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3-Phenylpyrazolium nitrate

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The title compound, $C_9H_9N_2^+\cdot NO_3^-$, is composed of discrete 3-phenylpyrazolium cations and nitrate anions. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds.

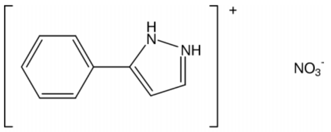
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.082
Data-to-parameter ratio = 14.6

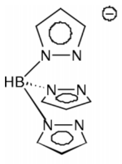
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

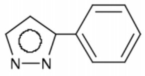
Recently, we have reported the X-ray crystal structure analysis of 3-phenylpyrazole, (2) (Haghiri *et al.*, 2002; Haghiri *et al.* 1993). We describe here the X-ray crystal structure analysis of 3-phenylpyrazolium nitrate, (I). Tris(1-pyrazolyl)borates ('scorpinates'), (1), were invented by Trofimenko more than 30 years ago and are today well established as ligands in coordination chemistry (Trofimenko, 2003), but only a limited number of transition metal complexes with 3-phenylpyrazole, (2), as ligand have been structurally characterized.



(I)



(1)



(2)

Therefore, we are interested in the synthesis of Fe^{III} complexes with (2) as ligand. Surprisingly, only protonation of (2) takes place in the reaction of $Fe(NO_3)_3$ with (2). Fe^{III} reacts as an acid in solution.

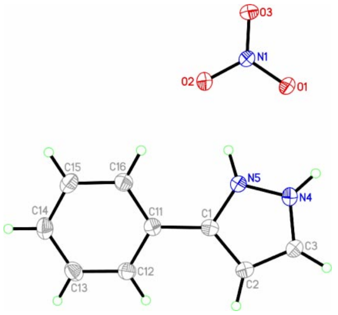


Figure 1
Perspective view of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

The crystal structure of (I) is composed of discrete 3-phenylpyrazolium cations and nitrate anions (Fig. 1). Geometric parameters are as expected. The dihedral angle between the phenyl ring and the pyrazole ring is 15.50 (7)°. Anions and cations are connected by bifurcated N—H···O hydrogen bonds. In addition, the crystal packing is stabilized by C—H···O hydrogen bonds (Fig. 2).

Experimental

X-ray quality crystals of (I) were obtained from a solution of 0.04 g (0.10 mmol) $Fe(NO_3)_3·9H_2O$ and 0.04 g (0.28 mmol) 3-phenylpyrazole in 5 ml CH_3OH at ambient temperature.

Crystal data

$C_9H_9N_2^+·NO_3^-$
M_r = 207.19
Monoclinic, $C2/c$
a = 16.8447 (16) Å
b = 13.392 (2) Å
c = 8.3779 (8) Å
β = 95.211 (8)°
V = 1882.2 (4) Å^3
Z = 8
D_x = 1.462 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5297 reflections
θ = 3.7–27.2°
μ = 0.11 mm^{-1}
T = 100 (2) K
Plate, colourless
0.22 × 0.16 × 0.12 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: none
8471 measured reflections
2106 independent reflections
1502 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.058
$\theta_{\rm max}$ = 27.3°
$h = -21 \rightarrow 21$
$k = -14 \rightarrow 17$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.082$
S = 0.90
2106 reflections
144 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1	0.90 (2)	2.40 (2)	2.9019 (17)	115.0 (16)
N4—H4···O1$^{\rm i}$	0.90 (2)	1.83 (2)	2.7233 (16)	169 (2)
N5—H5···O2	0.91 (2)	1.90 (2)	2.8060 (17)	177.0 (17)
N5—H5···O1	0.91 (2)	2.297 (18)	2.8802 (16)	121.7 (14)
C16—H16···O2	0.95	2.51	3.3643 (17)	149

Symmetry code: (i) $-x, 1-y, -z$.

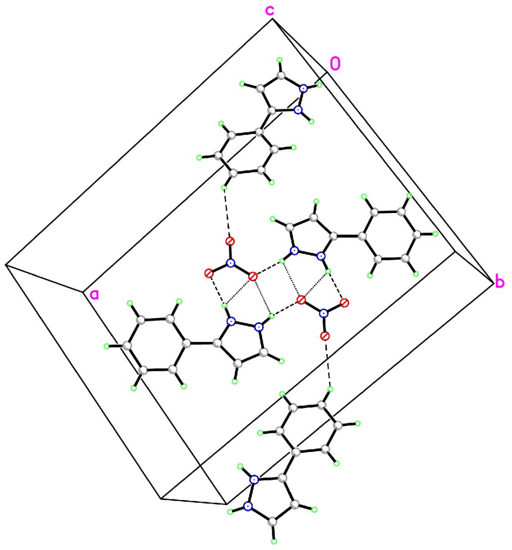


Figure 2
Hydrogen bonds (dashed lines) in the crystal structure of the title compound. Atom codes: C shaded black circles, H small open green circles, N blue shaded circles and O cross-hatched red circles.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)], using a riding model, with C—H = 0.95 Å. The H atoms bonded to N atoms were 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 and *PLATON* (Spek, 2003).

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