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References

FULLER, R. W. & WONG, D. T. (1977). *Fed. Proc. Fed. Am. Soc. Exp. Biol.* **36**, 2154–2158.

GOODLET, I., MIREYLEES, S. E. & SUGRUE, M. F. (1977). *Br. J. Pharmacol.* **59**, 4818–4828.

HORN, A. S. (1976). *Postgrad. Med. J.* **52**, 25–30.

KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.

POST, M. L. & HORN, A. S. (1977). *Acta Cryst.* **B33**, 2590–2595.

POST, M. L., KENNARD, O. & HORN, A. S. (1975). *Acta Cryst.* **B31**, 1008–1013.

SUGRUE, M. F., GOODLET, I. & MIREYLEES, S. E. (1976). *Eur. J. Pharmacol.* **40**, 121–130.

Acta Cryst. (1979). **B35**, 1515–1516

3,4-Seco-3,5-cyclo-1,2-dihydro-*N*(1),*N*(4)-dimethylstrictamine Methiodide

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Abstract. C₂₃H₃₁N₂O₂⁺·I⁻, monoclinic, *P*2₁, *a* = 6.577 (5), *b* = 12.194 (6), *c* = 13.342 (6) Å, β = 95.8 (1)°, *Z* = 2, final *R* value 9.7%. The *C* and *E* rings form a bicyclic (3.2.1) system with the six-membered ring in a chair conformation. Both rings are joined on their *endo* face by a three-atom segment. The 15*R* configuration was established using the anomalous dispersion of iodine.

Introduction. A crystal of 0.2 × 0.2 × 0.4 mm was mounted on a Philips PW 1100 diffractometer and 2547 reflections were collected using graphite-monochromated Cu *K*α radiation (λ = 1.5418 Å) for θ up to 68°. There were 401 Bijvoet pairs and 1891 independent reflections greater than 3σ(*I*) were finally retained for the calculations.

The position of the I anion was determined (*x* = 0.007, *y* = 0.500, *z* = 0.393) by an analysis of the Patterson function. Initial phases computed from the I, followed by successive full-matrix least-squares refinements of the three-dimensional electron density map led to the location of 28 atoms of the molecule. The O and N atoms were distinguished following simple mechanistic and biogenetic considerations. The H atoms directly attached to the C atom skeleton were introduced (C–H = 1.00 Å, C–C–H = 109 or 120°) and were assigned the *B* values of the bonded C atoms. Refinement was pursued considering all the atomic coordinates, isotropic temperature factors for the C, N, O atoms, and anisotropic temperature factors for iodine. After the anomalous dispersion was introduced, an unweighted agreement factor of 0.097 was obtained for the absolute configuration shown in Fig. 1. The enantiomer led to *R* = 0.104. At this stage the

difference Fourier map did not show any significant electron density which could be related to the H atoms of the methyl groups. The atomic coordinates are listed in Table 1, and the bond lengths and bond angles in Tables 2 and 3, respectively. Scattering factors for the non-hydrogen atoms were taken from Doyle & Turner (1968) while those for H atoms were from Stewart, Davidson & Simpson (1965).*

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34289 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

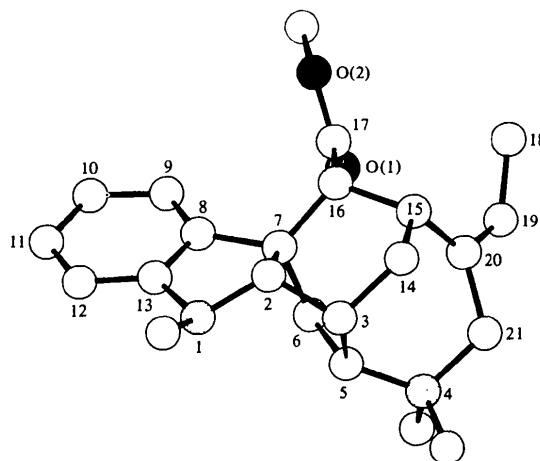


Fig. 1. Molecular structure and conformation.

Discussion. The title compound (1) is formed from the zinc-acetic acid reduction of the natural product deacetyldeformylpicraline (2) (Doe de Maindreville, 1976). Since it was not possible to conveniently determine the structure of this compound using classical methods, a methiodide of the permethylated derivative was prepared and submitted to single-crystal X-ray structure determination. The results of the X-ray study are summarized in Fig. 1. Rings *C* (atoms 2, 3, 5, 6, 7)

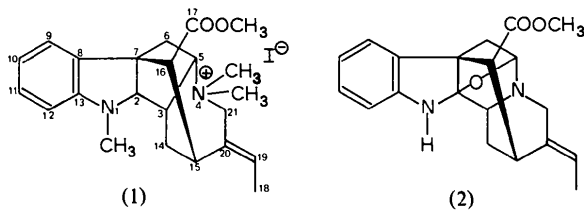


Table 3. *Intramolecular bond angles* (°)

The mean e.s.d. value is 2°.

C(2)—N(1)—C(13)	103	C(7)—C(8)—C(13)	106
C(2)—N(1)—Me(N1)	115	C(9)—C(8)—C(13)	121
C(13)—N(1)—Me(N1)	117	C(8)—C(9)—C(10)	120
N(1)—C(2)—C(3)	118	C(9)—C(10)—C(11)	119
N(1)—C(2)—C(7)	104	C(10)—C(11)—C(12)	120
C(3)—C(2)—C(7)	103	C(11)—C(12)—C(13)	119
C(2)—C(3)—C(5)	101	N(1)—C(13)—C(8)	113
C(2)—C(3)—C(14)	105	N(1)—C(13)—C(12)	126
C(5)—C(3)—C(14)	113	C(8)—C(13)—C(12)	121
C(5)—N(4)—C(21)	116	C(3)—C(14)—C(15)	113
C(5)—N(4)—Me(N4)	110	C(14)—C(15)—C(16)	109
C(5)—N(4)—Me2(N4)	106	C(14)—C(15)—C(20)	110
C(21)—N(4)—Me(N4)	109	C(16)—C(15)—C(20)	118
C(21)—N(4)—Me2(N4)	106	C(7)—C(16)—C(15)	110
Me(N4)—N(4)—Me2(N4)	108	C(7)—C(16)—C(17)	109
C(3)—C(5)—N(4)	116	C(15)—C(16)—C(17)	118
C(3)—C(5)—C(6)	105	C(16)—C(17)—O(1)	126
N(4)—C(5)—C(6)	115	C(16)—C(17)—O(2)	109
C(5)—C(6)—C(7)	105	O(1)—C(17)—O(2)	125
C(2)—C(7)—C(6)	104	C(18)—C(19)—C(20)	131
C(2)—C(7)—C(8)	101	C(15)—C(20)—C(19)	123
C(2)—C(7)—C(16)	108	C(15)—C(20)—C(21)	118
C(6)—C(7)—C(8)	110	C(19)—C(20)—C(21)	117
C(6)—C(7)—C(16)	113	N(4)—C(21)—C(20)	120
C(8)—C(7)—C(16)	120	C(17)—O(2)—Me(O)	117
C(7)—C(8)—C(9)	133		

and *E* (atoms 2, 3, 14, 15, 16, 7) form a bicyclic (3.2.1) system with the six-membered ring in a chair conformation. The two rings are joined by a three-atom bridge situated on the *endo* face between C(5) and C(15). Ring *A* is planar within 0.05 Å, while the five-membered ring *B* is in an envelope form. The dihedral angle H(2)—C(2)—C(3)—H(3) is 77° which rationalizes the small coupling constant ($J < 2$ Hz) observed between H(2) and H(3) in the ¹H NMR spectrum.

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References

- DOE DE MAINDREVILLE, M. (1976). Thèse, Reims (N° au CNRS AO 12821).
 DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* **A24**, 390–399.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Table 1. *Fractional coordinates* (×10⁴) for the non-hydrogen atoms

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	7272 (26)	6134 (15)	3152 (15)
C(2)	7361 (21)	5098 (27)	2575 (12)
C(3)	7750 (33)	4042 (19)	3169 (19)
N(4)	5149 (24)	2803 (13)	4034 (13)
C(5)	5676 (28)	3938 (16)	3690 (16)
C(6)	4009 (27)	4481 (16)	2944 (16)
C(7)	5171 (23)	4940 (22)	2082 (13)
C(8)	4539 (27)	6140 (16)	1871 (16)
C(9)	2988 (32)	6611 (19)	1250 (18)
C(10)	2689 (35)	7735 (20)	1272 (21)
C(11)	3922 (35)	8382 (20)	1975 (20)
C(12)	5508 (34)	7886 (19)	2598 (19)
C(13)	5799 (29)	6777 (17)	2537 (17)
C(14)	7994 (29)	3137 (17)	2366 (17)
C(15)	6032 (27)	2968 (15)	1621 (15)
C(16)	5244 (26)	4119 (16)	1205 (15)
C(17)	3207 (28)	4160 (17)	538 (17)
C(18)	2550 (35)	1578 (22)	458 (21)
C(19)	3039 (31)	1689 (19)	1582 (18)
C(20)	4488 (26)	2221 (16)	2102 (15)
C(21)	4967 (28)	1903 (17)	3224 (16)
Me(N1)	9273 (32)	6645 (20)	3465 (19)
Me(N4)	6667 (32)	2452 (19)	4886 (19)
Me2(N4)	2996 (32)	2893 (19)	4440 (18)
Me(O)	1746 (43)	4654 (23)	-1107 (25)
O(1)	1552 (24)	4025 (14)	816 (14)
O(2)	3547 (23)	4460 (14)	-388 (13)
I	9905 (2)	5005 (5)	6065 (1)

Table 2. *Intramolecular bond distances* (Å)

The mean e.s.d. value is 0.03 Å.

N(1)—C(2)	1.48	C(8)—C(13)	1.39
N(1)—C(13)	1.44	C(9)—C(10)	1.39
N(1)—Me(N1)	1.48	C(10)—C(11)	1.42
C(2)—C(3)	1.52	C(11)—C(12)	1.40
C(2)—C(7)	1.53	C(12)—C(13)	1.37
C(3)—C(5)	1.60	C(14)—C(15)	1.56
C(3)—C(14)	1.56	C(15)—C(16)	1.58
N(4)—C(5)	1.51	C(15)—C(20)	1.55
N(4)—C(21)	1.54	C(16)—C(17)	1.53
N(4)—Me(N4)	1.50	C(17)—O(1)	1.20
N(4)—Me2(N4)	1.57	C(17)—O(2)	1.33
C(5)—C(6)	1.55	C(18)—C(19)	1.51
C(6)—C(7)	1.55	C(19)—C(20)	1.29
C(7)—C(8)	1.54	C(20)—C(21)	1.55
C(7)—C(16)	1.54	Me(O)—O(2)	1.47
C(8)—C(9)	1.37		