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2,9-Dimethyl-1,10-phenanthrolinium nitrate

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The title compound, $C_{14}H_{13}N_2^+\cdot NO_3^-$, is a protonated amine salt compound. A nitrate ion acts as hydrogen-bond acceptor of N—H···O and C—H···O hydrogen bonds, connecting the cations and anions into layers running parallel to the *ab* plane. The packing is additionally stabilized by π–π interactions between phenanthroline ring systems.

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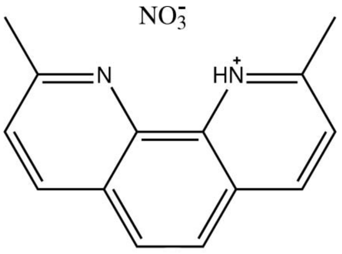
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.127
Data-to-parameter ratio = 10.3

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

Comment

Some metal complexes containing 1,10-phenanthroline are known to be good indicators for electrochemical probes (del Pozo *et al.*, 2005; Wang *et al.*, 1996). In our search for new probe indicators (Zhang *et al.*, 2005), the title compound, (I), was obtained as an intermediate. Compound (I) is an onium compound, in which the positive charge is localized at phenanthrolinium atom N2 (Fig. 1). This is also confirmed by the geometric parameters (Table 1) and the unambiguous location and refinement of all H atoms in the structure. All bond lengths and angles have usual values (Allen *et al.*, 1987).



(I)

The nitrate ion is linked to the cation and acts as a hydrogen-bond acceptor *via* N—H···O and C—H···O hydrogen bonds (Table 2). An intramolecular hydrogen bond, N2—H1*N*2···N1, forms a five-membered ring, which reinforces the planarity of the phenanthrolinium cation. In the crystal structure, the cations and anions are linked into two-dimensional layers parallel to the *bc* plane by N—H···O and C—H···O hydrogen bonds (Table 2 and Fig. 2). In the crystal packing, phenanthrolinium cations related by inversion symmetry have a separation of 3.601 Å [*Cg*2···*Cg*3$^{\rm iii}$, where *Cg*2 and *Cg*3 are the centroids of the N2/C8–C12 and C5–C8/C12/C13 rings; symmetry code: (iii) $-x$, $1 - y$ $1 - z$].

Experimental

In an ice-water bath, 2-methyl-8-aminoquinoline (124 g, 0.78 mol) and *o*-nitrophenol (55 g, 0.40 mol) were added to a solution of $AlCl_3$ (105 g, 0.85 mol) in hydrochloric acid (1200 ml, 30%); the mixture was stirred for 2 h at 273 K. Crotonaldehyde (67.2 g, 0.96 mol) was

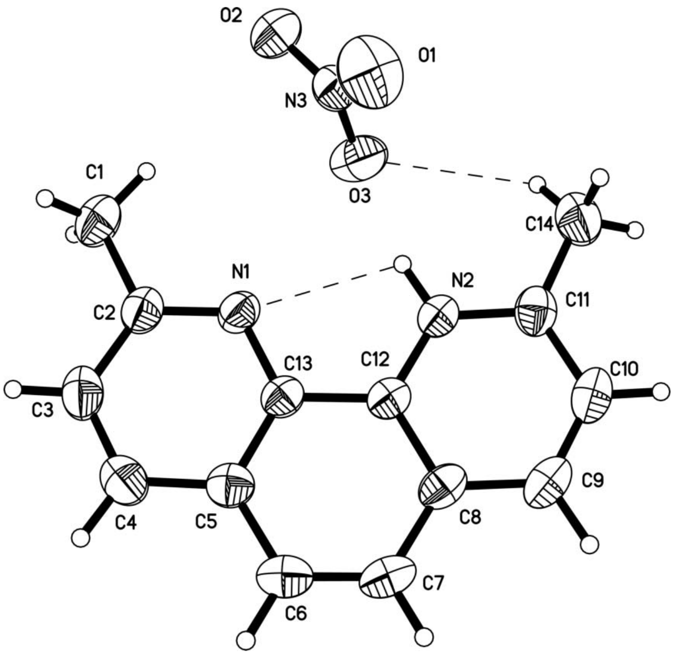


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

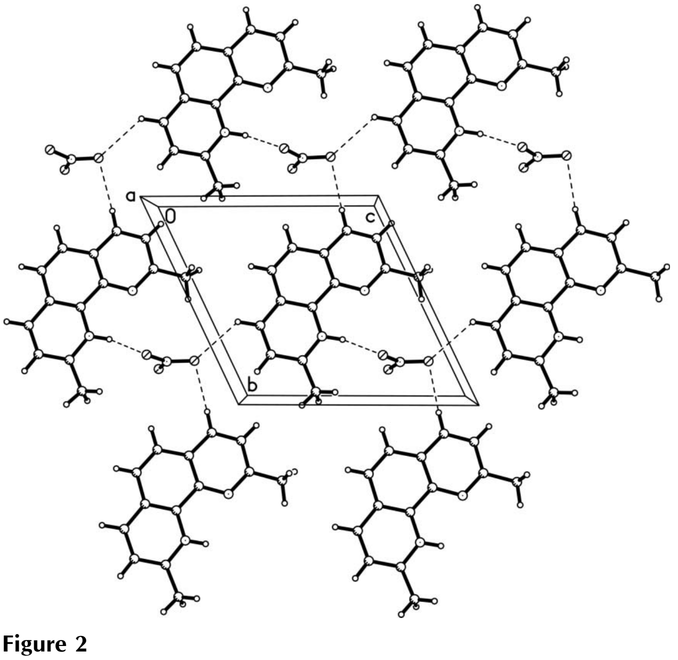


Figure 2
A view down the *a* axis showing the layer structure. Hydrogen bonds are indicated by dashed lines.

then added to the resulting solution and stirring continued for another 1 h. After cooling to room temperature, the reaction solution was poured into H_2O (50 ml) and NaOH was added until the solid dissolved. The mixture was filtered, giving 2,9-dimethyl-1,10-phenanthroline. To a solution of 2,9-dimethyl-1,10-phenanthroline (0.21 g, 1 mmol) in ethanol (10 ml), a solution of $Fe(NO_3)_3$ (0.24 g, 1 mmol) in distilled water (10 ml) was added. The mixture was stirred and reluxed for 5 h. The hot solution was filtered into another flask containing ethanol–water (1:1 *v*/*v*). Pale-yellow crystals appeared after a week by slow evaporation at room temperature.

Crystal data

$C_{14}H_{13}N_2^+\cdot NO_3^-$
$M_r = 271.27$
Triclinic, $P\overline{1}$
$a = 6.8971$ (10) Å
$b = 9.8366$ (14) Å
$c = 10.1800$ (14) Å
$\alpha = 64.443$ (2)°
$\beta = 86.796$ (2)°
$\gamma = 87.896$ (2)°
$V = 622.04$ (15) Å^3
$Z = 2$
$D_x = 1.448$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Plate, pale yellow
0.39 × 0.29 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.960$, $T_{\max} = 0.991$
3616 measured reflections
2396 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.011$
$\theta_{\max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.127$
$S = 1.02$
2396 reflections
233 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0703P)^2 + 0.0925P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—N3	1.236 (2)	C1—C2	1.493 (2)
O2—N3	1.2330 (19)	C11—C14	1.483 (3)
O3—N3	1.2455 (19)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O3	0.90 (2)	2.01 (2)	2.869 (2)	159 (2)
N2—H1*N*2···N1	0.90 (2)	2.36 (2)	2.730 (2)	104 (2)
C4—H4···O2$^{\rm i}$	0.95 (2)	2.46 (2)	3.312 (3)	150 (2)
C9—H9···O2$^{\rm ii}$	0.98 (2)	2.56 (2)	3.275 (2)	129 (2)
C14—H14*B*···O3	0.94 (3)	2.59 (3)	3.326 (2)	136 (2)

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

All H atoms were located in a difference Fourier map and were refined isotropically. The C—H and N—H distances are 0.91 (2)–0.99 (3) and 0.90 (2) Å, respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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