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10-Methyl-10*H*-phenothiazine

Parvathi Malikireddy,[a] Gouthaman Siddan,[b] Sugunalakshmi Madurai,[b] Suvasini Chandramouleeswaran[a] and Lakshmi Srinivasakannan[a]*

[a]Research Department of Physics, S.D.N.B. Vaishnav College for Women, Chromepet, Chennai 600 044, India, and [b]Industrial Chemistry Polymer Division, CSIR Central Leather Research Institute, Chennai 600 020, India. *Correspondence e-mail: lakssdnbvc@gmail.com

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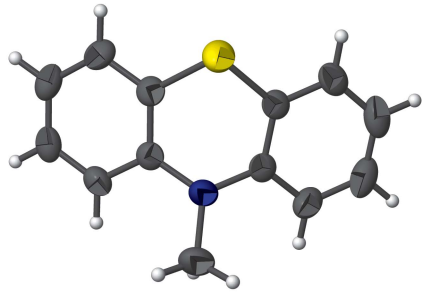
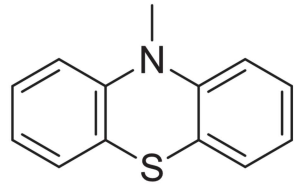
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound $C_{13}H_{11}NS$, the phenothiazine unit has a non-planar butterfly structure, and the central six-membered ring adopts a boat conformation. The dihedral angle between the two outer aromatic rings of the phenothiazine unit is 39.53 (10)°. In the crystal, a π–π interaction with a centroid–centroid distance of 3.6871 (12) Å is observed between the aromatic rings of neighbouring molecules.

3D view



Chemical scheme



Structure description

Phenothiazine derivatives possess anti-tumor, anti-bacterial, anti-plasmid and anti-tuberculosis activities (He *et al.*, 2015). Trifluoperazine, a phenothiazine derivative, is used for treating schizophrenia by minimizing hallucinations, delusions and disorganized thought and speech (Stanković *et al.*, 2015). The photodegradation of tricyclic cytosine, another phenothiazine derivative, finds application as a switching mechanism in DNA-based nanodevices (Preus *et al.*, 2013). Other phenothiazine derivatives are used in electrochromic devices (Grätzel, 2001) and act as donors in dye-sensitized solar cell fabrication (Marszalek *et al.*, 2012).

In the title compound, the phenothiazine moiety has a non-planar butterfly structure (Fig. 1). The central six-membered ring adopts a boat conformation [Q_T = 0.5994 (16) Å, θ= 96.86 (18), φ= 180.7 (2)°]. The dihedral angle between the two outer aromatic rings of the phenothiazine unit is 39.53 (10)°. The crystal packing exhibits a π–π interaction with a centroid-centroid distance of 3.6871 (12) Å between the benzene rings (C1–C6) of neighbouring molecules. The crystal packing is shown in Fig. 2.

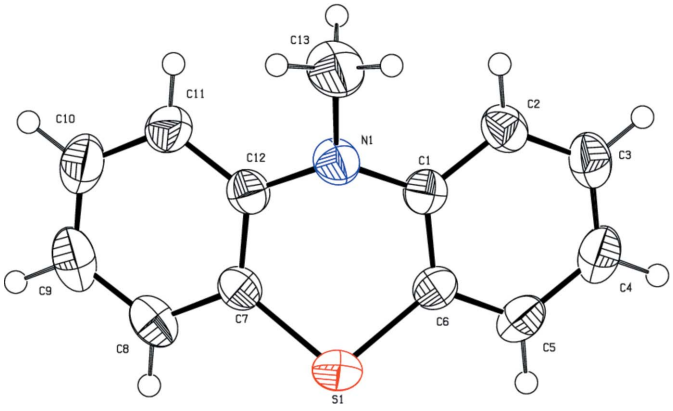


Figure 1
The molecular structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small sphere of arbitrary radius.

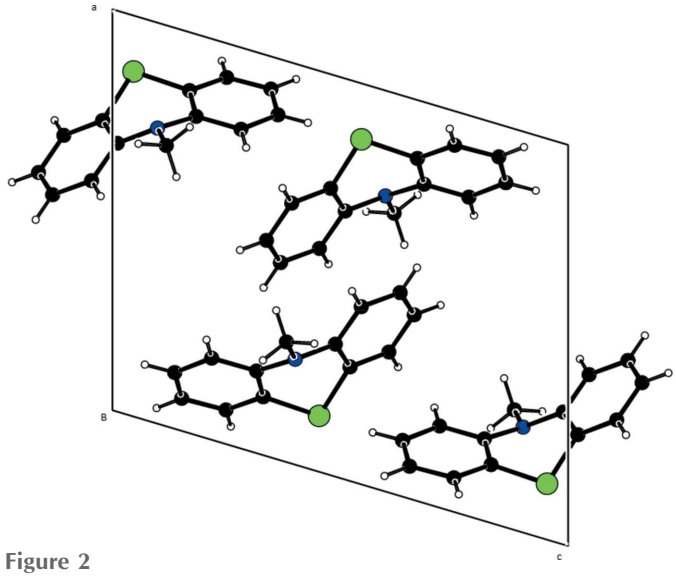


Figure 2
The packing of the title compound, viewed down the *b* axis.

Synthesis and crystallization

Phenothiazine (0.400 g, 0.002 mmol, 1 equiv.) was dissolved in DMF (5 ml). Sodium hydride (0.0964 g, 0.002 mmol, 2 equiv.) was added to the reaction mixture at 273 K within 15 min and stirred for 30 min at 273 K. Iodomethane (0.250 ml, 0.002 mmol, 2 equiv) was added slowly at 273 K and stirred for 2–3 h at room temperature. The completion of the reaction was monitored by TLC. The reaction mass was poured in ice and stirred, filtered and dried. The product was purified by column chromatography using silica gel 100–200 mesh and ethyl acetate: hexane (3:97) as eluent system. The crude product was recrystallized from a mixed solvent of DMF and DCM, yielding green block-shaped crystals.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{11}NS$
M_r	213.29
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	11.6245 (7), 6.9130 (4), 13.7792 (10)
β (°)	106.591 (2)
V (Å^3)	1061.20 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.27
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.691, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10676, 1868, 1567
R_{int}	0.018
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.097, 1.05
No. of reflections	1868
No. of parameters	137
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.21

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

The authors thank the single-crystal XRD facility, SAIF IIT Madras, Chennai, for the data collection.

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full crystallographic data

IUCrData (2016). **1**, x161299 [doi:10.1107/S2414314616012992]

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Crystal data

$C_{13}H_{11}NS$
M_r = 213.29
Monoclinic, $P2_1/c$
a = 11.6245 (7) Å
b = 6.9130 (4) Å
c = 13.7792 (10) Å
β = 106.591 (2)°
V = 1061.20 (12) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.335 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1567 reflections
θ = 3.1–29.4°
μ = 0.27 mm^{-1}
T = 296 K
Block, green
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Bruker axs kappa axes2 CCD Diffractometer scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.691, T_{max} = 0.746
10676 measured reflections
1868 independent reflections
1567 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°, θ_{min} = 3.1°
h = −13→13
k = −8→8
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097
S = 1.05
1868 reflections
137 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.6758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.86050 (5)	0.40501 (9)	0.04641 (4)	0.0510 (2)
N1	0.73700 (14)	0.7457 (2)	0.09898 (12)	0.0424 (4)
C6	0.71525 (17)	0.4834 (3)	−0.02138 (14)	0.0386 (4)
C7	0.85448 (15)	0.4638 (3)	0.16918 (14)	0.0377 (4)
C2	0.55714 (17)	0.7158 (3)	−0.04584 (15)	0.0435 (5)
H2	0.525007	0.826949	−0.025824	0.052*
C12	0.79493 (15)	0.6314 (3)	0.18380 (14)	0.0375 (4)
C1	0.66956 (16)	0.6503 (3)	0.01080 (14)	0.0363 (4)
C5	0.64954 (19)	0.3852 (3)	−0.10654 (15)	0.0482 (5)
H5	0.680226	0.272801	−0.126692	0.058*
C3	0.49300 (19)	0.6178 (3)	−0.13119 (15)	0.0487 (5)
H3	0.418167	0.663806	−0.168228	0.058*
C11	0.79623 (18)	0.6807 (3)	0.28215 (15)	0.0495 (5)
H11	0.754341	0.788976	0.293389	0.059*
C4	0.5380 (2)	0.4536 (4)	−0.16213 (15)	0.0523 (5)
H4	0.494181	0.388443	−0.219924	0.063*
C8	0.91483 (17)	0.3503 (3)	0.25088 (16)	0.0480 (5)
H8	0.952563	0.236993	0.240176	0.058*
C9	0.9189 (2)	0.4055 (4)	0.34792 (17)	0.0581 (6)
H9	0.961780	0.331961	0.402883	0.070*
C10	0.8596 (2)	0.5690 (4)	0.36327 (17)	0.0596 (6)
H10	0.862047	0.605223	0.428820	0.072*
C13	0.7003 (2)	0.9396 (3)	0.1182 (2)	0.0638 (7)
H13A	0.683800	1.014474	0.057068	0.096*
H13B	0.763616	1.000018	0.169713	0.096*
H13C	0.629368	0.932321	0.140436	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0417 (3)	0.0638 (4)	0.0492 (3)	0.0133 (2)	0.0158 (2)	−0.0065 (3)
N1	0.0440 (9)	0.0328 (8)	0.0465 (10)	0.0017 (7)	0.0065 (7)	−0.0023 (7)
C6	0.0394 (10)	0.0458 (11)	0.0346 (10)	0.0009 (8)	0.0171 (8)	0.0021 (8)
C7	0.0278 (9)	0.0429 (11)	0.0414 (10)	−0.0012 (8)	0.0085 (8)	−0.0006 (8)
C2	0.0444 (11)	0.0406 (11)	0.0446 (11)	0.0053 (9)	0.0113 (9)	0.0091 (9)
C12	0.0288 (9)	0.0412 (10)	0.0403 (10)	−0.0038 (8)	0.0067 (8)	−0.0030 (8)
C1	0.0396 (10)	0.0358 (10)	0.0356 (10)	−0.0015 (8)	0.0142 (8)	0.0051 (8)
C5	0.0557 (13)	0.0537 (13)	0.0388 (11)	−0.0004 (10)	0.0193 (9)	−0.0086 (9)
C3	0.0438 (11)	0.0613 (14)	0.0380 (11)	−0.0014 (10)	0.0070 (9)	0.0113 (10)
C11	0.0401 (11)	0.0613 (13)	0.0452 (12)	0.0002 (10)	0.0095 (9)	−0.0126 (10)
C4	0.0534 (13)	0.0681 (15)	0.0334 (11)	−0.0090 (11)	0.0094 (9)	−0.0032 (10)
C8	0.0348 (10)	0.0490 (12)	0.0552 (13)	0.0011 (9)	0.0048 (9)	0.0060 (10)
C9	0.0428 (12)	0.0780 (17)	0.0464 (13)	−0.0064 (11)	0.0015 (10)	0.0167 (12)
C10	0.0467 (12)	0.0927 (19)	0.0372 (11)	−0.0097 (13)	0.0086 (9)	−0.0066 (12)
C13	0.0721 (16)	0.0369 (12)	0.0734 (16)	0.0045 (11)	0.0061 (13)	−0.0066 (11)

Geometric parameters (Å, º)

S1—C7	1.760 (2)	C5—H5	0.9300
S1—C6	1.7652 (19)	C3—C4	1.368 (3)
N1—C1	1.408 (2)	C3—H3	0.9300
N1—C12	1.412 (2)	C11—C10	1.385 (3)
N1—C13	1.454 (3)	C11—H11	0.9300
C6—C5	1.382 (3)	C4—H4	0.9300
C6—C1	1.395 (3)	C8—C9	1.378 (3)
C7—C8	1.387 (3)	C8—H8	0.9300
C7—C12	1.394 (3)	C9—C10	1.371 (4)
C2—C3	1.377 (3)	C9—H9	0.9300
C2—C1	1.393 (3)	C10—H10	0.9300
C2—H2	0.9300	C13—H13A	0.9600
C12—C11	1.393 (3)	C13—H13B	0.9600
C5—C4	1.387 (3)	C13—H13C	0.9600
C7—S1—C6	98.23 (8)	C4—C3—H3	119.6
C1—N1—C12	117.96 (15)	C2—C3—H3	119.6
C1—N1—C13	117.97 (17)	C10—C11—C12	120.3 (2)
C12—N1—C13	117.42 (17)	C10—C11—H11	119.9
C5—C6—C1	120.53 (18)	C12—C11—H11	119.9
C5—C6—S1	120.80 (16)	C3—C4—C5	119.4 (2)
C1—C6—S1	118.64 (14)	C3—C4—H4	120.3
C8—C7—C12	120.65 (19)	C5—C4—H4	120.3
C8—C7—S1	119.99 (16)	C9—C8—C7	120.0 (2)
C12—C7—S1	119.23 (14)	C9—C8—H8	120.0
C3—C2—C1	120.74 (19)	C7—C8—H8	120.0
C3—C2—H2	119.6	C10—C9—C8	119.9 (2)
C1—C2—H2	119.6	C10—C9—H9	120.1
C11—C12—C7	118.41 (18)	C8—C9—H9	120.1
C11—C12—N1	122.60 (18)	C9—C10—C11	120.7 (2)
C7—C12—N1	118.99 (17)	C9—C10—H10	119.7
C2—C1—C6	118.19 (18)	C11—C10—H10	119.7
C2—C1—N1	122.26 (17)	N1—C13—H13A	109.5
C6—C1—N1	119.55 (16)	N1—C13—H13B	109.5
C6—C5—C4	120.3 (2)	H13A—C13—H13B	109.5
C6—C5—H5	119.9	N1—C13—H13C	109.5
C4—C5—H5	119.9	H13A—C13—H13C	109.5
C4—C3—C2	120.8 (2)	H13B—C13—H13C	109.5
C7—S1—C6—C5	−144.50 (17)	S1—C6—C1—N1	−3.4 (2)
C7—S1—C6—C1	37.60 (17)	C12—N1—C1—C2	136.84 (18)
C6—S1—C7—C8	146.79 (16)	C13—N1—C1—C2	−13.9 (3)
C6—S1—C7—C12	−37.18 (16)	C12—N1—C1—C6	−42.6 (2)
C8—C7—C12—C11	−0.8 (3)	C13—N1—C1—C6	166.72 (19)
S1—C7—C12—C11	−176.84 (14)	C1—C6—C5—C4	1.1 (3)
C8—C7—C12—N1	178.45 (17)	S1—C6—C5—C4	−176.76 (16)

S1—C7—C12—N1	2.4 (2)	C1—C2—C3—C4	0.2 (3)
C1—N1—C12—C11	−137.67 (19)	C7—C12—C11—C10	2.6 (3)
C13—N1—C12—C11	13.2 (3)	N1—C12—C11—C10	−176.63 (19)
C1—N1—C12—C7	43.1 (2)	C2—C3—C4—C5	0.1 (3)
C13—N1—C12—C7	−166.04 (18)	C6—C5—C4—C3	−0.8 (3)
C3—C2—C1—C6	0.1 (3)	C12—C7—C8—C9	−1.6 (3)
C3—C2—C1—N1	−179.31 (18)	S1—C7—C8—C9	174.34 (16)
C5—C6—C1—C2	−0.8 (3)	C7—C8—C9—C10	2.3 (3)
S1—C6—C1—C2	177.16 (14)	C8—C9—C10—C11	−0.5 (3)
C5—C6—C1—N1	178.68 (18)	C12—C11—C10—C9	−2.0 (3)