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3,3,6-Tribromo-1-methyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

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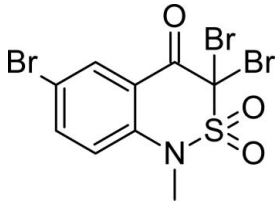
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.035; wR factor = 0.071; data-to-parameter ratio = 18.9.

In the title compound, $C_9H_6Br_3NO_3S$, a halogenated benzothiazine derivative, the thiazine ring adopts a sofa conformation. The crystal studied was a racemic twin with a contribution of 72 (1)% of the major domain.

Related literature

For the synthesis and related structures, see: Shafiq *et al.* (2009*a*,*b*).



Experimental

Crystal data

$C_9H_6Br_3NO_3S$
M_r = 447.94
Orthorhombic, $Pna2_1$
a = 14.922 (1) Å
b = 12.1310 (8) Å
c = 7.0811 (4) Å
V = 1281.81 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 9.60 mm^{-1}
T = 296 K
0.28 × 0.21 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.096, T_{max} = 0.144
7900 measured reflections
2941 independent reflections
2221 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.071
S = 0.99
2941 reflections
156 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Absolute structure: Flack (1983), 1242 Friedel pairs
Flack parameter: 0.00 (3)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5404).

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supplementary materials

Acta Cryst. (2010). E**66**, o3109 [**doi:10.1107/S1600536810045125**]

3,3,6-Tribromo-1-methyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

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Comment

The title compound, (I), is structurally related to the already reported crystal structures of 3,3-dichloro-1-ethyl-1*H*-2,1-benzothiazin-4 (3*H*)-one 2,2-dioxide, (II), (Shafiq *et al.*, 2009*a*) and 6-bromo-3,3-dichloro-1-methyl-1*H*-2,1-benzothiazin-4 (3*H*)-one 2,2-dioxide, (III), (Shafiq *et al.*, 2009*b*).

Like (II) and (III) the thiazine (C1/C6/C7/C8/S1/N1) ring in the crystal structure adopted a sofa form.

Experimental

The title compound was prepared following the already reported procedure (Shafiq *et al.*, 2009*b*).

Refinement

All H-atoms were positioned with idealized geometry with C—H = 0.93 Å and C—H = 0.96 Å and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C) or U_{iso}(H) = 1.5 U_{eq}(C_{methyl}). The crystal turned out to be a racemic twin with a contribution of 72 (1)% of the major domain.

Figures

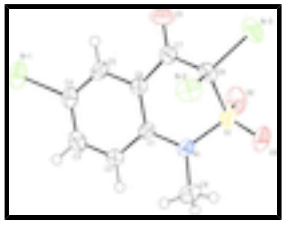

Fig. 1. Perspective view of the title compound with displacement ellipsoids drawn at the 50% probability level.

3,3,6-Tribromo-1-methyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

Crystal data

$C_9H_6Br_3NO_3S$	$F(000)$ = 848
M_r = 447.94	D_x = 2.321 Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 2326 reflections
a = 14.922 (1) Å	θ = 3.3–24.8°
b = 12.1310 (8) Å	μ = 9.60 mm^{-1}
c = 7.0811 (4) Å	T = 296 K
V = 1281.81 (14) Å^3	Needle, light brown

$Z = 4$	0.28 × 0.21 × 0.12 mm

Data collection

Bruker Kappa APEXII CCD diffractometer	2941 independent reflections
Radiation source: fine-focus sealed tube	2221 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.033$
φ and ω scans	$\theta_{max} = 28.2°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)	$h = -15\rightarrow19$
$T_{min} = 0.096$, $T_{max} = 0.144$	$k = -16\rightarrow10$
7900 measured reflections	$l = -8\rightarrow9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0309P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
2941 reflections	$\Delta\rho_{max} = 0.54$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.55$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 1242 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.00 (3)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
Br1	0.56408 (4)	0.70021 (5)	0.24956 (8)	0.05225 (17)
S1	0.21026 (9)	0.32623 (11)	0.3603 (2)	0.0368 (3)
O1	0.2102 (3)	0.6272 (3)	0.2328 (8)	0.0631 (13)
N1	0.3103 (2)	0.3077 (3)	0.2744 (6)	0.0341 (9)

C1	0.3690 (3)	0.3998 (3)	0.2654 (7)	0.0269 (10)
Br2	0.17819 (4)	0.39549 (5)	−0.05254 (9)	0.05073 (17)
O2	0.2124 (3)	0.3737 (3)	0.5431 (5)	0.0505 (11)
C2	0.4609 (3)	0.3843 (3)	0.2690 (8)	0.0352 (11)
H2	0.4841	0.3132	0.2754	0.042*
Br3	0.05403 (4)	0.47720 (5)	0.28558 (9)	0.05946 (19)
O3	0.1596 (3)	0.2288 (3)	0.3279 (6)	0.0569 (11)
C3	0.5178 (3)	0.4721 (4)	0.2632 (8)	0.0385 (11)
H3	0.5794	0.4605	0.2657	0.046*
C4	0.4844 (3)	0.5789 (4)	0.2536 (7)	0.0346 (11)
C5	0.3947 (3)	0.5960 (3)	0.2469 (7)	0.0333 (11)
H5	0.3727	0.6676	0.2400	0.040*
C6	0.3348 (3)	0.5074 (3)	0.2503 (7)	0.0279 (10)
C7	0.2389 (3)	0.5345 (4)	0.2336 (6)	0.0309 (10)
C8	0.1729 (3)	0.4378 (4)	0.2090 (6)	0.0325 (11)
C9	0.3419 (4)	0.1959 (4)	0.2311 (10)	0.0553 (16)
H9C	0.3840	0.1989	0.1287	0.083*
H9B	0.2919	0.1506	0.1958	0.083*
H9A	0.3705	0.1652	0.3405	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0503 (4)	0.0549 (3)	0.0516 (3)	−0.0277 (3)	0.0009 (3)	−0.0028 (3)
S1	0.0342 (8)	0.0388 (7)	0.0376 (6)	−0.0059 (6)	0.0022 (6)	0.0082 (6)
O1	0.039 (2)	0.0306 (19)	0.120 (4)	0.0096 (17)	−0.002 (3)	−0.006 (3)
N1	0.029 (2)	0.0234 (19)	0.050 (3)	−0.0030 (16)	0.001 (2)	0.002 (2)
C1	0.031 (3)	0.022 (2)	0.027 (2)	0.0008 (18)	0.004 (2)	0.004 (2)
Br2	0.0663 (4)	0.0518 (3)	0.0341 (2)	0.0025 (3)	−0.0111 (3)	−0.0066 (3)
O2	0.053 (3)	0.069 (3)	0.029 (2)	−0.009 (2)	0.0066 (18)	0.0041 (18)
C2	0.029 (3)	0.029 (2)	0.048 (3)	0.0052 (19)	0.005 (3)	0.007 (3)
Br3	0.0275 (3)	0.0677 (4)	0.0831 (5)	0.0025 (3)	0.0073 (3)	−0.0042 (4)
O3	0.047 (2)	0.043 (2)	0.081 (3)	−0.0203 (19)	0.005 (2)	0.009 (2)
C3	0.025 (3)	0.049 (3)	0.041 (3)	0.000 (2)	0.002 (3)	0.002 (3)
C4	0.031 (3)	0.039 (3)	0.033 (2)	−0.012 (2)	0.000 (2)	0.000 (3)
C5	0.041 (3)	0.022 (2)	0.037 (3)	−0.0056 (19)	0.001 (2)	−0.002 (2)
C6	0.029 (3)	0.028 (2)	0.027 (2)	−0.0008 (18)	0.000 (2)	−0.001 (2)
C7	0.030 (3)	0.028 (2)	0.034 (2)	−0.001 (2)	0.000 (2)	−0.001 (2)
C8	0.025 (3)	0.039 (3)	0.033 (3)	0.000 (2)	0.001 (2)	−0.007 (2)
C9	0.063 (4)	0.027 (3)	0.076 (4)	−0.002 (3)	0.014 (4)	−0.006 (3)

Geometric parameters (Å, °)

Br1—C4	1.892 (4)	C2—H2	0.9300
S1—O2	1.417 (4)	Br3—C8	1.916 (5)
S1—O3	1.422 (4)	C3—C4	1.390 (6)
S1—N1	1.627 (4)	C3—H3	0.9300
S1—C8	1.814 (5)	C4—C5	1.356 (7)
O1—C7	1.204 (5)	C5—C6	1.398 (6)

N1—C1	1.421 (5)	C5—H5	0.9300
N1—C9	1.469 (6)	C6—C7	1.473 (6)
C1—C2	1.384 (6)	C7—C8	1.542 (6)
C1—C6	1.406 (5)	C9—H9C	0.9600
Br2—C8	1.923 (4)	C9—H9B	0.9600
C2—C3	1.362 (6)	C9—H9A	0.9600
O2—S1—O3	119.9 (2)	C4—C5—C6	120.9 (4)
O2—S1—N1	112.1 (2)	C4—C5—H5	119.6
O3—S1—N1	108.2 (2)	C6—C5—H5	119.6
O2—S1—C8	104.1 (2)	C5—C6—C1	118.9 (4)
O3—S1—C8	111.2 (2)	C5—C6—C7	116.6 (4)
N1—S1—C8	99.4 (2)	C1—C6—C7	124.5 (4)
C1—N1—C9	121.2 (4)	O1—C7—C6	123.7 (4)
C1—N1—S1	118.3 (3)	O1—C7—C8	118.9 (4)
C9—N1—S1	120.0 (3)	C6—C7—C8	117.4 (4)
C2—C1—C6	119.2 (4)	C7—C8—S1	107.7 (3)
C2—C1—N1	120.3 (4)	C7—C8—Br3	111.7 (3)
C6—C1—N1	120.6 (4)	S1—C8—Br3	107.7 (2)
C3—C2—C1	120.7 (4)	C7—C8—Br2	106.6 (3)
C3—C2—H2	119.6	S1—C8—Br2	110.9 (2)
C1—C2—H2	119.6	Br3—C8—Br2	112.2 (2)
C2—C3—C4	120.5 (4)	N1—C9—H9C	109.5
C2—C3—H3	119.8	N1—C9—H9B	109.5
C4—C3—H3	119.8	H9C—C9—H9B	109.5
C5—C4—C3	119.9 (4)	N1—C9—H9A	109.5
C5—C4—Br1	120.1 (4)	H9C—C9—H9A	109.5
C3—C4—Br1	120.0 (4)	H9B—C9—H9A	109.5
O2—S1—N1—C1	−54.7 (4)	C2—C1—C6—C7	176.2 (5)
O3—S1—N1—C1	170.9 (4)	N1—C1—C6—C7	−3.0 (7)
C8—S1—N1—C1	54.8 (4)	C5—C6—C7—O1	−6.2 (8)
O2—S1—N1—C9	117.5 (5)	C1—C6—C7—O1	175.1 (5)
O3—S1—N1—C9	−16.9 (5)	C5—C6—C7—C8	171.7 (4)
C8—S1—N1—C9	−133.0 (5)	C1—C6—C7—C8	−7.1 (7)
C9—N1—C1—C2	−18.4 (7)	O1—C7—C8—S1	−142.5 (5)
S1—N1—C1—C2	153.6 (4)	C6—C7—C8—S1	39.5 (5)
C9—N1—C1—C6	160.7 (5)	O1—C7—C8—Br3	−24.5 (6)
S1—N1—C1—C6	−27.2 (6)	C6—C7—C8—Br3	157.6 (3)
C6—C1—C2—C3	1.8 (8)	O1—C7—C8—Br2	98.3 (5)
N1—C1—C2—C3	−179.1 (5)	C6—C7—C8—Br2	−79.6 (4)
C1—C2—C3—C4	0.0 (8)	O2—S1—C8—C7	57.1 (4)
C2—C3—C4—C5	−1.1 (8)	O3—S1—C8—C7	−172.5 (3)
C2—C3—C4—Br1	179.0 (4)	N1—S1—C8—C7	−58.7 (3)
C3—C4—C5—C6	0.3 (8)	O2—S1—C8—Br3	−63.5 (3)
Br1—C4—C5—C6	−179.8 (4)	O3—S1—C8—Br3	66.9 (3)
C4—C5—C6—C1	1.6 (7)	N1—S1—C8—Br3	−179.3 (2)
C4—C5—C6—C7	−177.3 (4)	O2—S1—C8—Br2	173.4 (3)
C2—C1—C6—C5	−2.6 (7)	O3—S1—C8—Br2	−56.2 (3)
N1—C1—C6—C5	178.3 (4)	N1—S1—C8—Br2	57.6 (3)

Fig. 1