organic papers

2-[2-(Methylammonio)ethyliminiomethyl]-4-nitrophenolate nitrate

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The title compound, $C_{10}H_{14}N_3O_3{}^+ \cdot NO_3{}^-$, is a nitrate salt of the Schiff base 4-nitro-2-[2-(methylamino)ethyliminomethyl]-phenol. The molecule adopts a *trans* configuration about the C=N double bond. In the crystal structure, molecules are linked through intermolecular N—H···O hydrogen bonds, forming layers parallel to the *ab* plane.

Comment

Schiff base compounds derived from salicylaldehyde and its derivatives have been of great interest in coordination chemistry (Nishijima *et al.*, 1995; Archer & Wang, 1990). They can easily coordinate to the transition metal ions through the deprotonated phenolate O atoms and the imine N atoms. Recently, the author has reported the Schiff base compound 2-(cyclopropyliminomethyl)-4-nitrophenol (Zhao, 2005). As further investigation of the crystal structures of such ligands, the structure of the title compound, (I), is reported here.
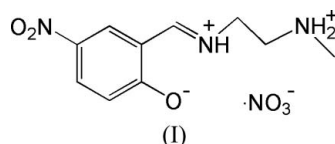
Compound (I) is a Schiff base compound (Fig. 1), which is derived from the condensation of equimolar 5-nitro-salicylaldehyde and *N*-methyl-1,2-diaminoethane in methanol.
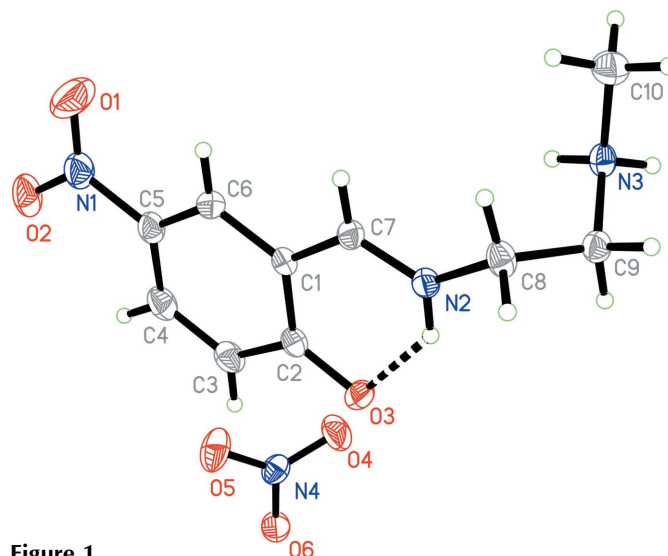
Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

All the bond lengths in the compound are within normal ranges (Allen *et al.*, 1987). As expected, atoms in the C7/N2/C8–C9/N3/C10 chain adopts a *trans* configuration to minimize steric effects. The dihedral angle between the benzene ring and the O1/N1/O2 nitro plane is 5.8 (3)°.

In the crystal structure, molecules are linked through intermolecular N—H⋯O hydrogen bonds (Table 2), forming layers parallel to the *ab* plane (Fig. 2).

Experimental

5-Nitrosalicylaldehyde (0.1 mmol, 16.8 mg), *N*-methyl-1,2-diaminoethane (0.1 mmol, 7.4 mg) and HNO$_3$ (0.1 mmol in 1 ml distilled water) were dissolved in MeOH (10 ml). The mixture was stirred for 30 min to give a yellow solution. The solution was left to stand in air for 5 d, whereupon yellow plate-shaped crystals formed.

Crystal data

$C_{10}H_{14}N_3O_3{}^+ \cdot NO_3{}^-$	$Z = 8$
$M_r = 286.25$	$D_x = 1.527$ Mg m^{-3}
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 7.119$ (2) Å	$\mu = 0.13$ mm^{-1}
$b = 16.922$ (4) Å	$T = 298$ (2) K
$c = 20.671$ (5) Å	Plate, yellow
$V = 2490.2$ (11) Å^3	$0.16 \times 0.10 \times 0.05$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	13625 measured reflections
ω scans	2564 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1430 reflections with $I > 2\sigma(I)$
$T_{min} = 0.980$, $T_{max} = 0.994$	$R_{int} = 0.103$
	$\theta_{max} = 26.5°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0371P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.071$	$+ 2.2208P]$
$wR(F^2) = 0.164$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2564 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
186 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3A⋯O6^i	0.90	2.00	2.838 (5)	155
N3—H3A⋯O2^{ii}	0.90	2.45	3.033 (4)	123
N3—H3B⋯O3^{iii}	0.90	1.95	2.824 (4)	165
N2—H2⋯O3	0.91 (3)	1.88 (3)	2.611 (4)	137 (4)

Symmetry codes: (i) $-x + \frac{3}{2}, -y, z - \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x - \frac{1}{2}, y, -z + \frac{1}{2}$.
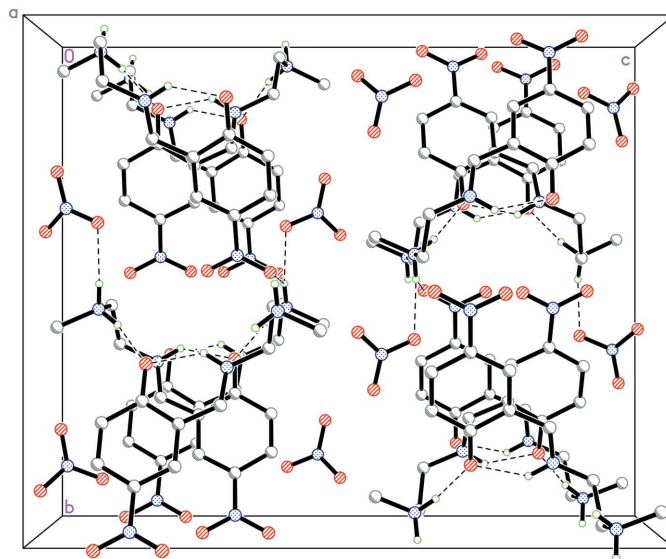
Figure 2
The crystal packing of (I), viewed along the *a* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Atom H2 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å, N—H = 0.90 Å and U_{iso}(H) = 1.2 or 1.5U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–83.
Archer, R. D. & Wang, B. (1990). *Inorg. Chem.* **29**, 39–43.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Nishijima, K., Nozaki, T., Miyasaka, H., Mago, G., Matsumoto, N. & Okawa, H. (1995). *Inorg. Chim. Acta*, **234**, 131–137.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Zhao, L.-F. (2005). *Acta Cryst.* E**61**, o1670–o1671.