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A new *Cryptocarya* lactone

Jeffrey R. Deschamps,[a]* Clifford George,[a] Judith L. Flippen-Anderson[a] and Gayland Spencer[b]

[a]Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA, and [b]National Center for Agricultural Utilization Research, Agriculture Research Service, United States Department of Agriculture, 1815 N. University St., Peoria, IL 61604, USA

Correspondence e-mail: deschamp@harker.nrl.navy.mil

Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.164
Data-to-parameter ratio = 5.9

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

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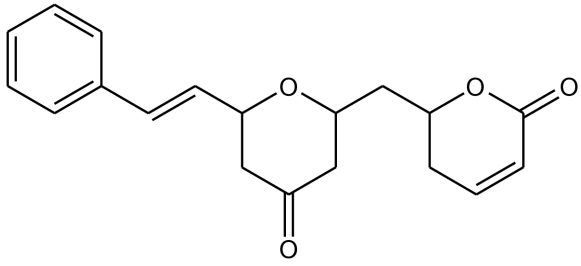
The title compound 6-({4-oxo-6-[(1*E*)-2-phenylvinyl]-2*H*-3,5,6-trihydropyran-2-yl}methyl)-5*H*-6-hydropyran-2-one, $C_{19}H_{24}O_4$, is a germination inhibitor isolated from the seeds of *Cryptocarya wightiana*.

Comment

Some extracts of seeds of plants in the genera *Cryptocarya* have antigermination properties. A study by Spencer *et al.* (1984) described (−)-cryptocaryalactone {1-[(6-oxo-2*H*-3-hydropyran-2-yl)methyl]-3-phenylpropyl acetate} and (−)-deacetyl-cryptocaryalactone [6-(2-hydroxy-4-phenylbutyl)-5*H*-6-hydropyran-2-one], germination inhibitors from *Cryptocarya moschata* seeds (*i.e.* Brazilian nutmeg). Both (−)-cryptocaryalactone and (−)-deacetylcryptocaryalactone inhibit the germination of *Abutilon theophrasti* (velvetleaf), with the deacetyl compound being more effective. Under conditions that were lethal to velvetleaf (*i.e.* 94% inhibition of germination), corn was virtually unaffected, and soybeans were only minimally affected (*i.e.* 21% inhibition of germination). Based on these results, a project was initiated to identify other natural products that inhibit seed germination of problem weed seeds (such as velvetleaf).

Extracts were prepared from a series of *Cryptocarya* seeds collected in Sri Lanka. An extract from the seeds of *Cryptocarya wightiana* showed antigermination properties. This extract was fractionated further and the active component identified as 6-({4-oxo-6-[(1*E*)-2-phenylvinyl]-2*H*-3,5,6-trihydropyran-2-yl}methyl)-5*H*-6-hydropyran-2-one, (I).



(I)

Two molecules comprise the asymmetric unit of (I) (Fig. 1). Both molecules have disordered phenyl rings with the two alternative positions at approximately 90° to each other and approximately equal occupancy of the two positions.

Experimental

The *Cryptocarya wightiana* seed extract was fractionated by chromatography on silica eluted with mixtures of hexane and ethyl

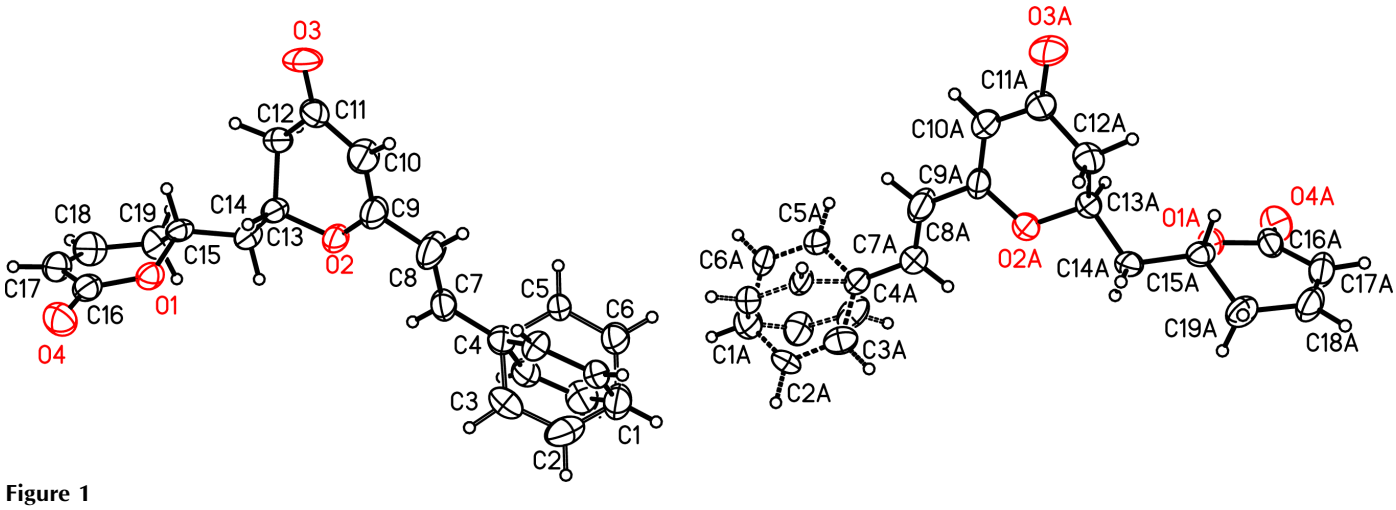


Figure 1
Displacement ellipsoid plot (Bruker, 1997) of (I) showing the ellipsoids at the 30% probability level. H atoms are shown as small circles of arbitrary radii. Atom labels for the alternative conformation of the disordered phenyl rings have been omitted for clarity.

acetate. The ethyl acetate fraction was further separated by preparative HPLC on ODS with CH_3CN/H_2O/EtOH (1/1/4).

Crystal data

$C_{19}H_{18}O_4$
M_r = 310.33
Monoclinic, $P2_1$
a = 5.2472 (1) Å
b = 5.2528 (1) Å
c = 58.726 (1) Å
β = 92.543 (1)°
V = 1617.04 (5) Å^3
Z = 4
D_x = 1.275 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 5137 reflections
θ = 3.8–56.4°
μ = 0.73 mm^{-1}
T = 153 (2) K
Prism, colorless
0.84 × 0.35 × 0.09 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Bruker, 2000)
T_{min} = 0.712, T_{max} = 0.937
5150 measured reflections
2890 independent reflections
2868 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 56.4°
$h = -5 \rightarrow 4$
$k = -4 \rightarrow 5$
$l = -58 \rightarrow 62$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.164
S = 0.99
2890 reflections
489 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1336P)^2 + 0.581P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.015
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983); 906 Friedel pairs
Flack parameter = 0.0 (4)

Data were collected at three settings of 2θ. With the detector at 95°, 2θ data extend to about 113° [*i.e.* $\sin(\theta_{max})/\lambda$ = 0.5402]. Unfortunately, the data crystal for this compound is no longer available so additional data can not be collected. The experimental restriction accounts for, in part, the relatively low data/parameter ratio. H atoms were refined with the riding model.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *XS* (Sheldrick, 1990); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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