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10-(Prop-1-yn-1-yl)-10*H*-phenothiazine

Satoru Umezono and Tsunehisa Okuno*

Department of Material Science and Chemistry, Wakayama University, Sakaedani, Wakayama 640-8510, Japan
Correspondence e-mail: okuno@center.wakayama-u.ac.jp

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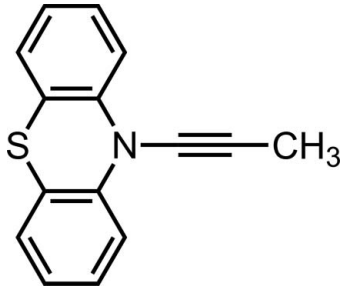
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.065; data-to-parameter ratio = 17.1.

In the title compound, $C_{15}H_{11}NS$, the asymmetric unit comprises one half-molecule; a mirror plane passes through the S atom, the ynamine fragment, the methyl C atom and one methyl H atom. The phenothiazine moiety has a butterfly conformation and the central six-membered ring has a boat conformation. The dihedral angle between the benzene rings is 149.40 (4)°. The crystal structure is stabilized by van der Waals interactions.

Related literature

For related structures of phenothiazine compounds, see: Okuno *et al.* (2006); Tabata & Okuno (2012). For the preparation of the title compound, see: Zaugg *et al.* (1958).



Experimental

Crystal data

$C_{15}H_{11}NS$
M_r = 237.31
Orthorhombic, $Cmc2_1$
a = 14.717 (6) Å
b = 10.631 (4) Å
c = 7.375 (3) Å
V = 1153.9 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 93 K
0.12 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn724+ diffractometer
Absorption correction: numerical (*NUMABS*; Rigaku, 1999)
T_{min} = 0.959, T_{max} = 0.980
4889 measured reflections
1486 independent reflections
1441 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.08
1486 reflections
87 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 670 Friedel pairs
Flack parameter: −0.01 (6)

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2425).

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supporting information

Acta Cryst. (2012). E**68**, o2790 [doi:10.1107/S1600536812036537]

10-(Prop-1-yn-1-yl)-10*H*-phenothiazine

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S1. Comment

Ynamines, where amino groups connect to acetylene groups, are known to be unstable because of their high reactivity. The title compound, $C_{15}H_{11}NS$, is the first ynamine compound which was prepared accidentally (Zaugg *et al.*, 1958). The asymmetric unit comprises one half-molecule; a mirror plane passes through the S, ynamine fragment, one C atom of methyl group and one H atom of methyl group. The bond distances and angles are comparable with others reported ynamines (Okuno *et al.*, 2006; Tabata *et al.*, 2012). The crystal structure is stabilized by van der Waals interactions. The phenothiazine moiety has a butterfly conformation, and the central six-membered ring has a boat conformation. The dihedral angle between two benzene rings is 149.40 (4)°.

S2. Experimental

The title compound was prepared according to a published procedure (Zaugg *et al.*, 1958). The single crystals with sufficient quality for X-ray analysis were obtained by slow concentration of a methanol solution.

S3. Refinement

The C-bound H atoms were placed at ideal positions and were refined as riding on their parent C atoms. U_{iso}(H) values of H2—H5 atoms were set at 1.2U_{eq}(parent atom).

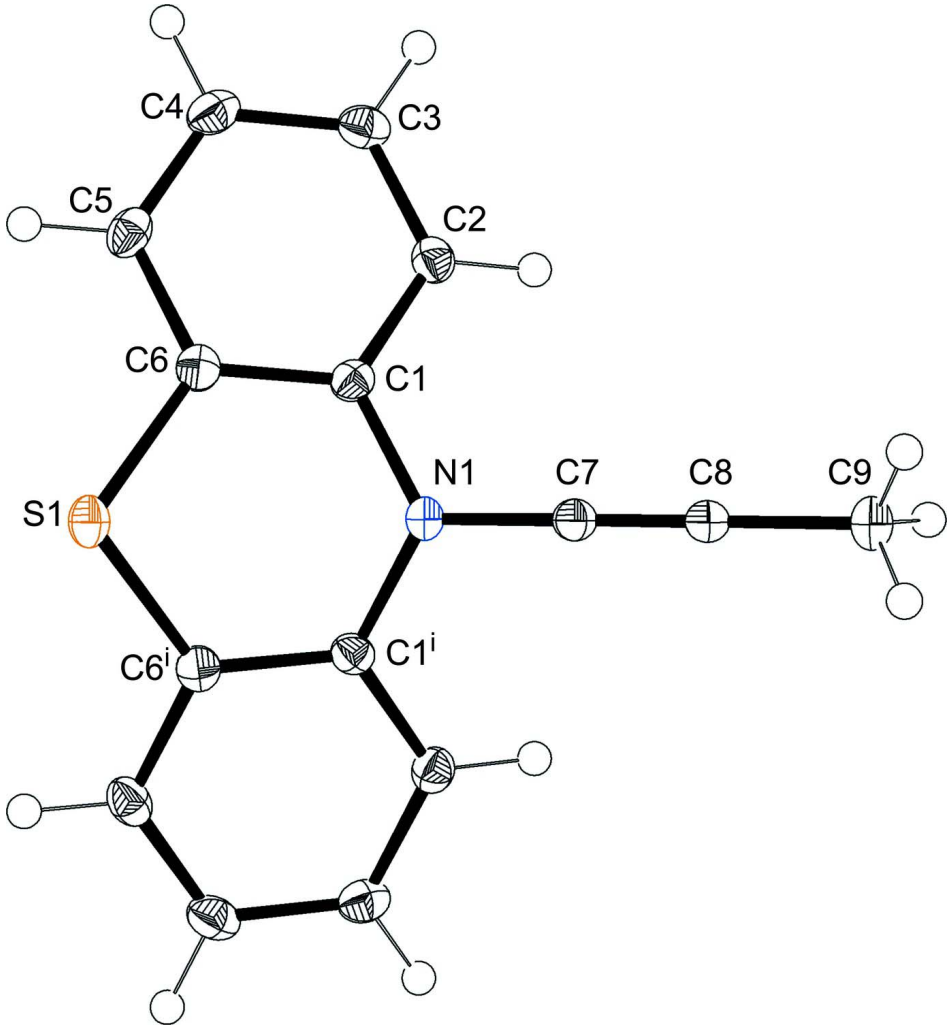

Figure 1

ORTEP view of the title compound with atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres. [Symmetry code: (i) -*x*, *y*, *z*.]

10-(Prop-1-yn-1-yl)-10*H*-phenothiazine

Crystal data

$C_{15}H_{11}NS$
$M_r = 237.31$
Orthorhombic, $Cmc2_1$
Hall symbol: C 2c -2
$a = 14.717\ (6)$ Å
$b = 10.631\ (4)$ Å
$c = 7.375\ (3)$ Å
$V = 1153.9\ (8)$ Å^3
$Z = 4$
$F(000) = 496$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 2342 reflections
$\theta = 2.4–31.2°$
$\mu = 0.25$ mm^{-1}
$T = 93$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn724+ diffractometer
Detector resolution: 28.445 pixels mm^{-1}
ω scans
Absorption correction: numerical (*NUMABS*; Rigaku, 1999)
T_{min} = 0.959, T_{max} = 0.980
4889 measured reflections
1486 independent reflections
1441 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.5°, θ_{min} = 2.4°
h = −19→16
k = −14→12
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.08
1486 reflections
87 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.514P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 670 Friedel pairs
Absolute structure parameter: −0.01 (6)

Special details

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.0000	0.03514 (4)	−0.00174 (4)	0.01839 (11)
N1	0.0000	0.28986 (13)	0.1771 (2)	0.0165 (3)
C1	0.08381 (8)	0.22366 (10)	0.19225 (16)	0.0153 (2)
C2	0.15834 (8)	0.27842 (11)	0.27878 (17)	0.0176 (2)
H2	0.1524	0.3585	0.3346	0.021*
C3	0.24154 (8)	0.21576 (12)	0.28336 (18)	0.0201 (3)
H3	0.2926	0.2542	0.3393	0.024*
C4	0.24978 (8)	0.09669 (12)	0.20594 (18)	0.0212 (3)
H4	0.3067	0.0545	0.2075	0.025*
C5	0.17460 (9)	0.03964 (11)	0.12637 (17)	0.0189 (3)
H5	0.1797	−0.0430	0.0783	0.023*
C6	0.09172 (8)	0.10329 (11)	0.11690 (15)	0.0163 (2)
C7	0.0000	0.41727 (16)	0.1900 (2)	0.0164 (3)
C8	0.0000	0.52987 (15)	0.1916 (3)	0.0171 (3)
C9	0.0000	0.66718 (16)	0.1911 (3)	0.0218 (4)
H9A	0.0000	0.7026	0.3042	0.045 (8)*
H9B	−0.0435	0.7017	0.1183	0.050 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0226 (2)	0.01654 (18)	0.01607 (19)	0.000	0.000	−0.00403 (15)
N1	0.0160 (7)	0.0124 (6)	0.0212 (7)	0.000	0.000	−0.0023 (5)
C1	0.0150 (5)	0.0154 (5)	0.0155 (5)	0.0004 (4)	0.0028 (4)	0.0021 (4)
C2	0.0188 (6)	0.0159 (5)	0.0180 (5)	−0.0016 (5)	0.0009 (5)	0.0001 (4)
C3	0.0165 (6)	0.0225 (6)	0.0212 (6)	−0.0021 (5)	0.0001 (5)	0.0039 (4)
C4	0.0178 (5)	0.0209 (6)	0.0249 (6)	0.0043 (5)	0.0040 (5)	0.0060 (5)
C5	0.0218 (6)	0.0159 (5)	0.0189 (6)	0.0030 (4)	0.0061 (5)	0.0021 (4)
C6	0.0183 (5)	0.0160 (5)	0.0144 (5)	−0.0007 (4)	0.0027 (4)	0.0011 (4)
C7	0.0146 (7)	0.0172 (8)	0.0175 (7)	0.000	0.000	−0.0018 (6)
C8	0.0143 (7)	0.0165 (8)	0.0206 (8)	0.000	0.000	−0.0014 (6)
C9	0.0232 (9)	0.0142 (7)	0.0278 (9)	0.000	0.000	−0.0011 (7)

Geometric parameters (Å, º)

S1—C6^{i}	1.7642 (13)	C3—H3	0.9500
S1—C6	1.7643 (13)	C4—C5	1.3914 (19)
N1—C7	1.358 (2)	C4—H4	0.9500
N1—C1	1.4246 (14)	C5—C6	1.3966 (17)
N1—C1^{i}	1.4246 (14)	C5—H5	0.9500
C1—C2	1.3961 (17)	C7—C8	1.197 (3)
C1—C6	1.3999 (17)	C8—C9	1.460 (2)
C2—C3	1.3944 (17)	C9—H9A	0.9152
C2—H2	0.9500	C9—H9B	0.9126
C3—C4	1.3940 (18)		
C6^{i}—S1—C6	99.84 (8)	C5—C4—H4	120.0
C7—N1—C1	119.16 (7)	C3—C4—H4	120.0
C7—N1—C1^{i}	119.16 (7)	C4—C5—C6	120.29 (11)
C1—N1—C1^{i}	119.97 (14)	C4—C5—H5	119.9
C2—C1—C6	119.82 (11)	C6—C5—H5	119.9
C2—C1—N1	120.67 (11)	C5—C6—C1	119.71 (11)
C6—C1—N1	119.50 (11)	C5—C6—S1	119.61 (9)
C3—C2—C1	120.12 (11)	C1—C6—S1	120.57 (9)
C3—C2—H2	119.9	C8—C7—N1	176.53 (19)
C1—C2—H2	119.9	C7—C8—C9	179.3 (2)
C4—C3—C2	120.02 (11)	C8—C9—H9A	114.2
C4—C3—H3	120.0	C8—C9—H9B	113.8
C2—C3—H3	120.0	H9A—C9—H9B	111.8
C5—C4—C3	119.96 (11)		
C7—N1—C1—C2	21.9 (2)	C4—C5—C6—S1	174.51 (9)
C1^{i}—N1—C1—C2	−143.06 (11)	C2—C1—C6—C5	−1.00 (17)
C7—N1—C1—C6	−157.02 (14)	N1—C1—C6—C5	177.97 (12)
C1^{i}—N1—C1—C6	38.0 (2)	C2—C1—C6—S1	−177.19 (9)
C6—C1—C2—C3	2.76 (17)	N1—C1—C6—S1	1.78 (15)

N1—C1—C2—C3	−176.20 (12)	C6^{i}—S1—C6—C5	152.23 (7)
C1—C2—C3—C4	−1.81 (18)	C6^{i}—S1—C6—C1	−31.58 (13)
C2—C3—C4—C5	−0.91 (18)	C1—N1—C7—C8	97.44 (13)
C3—C4—C5—C6	2.68 (18)	C1^{i}—N1—C7—C8	−97.44 (13)
C4—C5—C6—C1	−1.72 (18)	N1—C7—C8—C9	0.00 (5)

Symmetry code: (i) −*x*, *y*, *z*.