

initially refined [with their sum fixed at 1 and  $U(C) = 0.1 \text{ \AA}^2$ ] and then fixed with the isotropic temperature factors allowed to refine. Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.\* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

**Related literature.** For the preparation of the compound *via* a tandem-Knoevenagel-hetero-Diels-Alder reaction see Tietze, Bachmann & Schul (1988) and Tietze (1984). For structures of indoloquinolizine derivatives see Harms, Sheldrick, Schul & Tietze (1986) and Sawyer, Shariff & McLean (1985).

\* 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 44975 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

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*Acta Cryst.* (1988). **C44**, 1681–1682

## 1,2,3,4-Tetrahydro-1,4-dimethylisoquinolinium Picrate

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(Received 11 November 1987; accepted 25 April 1988)

**Abstract.**  $C_{11}H_{16}N^+ \cdot C_6H_2N_3O_7^-$ ,  $M_r = 390.36$ , monoclinic,  $P2_1/n$ ,  $a = 13.266$  (3),  $b = 8.127$  (2),  $c = 16.744$  (4) Å,  $\beta = 92.00$  (2)°,  $V = 1804.1$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.44$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 1.06$  cm<sup>-1</sup>,  $F(000) = 816$ ,  $T = 293$  K,  $R = 0.0560$  for 1537 reflections. The six-membered heterocyclic ring of the isoquinoline cation is in a half-chair conformation with *syn*-methyl groups at C(1) and C(4) occupying pseudo-axial and equatorial positions, respectively. The relevant torsion angles are C(10)C(9)C(1)CH<sub>3</sub> =  $-104.8$  (3) and C(9)C(10)C(4)CH<sub>3</sub> =  $139.9$  (3)°. A nitrogen proton [H(2a'),  $\bar{x}$ ,  $1-y$ ,  $1-z$ ] from the heterocyclic ring forms a bifurcated hydrogen bond to the phenolic O atom [N(2')...O(13) =  $3.130$  (4) Å] and to a nitro group O atom [N(2')...O(14a) =  $2.943$  (4) Å] of an adjacent picrate ion. The 1,2,3,4-tetrahydro-1,4-dimethylisoquinoline molecule is the result of an unusual, highly stereoselective alkylation of a boron-activated enamine.

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**Experimental.** Yellow, transparent crystal of dimensions  $0.25 \times 0.50 \times 1.00$  mm; Nicolet R3m/ $\mu$  update of  $P2_1$  diffractometer; data collected in Wyckoff mode ( $3 \leq 2\theta \leq 45^\circ$ ,  $2\theta$  fixed,  $\omega$  varied), scan rate  $4-29.3^\circ \text{ min}^{-1}$ , graphite-monochromated Mo  $K\alpha$  radiation; lattice parameters from a least-squares refinement of 23 reflections ( $26.81 \leq 2\theta \leq 42.85^\circ$ ), angles measured by a centering routine; systematic absences ( $h0l$ ,  $h+l=2n+1$ ,  $0k0$ ,  $k=2n+1$ ) and Laue symmetry  $2/m$  consistent with space group  $P2_1/n$ ; monitored reflections 110 and  $\bar{1}3\bar{1}$  showed only statistical variations in intensities; 1562 independent reflections measured ( $-14 \leq h \leq 14$ ,  $0 \leq k \leq 8$ ,  $0 \leq l \leq 18$ );  $1537 \geq 3\sigma(I)$ ; Lorentz-polarization corrections,  $\psi$ -scan empirical absorption correction (transmission factors 0.902–0.930); structure solved by direct methods, block-cascade least-squares refinement, H-atom positional parameters refined, single refined isotropic temperature factor; final  $R = 0.0560$ ,  $wR = 0.0301$  for 308 parameters and 1537 reflections,  $S = 1.519$ ,  $(\Delta/\sigma)_{\text{max}} = 0.028$ ,  $(\Delta/\sigma)_{\text{av}} = 0.007$ ; largest peaks in the final difference map of  $0.20$  and  $-0.20 \text{ e \AA}^{-3}$ ,  $\sum w(|F_o| - |F_c|)^2$  minimized with  $w =$

Table 1. Atomic coordinates (10<sup>4</sup>) and equivalent isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>)Equivalent isotropic *U* defined as one third of the trace of the orthogonalized *U<sub>ij</sub>* tensor.

	x	y	z	<i>U<sub>eq</sub></i>
C(1)	768 (2)	7630 (4)	8133 (2)	40 (1)
N(2)	1754 (2)	6701 (4)	8221 (2)	42 (1)
C(3)	1772 (2)	5599 (4)	8940 (2)	45 (1)
C(4)	1017 (2)	4225 (4)	8815 (2)	41 (1)
C(5)	-859 (3)	3881 (4)	8550 (2)	51 (1)
C(6)	-1779 (3)	4437 (5)	8269 (2)	60 (2)
C(7)	-1876 (3)	5974 (5)	7941 (2)	60 (2)
C(8)	-1038 (3)	6963 (5)	7889 (2)	47 (1)
C(9)	-97 (2)	6425 (4)	8181 (2)	34 (1)
C(10)	7 (2)	4864 (4)	8518 (2)	37 (1)
C(11)	739 (3)	9020 (4)	8723 (2)	57 (1)
C(12)	982 (3)	3265 (4)	9596 (2)	60 (1)
C(13)	-841 (3)	5574 (4)	3513 (2)	40 (1)
O(13)	-1697 (2)	5820 (3)	3193 (1)	56 (1)
C(14)	-541 (2)	4054 (4)	3903 (2)	37 (1)
N(14)	-1295 (2)	2765 (4)	3993 (2)	54 (1)
O(14a)	-1986 (2)	2655 (3)	3492 (1)	67 (1)
O(14b)	-1199 (2)	1838 (3)	4565 (1)	83 (1)
C(15)	406 (3)	3717 (4)	4196 (2)	41 (1)
C(16)	1152 (2)	4877 (4)	4128 (2)	41 (1)
N(16)	2181 (2)	4469 (4)	4395 (2)	60 (1)
O(16a)	2309 (2)	3193 (3)	4770 (2)	97 (1)
O(16b)	2852 (2)	5402 (4)	4226 (2)	95 (1)
C(17)	945 (2)	6394 (4)	3798 (2)	42 (1)
C(18)	-24 (2)	6745 (4)	3530 (2)	38 (1)
N(18)	-177 (2)	8412 (4)	3225 (2)	58 (1)
O(18a)	-1027 (2)	8907 (3)	3108 (2)	98 (1)
O(18b)	535 (2)	9269 (4)	3099 (2)	124 (2)

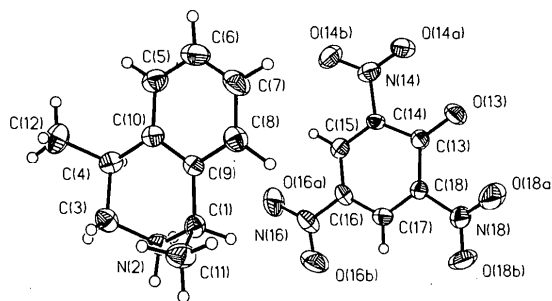


Fig. 1. Drawing of the title compound with thermal ellipsoids at the 40% level. H atoms are represented by spheres of arbitrary size.

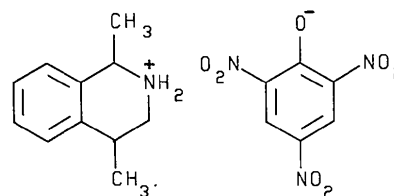
$1/\sigma^2(F_o)$ . All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microclipline and Nova 4/C configurations; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional parameters, Table 2 gives interatomic distances and valence angles, and Fig. 1 is a drawing of the title compound.\*

**Related literature.** A related crystal structure has been reported by Hara, Shirai, Hoshino, Umezawa & Itaka (1983). The title compound (1) was prepared from isoquinoline using a reaction similar to that reported by Brooks, Dowell, Minter & Villarreal (1984).

Table 2. Bond lengths (Å) and bond angles (°)

C(1)–N(2)	1.513 (4)	C(1)–C(9)	1.512 (4)
C(1)–C(11)	1.503 (5)	N(2)–C(3)	1.500 (4)
C(3)–C(4)	1.510 (4)	C(4)–C(10)	1.505 (4)
C(4)–C(12)	1.525 (5)	C(5)–C(6)	1.369 (5)
C(5)–C(10)	1.401 (5)	C(6)–C(7)	1.369 (6)
C(7)–C(8)	1.377 (5)	C(8)–C(9)	1.395 (5)
C(9)–C(10)	1.394 (4)	C(13)–O(13)	1.254 (4)
C(13)–C(14)	1.447 (5)	C(13)–C(18)	1.442 (5)
C(14)–N(14)	1.460 (4)	C(14)–C(15)	1.360 (4)
N(14)–O(14a)	1.225 (4)	N(14)–O(14b)	1.222 (4)
C(15)–C(16)	1.374 (5)	C(16)–N(16)	1.459 (4)
C(16)–C(17)	1.376 (5)	N(16)–O(16a)	1.221 (4)
N(16)–O(16b)	1.211 (4)	C(17)–C(18)	1.376 (4)
C(18)–N(18)	1.460 (4)	N(18)–O(18a)	1.207 (4)
N(18)–O(18b)	1.199 (4)		

N(2)–C(1)–C(9)	109.1 (2)	N(2)–C(1)–C(11)	110.6 (3)
C(9)–C(1)–C(11)	114.5 (3)	C(1)–N(2)–C(3)	111.5 (2)
N(2)–C(3)–C(4)	109.8 (3)	C(3)–C(4)–C(10)	111.7 (3)
C(3)–C(4)–C(12)	107.4 (3)	C(10)–C(4)–C(12)	114.0 (3)
C(6)–C(5)–C(10)	121.4 (3)	C(5)–C(6)–C(7)	120.6 (3)
C(6)–C(7)–C(8)	119.5 (3)	C(7)–C(8)–C(9)	120.6 (3)
C(1)–C(9)–C(8)	116.7 (3)	C(1)–C(9)–C(10)	123.1 (3)
C(8)–C(9)–C(10)	120.1 (3)	C(4)–C(10)–C(5)	120.8 (3)
C(4)–C(10)–C(9)	121.5 (3)	C(5)–C(10)–C(9)	117.7 (3)
O(13)–C(13)–C(14)	124.1 (3)	O(13)–C(13)–C(18)	124.8 (3)
C(14)–C(13)–C(18)	111.1 (3)	C(13)–C(14)–N(14)	118.7 (3)
C(13)–C(14)–C(15)	124.8 (3)	N(14)–C(14)–C(15)	116.4 (3)
C(14)–N(14)–O(14a)	118.7 (3)	C(14)–N(14)–O(14b)	118.1 (3)
C(14)–C(15)–C(16)	119.3 (3)	C(15)–C(16)–C(17)	121.0 (3)
C(15)–C(16)–C(17)	119.9 (3)	C(16)–N(16)–O(16a)	117.6 (3)
N(16)–C(16)–O(16a)	118.3 (3)	O(16a)–N(16)–O(16b)	124.1 (3)
C(16)–C(17)–C(18)	119.2 (3)	C(13)–C(18)–C(17)	124.2 (3)
C(13)–C(18)–N(18)	120.7 (3)	C(17)–C(18)–N(18)	115.0 (3)
C(18)–N(18)–O(18a)	118.9 (3)	C(18)–N(18)–O(18b)	120.0 (3)
O(18a)–N(18)–O(18b)	121.0 (3)		



(1)

We thank The Welch Foundation (P-074) and the TCU Research Fund for financial support.

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