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(2*Z*)-2-(*H*ydroximino)-3-methoxy-3-phenyl-2,3-dihydro-1*H*-imidazo[1,2-*a*]pyrimidin-4-ium perchlorate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.128
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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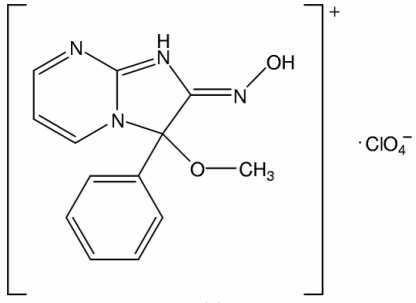
The title salt, $C_{13}H_{13}N_4O_2^+\cdot ClO_4^-$, contains an imidazo[1,2-*a*]pyrimidinium moiety carrying a hydroxyimine group, a methoxy group and a phenyl ring. The charge is balanced by a perchlorate anion. The 2-hydroximinoimidazo[1,2-*a*]pyrimidinium moiety is essentially planar and the hydroxyimine group is *cis* to the N atom of the heterocycle.

Comment

Functionalized imidazo[1,2-*a*]pyrimidines (IPM) are of great interest with regard to their potential biological activities (Anaflous *et al.*, 2004). Antimicrobial screening of imidazo[1,2-*a*]pyrimidine derivatives has been undertaken. The studies showed that compounds bearing a formyl, hydroxy or nitroso side chain in the 3-position are highly active (Benchat *et al.*, 2001). From general structure–biological activity correlations, it appears that functionalized side chains characterized by arms such as $[X-(C)_n-Y]$, where X, Y = O, N, with n = 2 or 3, are essential for such activity. These atoms or centres that have critical interactions with the bacterial cell receptor constitute the pharmacophore and are vital for antimicrobial activity. These interactions have typically precise geometric requirements that are readily described in terms of the distances between the atoms and their orientation in the pharmacophore. In continuation of this line of investigation, we have synthesized the title compound, (I). Compound (I) is stable at ambient temperature. Its composition and atomic connectivity have been determined by IR, MS and NMR (^{1}H and ^{13}C) spectroscopy. Since these techniques did not provide sufficient information about the conformation of the reaction product, we carried out the X-ray structure analysis described in this paper.



(I)

The bond lengths and angles of (I) do not show any unusual values. Whereas the hydroxyimine group is almost coplanar with the imidazo[1,2-*a*]pyrimidine moiety (Table 1), the phenyl ring makes a dihedral angle of 78.02 (6)° with it. The hydroxyl group forms a hydrogen bond with the perchlorate ion (Fig. 1), and the NH group forms a hydrogen bond to the N

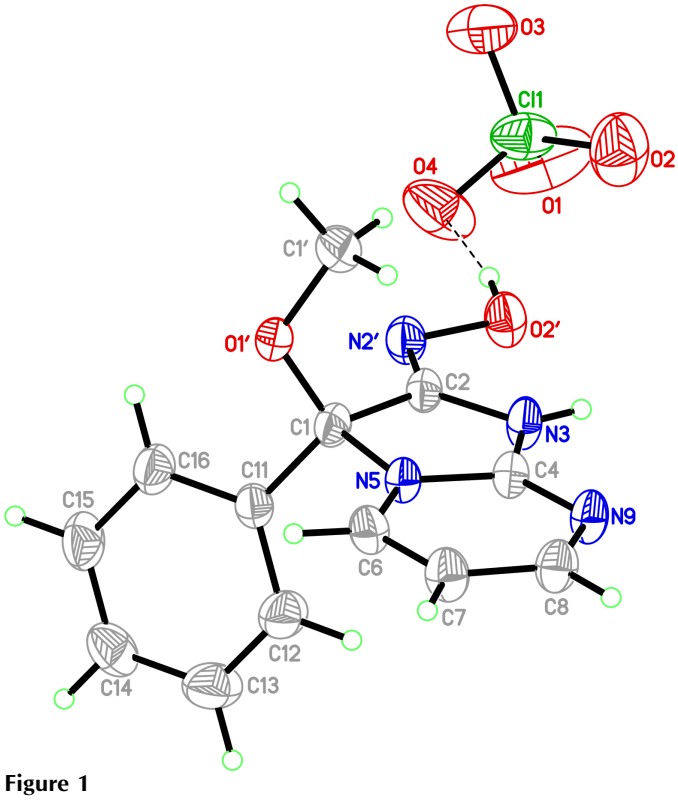


Figure 1
Perspective view of the title compound, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. The hydrogen bond is shown as a dashed line. H atoms are represented by small spheres.

atom of a symmetry-equivalent molecule. A short C6···O3($1+x$, $y-1$, z) contact [2.878 (3) Å] exists in the structure.

Experimental

The synthesis of (I) involved at first the condensation of the unsubstituted 2-aminopyrimidine intermediate (6.84 g, 72 mmol) with the chlorinated precursor $ClCH_2COC_6H_5$ (11.59 g, 75 mmol) in boiling ethanol. The reaction gave a good yield (70–75%) of 2-phenylimidazo[1,2-*a*]pyrimidine. This derivative (1 g, 5.13 mmol) was then functionalized with a nitroso group at the 3-position by treatment with 20 ml of an aqueous solution of sodium nitrite (0.22 mol l^{-1}) in acetic acid (10 ml) (Grassy & Rival, 1985; Rival *et al.*, 1991). Treatment of the latter compound (1 g, 0.0041 mol) with 20 ml of a hot methanol solution of perchloric acid (0.02 mol l^{-1}) leads to (I) in good yield (56%). Spectroscopic characterization: ^{1}H NMR (δ p.p.m.): 3.92 (*s*, 3H, OCH_3); 6.90 (*m*, 2H, H Ph, H6); 7.47 (*dd*, 2H Ph); 7.56 (*dd*, 2H Ph); 7.63 (*s*, NH); 7.93 (*dd*, 1H, H7).

Crystal data

$C_{13}H_{13}N_4O_2^+\cdot ClO_4^-$
M_r = 356.72
Triclinic, $P\overline{1}$
a = 7.9391 (16) Å
b = 8.7838 (18) Å
c = 11.611 (2) Å
α = 71.05 (3)°
β = 87.16 (3)°
γ = 89.42 (3)°
V = 764.9 (3) Å^3
Z = 2
D_x = 1.549 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 490 reflections
θ = 4.3–27.5°
μ = 0.29 mm^{-1}
T = 173 (2) K
Block, red
0.40 × 0.36 × 0.33 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.893, T_{max} = 0.910
12 375 measured reflections
3472 independent reflections
2770 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.06
3472 reflections
226 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.2799P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N2′	1.265 (2)	N2′—O2′	1.405 (2)
C2—N2′—O2′	109.37 (15)		
N3—C2—N2′—O2′	−0.4 (3)	C1—C2—N2′—O2′	179.93 (16)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N9^{i}	0.79 (3)	2.08 (3)	2.860 (3)	174 (3)
O2′—H2′···O4	0.90 (3)	1.80 (3)	2.688 (2)	170 (3)
O2′—H2′···O2	0.90 (3)	2.53 (3)	3.144 (3)	126 (3)
C6—H6···O3^{ii}	0.95	2.51	2.877 (3)	103

Symmetry codes: (i) $1-x, 2-y, -z$; (ii) $1+x, y-1, z$.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C) or U_{iso}(H) = 1.5 U_{eq}(C_{methyl})] using a riding model, with C—H = 0.95 and 0.98 Å for $C_{aromatic}$ and C_{methyl}, respectively; the methyl group was allowed to rotate but not to tip. The H atoms bonded to atoms N3 and O2′ were located in a difference map and refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

References

Anaflous, A., Benchat, N., Ben-Hadda, N., El Bali, B. & Bolte, M. (2004). *Acta Cryst.* E**60**, o1131–o1132.
Benchat, N., El Bali, B., Abouricha, S., Moueqqit, M., Mimouni, M. & Ben-Hadda, T. (2001). CPS: medichem/0301001. Available from http://preprint.chemweb.com/medichem/0301001.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Grassy, G. & Rival, Y. (1985). *Eur. J. Med. Chem. Chim.* Ther. **20**, 199–206.
Rival, Y., Grassy, G., Tandou, A. & Escalle, R. (1991). *Eur. J. Med. Chem.* **26**, 13–18.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.