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Structure of (*R**,*R**)-3-Methyl-1-(4-methyl-5-oxo-2-pyrrolidinyl)-3-pyrrolin-2-one

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Abstract. C₁₀H₁₄N₂O₂, *M_r* = 194.23, orthorhombic, *P*2₁2₁2₁, *a* = 5.977 (5), *b* = 10.067 (8), *c* = 17.406 (14) Å, *V* = 1047.4 (1.5) Å³, *Z* = 4, *D_m* = 1.2 (1), *D_x* = 1.23 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ = 0.676 mm⁻¹, *F*(000) = 416, room temperature, *R* = 0.053 for 725 observed reflections. The unsaturated lactam ring is planar, the saturated ring has an envelope conformation with C(24) out of plane. Ring substituents can be described as O(27) equatorial, C(26) pseudo-equatorial and N(11) pseudo-axial. Only the relative configurations of the chiral centres were established. There is an intermolecular hydrogen bond N(21)–H(22)⋯O(27') [(i) *x*–0.5, 0.5–*y*, –*z*] with N⋯O = 2.916 (5), H⋯O = 1.91 Å, N–H⋯O = 154°.

Experimental. The title compound was isolated from *Lilium candidum* L. (Liliaceae) by Haladová, Eisenreichová, Bučková, Tomko & Uhrin (1988). Colourless crystal 0.6 × 0.1 × 0.07 mm, *D_m* by flotation. Space group from Weissenberg photographs. Syntex *P*2₁ diffractometer, graphite monochromator, lattice

Table 1. Final atomic coordinates (× 10⁴) with *e.s.d.*'s in parentheses and equivalent isotropic thermal parameters

$$B_{\text{eq}} = \frac{1}{3} \sum_i \beta_i a_i^2.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i> (Å ²)
N(11)	7680 (6)	5082 (4)	1442 (2)	4.94
C(12)	6172 (7)	6069 (5)	1572 (3)	5.24
C(13)	7034 (8)	7281 (4)	1189 (3)	5.49
C(14)	8909 (9)	6969 (5)	842 (3)	6.15
C(15)	9541 (8)	5541 (5)	968 (3)	5.64
C(16)	5763 (11)	8560 (6)	1243 (3)	7.73
O(17)	4429 (6)	5936 (3)	1935 (2)	6.91
N(21)	6734 (6)	2922 (4)	956 (2)	4.79
C(22)	8345 (7)	2097 (4)	724 (2)	4.19
C(23)	10289 (7)	2188 (4)	1282 (3)	4.56
C(24)	9317 (8)	2957 (5)	1966 (3)	5.56
C(25)	7259 (7)	3692 (5)	1647 (3)	4.85
C(26)	11286 (10)	853 (5)	1470 (4)	7.82
O(27)	8258 (5)	1390 (3)	151 (2)	4.99

parameters from 15 reflections 22 < 2θ < 50°, ω–2θ technique, scan rate 4.88–29.3° 2θ min⁻¹, scan range 2.0° plus Kα₁, Kα₂ difference, background at each end of the scan for half of the scan time, two standards (014, 122) monitored every 98 reflections did not vary significantly, 902 reflections measured for 2θ < 120° (*h* = 0 to 6, *k* = 0 to 10, *l* = 0 to 18), (sinθ/λ)_{max} = 0.546 Å⁻¹, 844 unique, 119 unobserved with *I* < 1.96σ(*I*). Lorentz and polarization but no absorption or extinction corrections. Structure solved by *MULTAN80* (Main *et al.*, 1980), refinement on |*F_o*| by

Table 2. Interatomic distances (Å) and angles (°) with *e.s.d.*'s in parentheses

N(11)–C(12)	1.361 (6)	C(21)–C(22)	1.333 (5)
N(11)–C(15)	1.461 (6)	N(21)–C(25)	1.466 (6)
N(11)–C(25)	1.466 (6)	C(22)–C(23)	1.517 (6)
C(12)–C(13)	1.483 (7)	C(23)–O(27)	1.227 (5)
C(12)–O(17)	1.226 (6)	C(23)–C(24)	1.534 (6)
C(13)–C(14)	1.311 (7)	C(23)–C(26)	1.505 (7)
C(13)–C(16)	1.498 (8)	C(24)–C(25)	1.539 (7)
C(14)–C(15)	1.502 (7)		
C(12)–N(11)–C(15)	111.5 (4)	C(22)–N(21)–C(25)	115.0 (4)
C(12)–N(11)–C(25)	122.9 (4)	N(21)–C(22)–C(23)	108.8 (4)
C(15)–N(11)–C(25)	124.8 (4)	N(21)–C(22)–O(27)	125.2 (4)
N(11)–C(12)–O(17)	124.7 (4)	C(23)–C(22)–O(27)	126.0 (4)
N(11)–C(12)–C(13)	107.2 (4)	C(22)–C(23)–C(24)	103.7 (4)
C(13)–C(12)–O(17)	128.1 (4)	C(24)–C(23)–C(26)	115.6 (4)
C(12)–C(13)–C(14)	107.9 (4)	C(22)–C(23)–C(26)	112.9 (4)
C(12)–C(13)–C(16)	120.2 (4)	C(23)–C(24)–C(25)	105.4 (4)
C(14)–C(13)–C(16)	132.0 (5)	N(11)–C(25)–N(21)	110.0 (4)
C(13)–C(14)–C(15)	112.2 (5)	N(11)–C(25)–C(24)	114.2 (4)
N(11)–C(15)–C(14)	101.2 (4)	N(21)–C(25)–C(24)	102.3 (4)
N(11)–C(12)–C(13)–C(14)	1.7 (5)	C(13)–C(14)–C(15)–N(11)	0.6 (6)
N(11)–C(12)–C(13)–C(16)	–178.6 (4)	C(13)–C(12)–N(11)–C(25)	–171.8 (4)
N(21)–C(22)–C(23)–C(24)	12.4 (4)	C(14)–C(15)–N(11)–C(12)	0.6 (5)
N(21)–C(22)–C(23)–C(26)	138.2 (4)	C(14)–C(15)–N(11)–C(25)	170.8 (4)
O(17)–C(12)–C(13)–C(14)	–177.5 (5)	C(15)–N(11)–C(12)–C(13)	–1.4 (5)
O(17)–C(12)–C(13)–C(16)	2.2 (8)	C(15)–N(11)–C(25)–C(24)	49.5 (6)
O(17)–C(12)–N(11)–C(25)	7.5 (7)	C(15)–N(11)–C(25)–N(21)	–64.8 (5)
O(27)–C(23)–C(24)–C(25)	–167.9 (4)	C(16)–C(13)–C(14)–C(15)	178.9 (5)
O(27)–C(23)–C(24)–C(26)	–42.1 (6)	C(22)–C(23)–C(24)–C(25)	–20.6 (4)
O(27)–C(22)–N(21)–C(25)	–178.2 (4)	C(23)–C(24)–C(25)–N(21)	21.0 (4)
C(12)–C(13)–C(14)–C(15)	–1.4 (6)	C(26)–C(23)–C(24)–C(25)	–144.6 (4)
C(12)–N(11)–C(25)–C(24)	–141.4 (4)	C(24)–C(25)–N(21)–C(22)	–14.5 (5)
C(12)–N(11)–C(25)–N(21)	104.3 (5)	C(25)–N(21)–C(22)–C(23)	1.5 (5)
		O(17)–C(12)–N(11)–C(15)	177.9 (4)

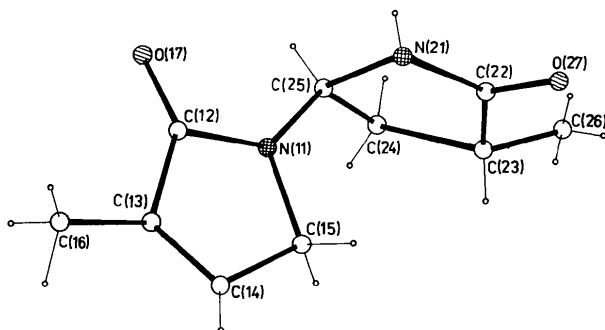


Fig. 1. The molecular structure of $C_{10}H_{14}N_2O_2$ showing the numbering scheme of the atoms.

block-diagonal least squares, H atoms at calculated positions not refined, fixed isotropic temperature parameter of 4.0 \AA^2 , anisotropic thermal parameters for heavy atoms, weighting scheme $w^{-1} = \sigma^2(F) + (0.02|F_o|)^2$, $\sigma(F)$ from counting statistics, $(\Delta/\sigma)_{\max} = 0.09$, $R = 0.053$ and 0.063 , $wR = 0.067$ and 0.069 for observed and all reflections, respectively, $S = 2.01$, $(\Delta\rho)_{\max} = 0.13$, $(\Delta\rho)_{\min} = -0.19 \text{ e \AA}^{-3}$. Scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). All calculations with XRC83 (Pavelčík, Kettmann & Majer, 1985) on an ES-1045 computer. Final atomic coordinates are given in Table

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Structure of Recinine*

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Abstract. 4-Methoxy-1-methyl-2-oxo-3-pyridine-carbonitrile, $C_{16}H_{16}N_4O_4$, $M_r = 328.3$, monoclinic, $P2_1/n$, $a = 7.357$ (3), $b = 14.620$ (6), $c = 14.838$ (6) \AA , $\beta = 94.15$ (3) $^\circ$, $V = 1592$ (1) \AA^3 , $Z = 8$, $D_x = 1.37 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.094 \text{ mm}^{-1}$, $F(000) = 688$, $T = 293 \text{ K}$, $R = 0.059$ for 1787 observed reflections. The X-ray study confirms that the structure of recinine is as inferred from chemical evidence. There are two crystallographically independent molecules and in both cases the six-membered ring is planar to within 0.012 \AA and the $-\text{C}-\text{C}\equiv\text{N}$

fragments are linear. The largest bond-length discrepancy between molecules *A* and *B* is 0.016 \AA (4σ) for $\text{C}(4)-\text{O}(2)$. The two $\text{C}=\text{O}$ carbonyl distances [mean 1.232 (4) \AA] are both slightly longer than normal. The methoxy groups at $\text{C}(4)$ are coplanar with the six-membered ring. The molecules in the crystal are packed at normal van der Waals distances. Five $\text{C}-\text{H}\cdots\text{O}/\text{N}$ intermolecular contacts $< 3.4 \text{ \AA}$ are noted.

Experimental. In the state of Oaxaca, Mexico, castor oil is obtained by crushing the seeds of the *Recinus communis* or castor-oil plant using stone mills. A by-product of this process is a paste that is sold at a low

1.* Interatomic distances, bond angles and torsion angles are presented in Table 2. Fig. 1 shows the molecule and the atom-numbering scheme.

Related literature. An overview of the conformations of pyrrolidine derivatives has been published by Pfafferott, Oberhammer, Boggs & Caminati (1985).

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51600 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

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