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Promazinium picrate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.046
wR factor = 0.128
Data-to-parameter ratio = 15.7

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

In the title compound [systematic name: 3-(phenothiazin-10-yl)propanaminium 2,4,6-trinitrophenolate], $C_{17}H_{21}N_2S^+\cdot C_6H_2N_3O_7^-$, the dihedral angle between the two outer aromatic rings of the phenothiazine unit is 37.76 (6)°. The crystal packing is stabilized by N—H···O hydrogen bonds and several weak C—H···O contacts.

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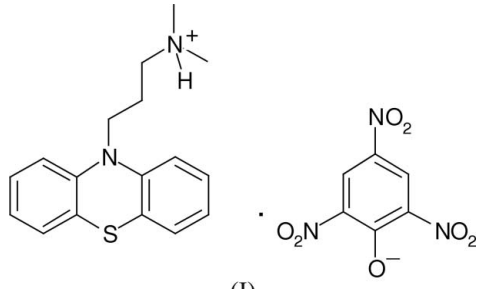
Comment

Promazine hydrochloride (promazinium chloride) is an important antipsychotic drug (Tarasiewicz & Basinska, 1974). The organic cation contains a distinctive tricyclic aromatic ring system containing S and N atoms in the central ring. As part of our studies of these systems, we describe here the synthesis and structure of the title salt, (I) (Fig. 1), containing the promazinium cation, accompanied by picrate anions.



(I)

The bond lengths and angles for (I) can be regarded as normal [Cambridge Structural Database, Version 5.28, November 2006 (Allen, 2002); *Mogul* Version 1.1 (Bruno *et al.*,

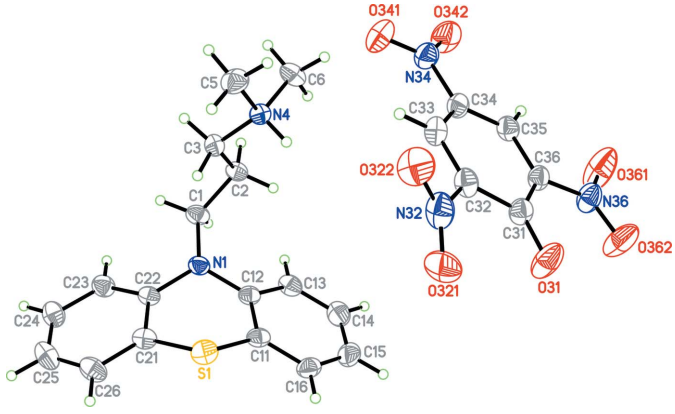


Figure 1
View of the molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). Only the major orientation of the disordered N32 nitro group is shown.

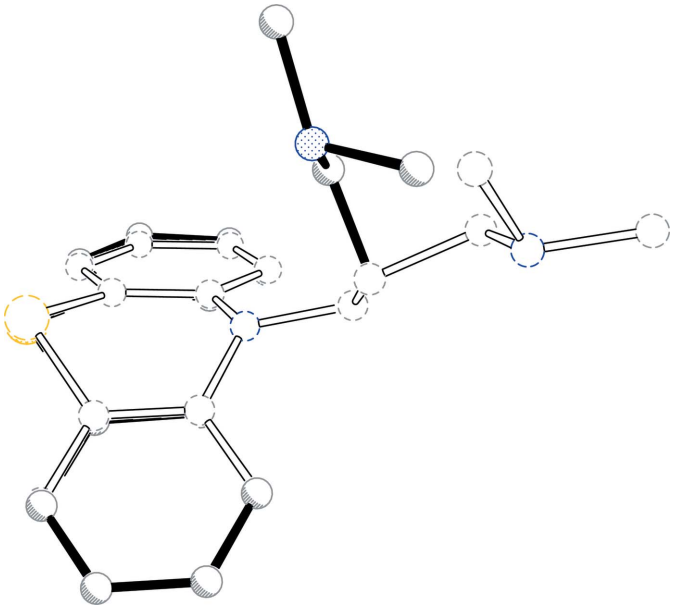


Figure 2
Least-squares fit of the promazinium cations in (I) (full bonds) and (II) (open bonds). H atoms have been omitted.

2004)]. The dihedral angle between the two aromatic rings of the phenothiazine ring system is 37.76 (6)°. Crystallographic studies of promazine (Falkenberg & Ringertz, 1967) and promazine hydrochloride, (II) (David *et al.*, 1998) have been reported, but atomic coordinates (without standard uncertainties) are available only for the latter. A least-squares comparison of the promazinium cations of (I) and (II), fitting only the phenothiazine units (r.m.s. deviation 0.070Å), is shown in Fig. 2. The cations differ only in two side-chain torsion angles. Whereas the conformation of N1—C1—C2—C3 is anticlinal in (I) (Table 1), it is antiperiplanar (−166.3°) in (II). On the other hand, C2—C3—N4—C6 is synclinal in (I), but anticlinal (−76.7°) in (II). The conformation of C1—C2—C3—N4 and C2—C3—N4—C5 is antiperiplanar in both structures. The crystal packing for (I) is stabilized by N—H···O hydrogen bonds and several weak C—H···O contacts (Table 2).

Experimental

Promazine hydrochloride (1.605 g, 0.05 *M*) and picric acid (1.147 g, 0.05 *M*) were mixed and stirred in a beaker. The precipitated bright brick-red salt was filtered off and washed thoroughly with doubly distilled water and dried over P_2O_5 in a vacuum desiccator. The compound was recrystallized from ethanol by slow evaporation (m.p. 415–417 K), yielding red plates of (I).

Crystal data

$C_{17}H_{21}N_2S^+\cdot C_6H_2N_3O_7^-$
M_r = 513.52
Triclinic, $P\overline{1}$
a = 10.0611 (6) Å
b = 10.2518 (7) Å
c = 11.9841 (8) Å
α = 85.649 (6)°
β = 78.414 (5)°
γ = 82.040 (5)°
V = 1197.77 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 173 (2) K
0.42 × 0.36 × 0.19 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.915, T_{max} = 0.955
22432 measured reflections
5489 independent reflections
5037 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.04
5489 reflections
349 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

Table 1
Selected torsion angles (°).

N1—C1—C2—C3	−72.28 (15)	C2—C3—N4—C6	50.38 (16)
C1—C2—C3—N4	161.37 (11)	C2—C3—N4—C5	174.58 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O31^{i}	0.93 (2)	1.78 (2)	2.6735 (17)	162 (2)
N4—H4···O362^{i}	0.93 (2)	2.41 (2)	2.9804 (18)	119.8 (17)
C2—H2*B*···O322^{ii}	0.99	2.53	3.280 (2)	132
C5—H5*A*···O362^{i}	0.98	2.46	3.008 (2)	115
C5—H5*C*···O341	0.98	2.57	3.439 (2)	148
C6—H6*B*···O341	0.98	2.50	3.389 (2)	150
C13—H13···O321^{ii}	0.95	2.59	3.336 (3)	136

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

The N-bound H atom was located in a difference map. Its position and U_{iso} value were freely refined. The C-bound H atoms were found in a difference map, repositioned in idealized locations (C—H = 0.95–0.99 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The O atoms of one of the picrate nitro groups are disordered over two positions with site occupation factors of 0.662 (4) and 0.338 (4) (sum constrained to unity).

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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