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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1438). Services for accessing these data are described at the back of the journal. Synthesis and spectroscopic analysis details have also been deposited.

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10-Ethyl-3-nitrophenothiazine

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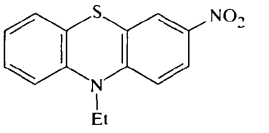
Abstract

The title compound, $C_{14}H_{12}N_2O_2S$ (ENP), is a precursor in the synthesis of antipsychotic drugs based on 3-nitrophenothiazines. The structure analysis confirms the expected 3-nitro derivative. The nitro group flattens the tricycle; the dihedral angle between the phenyl rings is 157.2 (1)°, compared with a value of 135.2 (1)° in the non-nitrated compound.

Comment

Phenothiazines having different substituents have been systematically synthesized in order to find new antipsychotic drugs. The title compound, ENP, is an intermediate in the synthesis of active 3-nitrophenothiazinic compounds.



ENP

The structural study of ENP was undertaken in order to confirm the position of the nitro group and the transposition of the phenyl group corresponding to the Smiles rearrangement (Levy *et al.*, 1931).

The folding of the molecule is characterized by the angle between the phenyl planes, whose value is 157.2 (1)°. In the non-nitrated compound, 10-ethylphenothiazine (Chu & van der Helm, 1975), the corresponding angle is 135.2 (1)°, which suggests that addition of groups to the phenyl rings flattens the tricycle. This tendency is also observed in the structure of the dibromo-substituted compound, 3,7-dibromo-10-ethylphenothiazine (Meester *et al.*, 1986), in which the folding angles are 156.3 (2) and 145.3 (2)° in the two independent molecules.

In ENP, the ethyl group is almost orthogonal to the thiazine ring, the torsion angle C9—N10—C11—C12 being −84.9 (2)°; the corresponding angle is 146.1 (4)° in the non-nitrated compound (Chu & van der Helm, 1975). The nitro group is almost coplanar with its

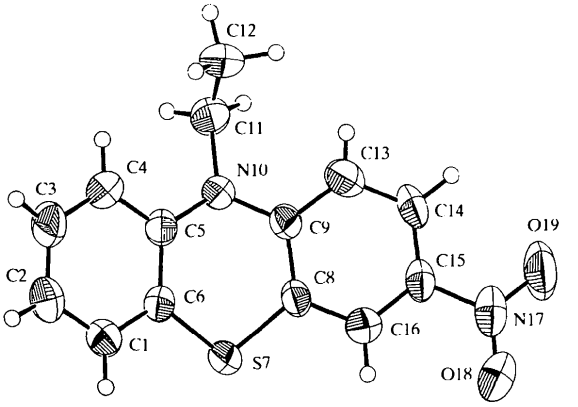


Fig. 1. The molecular structure of ENP shown with 50% probability displacement ellipsoids.

attached phenyl ring, the torsion angle C14—C15—N17—O19 being −3.1 (3)°. The crystal packing is characterized by van der Waals interactions.

Experimental

3-Nitrophenothiazine was prepared by the reaction of mercaptoaniline with chloro-2,4-dinitrobenzene in ethanol containing sodium acetate (Gupta, 1988; Saraswat *et al.*, 1993). Phase transfer catalysis was used for the alkylation of the N10 atom, with toluene as solvent and triethylbenzylammonium chloride as dispersant, the aqueous phase being a 50% potassium hydroxide solution. Purple square-needle-shaped crystals were obtained by evaporation of a water–methanol (80:20) solution.

Crystal data

$C_{14}H_{12}N_2O_2S$
M_r = 272.32
Orthorhombic
Pnaa
a = 7.654 (1) Å
b = 11.282 (2) Å
c = 29.404 (4) Å
V = 2539.1 (7) Å^3
Z = 8
D_x = 1.425 Mg m^{-3}
D_m = 1.41 (2) Mg m^{-3}
D_m measured by flotation in benzene–chloroform
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9–16°
μ = 0.254 mm^{-1}
T = 293 (2) K
Square prism cut from a needle
0.41 × 0.32 × 0.29 mm
Purple

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3271 measured reflections
3271 independent reflections
2342 reflections with $I > 2\sigma(I)$
θ_{max} = 30.16°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 41$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.082
S = 0.992
3271 reflections
208 parameters
H-atom coordinates refined with $U = 1.2U_{eq}$(parent atom)
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.213 e Å^{-3}
$\Delta\rho_{min}$ = −0.240 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C5—N10	1.403 (2)	C9—N10	1.391 (2)
C6—S7	1.7600 (14)	N10—C11	1.475 (2)
S7—C8	1.7465 (14)		
C8—S7—C6	100.15 (7)	C9—N10—C11	117.72 (14)
C9 N10 C5	122.84 (13)	C5 N10 C11	118.23 (13)
N10—C5—C6—S7	−5.7 (2)	C5—N10—C11—C12	82.9 (2)
S7—C8—C9—N10	8.0 (2)	C14—C15—N17—O19	−3.1 (3)
C9—N10—C11—C12	−84.9 (2)		

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *DATARED* (Pèpe, 1979). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1230). Services for accessing these data are described at the back of the journal.

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(Z)-5-(1-Methoxy-2-naphthylmethylene)-4-oxo-2-thioxo-1,3-thiazolidine-3-acetic Acid Dimethyl Sulfoxide Solvate

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Abstract

The title compound (1-OCH_3-NOTA) is a potent aldose reductase (AR) inhibitor. It is found to co-crystallize with the solvent dimethyl sulfoxide (DMSO), *i.e.* $C_{17}H_{13}NO_4S_2.C_2H_6OS$. The skeleton of 1-$OCH_3$-NOTA is highly planar, except for the acetic acid group