The computations were made with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), *PARST* (Nardelli, 1983) and *PESOS* (Martínez-Ripoll & Cano, 1975) programs on a VAX 11/750 computer. Scattering and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974). The atomic coordinates are given in Table 1* and bond lengths and angles in Table 2. Fig. 1 shows the structure with the numbering scheme used and Fig. 2 a stereoview of the structure. Asymmetric parameters for the six-membered heterocyclic ring are: $q_2 = 0{\cdot}393$ (3), $q_3 = 0{\cdot}240$ (2) Å, $\varphi = -77{\cdot}9$ (3), $\theta = 58{\cdot}6$ (3)°, $Q_T = 0{\cdot}461$ (3) Å (Cremer & Pople, 1975).

Related literature. The synthesis of the title compound and other related compounds is discussed by de March, Moreno-Manas, Ripoll, Florencio, García-Blanco & Martínez-Carrera, 1986). Structure determinations of related compounds are reported by Mascarenhas & Gottlieb (1977), Okuyama, Yamazaki, Kobayashi & Sakurai (1983) and Florencio & García-Blanco (1987).

* Lists of structure factors, anisotropic thermal parameters, bond distances, bond angles, torsion angles, least-squares planes, asymmetry parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44063 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(–)-Platynecine, a Pyrrolizidine Necine Base

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Abstract. $C_8H_{15}NO_2$, $M_r = 157{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}810$ (1), $b = 8{\cdot}348$ (1), $c = 12{\cdot}459$ (1) Å, $V = 812{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}28$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}07$ cm^{-1}, $F(000) = 344$, $T = 291$ K, final $R = 0{\cdot}030$ for 766 observed reflections. (–)-Platynecine adopts an *exo–endo* conformation with ring *A* *exo*-buckled with a puckering angle of 35·1 (3)° whilst the *endo* ring, *B*, has a puckering angle of 150·2 (4)°. These ring pucker values, as well as the dihedral angle of 125·6 (4)° between the planes defined by atoms C(1), C(8), N(4), C(3) and C(5), N(4), C(8), C(7), are in close agreement with values reported for other pyrrolizidine alkaloid nuclei. The hydroxyl group on C(9) is involved in two intermolecular hydrogen bonds: O(1)···O(2) 2·835 (3) and O(1)···N(4) 2·777 (3) Å. Their respective hydrogen-bond angles are O(1)···H(O2)–O(2) 165 (6)° and O(1)–H(O1)···O(2) 168 (9)°.

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Experimental. Hydrolysis of retrorsine, obtained from *Senecio isatideus* plants, yielded retronecine (2) (Robins & Sweeney, 1981). Hydrogenation of retronecine in ethanol using Adam's catalyst afforded (–)-platynecine (1) (*cf.* Adams & Rogers, 1941). Colourless, needle-shaped crystals grown by slow evaporation from aqueous acetone, crystal *ca* 0·6 × 0·3 × 0·2 mm used in data collection, CAD-4 diffractometer. Systematic absences from Weissenberg photographs indicated the crystals to be orthorhombic, $P2_12_12_1$. 989 independent intensities, θ limit 75°, $\omega/2\theta$ scan. Two standard

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Table 1. *Final positional parameters and equivalent isotropic thermal parameters* (Å²) *with e.s.d.'s in parentheses*

$$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	−0·2425 (2)	−0·3208 (3)	−0·8099 (2)	0·040
O(2)	−0·0513 (2)	0·0035 (3)	−0·6269 (2)	0·041
N(4)	−0·4340 (3)	0·0926 (2)	−0·6182 (2)	0·031
C(1)	−0·3641 (3)	−0·1823 (3)	−0·6544 (2)	0·031
C(2)	−0·3662 (4)	−0·1584 (3)	−0·5328 (2)	0·038
C(3)	−0·4685 (4)	−0·0047 (4)	−0·5212 (2)	0·046
C(5)	−0·3275 (4)	0·2365 (3)	−0·6003 (3)	0·044
C(6)	−0·2006 (3)	0·2384 (3)	−0·6923 (2)	0·039
C(7)	−0·1631 (3)	0·0611 (3)	−0·7083 (2)	0·032
C(8)	−0·3422 (3)	−0·0116 (3)	−0·6971 (2)	0·027
C(9)	−0·2391 (3)	−0·3057 (3)	−0·6962 (2)	0·036

Table 2. *Bond distances* (Å) *and bond angles* (°)

O(1)–C(9)	1·423 (4)	O(2)–C(7)	1·422 (4)
N(4)–C(3)	1·480 (4)	N(4)–C(5)	1·477 (4)
N(4)–C(8)	1·496 (3)	C(1)–C(2)	1·528 (4)
C(1)–C(8)	1·531 (4)	C(1)–C(9)	1·511 (4)
C(2)–C(3)	1·518 (5)	C(5)–C(6)	1·515 (5)
C(6)–C(7)	1·522 (4)	C(7)–C(8)	1·531 (4)

C(3)–N(4)–C(5)	115·2 (3)	C(3)–N(4)–C(8)	107·8 (2)
C(5)–N(4)–C(8)	107·6 (2)	C(2)–C(1)–C(8)	103·0 (2)
C(2)–C(1)–C(9)	116·0 (3)	C(8)–C(1)–C(9)	116·3 (2)
C(1)–C(2)–C(3)	102·1 (3)	N(4)–C(3)–C(2)	106·9 (3)
N(4)–C(5)–C(6)	105·2 (3)	C(5)–C(6)–C(7)	102·4 (3)
O(2)–C(7)–C(6)	110·7 (3)	O(2)–C(7)–C(8)	111·2 (2)
C(6)–C(7)–C(8)	101·4 (2)	N(4)–C(8)–C(1)	105·0 (2)
N(4)–C(8)–C(7)	105·5 (2)	C(1)–C(8)–C(7)	120·1 (2)
O(1)–C(9)–C(1)	113·1 (3)		

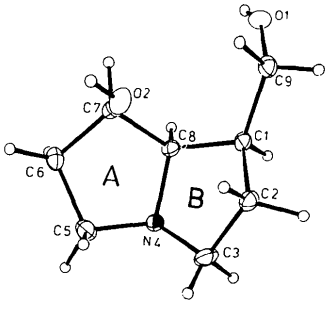
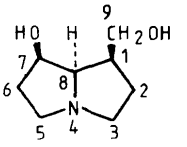
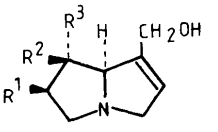


Fig. 1. A perspective view of the molecule showing the numbering scheme.

intensities used to monitor variations in intensity data: < 3% variation observed. Least-squares refinement of 24 reflections, $\theta > 20°$, used to determine lattice parameters. No absorption correction, h 0 to 9, k 0 to 10, l 0 to 15. Structure solutions by direct methods with *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for all non-H atoms converged to R and wR of 0·030 and 0·037 with $w = 1/\sigma^2(F_o)$. H-atom coordinates located from difference Fourier maps were included and refined isotropically in the final three cycles of least squares. Isotropic extinction coefficient (Larson, 1970), $r = 0·348$. 766 reflections with $I \geq 3·0\sigma(I)$ used. $(\Delta/\sigma)_{max} = 0·08$ for non-H atoms, 0·24 for H atoms; max. and min. heights in final difference synthesis = 0·21 and −0·33 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using Glasgow *GX* package (Mallinson & Muir, 1985). Final positional and equivalent isotropic thermal parameters are given in Table 1* while bond lengths and angles with their standard deviations are given in Table 2. An *ORTEP*II (Johnson, 1976) diagram, Fig. 1, illustrates the numbering scheme and absolute configuration (Warren, 1966) for the molecule.



(1)



(2) $R^1 = R^3 = H$, $R^2 = OH$

(3) $R^1 = R^2 = H$, $R^3 = OH$

(4) $R^1 = R^2 = OH$, $R^3 = H$

Related literature. Alkaloids containing platynecine (1) are comparatively rare (Robins, 1982). They usually occur as macrocyclic diesters, but they are not hepatotoxic as they do not possess a 1,2 double bond in the pyrrolizidine nucleus (Mattocks, 1986). X-ray studies of alkaloids containing platynecine have been restricted to the 12-membered dilactones platyphylline (Röder & Wiedenfeld, 1982) and hygrophylline (Culvenor, Mackay & Mitrprachachon, 1985), and the 13-membered compound bulgarsenine (Stoeckli-Evans, 1980). Recently, X-ray structures of several pyrrolizidine bases have been reported, including retronecine (2), heliotridine (3) (Gelbaum, Glinski, Van Derveer & Zalkow, 1985), and crotanecine (4) (Culvenor & Richardson, 1985). The absolute configurations of the bases (1)–(3) were established by chemical interconversions and degradations (Warren, 1966).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44036 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 6-Hydroxy-2,5-dinitro-2,5-diazacyclohexyl Acetate

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(*Received* 17 *March* 1987; *accepted* 26 *May* 1987)

Abstract. $C_6H_{10}N_4O_7$, $M_r = 250{\cdot}17$, monoclinic, $P2_1/c$, $a = 13{\cdot}588$ (2), $b = 7{\cdot}276$ (1), $c = 10{\cdot}830$ (1) Å, $\beta = 109{\cdot}75$ (1)°, $V = 1007{\cdot}66$ (19) Å^3, $Z = 4$, $D_x = 1{\cdot}65$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1{\cdot}28$ mm^{-1}, $F(000) = 520$, $T = 295$ K, final $R = 0{\cdot}029$, $wR = 0{\cdot}028$ for 1230 observed reflections. The ring has a normal chair conformation. Both ring N atoms are pyramidal; the angles between the exocyclic N–N bonds and the CNC planes are 32·9 and 36·6°. There is an intermolecular hydrogen bond (2·998 Å) between the hydroxy oxygen and one of the nitro-group oxygens.

Experimental. Colorless 0·1 × 0·15 × 0·4 mm crystal. Synthesized by C. Coon of Lawrence Livermore Laboratory, Livermore, CA, USA. Automated Nicolet *R*3*M* diffractometer with incident-beam graphite monochromator, 25 centered reflections within $15 < 2\theta < 81°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{max} = 115°$, range of hkl $-14 \le h \le 14$, $0 \le k \le 7$, $0 \le l \le 10$. Standards $\bar{1}3,0,0$, $\bar{2}40$, 008, monitored every 60 reflections with random variation 2·0% over data collection, θ–2θ mode, scan width $(2{\cdot}0 + \Delta_{\alpha 1 \alpha 2})$, scan rate a function of count rate (10° min^{-1} minimum, 30° min^{-1} maximum), 1686 reflections measured, 1551 unique, $R_{int} = 0{\cdot}008$, 1230 observed $[F_o > 3\sigma(|F_o|)]$.

Structure solved by direct methods. Full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1{\cdot}0$, isotropic secondary extinction value = $2{\cdot}3 \times 10^{-6}$. 185 parameters refined: atom coordinates and anisotropic temperature factors for all non-H atoms, coordinates for H atoms. Isotropic temperature factors for H atoms set at $1{\cdot}1 \times U_{eq}$ of covalently bonded atoms. $(\Delta/\sigma)_{max} = 0{\cdot}004$, $R = 0{\cdot}029$, $wR = 0{\cdot}028$, $S = 0{\cdot}6$. Final difference Fourier excursions, 0·13 and −0·18 e Å^{-3}. All calculations performed with *SDP* system of programs (Enraf–Nonius, 1985).

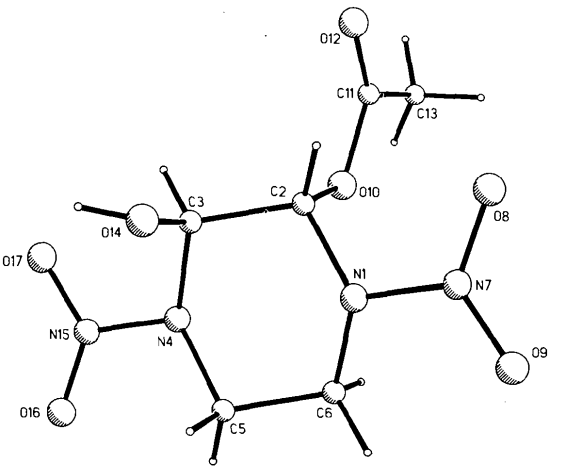

Fig. 1. A perspective drawing illustrating the results of the X-ray study on the title compound.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atom numbering for Tables 1 and 2, which report atom coordinates, bond distances and angles, follows that shown in Fig. 1.* The hydrogen-bond parameters are: H14···O8 =

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44086 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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