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(+/-)-4-(2-Oxo-1,2,3,4,4a,5,6,7-octa-
hydroquinolin-8-yl)butan-2-one. A
Michael-reaction adduct from acid-cata-
lyzed alkylation of a bicyclic enamide

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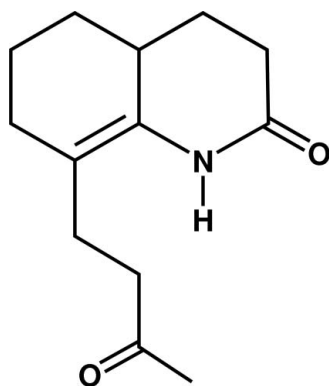
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.042; wR factor = 0.115; data-to-parameter ratio = 13.4.

The title racemate, $\text{C}_{13}\text{H}_{19}\text{NO}_2$, was isolated from the Michael condensation of methyl vinyl ketone with 3,4,5,6,7,8-hexahydro-2-quinolone under acidic conditions. The compound aggregates as dimers by centrosymmetric hydrogen-bonded pairing of the carboxamide groups [$\text{N}\cdots\text{O} = 2.9321$ (16) Å and $\text{N}-\text{H}\cdots\text{O} = 174$ (2)°]. The packing includes three close intermolecular dipolar contacts, involving both O atoms.

Related literature

For related literature, see: Davison *et al.* (2004); Elad & Ginsburg (1953); El-Barbary *et al.* (1982); Lalancette *et al.* (2002); Lions (1938); Murahashi *et al.* (1993); Ninomiya *et al.* (1971); Steiner (1997); Stork (1968); Stork *et al.* (1968); Thompson *et al.* (2006); Trushina *et al.* (1993); Zewge *et al.* (1998, 2006); Zwege *et al.* (1999).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{19}\text{NO}_2$
 $M_r = 221.29$
Triclinic, $P\bar{1}$
 $a = 5.4984$ (2) Å
 $b = 10.4062$ (3) Å
 $c = 10.7217$ (3) Å
 $\alpha = 104.6939$ (11)°
 $\beta = 97.7636$ (14)°
 $\gamma = 91.1473$ (12)°
 $V = 587.02$ (3) Å³
 $Z = 2$
Cu $K\alpha$ radiation
 $\mu = 0.67$ mm⁻¹
 $T = 100$ (2) K
 $0.50 \times 0.18 \times 0.13$ mm

Data collection

Bruker SMART CCD APEX II
area-detector diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2001)
 $T_{\min} = 0.731$, $T_{\max} = 0.918$
8172 measured reflections
2022 independent reflections
1913 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.115$
 $S = 1.05$
2022 reflections
151 parameters
H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\text{max}} = 0.52$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond and close-contact geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.89 (2)	2.04 (2)	2.9321 (16)	174 (2)
$\text{C9}-\text{H9B}\cdots\text{O1}^i$	0.99	2.47	3.1517 (18)	126
$\text{C10}-\text{H10B}\cdots\text{O1}^i$	0.99	2.56	3.0568 (17)	111
$\text{C3}-\text{H3B}\cdots\text{O2}^{ii}$	0.99	2.59	3.387 (2)	137

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 2, -y + 1, -z + 1$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2004); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2489).

References

- Bruker (2005). *SAINT*. Version 7.23a. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Version 2.0-2. Bruker AXS Inc., Madison, Wisconsin, USA.
Davison, M., Thompson, H. W. & Lalancette, R. A. (2004). *Acta Cryst.* **C60**, o242–o244.
Elad, D. & Ginsburg, D. (1953). *J. Chem. Soc.* pp. 4137–4139.
El-Barbary, A. A., Carlsson, S. & Lawesson, S. O. (1982). *Tetrahedron*, **38**, 405–412.
Lalancette, R. A., Brunskill, A. P. J. & Thompson, H. W. (2002). *Acta Cryst.* **C58**, o491–o493.
Lions, F. (1938). *J. Proc. R. Soc. N. S. Wales*, **71**, 192–208.
Murahashi, S., Sasao, S., Saito, E. & Naota, T. (1993). *Tetrahedron*, **49**, 8805–8826.

- Ninomiya, I., Naito, T., Higuchi, S. & Mori, T. (1971). *Chem. Commun.* pp. 457–458.
- Sheldrick, G. M. (2001). *SADABS*. Version 2. University of Göttingen, Germany.
- Sheldrick, G. M. (2004). *SHELXTL*. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
- Steiner, T. (1997). *Chem. Commun.* pp. 727–734.
- Stork, G. (1968). *Pure Appl. Chem.* **17**, 383–401.
- Stork, G., Kretchmer, R. A. & Schlessinger, R. H. (1968). *J. Am. Chem. Soc.* **90**, 1647–1648.
- Thompson, H. W., Kikolski, E. M. & Lalancette, R. A. (2006). *Acta Cryst.* **E62**, o4342–o4344.
- Trushina, E. V., Bel'skii, V. K., Zavodnik, V. E., Stash, A. I. & Klimenko, S. K. (1993). *Kristallografiya*, **38**, 63–68.
- Zewge, D., Brunskill, A. P. J., Lalancette, R. A. & Thompson, H. W. (1998). *Acta Cryst.* **C54**, 1651–1653.
- Zewge, D., Thompson, H. W., Lalancette, R. A. & Brunskill, A. P. J. (2006). *Acta Cryst.* **E62**, o11–o12.
- Zewge, D., Thompson, H. W., Brunskill, A. P. J. & Lalancette, R. A. (1999). *Acta Cryst.* **C55**, 1370–1373.

supplementary materials

Acta Cryst. (2007). E63, o4007-o4008 [doi:10.1107/S160053680704319X]

(+/-)-4-(2-Oxo-1,2,3,4,4a,5,6,7-octahydroquinolin-8-yl)butan-2-one. A Michael-reaction adduct from acid-catalyzed alkylation of a bicyclic enamide

D. Zewge, A. P. J. Brunskill, H. W. Thompson and R. A. Lalancette

Comment

An interest in keto acids containing the naphthalene skeleton led us to alkylations and annulations of several simple compounds derivable directly from cyclohexanone (Lalancette *et al.*, 2002; Davison *et al.*, 2004; Thompson *et al.*, 2006; Zewge *et al.*, 1998, 2006; Zwege *et al.*, 1999). One such acid-catalyzed reaction, employing 3,4,5,6,7,8-hexahydro-2-quinolone, yielded the title compound.

Fig. 1 illustrates the asymmetric unit and the staggered conformation of the oxobutyl chain, which extends away from the plane of the bicyclic system, utilizing the molecular face that bears the 4a methine hydrogen. Although the molecule's bicyclic portion is quite flat, extension of the chain in this direction avoids an apparently serious interaction with the pseudo-axial C7 hydrogen on the opposite face.

Fig. 2 shows the dimers (Table 1) formed by typical centrosymmetric H-bonded carboxamide pairing (Zwege *et al.*, 1999), the same pattern reported by Trushina *et al.* (1993) for two different crystal phases of our starting material. Since $Z = 2$, the relationship among these dimer units in the packing is entirely translational.

The double-bond placement is of interest with respect to its position in the starting material, as well as regarding the mechanism of our alkylation. Our starting material was first reported in 1938 (Lions) and is obtainable by direct base-catalyzed alkylation of cyclohexanone with acrylamide (Elad & Ginsburg, 1953), albeit in poor yield. It later became easily available *via* the enamine (Stork, 1968; Stork *et al.*, 1968; Ninomiya *et al.*, 1971). In 1982, El-Barbary *et al.* reported isolating a mixture including the less-substituted 3,4,4a,5,6,7-hexahydro enamide isomer. Subsequent careful work by Murahashi *et al.* (1993) showed that these two isomers are readily equilibrated by acid; their equilibrium, approached from both sides, contained an 85:15 ratio of the two, separable by column chromatography. This ratio represents a free-energy difference of *ca* 1.0 kcal/mol. Thus, for this system, precedent is well established for the existence of double-bond isomers accessed *via* acid mechanisms. We assume that our alkylation is a normal acid-catalyzed Michael condensation, occurring *via* the less substituted isomer, and that the double-bond position is equilibrated in the product, where this position is presumably stabler because of the added substituent at C8.

Within the 2.6 Å range we survey for non-bonded C—H···O and C—H···N packing interactions (Steiner, 1997), three close intermolecular contacts were found, involving both O atoms (Table 1).

Experimental

The title compound, previously unreported, was isolated from a reaction in which methanolic MVK was added slowly at 298 K to an equimolar amount of 3,4,5,6,7,8-hexahydro-2-quinolone in a stirred benzene solution containing 0.7 equiv. of H₂SO₄. Overnight stirring and the usual workup led, upon refrigeration with CH₂Cl₂/diethyl ether, to an 18% yield of

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the title compound, mp 363–387 K. Recrystallization from the same solvent mixture gave crystals suitable for X-ray, mp 388–390 K.

Refinement

All H atoms were found in electron density difference maps. The amide H was fully refined. The methyl H atoms were put in ideally staggered positions with C—H distances of 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The methylene and methine Hs were placed in geometrically idealized positions and constrained to ride on their parent C atoms with C—H distances of 0.99 and 1.00 Å, respectively, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

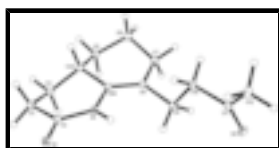


Fig. 1. A view of the asymmetric unit with its numbering. Displacement ellipsoids are drawn at the 20% probability level.

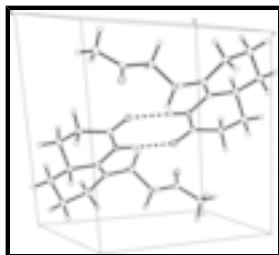


Fig. 2. A partial packing diagram, illustrating the centrosymmetric dimerization of the asymmetric unit around 1/2, 1/2, 1/2 in the chosen cell. Displacement ellipsoids are drawn at the 20% probability level. Hydrogen bonds are shown as dashed lines.

'(+/-)-4-(2-Oxo-1,2,3,4,4a,5,6,7-octahydroquinolin-8-yl)butan-2-one'

Crystal data

$\text{C}_{13}\text{H}_{19}\text{NO}_2$	$Z = 2$
$M_r = 221.29$	$F_{000} = 240$
Triclinic, $P\bar{1}$	$D_x = 1.252 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Melting point: 388 K
$a = 5.4984 (2) \text{ \AA}$	Cu $K\alpha$ radiation
$b = 10.4062 (3) \text{ \AA}$	$\lambda = 1.54178 \text{ \AA}$
$c = 10.7217 (3) \text{ \AA}$	Cell parameters from 8172 reflections
$\alpha = 104.6939 (11)^\circ$	$\theta = 4.4\text{--}68.2^\circ$
$\beta = 97.7636 (14)^\circ$	$\mu = 0.67 \text{ mm}^{-1}$
$\gamma = 91.1473 (12)^\circ$	$T = 100 (2) \text{ K}$
$V = 587.02 (3) \text{ \AA}^3$	Needle, colourless
	$0.50 \times 0.18 \times 0.13 \text{ mm}$

Data collection

Bruker SMART CCD APEX II area-detector diffractometer	2022 independent reflections
Radiation source: fine-focus sealed tube	1913 reflections with $I > 2\sigma(I)$

Monochromator: graphite $R_{\text{int}} = 0.017$
 $T = 100(2)$ K $\theta_{\text{max}} = 68.2^\circ$
 φ and ω scans $\theta_{\text{min}} = 4.4^\circ$
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2001) $h = -6 \rightarrow 6$
 $T_{\text{min}} = 0.731$, $T_{\text{max}} = 0.918$ $k = -12 \rightarrow 12$
 8172 measured reflections $l = -12 \rightarrow 12$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.042$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.115$ $w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.3425P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.05$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 2022 reflections $\Delta\rho_{\text{max}} = 0.52 \text{ e } \text{\AA}^{-3}$
 151 parameters $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: ?

Special details

Experimental. 'crystal mounted on cryoloop using Paratone-N'

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.4797 (2)	0.36581 (10)	0.56584 (10)	0.0247 (3)
N1	0.6993 (2)	0.36593 (12)	0.40269 (12)	0.0208 (3)
H1	0.650 (4)	0.448 (2)	0.4076 (19)	0.030 (5)*
O2	0.8942 (2)	0.79200 (10)	0.23977 (11)	0.0299 (3)
C2	0.6144 (3)	0.30732 (14)	0.48882 (14)	0.0207 (3)
C3	0.6883 (3)	0.16837 (14)	0.48849 (15)	0.0237 (4)
H3A	0.5474	0.1046	0.4455	0.028*
H3B	0.7268	0.1624	0.5797	0.028*
C4A	0.8779 (3)	0.16100 (14)	0.28774 (15)	0.0231 (4)

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H4AA	0.7224	0.1133	0.2361	0.028*
C4	0.9101 (3)	0.12817 (15)	0.41878 (15)	0.0274 (4)
H4A	1.0609	0.1759	0.4732	0.033*
H4B	0.9293	0.0315	0.4059	0.033*
C5	1.0858 (3)	0.11430 (15)	0.21097 (15)	0.0270 (4)
H5A	1.2450	0.1442	0.2675	0.032*
H5B	1.0768	0.0158	0.1836	0.032*
C6	1.0741 (3)	0.16785 (16)	0.09162 (16)	0.0299 (4)
H6A	0.9150	0.1387	0.0348	0.036*
H6B	1.2076	0.1326	0.0414	0.036*
C7	1.1011 (3)	0.31919 (15)	0.13364 (15)	0.0251 (4)
H7A	1.2768	0.3468	0.1649	0.030*
H7B	1.0505	0.3538	0.0567	0.030*
C8	0.9520 (3)	0.38189 (14)	0.23976 (14)	0.0208 (3)
C8A	0.8484 (3)	0.30845 (14)	0.30658 (14)	0.0203 (3)
C9	0.9346 (3)	0.53031 (14)	0.26331 (14)	0.0206 (3)
H9A	1.0909	0.5695	0.2492	0.025*
H9B	0.9129	0.5686	0.3553	0.025*
C10	0.7225 (3)	0.56887 (14)	0.17467 (14)	0.0215 (3)
H10A	0.7363	0.5239	0.0829	0.026*
H10B	0.5654	0.5362	0.1943	0.026*
C11	0.7162 (3)	0.71720 (14)	0.18933 (14)	0.0220 (3)
C12	0.4807 (3)	0.76665 (16)	0.13491 (17)	0.0283 (4)
H12A	0.5010	0.8628	0.1455	0.042*
H12B	0.3482	0.7481	0.1816	0.042*
H12C	0.4393	0.7213	0.0421	0.042*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0288 (6)	0.0232 (5)	0.0254 (6)	0.0053 (4)	0.0102 (5)	0.0089 (4)
N1	0.0256 (7)	0.0159 (6)	0.0225 (6)	0.0037 (5)	0.0058 (5)	0.0065 (5)
O2	0.0315 (7)	0.0202 (5)	0.0364 (6)	-0.0029 (5)	0.0010 (5)	0.0068 (5)
C2	0.0213 (8)	0.0202 (7)	0.0198 (7)	-0.0014 (6)	0.0002 (6)	0.0055 (5)
C3	0.0305 (9)	0.0196 (7)	0.0228 (7)	0.0017 (6)	0.0046 (6)	0.0085 (6)
C4A	0.0265 (9)	0.0175 (7)	0.0255 (8)	0.0021 (6)	0.0052 (6)	0.0054 (6)
C4	0.0354 (10)	0.0217 (7)	0.0260 (8)	0.0054 (6)	0.0046 (7)	0.0075 (6)
C5	0.0358 (10)	0.0197 (7)	0.0254 (8)	0.0055 (6)	0.0073 (7)	0.0040 (6)
C6	0.0381 (10)	0.0247 (8)	0.0283 (8)	0.0047 (7)	0.0125 (7)	0.0049 (6)
C7	0.0284 (9)	0.0225 (8)	0.0255 (8)	0.0001 (6)	0.0083 (7)	0.0059 (6)
C8	0.0220 (8)	0.0189 (7)	0.0204 (7)	-0.0003 (6)	0.0015 (6)	0.0040 (5)
C8A	0.0211 (8)	0.0189 (7)	0.0196 (7)	0.0017 (6)	0.0012 (6)	0.0033 (5)
C9	0.0226 (8)	0.0185 (7)	0.0206 (7)	-0.0018 (6)	0.0044 (6)	0.0043 (5)
C10	0.0244 (9)	0.0188 (7)	0.0209 (7)	-0.0015 (6)	0.0039 (6)	0.0043 (5)
C11	0.0269 (9)	0.0207 (7)	0.0192 (7)	-0.0003 (6)	0.0069 (6)	0.0050 (6)
C12	0.0284 (9)	0.0241 (8)	0.0337 (9)	0.0027 (6)	0.0057 (7)	0.0090 (6)

Geometric parameters (Å, °)

O1—C2	1.2368 (18)	C6—C7	1.523 (2)
N1—C2	1.3541 (19)	C6—H6A	0.9900
N1—C8A	1.4235 (19)	C6—H6B	0.9900
N1—H1	0.89 (2)	C7—C8	1.513 (2)
O2—C11	1.2121 (19)	C7—H7A	0.9900
C2—C3	1.509 (2)	C7—H7B	0.9900
C3—C4	1.527 (2)	C8—C8A	1.339 (2)
C3—H3A	0.9900	C8—C9	1.5076 (19)
C3—H3B	0.9900	C9—C10	1.529 (2)
C4A—C8A	1.5111 (19)	C9—H9A	0.9900
C4A—C5	1.515 (2)	C9—H9B	0.9900
C4A—C4	1.517 (2)	C10—C11	1.5130 (19)
C4A—H4AA	1.0000	C10—H10A	0.9900
C4—H4A	0.9900	C10—H10B	0.9900
C4—H4B	0.9900	C11—C12	1.502 (2)
C5—C6	1.514 (2)	C12—H12A	0.9800
C5—H5A	0.9900	C12—H12B	0.9800
C5—H5B	0.9900	C12—H12C	0.9800
C2—N1—C8A	126.69 (12)	H6A—C6—H6B	108.2
C2—N1—H1	114.2 (12)	C8—C7—C6	114.08 (13)
C8A—N1—H1	119.1 (12)	C8—C7—H7A	108.7
O1—C2—N1	121.04 (13)	C6—C7—H7A	108.7
O1—C2—C3	120.69 (13)	C8—C7—H7B	108.7
N1—C2—C3	118.26 (13)	C6—C7—H7B	108.7
C2—C3—C4	113.24 (12)	H7A—C7—H7B	107.6
C2—C3—H3A	108.9	C8A—C8—C9	124.11 (13)
C4—C3—H3A	108.9	C8A—C8—C7	121.14 (13)
C2—C3—H3B	108.9	C9—C8—C7	114.75 (12)
C4—C3—H3B	108.9	C8—C8A—N1	121.37 (13)
H3A—C3—H3B	107.7	C8—C8A—C4A	123.92 (13)
C8A—C4A—C5	111.49 (12)	N1—C8A—C4A	114.71 (12)
C8A—C4A—C4	109.87 (12)	C8—C9—C10	113.05 (12)
C5—C4A—C4	112.35 (13)	C8—C9—H9A	109.0
C8A—C4A—H4AA	107.6	C10—C9—H9A	109.0
C5—C4A—H4AA	107.6	C8—C9—H9B	109.0
C4—C4A—H4AA	107.6	C10—C9—H9B	109.0
C4A—C4—C3	110.49 (13)	H9A—C9—H9B	107.8
C4A—C4—H4A	109.6	C11—C10—C9	113.53 (12)
C3—C4—H4A	109.6	C11—C10—H10A	108.9
C4A—C4—H4B	109.6	C9—C10—H10A	108.9
C3—C4—H4B	109.6	C11—C10—H10B	108.9
H4A—C4—H4B	108.1	C9—C10—H10B	108.9
C6—C5—C4A	111.61 (13)	H10A—C10—H10B	107.7
C6—C5—H5A	109.3	O2—C11—C12	121.66 (13)
C4A—C5—H5A	109.3	O2—C11—C10	121.63 (14)
C6—C5—H5B	109.3	C12—C11—C10	116.69 (13)

supplementary materials

C4A—C5—H5B	109.3	C11—C12—H12A	109.5
H5A—C5—H5B	108.0	C11—C12—H12B	109.5
C5—C6—C7	109.48 (13)	H12A—C12—H12B	109.5
C5—C6—H6A	109.8	C11—C12—H12C	109.5
C7—C6—H6A	109.8	H12A—C12—H12C	109.5
C5—C6—H6B	109.8	H12B—C12—H12C	109.5
C7—C6—H6B	109.8		
C8A—N1—C2—O1	-177.87 (14)	C7—C8—C8A—N1	-177.32 (13)
C8A—N1—C2—C3	2.1 (2)	C9—C8—C8A—C4A	-176.49 (13)
O1—C2—C3—C4	-162.71 (14)	C7—C8—C8A—C4A	2.7 (2)
N1—C2—C3—C4	17.3 (2)	C2—N1—C8A—C8	-169.86 (15)
C8A—C4A—C4—C3	59.16 (17)	C2—N1—C8A—C4A	10.1 (2)
C5—C4A—C4—C3	-176.11 (13)	C5—C4A—C8A—C8	14.3 (2)
C2—C3—C4—C4A	-47.81 (18)	C4—C4A—C8A—C8	139.53 (16)
C8A—C4A—C5—C6	-46.50 (18)	C5—C4A—C8A—N1	-165.64 (13)
C4—C4A—C5—C6	-170.34 (13)	C4—C4A—C8A—N1	-40.41 (18)
C4A—C5—C6—C7	61.87 (18)	C8A—C8—C9—C10	-93.84 (18)
C5—C6—C7—C8	-43.8 (2)	C7—C8—C9—C10	86.89 (16)
C6—C7—C8—C8A	12.7 (2)	C8—C9—C10—C11	-175.09 (12)
C6—C7—C8—C9	-168.02 (14)	C9—C10—C11—O2	17.8 (2)
C9—C8—C8A—N1	3.5 (2)	C9—C10—C11—C12	-163.85 (13)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1 ⁱ	0.89 (2)	2.04 (2)	2.9321 (16)	174 (2)
C9—H9B \cdots O1 ⁱ	0.99	2.47	3.1517 (18)	126
C10—H10B \cdots O1 ⁱ	0.99	2.56	3.0568 (17)	111
C3—H3B \cdots O2 ⁱⁱ	0.99	2.59	3.387 (2)	137

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$.

Fig. 1

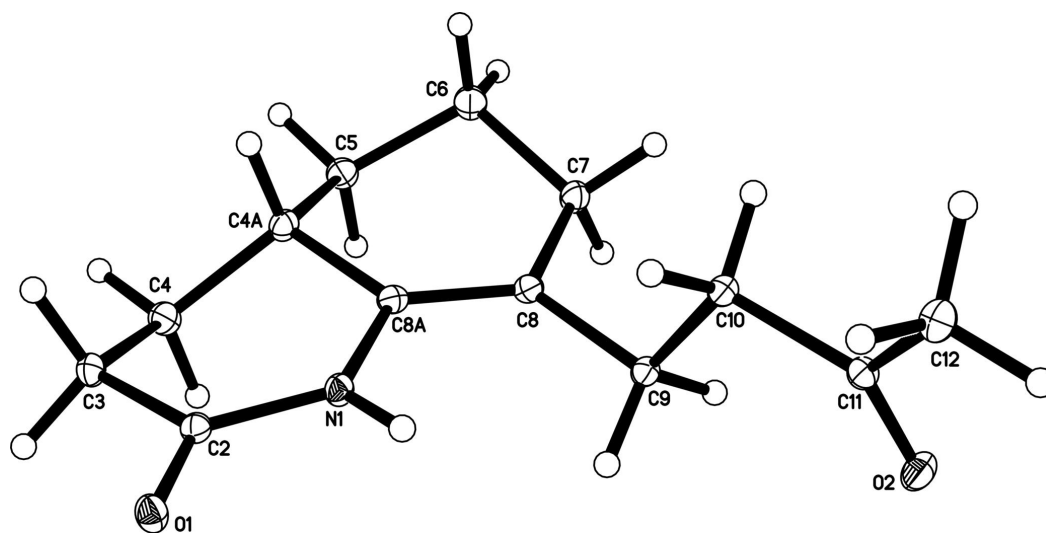


Fig. 2

