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Matricarin

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The structure of the sesquiterpene lactone matricarin, $C_{17}H_{20}O_2$, is composed of a seven-membered ring that adopts a chair conformation, fused to two five-membered rings, one of which is essentially planar and the other exhibits an envelope conformation. The structure is devoid of any classical hydrogen bonds.

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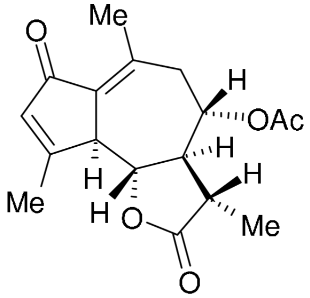
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.093
Data-to-parameter ratio = 12.2

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

Comment

We have isolated a sesquiterpene lactone, matricarin, (I), from *Achillea vermacularis* (Compositae), collected from Tehran area. Martinez *et al.* (1988) have reported the NMR spectroscopic data for (I) and some related guanianolides without quoting the source of (I). In this paper, we report the structure of (I), which has been established by X-ray crystallography.



(I)

The structure of (I) is presented in Fig. 1. The molecular dimensions lie within expected ranges (Orpen *et al.*, 1994) for the corresponding bond distances and angles with mean bond distances: Csp^3-Csp^3 1.527 (5), Csp^3-Csp^2 1.508 (11), Csp^2-Csp^2 1.480 (16), $O-Csp^3$ 1.455 (2), $O-Csp^2$ 1.354 (7), C=C 1.341 (6) and C=O 1.205 (15) Å. The seven-membered ring adopts a chair conformation wherein atoms C5/C6/C8/C9 are in a plane [maximum deviation 0.0096 (8) Å], with C7 0.744 (2) Å above and C1 and C10 1.012 (2) and 1.051 (2) Å, respectively, below this plane. The C1–C5 five-membered ring is essentially planar, with the maximum deviation of any atom being 0.0114 (10) Å. The other five-membered ring, O2/C6/C7/C11/C12, has a C7-envelope conformation with C7 0.608 (3) Å out of the plane of the remaining ring atoms. The structure is devoid of any classical hydrogen bonds.

Experimental

The plants of *Achillea vermacularis* (Compositae) were collected from Tehran area in July, 1997, and were shade-dried; ground whole plant material (6 kg) was extracted with methanol. The resulting gummy material (600 g) was partitioned into hexane, ethyl acetate and *n*-butanol soluble fractions. The ethyl acetate fraction was subjected to flash column chromatography using hexane/ethyl acetate (7:3) over silica gel, affording (I) as colorless needles suitable for X-ray diffraction analysis.

Crystal data

$C_{17}H_{20}O_5$
$M_r = 304.33$
Monoclinic, $P2_1$
$a = 10.2083$ (2) Å
$b = 7.5434$ (2) Å
$c = 11.0118$ (3) Å
$\beta = 109.677$ (1)°
$V = 798.45$ (3) Å^3
$Z = 2$

$D_x = 1.266$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2454 reflections
$\theta = 1.0$–30.0°
$\mu = 0.09$ mm^{-1}
$T = 173$ (2) K
Block, colorless
0.25 × 0.20 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*: Blessing, 1995, 1997)
$T_{\min} = 0.977$, $T_{\max} = 0.983$
4575 measured reflections
2477 independent reflections

2297 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 30.0°$
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$
Intensity decay: <0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.093$
$S = 1.07$
2477 reflections
203 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.2274 (18)	O4—C16	1.346 (2)
O2—C12	1.361 (2)	O4—C8	1.4531 (16)
O2—C6	1.4560 (16)	O5—C16	1.194 (2)
O3—C12	1.195 (2)		
C12—O2—C6	108.82 (11)	C16—O4—C8	117.07 (13)

The H atoms were located from a difference Fourier synthesis and were included in the refinement at geometrically idealized positions, with C—H distances in the range 0.95–1.00 Å, utilizing riding models and allowing isotropic displacement parameters 1.2 (non-methyl) and 1.5 (methyl) times the equivalent isotropic displacement parameters of the atoms to which they were bonded. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged and the absolute configuration cannot be determined from the crystallographic experiment. The absolute configuration depicted in the *Scheme* and shown in Fig. 1 was chosen arbitrarily.

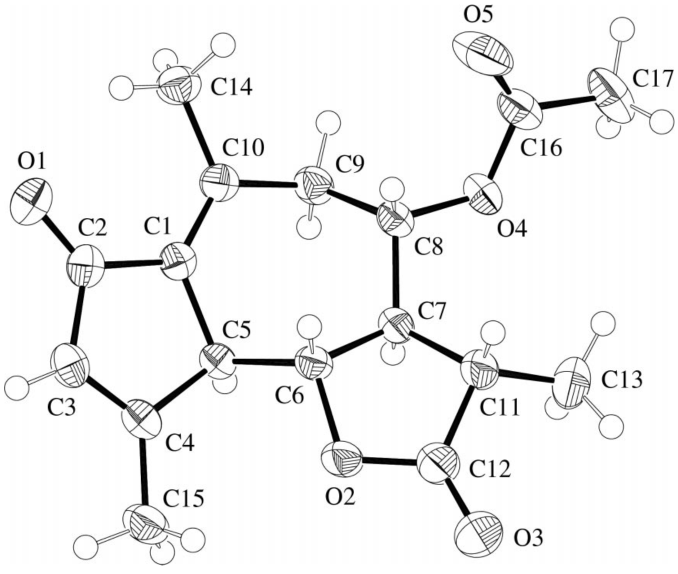


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) with displacement ellipsoids plotted at the 50% probability level.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1994); software used to prepare material for publication: *SHELXL*97.

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