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Crystal structure of pseudoguainolide

Noureddine Beghidja,[a]* Samir Benayache,[a] Fadila Benayache,[a] David W. Knight[b] and Benson M. Kariuki[b]*

[a]Unité de Recherche VARENBIOMOL, Constantine 1 University, Constantine 25000, Algeria, and [b]School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, Wales. *Correspondence e-mail: nourbeghidja@yahoo.fr, kariukib@cf.ac.uk

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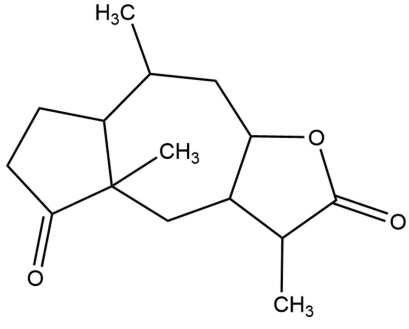
The lactone ring in the title molecule, $C_{15}H_{22}O_3$ (systematic name: 3,4a,8-trimethyldodecahydroazuleno[6,5-*b*]furan-2,5-dione), assumes an envelope conformation with the methine C atom adjacent to the the methine C atom carrying the methyl substituent being the flap atom. The other five-membered ring adopts a twisted conformation with the twist being about the methine–methylene C—C bond. The seven-membered ring is based on a twisted boat conformation. No specific interactions are noted in the the crystal packing.

Keywords: crystal structure; plant extract; *inula graveolens*.

CCDC reference: 1047797

1. Related literature

For background to *inula graveolens*, see: Chiappini & Fardella (1980); Rustaiyan *et al.* (1987). For related structures, see: Herz *et al.* (1982); Schmidt *et al.* (1996); Wu *et al.* (2012); Billodeaux *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{15}H_{22}O_3$
M_r = 250.33
Orthorhombic, $P2_12_12_1$
a = 7.4320 (3) Å
b = 11.9278 (3) Å
c = 15.3152 (6) Å
V = 1357.65 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 150 K
0.20 × 0.20 × 0.04 mm

2.2. Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.984, T_{max} = 0.997
9382 measured reflections
3098 independent reflections
2533 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.107
S = 1.08
3098 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5358).

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S1. Comment

Inula graveolens have consistently been the subject of research interest (Chiappini & Fardella, 1980; Rustaiyan *et al.*, 1987). Our interest is in the extracts from aerial parts of Algerian species such as stems, flowers and leaves. The asymmetric unit of the crystal structure consists of a single molecule (Fig. 1). In the molecule, the lactone ring assumes an envelope conformation. In the crystal structure, the planes of the lactone rings are approximately parallel. The molecules are arranged with the lactone rings stacked parallel to the *a* axis. Structures of some related compounds have been reported (Herz *et al.*, 1982; Schmidt *et al.*, 1996; Wu *et al.*, 2012; Billodeaux *et al.*, 2014).

S2. Experimental

The air-dried aerial parts of *inula graveolens* (500 g) were extracted with acetone/Et_2O (1:1) at room temperature. The solution was filtered off and concentrated under reduced pressure to give a pale-yellow gum (9 g). The gum was subjected to successive column chromatography (silica gel) and TLC (silica gel, PF254). Eleven fractions were obtained. Fraction 5 gave a material which crystallized as colourless crystals with a melting point of 152 ^{o}C.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.96–0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2–1.5U_{equiv}(C).

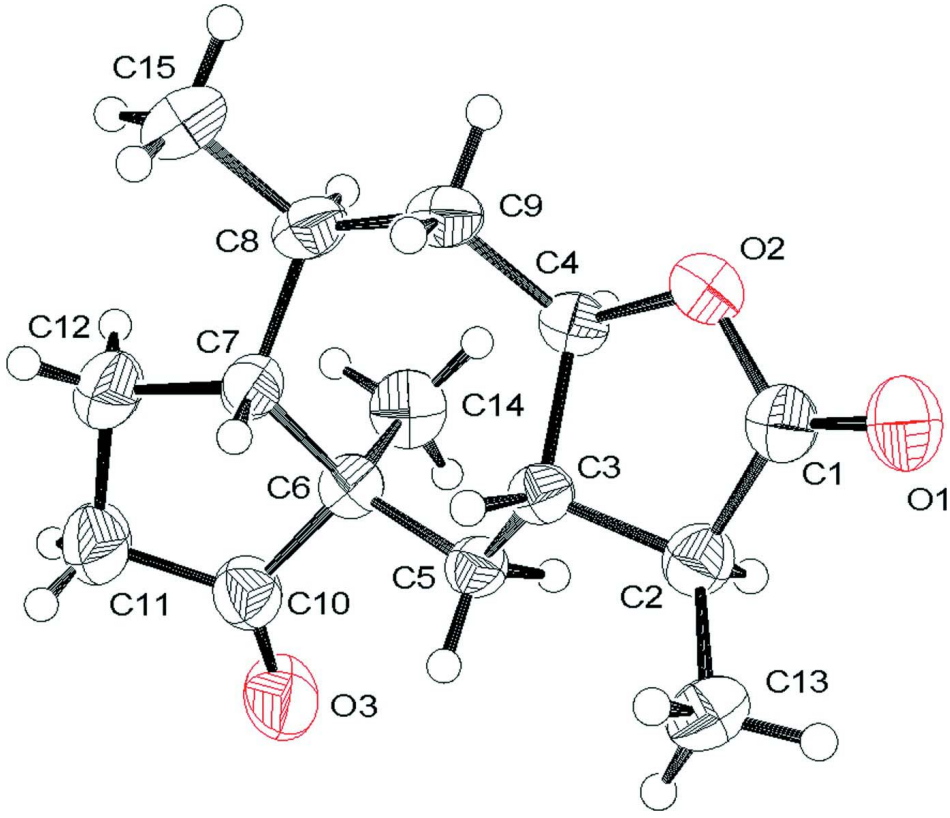

Figure 1

A molecule showing atom labels and 50% probability displacement ellipsoids for non-H atoms.

3,4a,8-Trimethyldodecahydroazuleno[6,5-*b*]furan-2,5-dione

Crystal data

$C_{15}H_{22}O_3$
$M_r = 250.33$
Orthorhombic, $P2_12_12_1$
$a = 7.4320$ (3) Å
$b = 11.9278$ (3) Å
$c = 15.3152$ (6) Å
$V = 1357.65$ (8) Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.225$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3098 reflections
$\theta = 3.1$–$27.5°$
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Plate, colourless
0.20 × 0.20 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD slices, ω and φ scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.984$, $T_{max} = 0.997$
9382 measured reflections
3098 independent reflections
2533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.107$
$S = 1.08$
3098 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.2031P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8136 (2)	0.24699 (16)	0.52812 (12)	0.0383 (4)
C2	0.8423 (3)	0.16380 (15)	0.45546 (12)	0.0407 (4)
H2	0.9722	0.1503	0.4522	0.049*
C3	0.7918 (2)	0.22980 (13)	0.37360 (11)	0.0318 (4)
H3	0.6620	0.2220	0.3642	0.038*
C4	0.8307 (2)	0.35109 (14)	0.40005 (11)	0.0338 (4)
H4	0.9552	0.3703	0.3852	0.041*
C5	0.8884 (2)	0.19024 (14)	0.29170 (12)	0.0346 (4)
H5A	0.8386	0.1183	0.2748	0.041*
H5B	1.0142	0.1785	0.3060	0.041*
C6	0.8781 (2)	0.26974 (15)	0.21218 (12)	0.0371 (4)
C7	0.7099 (3)	0.34762 (15)	0.20872 (12)	0.0388 (4)
H7	0.6065	0.3039	0.2292	0.047*
C8	0.7176 (3)	0.45618 (14)	0.26350 (13)	0.0429 (5)
H8	0.8326	0.4932	0.2513	0.052*
C9	0.7050 (3)	0.43722 (14)	0.36244 (12)	0.0396 (4)
H9A	0.7276	0.5081	0.3914	0.048*
H9B	0.5827	0.4152	0.3763	0.048*
C10	0.8618 (3)	0.20169 (18)	0.12824 (14)	0.0495 (5)
C11	0.7283 (4)	0.25562 (19)	0.06728 (14)	0.0630 (6)
H11A	0.6211	0.2099	0.0617	0.076*
H11B	0.7805	0.2665	0.0098	0.076*
C12	0.6842 (3)	0.36830 (18)	0.10993 (14)	0.0583 (6)
H12A	0.5613	0.3905	0.0973	0.070*
H12B	0.7651	0.4264	0.0893	0.070*

C13	0.7564 (3)	0.05319 (14)	0.47094 (13)	0.0453 (5)
H13A	0.6282	0.0623	0.4737	0.068*
H13B	0.7992	0.0226	0.5251	0.068*
H13C	0.7864	0.0032	0.4240	0.068*
C14	1.0567 (3)	0.33493 (19)	0.20250 (15)	0.0519 (5)
H14A	1.0488	0.3846	0.1533	0.078*
H14B	1.1536	0.2830	0.1937	0.078*
H14C	1.0786	0.3777	0.2545	0.078*
C15	0.5664 (3)	0.53701 (18)	0.23775 (17)	0.0650 (7)
H15A	0.5670	0.6004	0.2764	0.098*
H15B	0.4526	0.4992	0.2419	0.098*
H15C	0.5847	0.5620	0.1788	0.098*
O1	0.79726 (18)	0.22938 (12)	0.60507 (9)	0.0478 (3)
O2	0.80792 (16)	0.35200 (10)	0.49559 (7)	0.0390 (3)
O3	0.9465 (2)	0.11791 (15)	0.11224 (11)	0.0721 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0265 (8)	0.0451 (10)	0.0433 (11)	−0.0014 (8)	−0.0003 (8)	0.0008 (8)
C2	0.0416 (10)	0.0404 (9)	0.0400 (10)	−0.0014 (8)	0.0015 (8)	0.0040 (8)
C3	0.0265 (8)	0.0298 (8)	0.0392 (9)	−0.0018 (7)	0.0004 (7)	0.0008 (7)
C4	0.0296 (9)	0.0335 (8)	0.0384 (9)	−0.0040 (7)	0.0040 (7)	−0.0020 (7)
C5	0.0314 (9)	0.0331 (9)	0.0393 (10)	0.0025 (7)	−0.0013 (8)	−0.0011 (7)
C6	0.0344 (9)	0.0381 (9)	0.0387 (10)	−0.0011 (8)	0.0020 (8)	0.0015 (8)
C7	0.0381 (9)	0.0357 (8)	0.0426 (10)	0.0007 (8)	−0.0058 (8)	0.0050 (8)
C8	0.0453 (11)	0.0301 (8)	0.0534 (12)	0.0008 (9)	0.0005 (9)	0.0064 (8)
C9	0.0373 (10)	0.0303 (8)	0.0512 (11)	−0.0012 (8)	0.0023 (9)	−0.0031 (7)
C10	0.0526 (12)	0.0532 (12)	0.0426 (12)	0.0015 (10)	−0.0019 (9)	−0.0027 (9)
C11	0.0807 (17)	0.0660 (14)	0.0424 (12)	0.0121 (14)	−0.0134 (12)	−0.0037 (10)
C12	0.0731 (15)	0.0544 (12)	0.0472 (12)	0.0092 (12)	−0.0115 (12)	0.0079 (9)
C13	0.0473 (11)	0.0383 (10)	0.0502 (12)	0.0076 (9)	0.0064 (9)	0.0056 (8)
C14	0.0441 (11)	0.0595 (12)	0.0522 (13)	−0.0096 (10)	0.0101 (10)	0.0073 (10)
C15	0.0772 (17)	0.0419 (11)	0.0761 (17)	0.0178 (12)	−0.0123 (14)	0.0068 (11)
O1	0.0432 (8)	0.0636 (8)	0.0367 (8)	−0.0003 (7)	0.0028 (6)	0.0023 (6)
O2	0.0380 (7)	0.0401 (7)	0.0388 (7)	−0.0038 (6)	0.0016 (6)	−0.0055 (5)
O3	0.0840 (12)	0.0763 (11)	0.0560 (10)	0.0307 (10)	−0.0134 (9)	−0.0248 (9)

Geometric parameters (Å, º)

C1—O1	1.203 (2)	C8—C15	1.532 (3)
C1—O2	1.349 (2)	C8—C9	1.535 (3)
C1—C2	1.506 (3)	C8—H8	0.9800
C2—C13	1.485 (2)	C9—H9A	0.9700
C2—C3	1.527 (2)	C9—H9B	0.9700
C2—H2	0.9800	C10—O3	1.206 (2)
C3—C5	1.520 (2)	C10—C11	1.507 (3)
C3—C4	1.530 (2)	C11—C12	1.530 (3)

C3—H3	0.9800	C11—H11A	0.9700
C4—O2	1.473 (2)	C11—H11B	0.9700
C4—C9	1.503 (2)	C12—H12A	0.9700
C4—H4	0.9800	C12—H12B	0.9700
C5—C6	1.545 (3)	C13—H13A	0.9600
C5—H5A	0.9700	C13—H13B	0.9600
C5—H5B	0.9700	C13—H13C	0.9600
C6—C10	1.525 (3)	C14—H14A	0.9600
C6—C14	1.546 (3)	C14—H14B	0.9600
C6—C7	1.558 (3)	C14—H14C	0.9600
C7—C8	1.544 (3)	C15—H15A	0.9600
C7—C12	1.545 (3)	C15—H15B	0.9600
C7—H7	0.9800	C15—H15C	0.9600

O1—C1—O2	121.39 (17)	C9—C8—H8	107.9
O1—C1—C2	128.53 (18)	C7—C8—H8	107.9
O2—C1—C2	110.08 (15)	C4—C9—C8	116.18 (15)
C13—C2—C1	114.00 (15)	C4—C9—H9A	108.2
C13—C2—C3	118.92 (16)	C8—C9—H9A	108.2
C1—C2—C3	103.43 (14)	C4—C9—H9B	108.2
C13—C2—H2	106.6	C8—C9—H9B	108.2
C1—C2—H2	106.6	H9A—C9—H9B	107.4
C3—C2—H2	106.6	O3—C10—C11	124.8 (2)
C5—C3—C2	113.67 (14)	O3—C10—C6	124.83 (19)
C5—C3—C4	115.01 (14)	C11—C10—C6	110.31 (17)
C2—C3—C4	102.93 (13)	C10—C11—C12	104.58 (18)
C5—C3—H3	108.3	C10—C11—H11A	110.8
C2—C3—H3	108.3	C12—C11—H11A	110.8
C4—C3—H3	108.3	C10—C11—H11B	110.8
O2—C4—C9	107.71 (13)	C12—C11—H11B	110.8
O2—C4—C3	104.38 (13)	H11A—C11—H11B	108.9
C9—C4—C3	115.32 (15)	C11—C12—C7	104.55 (16)
O2—C4—H4	109.7	C11—C12—H12A	110.8
C9—C4—H4	109.7	C7—C12—H12A	110.8
C3—C4—H4	109.7	C11—C12—H12B	110.8
C3—C5—C6	115.87 (13)	C7—C12—H12B	110.8
C3—C5—H5A	108.3	H12A—C12—H12B	108.9
C6—C5—H5A	108.3	C2—C13—H13A	109.5
C3—C5—H5B	108.3	C2—C13—H13B	109.5
C6—C5—H5B	108.3	H13A—C13—H13B	109.5
H5A—C5—H5B	107.4	C2—C13—H13C	109.5
C10—C6—C5	109.98 (14)	H13A—C13—H13C	109.5
C10—C6—C14	104.76 (16)	H13B—C13—H13C	109.5
C5—C6—C14	109.96 (15)	C6—C14—H14A	109.5
C10—C6—C7	103.01 (15)	C6—C14—H14B	109.5
C5—C6—C7	115.63 (14)	H14A—C14—H14B	109.5
C14—C6—C7	112.70 (14)	C6—C14—H14C	109.5
C8—C7—C12	113.76 (15)	H14A—C14—H14C	109.5

C8—C7—C6	116.85 (15)	H14B—C14—H14C	109.5
C12—C7—C6	103.16 (15)	C8—C15—H15A	109.5
C8—C7—H7	107.5	C8—C15—H15B	109.5
C12—C7—H7	107.5	H15A—C15—H15B	109.5
C6—C7—H7	107.5	C8—C15—H15C	109.5
C15—C8—C9	107.59 (17)	H15A—C15—H15C	109.5
C15—C8—C7	111.13 (17)	H15B—C15—H15C	109.5
C9—C8—C7	114.24 (14)	C1—O2—C4	110.89 (13)
C15—C8—H8	107.9		