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10-Ethyl-3-(5-methyl-1,3,4-oxadiazol-2-yl)-10*H*-phenothiazine

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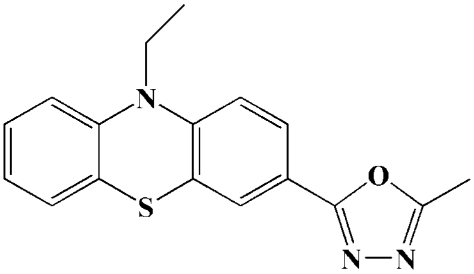
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.107; data-to-parameter ratio = 12.7.

In the title compound, $C_{17}H_{15}N_3OS$, the phenothiazine ring system is slightly bent, with a dihedral angle of 13.68 (7)° between the benzene rings. The dihedral angle between the oxadiazole ring and the adjacent benzene ring is 7.72 (7)°. In the crystal, a π–π interaction with a centroid–centroid distance of 3.752 (2) Å is observed between the benzene rings of neighbouring molecules.

Related literature

For general background to phenothiazine derivatives, see: Kim *et al.* (2011); Hagfeldt *et al.* (2010). For related structures, see: Chu & Van der Helm (1975); Hdii *et al.* (1998); Li, Hu *et al.* (2009); Li, Lv *et al.* (2009); Yu *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{15}N_3OS$	γ = 63.696 (3)°
M_r = 309.38	V = 731.92 (7) Å^3
Triclinic, $P\overline{1}$	Z = 2
a = 7.6752 (4) Å	Mo $K\alpha$ radiation
b = 8.2913 (4) Å	μ = 0.23 mm^{-1}
c = 12.9469 (8) Å	T = 293 K
α = 84.870 (4)°	0.15 × 0.15 × 0.10 mm
β = 82.569 (4)°	

Data collection

Bruker SMART APEX diffractometer	5348 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	2554 independent reflections
T_{min} = 0.968, T_{max} = 0.979	2217 reflections with $I > 2\sigma(I)$
	R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039	201 parameters
$wR(F^2)$ = 0.107	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
2554 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg & Putz, 2004) and *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5065).

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supplementary materials

Acta Cryst. (2012). E**68**, o649 [doi:10.1107/S160053681200462X]

10-Ethyl-3-(5-methyl-1,3,4-oxadiazol-2-yl)-10*H*-phenothiazine

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Comment

The derivatives of phenothiazine are a series important chemical intermediates in design of the dye-sensitized solar cells (DSSCs) (Kim *et al.*, 2011; Hagfeldt *et al.*, 2010). As part of our interest in these materials, here we report the crystal structure of the title compound $C_{17}H_{15}N_3OS$.

The title molecule is in a nonlplanar butterfly conformation with a dihedral angle of 13.68 (7)° between two benzene rings (Fig. 1). The crystal packing exhibits a π–π interaction with a centroid-centroid distance of 3.752 (2) Å between the benzene rings from the neighbouring molecules.

Experimental

A solution of 5-[3-(10-ethyl)phenothiazyl]-tetrazole (500 mg, 1.69 mmol) in 10 ml acetic anhydride was heated to reflux and stirred for 1 h. The excess acetic anhydride was evaporated and the residue solid was extracted three times with dichloromethane. Then the organic layer was washed with water and dried with anhydrous sodium sulfate. After removal of the solvent, the crude product was purified by chromatography on a silica gel column using dichloromethane-ethyl acetate (*v*/*v* = 10:1) as eluent and isolated as a yellow powder. Yield: 472 mg (90%). The yellow single crystals suitable for X-ray diffraction were obtained after several days by slow evaporation of a mixture solution of dichloromethane and petroleum ether.

Refinement

H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and treated as riding atoms, with $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$.

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2004) and *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

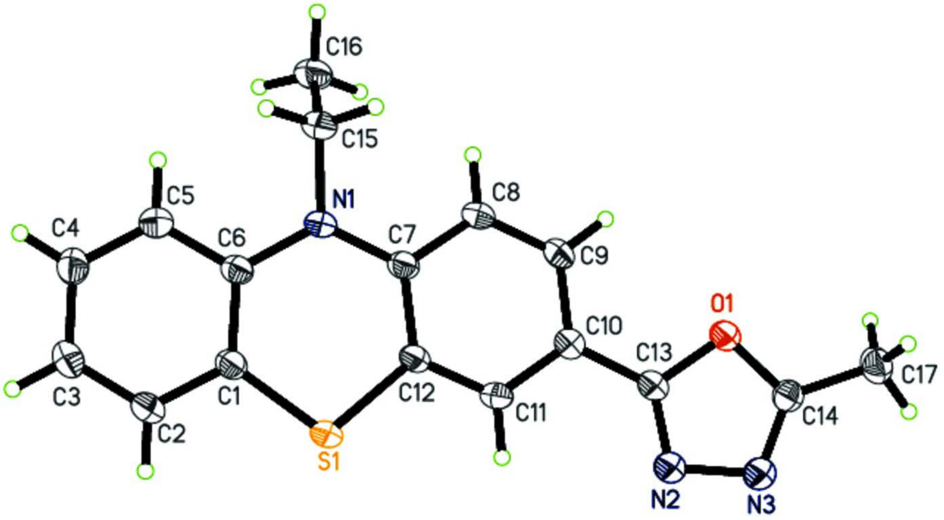

Figure 1

The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

10-Ethyl-3-(5-methyl-1,3,4-oxadiazol-2-yl)-10*H*-phenothiazine

Crystal data

$C_{17}H_{15}N_3OS$
$M_r = 309.38$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.6752$ (4) Å
$b = 8.2913$ (4) Å
$c = 12.9469$ (8) Å
$\alpha = 84.870$ (4)°
$\beta = 82.569$ (4)°
$\gamma = 63.696$ (3)°
$V = 731.92$ (7) Å^3

$Z = 2$
$F(000) = 324$
$D_x = 1.404$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2708 reflections
$\theta = 3.0$–31.6°
$\mu = 0.23$ mm^{-1}
$T = 293$ K
Block, yellow
0.15 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{\min} = 0.968$, $T_{\max} = 0.979$

5348 measured reflections
2554 independent reflections
2217 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.020$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.7°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.107$
$S = 1.05$
2554 reflections
201 parameters

0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.2148P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.37243 (8)	0.17365 (6)	0.11098 (4)	0.05078 (19)
O1	0.39396 (19)	0.69128 (15)	−0.27750 (9)	0.0430 (3)
C12	0.2887 (2)	0.3831 (2)	0.04285 (13)	0.0353 (4)
N1	0.0965 (2)	0.54971 (18)	0.19818 (11)	0.0410 (4)
C10	0.3234 (3)	0.5423 (2)	−0.11819 (13)	0.0388 (4)
C6	0.0893 (3)	0.3994 (2)	0.25572 (13)	0.0376 (4)
C7	0.1642 (2)	0.5454 (2)	0.09210 (13)	0.0371 (4)
C1	0.2039 (3)	0.2227 (2)	0.22245 (13)	0.0377 (4)
N2	0.5266 (2)	0.3956 (2)	−0.27726 (12)	0.0460 (4)
C13	0.4168 (3)	0.5350 (2)	−0.22376 (13)	0.0388 (4)
C11	0.3653 (3)	0.3829 (2)	−0.05932 (13)	0.0382 (4)
H11	0.4470	0.2736	−0.0898	0.046*
C9	0.1961 (3)	0.7029 (2)	−0.07199 (14)	0.0452 (5)
H9	0.1633	0.8110	−0.1103	0.054*
C5	−0.0285 (3)	0.4198 (2)	0.34965 (14)	0.0450 (4)
H5	−0.1070	0.5351	0.3734	0.054*
C15	0.0098 (3)	0.7238 (2)	0.24919 (15)	0.0454 (5)
H15A	0.0783	0.7936	0.2189	0.054*
H15B	0.0307	0.7016	0.3224	0.054*
N3	0.5841 (2)	0.4600 (2)	−0.37384 (12)	0.0477 (4)
C8	0.1174 (3)	0.7040 (2)	0.03038 (14)	0.0453 (5)
H8	0.0307	0.8133	0.0592	0.054*
C2	0.1976 (3)	0.0770 (2)	0.28143 (15)	0.0462 (5)
H2	0.2731	−0.0388	0.2577	0.055*
C14	0.5034 (3)	0.6320 (2)	−0.36985 (13)	0.0424 (4)
C4	−0.0320 (3)	0.2730 (3)	0.40882 (15)	0.0498 (5)
H4	−0.1109	0.2908	0.4716	0.060*
C16	−0.2063 (3)	0.8351 (3)	0.24062 (18)	0.0573 (6)
H16A	−0.2300	0.8537	0.1685	0.086*
H16B	−0.2488	0.9493	0.2713	0.086*
H16C	−0.2773	0.7728	0.2766	0.086*
C3	0.0812 (3)	0.1007 (3)	0.37469 (16)	0.0521 (5)
H3	0.0790	0.0020	0.4140	0.063*

C17	0.5164 (4)	0.7672 (3)	−0.44929 (15)	0.0558 (5)
H17A	0.5952	0.8184	−0.4272	0.084*
H17B	0.3878	0.8606	−0.4578	0.084*
H17C	0.5748	0.7102	−0.5145	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0589 (3)	0.0257 (3)	0.0468 (3)	−0.0033 (2)	0.0067 (2)	0.00176 (18)
O1	0.0540 (8)	0.0336 (6)	0.0387 (7)	−0.0177 (6)	−0.0011 (6)	0.0001 (5)
C12	0.0363 (9)	0.0266 (8)	0.0403 (9)	−0.0112 (7)	−0.0049 (7)	0.0004 (7)
N1	0.0469 (9)	0.0259 (7)	0.0425 (8)	−0.0103 (6)	0.0023 (7)	−0.0035 (6)
C10	0.0405 (10)	0.0356 (9)	0.0403 (9)	−0.0169 (8)	−0.0050 (8)	0.0008 (7)
C6	0.0380 (9)	0.0298 (9)	0.0413 (9)	−0.0111 (7)	−0.0060 (7)	−0.0005 (7)
C7	0.0352 (9)	0.0287 (8)	0.0438 (9)	−0.0110 (7)	−0.0022 (7)	−0.0016 (7)
C1	0.0412 (10)	0.0304 (9)	0.0393 (9)	−0.0134 (7)	−0.0066 (7)	0.0008 (7)
N2	0.0514 (10)	0.0370 (8)	0.0437 (8)	−0.0154 (7)	−0.0002 (7)	0.0004 (7)
C13	0.0407 (10)	0.0325 (9)	0.0427 (9)	−0.0156 (8)	−0.0067 (8)	0.0023 (7)
C11	0.0393 (10)	0.0281 (8)	0.0430 (9)	−0.0103 (7)	−0.0039 (8)	−0.0048 (7)
C9	0.0492 (11)	0.0290 (9)	0.0492 (10)	−0.0119 (8)	−0.0024 (9)	0.0070 (8)
C5	0.0437 (10)	0.0377 (10)	0.0460 (10)	−0.0118 (8)	0.0002 (8)	−0.0035 (8)
C15	0.0516 (11)	0.0325 (9)	0.0502 (10)	−0.0170 (8)	−0.0002 (9)	−0.0068 (8)
N3	0.0526 (10)	0.0434 (9)	0.0423 (8)	−0.0181 (8)	0.0011 (7)	−0.0023 (7)
C8	0.0472 (11)	0.0268 (9)	0.0505 (11)	−0.0078 (8)	0.0020 (9)	−0.0011 (7)
C2	0.0545 (12)	0.0309 (9)	0.0496 (10)	−0.0158 (8)	−0.0053 (9)	0.0009 (8)
C14	0.0471 (11)	0.0425 (10)	0.0374 (9)	−0.0197 (8)	−0.0031 (8)	−0.0014 (7)
C4	0.0507 (12)	0.0497 (11)	0.0452 (10)	−0.0212 (10)	0.0014 (9)	0.0039 (9)
C16	0.0530 (12)	0.0355 (10)	0.0696 (13)	−0.0089 (9)	0.0043 (10)	−0.0056 (9)
C3	0.0612 (13)	0.0424 (10)	0.0523 (11)	−0.0249 (10)	−0.0033 (10)	0.0093 (9)
C17	0.0736 (15)	0.0516 (12)	0.0451 (11)	−0.0321 (11)	−0.0024 (10)	0.0049 (9)

Geometric parameters (Å, º)

S1—C1	1.7541 (17)	C9—H9	0.9300
S1—C12	1.7583 (17)	C5—C4	1.388 (3)
O1—C14	1.362 (2)	C5—H5	0.9300
O1—C13	1.365 (2)	C15—C16	1.512 (3)
C12—C11	1.376 (2)	C15—H15A	0.9700
C12—C7	1.409 (2)	C15—H15B	0.9700
N1—C7	1.402 (2)	N3—C14	1.282 (2)
N1—C6	1.413 (2)	C8—H8	0.9300
N1—C15	1.472 (2)	C2—C3	1.379 (3)
C10—C9	1.387 (2)	C2—H2	0.9300
C10—C11	1.392 (2)	C14—C17	1.480 (2)
C10—C13	1.452 (2)	C4—C3	1.380 (3)
C6—C5	1.394 (2)	C4—H4	0.9300
C6—C1	1.407 (2)	C16—H16A	0.9600
C7—C8	1.402 (2)	C16—H16B	0.9600
C1—C2	1.387 (2)	C16—H16C	0.9600
N2—C13	1.287 (2)	C3—H3	0.9300

N2—N3	1.411 (2)	C17—H17A	0.9600
C11—H11	0.9300	C17—H17B	0.9600
C9—C8	1.383 (2)	C17—H17C	0.9600

C1—S1—C12	101.74 (8)	N1—C15—H15A	108.5
C14—O1—C13	102.81 (13)	C16—C15—H15A	108.5
C11—C12—C7	121.19 (15)	N1—C15—H15B	108.5
C11—C12—S1	116.40 (12)	C16—C15—H15B	108.5
C7—C12—S1	122.15 (13)	H15A—C15—H15B	107.5
C7—N1—C6	123.14 (14)	C14—N3—N2	106.14 (14)
C7—N1—C15	118.30 (14)	C9—C8—C7	122.01 (16)
C6—N1—C15	118.23 (14)	C9—C8—H8	119.0
C9—C10—C11	118.04 (16)	C7—C8—H8	119.0
C9—C10—C13	122.68 (16)	C3—C2—C1	121.15 (17)
C11—C10—C13	119.26 (15)	C3—C2—H2	119.4
C5—C6—C1	117.04 (16)	C1—C2—H2	119.4
C5—C6—N1	121.22 (15)	N3—C14—O1	112.57 (15)
C1—C6—N1	121.73 (15)	N3—C14—C17	129.09 (17)
C8—C7—N1	121.38 (15)	O1—C14—C17	118.33 (16)
C8—C7—C12	116.46 (15)	C3—C4—C5	120.19 (17)
N1—C7—C12	122.11 (15)	C3—C4—H4	119.9
C2—C1—C6	120.68 (16)	C5—C4—H4	119.9
C2—C1—S1	116.70 (13)	C15—C16—H16A	109.5
C6—C1—S1	122.48 (13)	C15—C16—H16B	109.5
C13—N2—N3	106.45 (14)	H16A—C16—H16B	109.5
N2—C13—O1	112.02 (15)	C15—C16—H16C	109.5
N2—C13—C10	128.50 (16)	H16A—C16—H16C	109.5
O1—C13—C10	119.47 (15)	H16B—C16—H16C	109.5
C12—C11—C10	121.51 (15)	C2—C3—C4	119.06 (17)
C12—C11—H11	119.2	C2—C3—H3	120.5
C10—C11—H11	119.2	C4—C3—H3	120.5
C8—C9—C10	120.70 (16)	C14—C17—H17A	109.5
C8—C9—H9	119.6	C14—C17—H17B	109.5
C10—C9—H9	119.6	H17A—C17—H17B	109.5
C4—C5—C6	121.87 (17)	C14—C17—H17C	109.5
C4—C5—H5	119.1	H17A—C17—H17C	109.5
C6—C5—H5	119.1	H17B—C17—H17C	109.5
N1—C15—C16	114.91 (16)		