angles are given in Table 3. The molecule is illustrated in Fig. 2.

Related literature. Structure of 2-chloroacetophenone: Grossert, Dubey, Gill, Cameron & Gardner (1984); structure of 2-chloro-1-(2,4-dihydroxyphenyl)ethanone: van Rooyen & Breytenbach (1988). Activation of α-bromo ketones by complexation with hard and soft Lewis acids: Laube, Weidenhaupt & Hunziker (1991). Cyclization of 2-chloro-2′-hydroxyacetophenone to coumaran-3-one: Bell, Earls & Timimi (1974).

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Structure of a Tricyclic Subunit of Manzamine A

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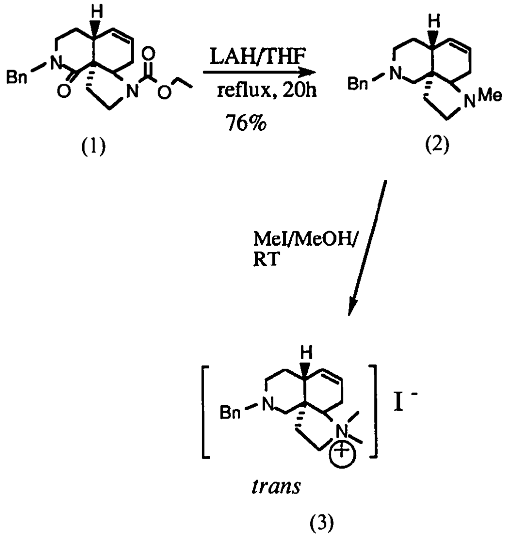
(*Received* 31 *October* 1991; *accepted* 21 *January* 1992)

Abstract. (4aα,7aβ,10aβ)-2-Benzyl-1,2,3,4,4a,7,7a,8,9,10-decahydro-8,8-dimethylpyrrolo[2,3-*i*]isoquinolinium iodide, $C_{20}H_{29}N_2{}^+.I^-$, $M_r = 424.37$, monoclinic, $P2_1/n$, $a = 9.441\,(4)$, $b = 10.378\,(4)$, $c = 20.023\,(9)$ Å, $\beta = 91.56\,(3)°$, $V = 1961\,(1)$ Å^3, $Z = 4$, $D_x = 1.44$ g cm^{-3} (188 K), λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 16.16$ cm^{-1}, $F(000) = 864$, $T = 188$ K, $R = 0.0288$ for 3070 reflections [$F_o \geq 4\sigma(F_o)$]. The crystal structure determination was undertaken in order to establish the configuration around C10a. The rings of the isoquinoline group are *trans*, with the pyrrole moiety *cis* fused. The *A* ring is in the chair conformation, while the cyclohexene ring, *B*, is in the boat conformation owing to the *cis* fusion of the five-membered pyrrole ring. The pyrrole ring, *C*, assumes the half-chair conformation. The C—N bonds of the quarternary N atom, N8, are longer than those of the tertiary N atom, N2 [1.517 (2) for N8 and 1.463 (2) Å for N2].

Experimental. Compound (1) was synthesized by an intramolecular Diels–Alder reaction of the corresponding substrate. Treatment of (1) with lithium aluminium hydride (LAH) yielded (2) which was then treated with methyl iodide in methanol resulting in the formation of (3). Synthetic details are described elsewhere (Martin, Rein & Liao, 1991). Crystals of (3) were obtained by slow evaporation from methanol. The data crystal was a colorless



(1)

(2)

trans

(3)

0108-2701/92/091703-03$06.00

needle of approximate dimensions 0.27 × 0.35 × 0.83 mm. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and equipped with a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 28 reflections with $19.2 < 2\theta < 23.8°$ The data were collected using the ω-scan technique, with 1.2° ω scans at 6–12° min^{-1} and a 2θ range of 4.0–50.0° ($h = 0 \rightarrow 11$, $k = 0 \rightarrow 12$, $l = -23 \rightarrow 23$). A total of 3932 reflections were collected, of which 3481 were unique, with $R_{\rm int} = 0.016$. Four reflections ($23\bar{8}$, $\bar{2}4\bar{2}$, $\bar{3}28$, 334) were remeasured every 196 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor range was 0.979–1.02. The data were also corrected for Lp effects and absorption. An analytical absorption correction was applied based on the crystal face measurements. The transmission factor range was 0.5272–0.6930. Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (411 reflections). Data reduction and decay correction were performed using *SHELXTL-Plus* (Sheldrick, 1991). The structure was solved by the heavy-atom method and refined by full-matrix least squares (Sheldrick, 1991). In all, 324 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The function, $\sum w(|F_o| - |F_c|)^2$, was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate); where 0.02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction for Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times (\text{scan rate})]$. Final $R = 0.0288$ for 3070 reflections, with $wR = 0.0385$ ($R_{\rm all} = 0.0341$, $wR_{\rm all} = 0.0400$), and goodness of fit = 1.484. The maximum $|\Delta/\sigma| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were -0.89 and 0.71 e Å^{-3}, respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors

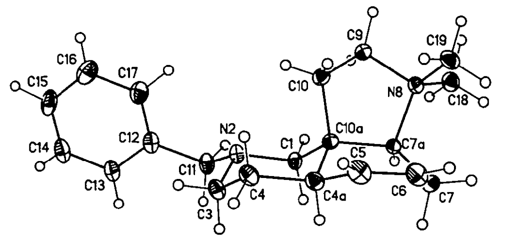


Fig. 1. View of (3) showing the atom-labelling scheme for the cation. The conformation of the isoquinoline moiety is apparent from this view. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size. The I$^-$ ion is not shown.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non-*H *atoms of* (3)

$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

	x	*y*	*z*	$U_{\rm eq}$
I1	0.53972 (2)	−0.07530 (2)	0.716040 (10)	0.03449 (8)
C1	0.7024 (3)	0.2819 (3)	0.62578 (14)	0.0247 (8)
N2	0.7498 (3)	0.2657 (3)	0.55694 (12)	0.0278 (7)
C3	0.6609 (3)	0.1735 (3)	0.5198 (2)	0.0326 (10)
C4	0.5072 (3)	0.2170 (3)	0.5168 (2)	0.0330 (10)
C4*a*	0.4554 (3)	0.2379 (3)	0.5878 (2)	0.0293 (9)
C5	0.3015 (3)	0.2732 (3)	0.5952 (2)	0.0394 (10)
C6	0.2579 (3)	0.2966 (3)	0.6555 (2)	0.0396 (10)
C7	0.3621 (3)	0.2920 (3)	0.7131 (2)	0.0343 (10)
C7*a*	0.5001 (3)	0.3606 (3)	0.69665 (14)	0.0238 (8)
N8	0.4903 (3)	0.5090 (2)	0.70550 (12)	0.0242 (7)
C9	0.5851 (3)	0.5596 (3)	0.6517 (2)	0.0282 (10)
C10	0.5521 (3)	0.4720 (3)	0.5929 (2)	0.0273 (9)
C10*a*	0.5514 (3)	0.3371 (3)	0.62458 (13)	0.0220 (8)
C11	0.8992 (3)	0.2311 (4)	0.5563 (2)	0.0334 (10)
C12	0.9633 (3)	0.2499 (3)	0.4886 (2)	0.0333 (10)
C13	1.0618 (3)	0.1627 (4)	0.4657 (2)	0.0363 (10)
C14	1.1268 (4)	0.1826 (4)	0.4050 (2)	0.0458 (13)
C15	1.0911 (4)	0.2875 (4)	0.3664 (2)	0.0474 (13)
C16	0.9937 (4)	0.3753 (5)	0.3887 (2)	0.0482 (13)
C17	0.9304 (4)	0.3569 (4)	0.4500 (2)	0.0421 (11)
C18	0.5452 (4)	0.5487 (3)	0.7735 (2)	0.0343 (11)
C19	0.3453 (3)	0.5651 (3)	0.6956 (2)	0.0349 (10)

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non-*H *atoms of* (3)

A	*B*	*C*	*A*—*B*	*A*—*B*—*C*
C3	N2	C1	1.462 (4)	111.5 (2)
C1	N2	C11	1.471 (4)	110.9 (2)
C11	N2	C3	1.455 (4)	112.2 (3)
C4	C3	N2	1.520 (5)	111.1 (3)
C4*a*	C4	C3	1.530 (5)	109.6 (3)
C5	C4*a*	C10*a*	1.510 (4)	110.2 (3)
C5	C4*a*	C4		117.3 (3)
C10*a*	C4*a*	C4	1.545 (4)	109.8 (2)
C6	C5	C4*a*	1.310 (5)	117.6 (3)
C7	C6	C5	1.495 (5)	119.3 (3)
C7*a*	C7	C6	1.528 (4)	111.5 (3)
C10*a*	C7*a*	N8	1.554 (4)	106.4 (2)
C10*a*	C7*a*	C7		114.6 (2)
N8	C7*a*	C7	1.553 (4)	112.6 (2)
C10	C10*a*	C1	1.537 (4)	109.4 (2)
C10	C10*a*	C4*a*		114.8 (2)
C10	C10*a*	C7*a*		104.2 (2)
C1	C10*a*	C4*a*	1.536 (4)	106.9 (2)
C1	C10*a*	C7*a*		110.9 (2)
C4*a*	C10*a*	C7*a*		110.7 (2)
C9	N8	C18	1.514 (4)	110.7 (2)
C9	N8	C19		109.1 (2)
C9	N8	C7*a*		103.0 (2)
C18	N8	C19	1.501 (4)	107.7 (3)
C18	N8	C7*a*		110.9 (2)
C19	N8	C7*a*	1.496 (4)	115.3 (2)
C10	C9	N8	1.513 (4)	103.4 (2)
C10*a*	C10	C9		103.3 (2)
N2	C1	C10*a*		109.5 (2)
C12	C11	N2	1.513 (4)	112.8 (2)
C13	C12	C17	1.385 (5)	118.9 (3)
C13	C12	C11		120.1 (3)
C17	C12	C11	1.382 (5)	121.0 (3)
C14	C13	C12	1.391 (5)	120.5 (3)
C15	C14	C13	1.372 (6)	120.2 (4)
C16	C15	C14	1.378 (6)	119.9 (3)
C17	C16	C15	1.393 (5)	120.0 (4)
C12	C17	C16		120.5 (4)

for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).* Fig. 1, showing the atom-labelling scheme, was generated using *SHELXTL-Plus* (Sheldrick, 1991). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. Synthesis of (3) was accomplished as part of an effort directed towards the total synthesis of manzamine A (Martin, Rein & Liao, 1991, and references therein).

Funding for this project was supplied by the Robert A. Welch Foundation (F-652) and the National Institutes of Health (G.M. 25439) to SFM.

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* Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving the H atoms, torsion angles, observed and calculated structure factors and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55072 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0569]

Acta Cryst. (1992). C**48**, 1705–1707

Structure of Mepirizole.H(mepirizole) Hexafluorophosphate

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(*Received* 28 *November* 1991; *accepted* 29 *January* 1992)

Abstract. $C_{11}H_{14}N_4O_2.C_{11}H_{15}N_4O_2^+.PF_6^-$, M_r = 614.5, monoclinic, $P2_1/c$, $a = 10.4097$ (7), $b = 22.186$ (2), $c = 12.742$ (1) Å, $\beta = 101.51$ (1)°, $V = 2883.6$ (5) Å^3, $Z = 4$, $D_x = 1.42$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.17$ mm^{-1}, $F(000) = 1272$, $T = 295$ K, full-matrix least-squares refinement based on 2736 reflections led to $R(F_o)$ and $wR(F_o)$ values of 0.040 and 0.039, respectively. Mepirizole (mep) is 4-methoxy-2-(5-methoxy-3-methyl-1*H*-pyrazol-1-yl)-6-methylpyrimidine. The asymmetric unit contains one neutral mep molecule and one protonated (Hmep)$^+$ ion associated in pairs by a hydrogen bond involving the additional proton. The proton is located on an N atom of the pyrimidine ring.

Experimental. Synthesis of the title compound was from an ethanol/water solution of mep and KPF_6 in acid medium. A colourless crystal of dimensions 0.50 × 0.50 × 0.20 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell dimensions were determined from setting angles of 25 reflections having $12 < \theta < 15°$. 6597 reflections were measured using $\omega/2\theta$ scan with 2θ from 3 to 54° ($0 \leq h \leq 13$, $0 \leq k \leq 28$, $-16 \leq l \leq 16$), scan range $(0.85 + 0.35\tan\theta)°$, and variable scan speed 0.97–8.24° min^{-1}. Intensities of three reflections ($41\bar{7}$, $\bar{4}$,10,2, $\bar{4}17$) measured every 2 h showed no significant variations. Corrections were made for Lp effects and for absorption using ψ scans (North, Phillips & Mathews, 1968), minimum relative transmission

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