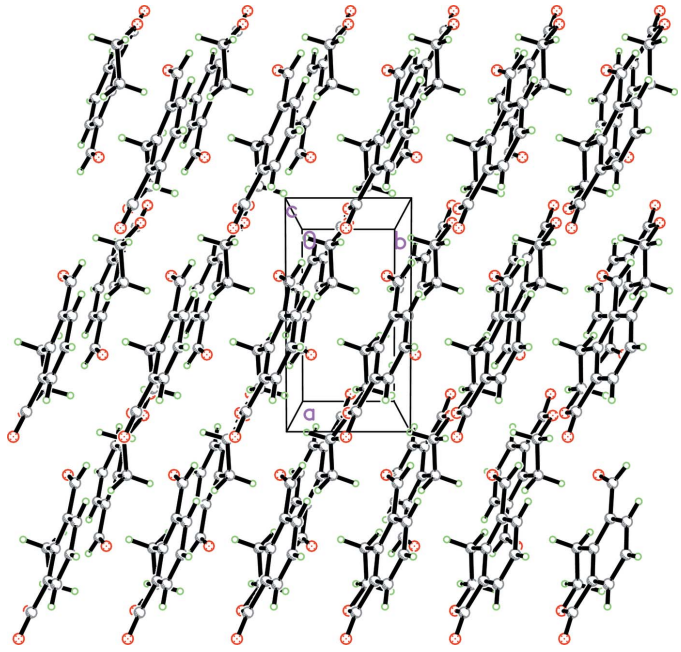


Figure 2
Molecular packing of (I), viewed along the *c* axis.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.143
S = 1.17
1573 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.2194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7*B*···O3	0.97	2.35	2.902 (3)	115

All H atoms were positioned geometrically and allowed to ride on their parent C atoms, with Csp^2—H = 0.93 Å and methylene C—H = 0.96 Å; U_{iso}(H) = 1.2U_{eq}(C).

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* and *PLATON* (Spek, 2003).

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