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).

* 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]

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).

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Structure of Mepirizole.H(mepirizole) Hexafluorophosphate

BY L. SOTO, J. GARCIA-LOZANO AND M. MARTINEZ

Departament de Quimica Inorganica, Facultat de Farmacia, Universitat de Valencia, Avda Blasco Ibañez 13, 46010 *Valencia, Spain*

AND F. DAHAN* AND J.-P. LEGROS

Laboratoire de Chimie de Coordination du CNRS, UPR n° 8241 *liée par conventions à l'Université Paul Sabatier et à l'Institut National Polytechnique*, 205 *route de Narbonne*, 31077 *Toulouse CEDEX, France*

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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, β = 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, μ = 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.

* To whom correspondence should be addressed.

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° ($0 \le h \le 13$, $0 \le k \le 28$, $-16 \le l \le 16$), 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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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* ($Å^2 \times 100$) *with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}$ trace **U**.

	x	y	z	U_{eq}
P	0.3729 (1)	0.34001 (4)	0.49083 (8)	5.4 (2)
F(1)	0.4255 (2)	0.4039 (1)	0.4652 (2)	9.3 (5)
F(2)	0.2712 (3)	0.3729 (1)	0.5468 (2)	11.4 (6)
F(3)	0.2736 (3)	0.3430 (1)	0.3799 (2)	10.3 (6)
F(4)	0.3211 (3)	0.2759 (1)	0.5146 (2)	11.0 (6)
F(5)	0.4738 (3)	0.3075 (1)	0.4337 (2)	12.1 (7)
F(6)	0.4738 (3)	0.3377 (1)	0.6007 (2)	10.8 (6)
C(33)	0.4045 (5)	0.7037 (2)	0.8493 (4)	9 (1)
O(32)	0.3602 (3)	0.6517 (1)	0.8987 (2)	6.9 (5)
C(31)	0.3970 (3)	0.6000 (2)	0.8602 (3)	5.0 (6)
N(27)	0.3583 (3)	0.5460 (1)	0.8971 (2)	4.9 (5)
N(28)	0.4030 (3)	0.4992 (1)	0.8432 (2)	5.0 (5)
C(29)	0.4685 (3)	0.5247 (2)	0.7765 (3)	4.8 (6)
C(30)	0.4685 (3)	0.5876 (2)	0.7839 (3)	5.3 (7)
C(34)	0.5290 (4)	0.4869 (2)	0.7031 (3)	7.0 (8)
C(22)	0.2831 (3)	0.5323 (2)	0.9741 (3)	5.3 (7)
N(23)	0.2567 (3)	0.5777 (2)	1.0332 (2)	6.9 (7)
C(24)	0.1839 (4)	0.5616 (3)	1.1035 (3)	9 (1)
C(25)	0.1403 (5)	0.5039 (3)	1.1125 (4)	10 (1)
C(26)	0.1766 (4)	0.4610 (2)	1.0505 (3)	8 (1)
N(21)	0.2496 (3)	0.4751 (2)	0.9778 (2)	6.6 (6)
O(35)	0.1512 (4)	0.6033 (2)	1.1697 (3)	12.4 (9)
C(36)	0.2144 (5)	0.6584 (2)	1.1763 (4)	11 (1)
C(37)	0.1420 (5)	0.3963 (2)	1.0581 (4)	12 (1)
C(13)	−0.2715 (3)	0.4366 (2)	0.4949 (3)	8.0 (9)
O(12)	−0.1472 (2)	0.4182 (1)	0.5611 (2)	6.1 (5)
C(11)	−0.0744 (3)	0.4640 (2)	0.6060 (3)	4.8 (6)
N(7)	0.0468 (2)	0.4520 (1)	0.6721 (2)	4.5 (5)
N(8)	0.1077 (3)	0.5049 (1)	0.7115 (2)	4.9 (5)
C(9)	0.0259 (3)	0.5475 (2)	0.6693 (3)	5.3 (6)
C(10)	−0.0891 (3)	0.5242 (2)	0.6037 (3)	5.4 (7)
C(14)	0.0585 (4)	0.6120 (2)	0.6914 (3)	7.6 (9)
C(2)	0.1062 (3)	0.3975 (1)	0.7031 (2)	4.1 (5)
N(3)	0.0479 (3)	0.3478 (1)	0.6642 (2)	5.0 (5)
C(4)	0.1092 (4)	0.2970 (2)	0.6972 (3)	5.7 (7)
C(5)	0.2270 (4)	0.2945 (2)	0.7697 (3)	6.3 (7)
C(6)	0.2842 (3)	0.3471 (2)	0.8062 (3)	5.3 (6)
N(1)	0.2214 (2)	0.3993 (1)	0.7717 (2)	4.4 (5)
O(15)	0.0548 (3)	0.2450 (1)	0.6596 (2)	8.3 (6)
C(16)	−0.0606 (5)	0.2479 (2)	0.5773 (4)	11 (1)
C(17)	0.4124 (4)	0.3522 (2)	0.8824 (3)	7.6 (8)

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

P—F(1)	1.578 (3)	P—F(4)	1.572 (3)
P—F(2)	1.569 (3)	P—F(5)	1.568 (3)
P—F(3)	1.578 (3)	P—F(6)	1.575 (3)
C(33)—O(32)	1.433 (5)	C(13)—O(12)	1.455 (4)
O(32)—C(31)	1.333 (4)	O(12)—C(11)	1.327 (4)
C(31)—N(27)	1.377 (5)	C(11)—N(7)	1.396 (4)
N(27)—N(28)	1.376 (4)	N(7)—N(8)	1.379 (4)
N(28)—C(29)	1.319 (5)	N(8)—C(9)	1.312 (4)
C(29)—C(30)	1.400 (5)	C(9)—C(10)	1.415 (5)
C(30)—C(31)	1.365 (5)	C(10)—C(11)	1.345 (5)
C(29)—C(34)	1.485 (5)	C(9)—C(14)	1.485 (5)
N(27)—C(22)	1.405 (5)	N(7)—C(2)	1.381 (4)
C(22)—N(23)	1.319 (5)	C(2)—N(3)	1.308 (4)
N(23)—C(24)	1.332 (6)	N(3)—C(4)	1.320 (4)
C(24)—C(25)	1.371 (8)	C(4)—C(5)	1.381 (5)
C(25)—C(26)	1.338 (8)	C(5)—C(6)	1.349 (5)
C(26)—N(21)	1.346 (6)	C(6)—N(1)	1.360 (4)
N(21)—C(22)	1.320 (5)	N(1)—C(2)	1.336 (4)
C(24)—O(35)	1.341 (7)	C(4)—O(15)	1.331 (4)
O(35)—C(36)	1.383 (7)	O(15)—C(16)	1.430 (5)
C(26)—C(37)	1.488 (7)	C(6)—C(17)	1.490 (5)
F(1)—P—F(2)	88.2 (1)	F(2)—P—F(6)	90.1 (2)
F(1)—P—F(3)	88.3 (1)	F(3)—P—F(4)	91.2 (1)
F(1)—P—F(4)	179.0 (2)	F(3)—P—F(5)	89.0 (2)
F(1)—P—F(5)	91.5 (2)	F(3)—P—F(6)	179.0 (2)
F(1)—P—F(6)	90.9 (1)	F(4)—P—F(5)	87.7 (2)
F(2)—P—F(3)	90.4 (1)	F(4)—P—F(6)	89.6 (1)
F(2)—P—F(4)	92.6 (2)	F(5)—P—F(6)	90.5 (2)
F(2)—P—F(5)	179.4 (2)		
C(33)—O(32)—C(31)	112.9 (3)	C(13)—O(12)—C(11)	113.5 (3)
O(32)—C(31)—C(30)	132.3 (3)	O(12)—C(11)—C(10)	134.3 (3)
O(32)—C(31)—N(27)	119.9 (3)	O(12)—C(11)—N(7)	119.0 (3)
C(30)—C(31)—N(27)	107.8 (3)	C(10)—C(11)—N(7)	106.7 (3)
C(31)—N(27)—N(28)	109.6 (3)	C(11)—N(7)—N(8)	110.7 (3)
N(27)—N(28)—C(29)	105.6 (3)	N(7)—N(8)—C(9)	104.5 (2)
N(28)—C(29)—C(30)	112.1 (3)	N(8)—C(9)—C(10)	112.5 (3)
C(29)—C(30)—C(31)	104.9 (3)	C(9)—C(10)—C(11)	105.7 (3)
C(31)—N(27)—C(22)	132.0 (3)	C(11)—N(7)—C(2)	129.7 (3)
N(28)—N(27)—C(22)	118.4 (3)	N(8)—N(7)—C(2)	119.6 (2)
N(28)—C(29)—C(34)	120.2 (3)	N(8)—C(9)—C(14)	120.8 (3)
C(30)—C(29)—C(34)	127.7 (3)	C(10)—C(9)—C(14)	126.7 (3)
N(27)—C(22)—N(21)	114.9 (3)	N(7)—C(2)—N(1)	116.9 (3)
N(27)—C(22)—N(23)	116.3 (3)	N(7)—C(2)—N(3)	118.9 (2)
N(21)—C(22)—N(23)	128.8 (4)	N(1)—C(2)—N(3)	124.2 (3)
C(22)—N(23)—C(24)	113.1 (4)	C(2)—N(3)—C(4)	116.1 (3)
N(23)—C(24)—C(25)	123.2 (5)	N(3)—C(4)—C(5)	123.7 (3)
C(24)—C(25)—C(26)	118.5 (5)	C(4)—C(5)—C(6)	117.9 (3)
C(25)—C(26)—N(21)	120.4 (5)	C(5)—C(6)—N(1)	118.3 (3)
C(26)—N(21)—C(22)	115.9 (4)	C(6)—N(1)—C(2)	119.8 (3)
N(23)—C(24)—O(35)	119.2 (5)	N(3)—C(4)—O(15)	118.7 (3)
C(25)—C(24)—O(35)	117.5 (5)	C(5)—C(4)—O(15)	117.6 (3)
C(24)—O(35)—C(36)	118.1 (4)	C(4)—O(15)—C(16)	117.4 (3)
C(25)—C(26)—C(37)	123.1 (5)	C(5)—C(6)—C(17)	124.6 (3)
N(21)—C(26)—C(37)	116.5 (4)	N(1)—C(6)—C(17)	117.1 (3)

0.95. 6265 data were unique; $R_{int} = 0.028$ for averaging redundant $0kl$ and $0k\bar{l}$ data. The structure was solved by direct methods followed by Fourier and least-squares techniques using 2736 reflections having $F_o^2 > 2\sigma(F_o^2)$, based on counting statistics. Full-matrix least-squares refinement minimized $\sum w(|F_o| - |F_c|)^2$, with anisotropic thermal parameters for non-H atoms. All H atoms were located by ΔF, introduced in constrained geometry (C—H = N—H = 0.97 Å), with isotropic U_H, first allowed to vary, then kept fixed at 0.130 $Å^2$ for methyl H atoms and at 0.069 $Å^2$ for other H atoms.

$R = 0.040$, $wR = 0.039$, 370 variables, unit weights. Maximum parameter shift = 0.002σ. Maximum and minimum heights in the final ΔF map were 0.18 and −0.17 e $Å^{-3}$. Scattering factors including real and imaginary parts of anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101, 149), and from Stewart, Davidson & Simpson (1965) for H atoms. Computations were performed on a MicroVAX 3400 DEC computer, with *MolEN* (Fair, 1990), *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1986), *ORFFE* (Busing, Martin & Levy, 1964) and *NRC* (Ahmed, Hall, Pippy & Huber, 1966) programs.

Related literature. The final positional and equivalent isotropic thermal parameters are listed in Table 1,* bond lengths and angles in Table 2. The thermal-ellipsoid plot of the neutral and protonated molecules (*ORTEP*; Johnson, 1965) is shown in Fig. 1 with atomic labelling chosen for consistency with

* Lists of structure factors, H-atom parameters, anisotropic thermal parameters and least-squares-planes equations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55112 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0273]

previous studies. Fig. 2 is a stereoview of the molecular packing (*PLUTO*; Motherwell & Clegg, 1978). The additional proton of the $(Hmep)^+$ ion is located on the N atom N(1) of the pyrimidine ring; however, there is no striking difference in bond lengths and angles between mep and $(Hmep)^+$ entities. The mep and $(Hmep)^+$ entities are associated in pairs through a hydrogen bond involving the additional proton and the N atom N(28) of the pyrazole ring of the neutral molecule. Both mep and $(Hmep)^+$ are in the usual conformation *A* (Soto, Garcia, Escriva & Legros, 1992), and in both cases the pyrazole and pyrimidine rings are almost coplanar.

Mepirizole has been found to possess analgesic and anti-inflammatory properties (Takabatake, Kodama, Tanaka, Dohmori, Tachizanawa & Naito, 1970). Although a number of crystal structures of mep-containing metal complexes have been published (Beneto, Soto, Garcia-Lozano, Escriva, Legros & Dahan, 1991), no structure determination of mepirizole itself has been reported to date. The only previous structural information available about free mepirizole was drawn from the structure of $[Cu_2(mepirizole)_2(C_2O_4)(H_2O)_2](PF_6)_2.mepirizole.3H_2O$ (Soto, Garcia, Escriva, Legros, Tuchagues, Dahan & Fuertes, 1989) which contains a molecule of non-coordinated mepirizole.

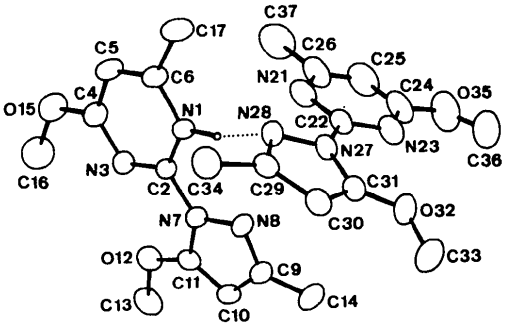

Fig. 1. *ORTEP* (Johnson, 1965) plot of mep and $(Hmep)^+$ molecules with atomic numbering, showing 30% probability thermal ellipsoids. H atoms except the additional proton of $(Hmep)^+$ are omitted for clarity. The hydrogen bond is drawn as a dotted line [N(1)···N(28) = 2.938 (4), H(N1)···N(28) = 2.02 Å, N(1)—H(N1)···N(28) = 158°].

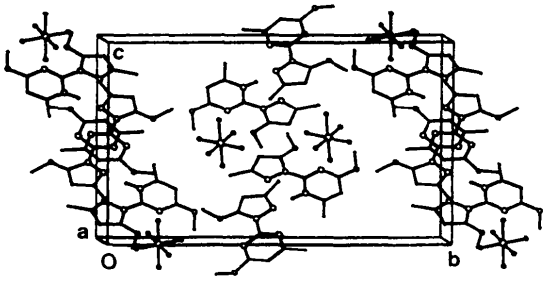

Fig. 2. Perspective view of the molecular packing.

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