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Structure of (3aR*,5S*,7aR*)-2,5-Dimethyl-1,3,3a,4,5,6,7,7a-octahydroisoindol-1-one at 163 K

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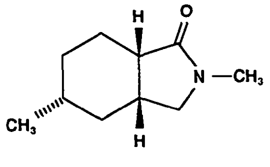
Abstract. $C_{10}H_{17}NO$, $M_r = 167{\cdot}25$, monoclinic, $P2_1/c$, $a = 15{\cdot}699\,(3)$, $b = 5{\cdot}2451\,(6)$, $c = 11{\cdot}7872\,(13)$ Å, $\beta = 106{\cdot}772\,(14)°$, $V = 929{\cdot}3\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}195$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}825$ cm^{-1}, $F(000) = 368$, $R = 0{\cdot}0415$ for 2122 reflections ($F \geq 4\sigma_F$). The six-membered ring is in a chair conformation. The constriction of the five-membered ring closes up the NCC angle at the carbonyl to 107·49 (10)°. The carbonyl and adjacent atoms are planar [maximum deviation: 0·008 (1) Å] whereas the N and adjacent atoms are slightly pyramidal [maximum deviation from least-squares plane: −0·048 (1) Å]. The C(carbonyl)–N distance of 1·3508 (15) Å is shortened by conjugation. The remaining bond lengths [ranges C—C, 1·524 (2)–1·539 (2); C—N, 1·453 (2), 1·461 (2); C=O, 1·230 (2) Å] and angles are normal.

Experimental. The title compound (1) was isolated by normal-phase HPLC from the two-component mixture produced by the catalytic hydrogenation of the intramolecular Diels–Alder condensate of *N*-allyl-*N*-methylsorbamide (Martin, Williamson, Gist & Smith, 1983). Crystals were grown by slow evaporation of a diethyl ether solution at room temperature. Table 1 summarizes data collection and structure refinement.



(1)

Structure solved by direct methods and refined by full-matrix least squares (*SHELX*76; Sheldrick, 1976). H atoms located in electron density difference map at $R = 0{\cdot}097$ as peaks of 0·68–0·91 e Å^{-3}. All atomic positions were refined; non-H atoms were refined anisotropically and H atoms isotropically. Scattering factors and anomalous-dispersion corrections taken from *International Tables for X-ray Crystallography* (1974); H from Stewart, Davidson & Simpson (1965).

Table 1. *Summary of data collection and structure refinement*

(A) Data collection (163 K)	
Mode	ω scan
Scan range	Symmetrically over 1·0° about $K\alpha_{1,2}$ max.
Background	Offset 1·0 and −1·0° in ω from $K\alpha_{1,2}$ max.
Scan rate (° min^{-1})	2·5–6·0
Exposure time (h)	44·3
Stability analysis	
Computed s	0·000178
t	−0·000003
Max. correction (on I)	0·26% (not applied)
2θ range (°)	4·0–60·0
Range in hkl, min.	0,0,−16
max.	22,7,15
Total refls, measd, unique	2715,2715
Crystal dimensions (mm)	0·39 × 0·29 × 0·27
Crystal volume (mm^3)	0·0352
Crystal faces	{001};{100};~−2,2,1;~2,−2,−1
Absorption coeff., $\mu(\text{Mo } K\alpha)$ (cm^{-1})	0·825
Transmission-factor range	0·976–0·981 (not applied)
(B) Structure refinement	
Instability factor p	0·04
Reflections used, m ($F \geq 4\,\sigma_F$)	2122
No. of variables, n	177
Goodness of fit, S	1·508
R, wR	0·0415, 0·0488
R for all data	0·0567
Max. shift/e.s.d.	0·010
Max., min. density in diff. map (e Å^{-3})	0·32, −0·22

Unit-cell parameters were obtained by least-squares refinement of the setting angles of 45 reflections with $23{\cdot}9 < 2\theta < 29{\cdot}6°$.

Syntex $P2_1$ autodiffractometer with a graphite monochromator and a Syntex LT-1 inert-gas (N_2) low-temperature delivery system. Data reduction was carried out as described by Riley & Davis (1976). Crystal and instrument stability were monitored by re-measurement of four check reflections after every 96 reflections. As detailed by Henslee & Davis (1975), these data were analyzed to relate intensity to exposure time by the equation $y = 1{\cdot}0 + sx + tx^2$ where x is exposure time (h), y is fractional intensity relative to $x = 0$ and s and t are coefficients determined by least-squares fit.

Function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{pk} + N_{bg1} + N_{bg2} + (pI)^2]^{1/2}$.

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Atomic parameters are given in Table 2,* bond lengths and bond angles in Table 3. The atom labeling is given in Fig. 1 and a view of the packing in Fig. 2. Principal computer programs are given by Gadol & Davis (1982); program for least-squares-planes calculations from Cordes (1983).

Table 2. *Atomic positions and U/U_{eq} for the title compound*

	x	y	z	U/U_{eq}*(Å^2)
O(1)	0·38246 (6)	−0·2500 (2)	0·10909 (8)	0·0301 (3)
C(1)	0·35565 (7)	−0·1055 (2)	0·17349 (10)	0·0209 (3)
N(2)	0·38527 (6)	−0·0966 (2)	0·29300 (8)	0·0214 (3)
C(3)	0·34090 (8)	0·0978 (2)	0·34442 (10)	0·0224 (3)
C(4)	0·18306 (7)	−0·0491 (2)	0·25325 (9)	0·0196 (3)
C(5)	0·10785 (7)	−0·0861 (2)	0·13833 (10)	0·0202 (3)
C(6)	0·14754 (8)	−0·1630 (2)	0·03905 (10)	0·0221 (3)
C(7)	0·21241 (8)	0·0373 (3)	0·01949 (10)	0·0239 (4)
C(8)	0·28490 (8)	0·1010 (2)	0·13324 (9)	0·0203 (3)
C(9)	0·25434 (7)	0·1439 (2)	0·24465 (10)	0·0193 (3)
C(10)	0·46216 (8)	−0·2364 (3)	0·36384 (12)	0·0267 (4)
C(11)	0·04135 (9)	−0·2840 (3)	0·15540 (13)	0·0273 (4)
H(3)*A*	0·3312 (10)	0·033 (3)	0·4194 (13)	0·028 (4)
H(3)*B*	0·3786 (10)	0·254 (3)	0·3664 (13)	0·030 (4)
H(4)*A*	0·2127 (9)	−0·218 (3)	0·2767 (12)	0·018 (3)
H(4)*B*	0·1591 (9)	0·005 (3)	0·3196 (12)	0·023 (3)
H(5)	0·0782 (9)	0·084 (3)	0·1158 (13)	0·027 (4)
H(6)*A*	0·1794 (10)	−0·332 (3)	0·0596 (12)	0·026 (4)
H(6)*B*	0·0998 (10)	−0·192 (3)	−0·0345 (14)	0·029 (4)
H(7)*A*	0·2404 (10)	−0·024 (3)	−0·0396 (14)	0·034 (4)
H(7)*B*	0·1776 (10)	0·198 (3)	−0·0117 (13)	0·030 (4)
H(8)	0·3177 (10)	0·255 (3)	0·1218 (13)	0·033 (4)
H(9)	0·2320 (10)	0·323 (3)	0·2487 (13)	0·031 (4)
H(10)*A*	0·4498 (10)	−0·308 (3)	0·4361 (14)	0·029 (4)
H(10)*B*	0·4736 (10)	−0·376 (3)	0·3144 (14)	0·033 (4)
H(10)*C*	0·5150 (11)	−0·127 (3)	0·3845 (14)	0·041 (4)
H(11)*A*	0·0705 (11)	−0·449 (3)	0·1771 (14)	0·044 (5)
H(11)*B*	−0·0059 (11)	−0·307 (3)	0·0814 (15)	0·040 (4)
H(11)*C*	0·0136 (12)	−0·229 (3)	0·2193 (14)	0·041 (4)

* For non-hydrogen atoms, the U value is U_{eq}, calculated as $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*A_{ij}$, where A_{ij} is the dot product of the ith and jth direct-space unit-cell vectors.

Table 3. *Bond lengths* (Å) *and bond angles* (°)

1	2	3	1–2	1–2–3
N(2)	C(1)	C(8)	1·3508 (15)	107·49 (10)
C(8)	C(1)	O(1)	1·525 (2)	126·42 (10)
O(1)	C(1)	N(2)	1·230 (2)	126·07 (10)
C(3)	N(2)	C(10)	1·461 (2)	122·03 (9)
C(3)	N(2)	C(1)		113·25 (9)
C(10)	N(2)	C(1)	1·453 (2)	124·12 (11)
C(9)	C(3)	N(2)	1·5381 (15)	102·38 (9)
C(6)	C(5)	C(11)	1·530 (2)	111·50 (10)
C(11)	C(5)	C(4)	1·526 (2)	110·59 (10)
C(4)	C(5)	C(6)	1·5308 (14)	109·26 (10)
C(7)	C(6)	C(5)	1·526 (2)	111·76 (10)
C(8)	C(7)	C(6)	1·5244 (15)	112·28 (10)
C(9)	C(8)	C(1)	1·539 (2)	102·52 (9)
C(9)	C(8)	C(7)		116·43 (10)
C(1)	C(8)	C(7)		114·20 (10)
C(4)	C(9)	C(3)	1·534 (2)	110·24 (9)
C(4)	C(9)	C(8)		111·88 (9)
C(3)	C(9)	C(8)		101·84 (10)
C(5)	C(4)	C(9)		114·59 (9)

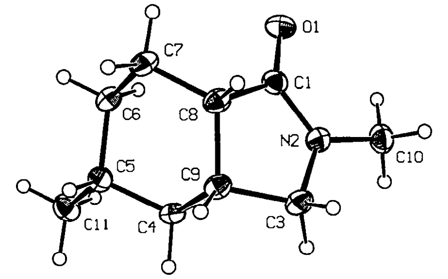

Fig. 1. Perspective drawing of (1) indicating atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

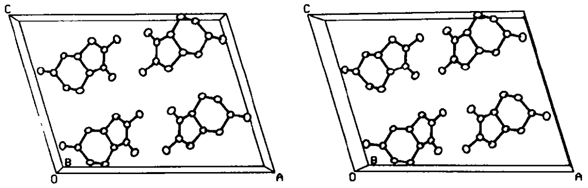

Fig. 2. Stereoview of the molecular packing as viewed down the b axis. No intermolecular contacts are shorter than van der Waals contacts.

Related literature. Synthesis and related compounds are discussed by Martin *et al.* (1983). Structures of two hydroindoles are reported by Martin, Tu, Kimura & Simonsen (1982).

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44031 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.