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N—H···O hydrogen bonding in the adduct of *N*-(2-aminoethyl)-2-hydroxybenzamide with picric acid

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.142
Data-to-parameter ratio = 11.9

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

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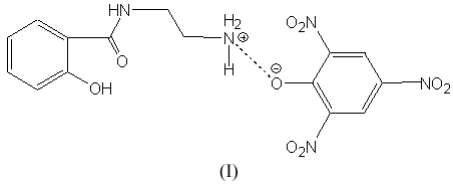
The title adduct *N*-(2-ammonioethyl)-2-hydroxybenzamide picrate, $C_9H_{13}N_2O_2^+\cdot C_6H_2N_3O_7^-$, contains an asymmetric N—H···O hydrogen bonds involving one N and three O atoms. The unit-cell packing is influenced by a network of intermolecular hydrogen bonds and π–π stacking of the aromatic rings.

Comment

Schiff bases are important ligands in coordination chemistry because they have applications in catalysis, medicine and magnetism, and they show biological activity (Vigato & Fenton, 1987; Yao, 2000). The amido group (HN—C═O) also has special biological activity and there are analogies between the amide and Schiff bases. By combining a Schiff base and an amide in one reaction, Yao Ke-Min and co-workers suggested that the differences in N properties could lead to some mixed or polymeric Schiff bases (Wu *et al.*, 2001). Therefore, we selected *N*-(2-aminoethyl)-2-hydroxybenzamide for the synthesis of this type of amine. Instead of KU-2 cation-exchange resin (Isagulyants *et al.*, 1972) and chromatographic resolution (Lutz *et al.*, 1996), we made use of the amine salt to purify the complex.



(I)

The crystal structure of the title compound, (I), showed it to be a 1:1 adduct. A view of this adduct is shown in Fig. 1. The two aromatic rings are approximately perpendicular to one another; the dihedral angle between mean planes is 75.9 (4)°. There are two intramolecular hydrogen bonds in the *N*-(2-ammonioethyl)-2-hydroxybenzamide fragment, *viz*. O1—H1···O2 of 2.618 (3) Å and N2—H2*C*···O2 of 3.045 (40) Å. A difference map showed that the H atom in the hydrogen bond between phenoxy atom O3 and amino atom N2 was localized on the N atom, indicating that the N atom is positively charged and the O atom negatively charged. The interaction between the ions is completed by four additional N—H···O hydrogen bonds. Geometric parameters for the hydrogen bonds, including two C—H···O bonds, are presented in Table 1.

In the crystal structure, the benzene rings of *N*-(2-ammonioethyl)-2-hydroxybenzamide are interlaced with the benzene rings of picric acid, to form columns along the [110] direction. This is illustrated in the packing diagram in Fig. 2. The interplanar distances between adjacent rings are about

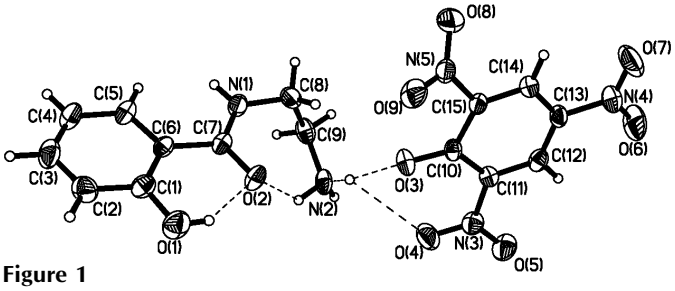


Figure 1
A view of the molecular structure of the adduct, with the atom-numbering scheme and 30% displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

2.96 and 3.36 Å, respectively. Each benzene ring is linked by N—H···O interactions to two adjacent nearly perpendicular aromatic ring columns related by a translation along *c* to give the appearance of a zigzag chain. The chains are linked through N—H···O and C—H···O interactions to complete a three-dimensional network.

Experimental

Methyl salicylate (1.5 g, 10 mmol) was dissolved in neat ethylenediamine (3.0 g, 50 mmol) and refluxed for 4 h. The solution was concentrated under vacuum, yielding a light brown oil. The oil was dissolved in 20 ml ethanol and added dropwise to an ethanol solution (20 ml) of 2,4,6-trinitrophenol (2.3 g, 10 mmol). The resulting solution was refluxed for 1 h and concentrated to 20 ml. The mixture was filtered hot. The filtrate was then kept at room temperature and yellow crystals were obtained. Analysis calculated for $C_{15}H_{15}N_5O_9$: C 44.01, H 3.70, N 17.11; found: C 43.70, H 3.88, N 17.00%.

Crystal data

$C_{15}H_{15}N_5O_9$	D_x = 1.518 Mg m^{-3}
M_r = 409.32	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 27 reflections
a = 12.761 (3) Å	θ = 3.0–13.2°
b = 8.005 (2) Å	μ = 0.13 mm^{-1}
c = 17.744 (6) Å	T = 295 (2) K
β = 98.84 (3)°	Block, yellow
V = 1790.9 (8) Å^3	0.56 × 0.44 × 0.40 mm
Z = 4	

Data collection

Siemens *P*4 diffractometer	θ_{max} = 25.0°
ω scans	$h = 0 \rightarrow 15$
Absorption correction: none	$k = 0 \rightarrow 9$
3305 measured reflections	$l = -21 \rightarrow 20$
3154 independent reflections	3 standard reflections
1801 reflections with $I > 2\sigma(I)$	every 97 reflections
R_{int} = 0.020	intensity decay: 2.8%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.074P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.142	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
S = 0.94	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
3154 reflections	Extinction correction: *SHELXL*97
265 parameters	Extinction coefficient: 0.0056 (13)
H-atom parameters constrained	

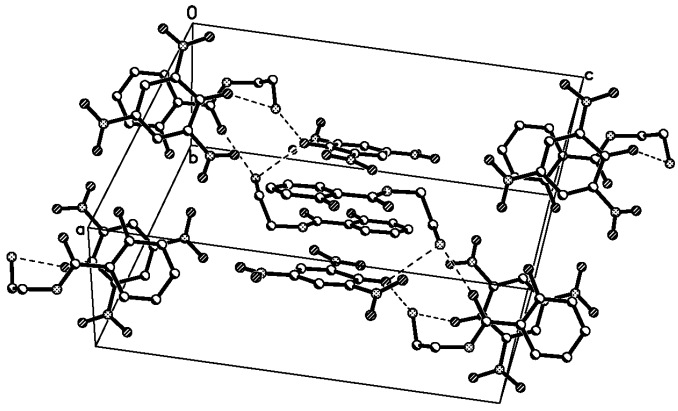


Figure 2
View of the crystal packing of *N*-(2-ammonioethyl)-2-hydroxybenzamide picrate down the *a* axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.90	2.618 (3)	146
N2—H2*B*···O2^{i}	0.89	1.99	2.865 (3)	165
N2—H2*A*···O3	0.89	1.90	2.779 (3)	171
N1—H1*N*···O5^{ii}	0.86	2.31	3.078 (8)	150
N2—H2*A*···O4	0.89	2.59	3.093 (2)	117
N2—H2*C*···O2	0.89	2.47	3.045 (4)	123
N2—H2*C*···O3^{i}	0.89	2.26	2.832 (4)	122
N2—H2*C*···O9^{i}	0.89	2.30	3.035 (1)	141
C2—H2···O8^{iii}	0.93	2.44	3.360 (6)	174
C9—H9*B*···O7^{iv}	0.97	2.31	3.217 (7)	154

Symmetry codes: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $\frac{1}{2} + x, -\frac{1}{2} - y, z - \frac{1}{2}$; (iv) $1 - x, -y, 2 - z$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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