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Ethopropazinium picrate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.077
wR factor = 0.212
Data-to-parameter ratio = 12.7

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

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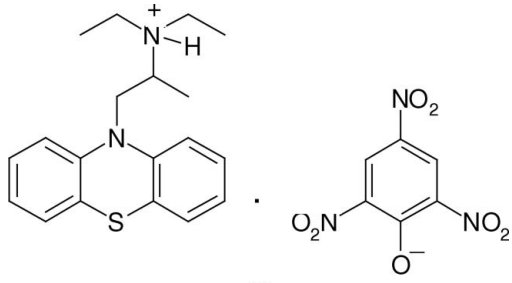
The title compound [systematic name: 10-[2-(diethylamino)-propyl]phenothiazinium 2,4,6-trinitrophenolate], $C_{19}H_{25}N_2S^+\cdot C_6H_2N_3O_7^-$, is a pharmacologially active compound. The dihedral angle between the two outer aromatic rings of the phenothiazine unit is 38.64 (12)°. The crystal packing is stabilized by N—H···O hydrogen bonds and several weak C—H···O contacts. The molecular conformation of the cation does not change significantly when it is crystallized with chloride or perrhenate as the anion.

Comment

Ethopropazine is an anticholinergic agent with some anti-histaminic and ganglionic blocking activity (Bratfos & Haug, 1979). The present work results in the formation of a salt by the interaction between ethopropazinium hydrochloride and 2,4,6-trinitrophenol in an aqueous medium.



(I)

A perspective view of the title compound, (I), is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.28, November 2006; *MOGUL*, Version 1.1; Allen, 2002; Bruno *et al.*, 2004). The dihedral angle between the two aromatic rings of the phenothiazine unit is 38.64 (12)°. Crystallographic data for ethopropazinium chloride, (I*a*) (Marsau & Calas, 1971; Klein *et al.*, 1994), and ethopropazinium perrhenate, (I*b*) (Gowda *et al.*, 1994), have been published, but since there is a coordinate error in the report by Marsau & Calas (1971), the structure given by Klein *et al.* (1994) is employed for comparison. Least-squares overlays of the ethopropazinium cations of (I) and (I*a*) (r.m.s. deviation 0.066 Å) as well as of (I) and (I*b*) (r.m.s. deviation 0.120 Å), fitting only the phenothiazine units, are shown in Figs. 2 and 3. As can be seen, the molecular conformation is not significantly different in the compared structures. The crystal packing is stabilized by N—H···O hydrogen bonds and several weak C—H···O contacts (Table 1).

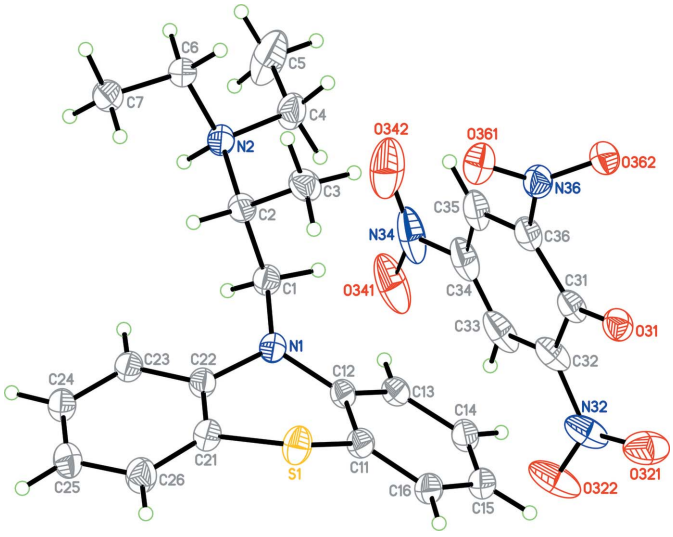


Figure 1
The structure of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 30% probability level.

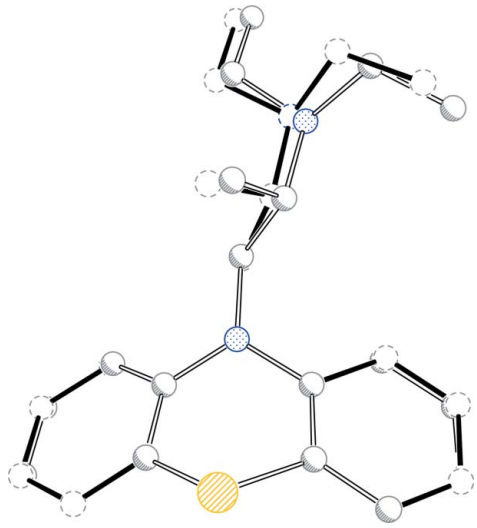


Figure 2
Least-squares fit of the ethopropazinium cations in (I) (full bonds) and (I*a*) (open bonds). H atoms have been omitted.

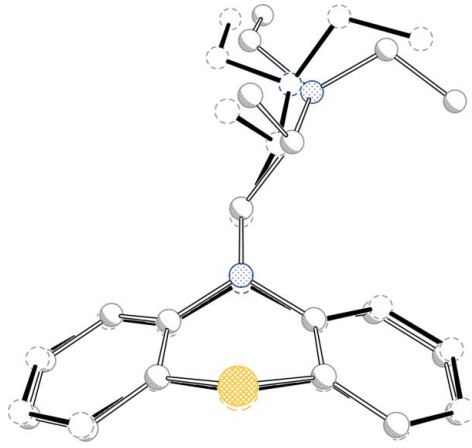


Figure 3
Least-squares fit of the ethopropazinium cations in (I) (full bonds) and (I*b*) (open bonds). H atoms have been omitted.

Experimental

Profenamine hydrochloride (0.7010 g, 0.02 *M*) and picric acid (0.4610 g, 0.02 *M*) were dissolved in distilled water (100 ml), mixed and stirred in a beaker at room temperature. The separated yellow salt was washed well with distilled water, filtered and dried in a vacuum desiccator over phosphorus pentoxide. The complex was recrystallized from acetonitrile (m.p. 378 K).

Crystal data

$C_{19}H_{25}N_2S^+\cdot C_6H_2N_3O_7^-$
M_r = 541.58
Monoclinic, $C2/c$
a = 36.876 (3) Å
b = 8.4622 (4) Å
c = 16.5727 (11) Å
β = 104.163 (6)°
V = 5014.3 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 173 (2) K
0.29 × 0.26 × 0.25 mm

Data collection

Stoe IPDS-II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.938, T_{max} = 0.945
22001 measured reflections
4421 independent reflections
3505 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.212
S = 1.06
4421 reflections
347 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O31^{i}	0.95 (5)	1.92 (5)	2.841 (4)	161 (4)
C1—H1*A*···O31^{i}	0.99	2.45	3.234 (4)	136
C23—H23···O31^{i}	0.95	2.37	3.316 (4)	175

Symmetry code: (i) $x, -y+1, z-\frac{1}{2}$.

H atoms were found in a difference map, but the C-bound H atoms were refined using a riding model, with C—H ranging from 0.95 to 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). The H atom bonded to N was freely refined.

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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