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3-Methyl-6-trichloromethyl-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazole

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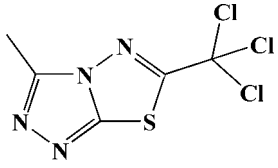
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.113; data-to-parameter ratio = 18.3.

In the crystal structure of the title compound, $C_5H_3Cl_3N_4S$, two molecules related by a centre of symmetry demonstrate extremely short intermolecular S···N contacts of 2.783 (2) Å. The crystal packing also exhibits π–π interactions indicated by a short distance of 3.340 (1) Å between the centroids of the triazole rings of neighbouring molecules.

Related literature

For the antimicrobial and anti-inflammatory activity of 1,2,4-triazole and 1,3,4-thiodiazole derivatives, see: Karabasanagouda *et al.* (2007); Mathew *et al.* (2007); For related structures, see: Du *et al.* (2008); Khan *et al.* (2009); Haugwitz *et al.* (1977).



Experimental

Crystal data

$C_5H_3Cl_3N_4S$ M_r = 257.52
Monoclinic, $P2_1/n$
a = 5.8732 (12) Å
b = 9.4164 (19) Å
c = 16.750 (3) Å
β = 91.82 (3)°
V = 925.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.17 mm^{-1}
T = 153 K
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.721, T_{max} = 0.892
9841 measured reflections
2196 independent reflections
1934 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.113
S = 1.24
2196 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Data collection: *CrystalClear* (Rigaku/MSC, 2005); 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: *SHELXTL* (Sheldrick, 2008); 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: CV5070).

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supporting information

Acta Cryst. (2011). E**67**, o1093 [doi:10.1107/S1600536811012748]

3-Methyl-6-trichloromethyl-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazole

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S1. Comment

1,2,4-Triazole and 1,3,4-thiodiazole derivatives demonstrate various activities such as antimicrobial (Karabasanagouda *et al.*, 2007) and anti-inflammatory (Mathew *et al.*, 2007) activities. Herewith we report the synthesis and crystal structure of the title compound (I), a new derivative from the aforementioned family.

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in the related structures (Du *et al.*, 2008; Khan *et al.*, 2009). The triazolothiadiazole ring system is essentially planar with an r.m.s derivation of 0.0087 (2)Å and maximum deviation of 0.0037 (2)Å for atom C2. In the crystal structure, π-π interactions (Table 1) consolidate the crystal packing, which exhibits short intermolecular S···N contacts of 2.783 (2) Å observed eralier in the related structure (Haugwitz *et al.*, 1977).

S2. Experimental

The title compound was synthesized by the reaction of 4-amino-3-methyl-4*H*-1,2,4-triazole-5-thiol (2.0 mmol) and trichloroacetic acid (2.0 mmol) in phosphoryl trichloride for 24 h. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform-ethanol (1:1).

S3. Refinement

H atoms were positioned geometrically (C—H = 0.98 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(parent).

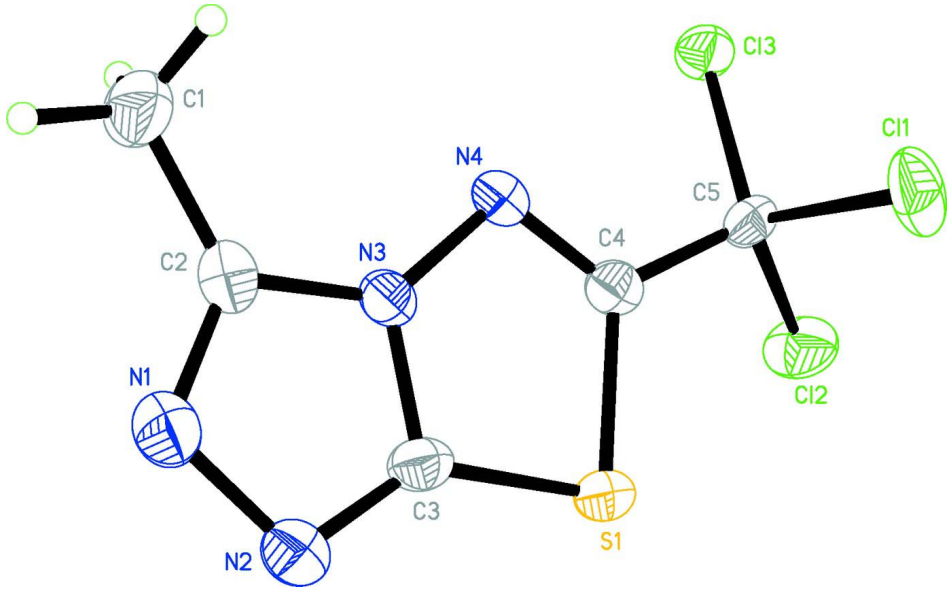

Figure 1

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 60% probability level.

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Crystal data

$C_5H_3Cl_3N_4S$
$M_r = 257.52$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.8732$ (12) Å
$b = 9.4164$ (19) Å
$c = 16.750$ (3) Å
$\beta = 91.82$ (3)°
$V = 925.9$ (3) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.847$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2918 reflections
$\theta = 3.3$–27.9°
$\mu = 1.17$ mm^{-1}
$T = 153$ K
Prism, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 7.31 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.721$, $T_{max} = 0.892$
9841 measured reflections
2196 independent reflections
1934 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.4°$
$h = -7\rightarrow7$
$k = -10\rightarrow12$
$l = -22\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.113$
$S = 1.24$
2196 reflections
120 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
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.071 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.13777 (9)	0.52705 (5)	0.13657 (3)	0.01622 (19)
Cl1	0.55694 (10)	0.67616 (5)	0.27560 (3)	0.0242 (2)
Cl2	0.15047 (9)	0.53079 (6)	0.32341 (3)	0.02410 (19)
Cl3	0.58496 (8)	0.38759 (5)	0.33267 (3)	0.01648 (18)
N1	0.3533 (3)	0.30847 (19)	−0.04370 (11)	0.0193 (4)
N2	0.1853 (3)	0.40411 (19)	−0.01768 (11)	0.0191 (4)
N3	0.4320 (3)	0.35972 (16)	0.08095 (10)	0.0134 (4)
N4	0.5132 (3)	0.37661 (17)	0.15796 (10)	0.0133 (4)
C1	0.7018 (4)	0.1903 (2)	0.01481 (13)	0.0223 (5)
H1A	0.7271	0.1594	−0.0401	0.033*
H1B	0.6776	0.1070	0.0486	0.033*
H1C	0.8353	0.2430	0.0352	0.033*
C2	0.4981 (3)	0.2833 (2)	0.01591 (12)	0.0158 (4)
C3	0.2385 (3)	0.4306 (2)	0.05699 (12)	0.0151 (4)
C4	0.3744 (3)	0.46089 (19)	0.19257 (12)	0.0135 (4)
C5	0.4165 (3)	0.5096 (2)	0.27709 (11)	0.0136 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0153 (3)	0.0166 (3)	0.0165 (3)	0.00419 (18)	−0.0027 (2)	−0.00059 (17)
Cl1	0.0359 (4)	0.0136 (3)	0.0227 (3)	−0.0086 (2)	−0.0055 (2)	0.00025 (18)
Cl2	0.0186 (3)	0.0337 (4)	0.0202 (3)	0.0075 (2)	0.0050 (2)	−0.0021 (2)
Cl3	0.0186 (3)	0.0164 (3)	0.0143 (3)	0.00304 (18)	−0.00199 (19)	0.00178 (16)
N1	0.0204 (9)	0.0206 (9)	0.0169 (9)	−0.0011 (7)	−0.0001 (7)	−0.0020 (7)
N2	0.0201 (9)	0.0214 (9)	0.0157 (9)	0.0021 (7)	−0.0023 (7)	−0.0011 (7)
N3	0.0142 (8)	0.0127 (8)	0.0133 (8)	−0.0010 (6)	−0.0022 (6)	0.0002 (6)
N4	0.0132 (8)	0.0145 (8)	0.0122 (8)	−0.0015 (6)	−0.0015 (6)	0.0002 (6)
C1	0.0240 (12)	0.0254 (11)	0.0176 (10)	0.0059 (9)	0.0015 (8)	−0.0031 (8)

C2	0.0180 (10)	0.0155 (9)	0.0142 (9)	−0.0029 (8)	0.0017 (7)	−0.0013 (7)
C3	0.0133 (10)	0.0135 (9)	0.0184 (10)	0.0013 (8)	−0.0018 (8)	0.0022 (7)
C4	0.0145 (10)	0.0121 (9)	0.0136 (9)	−0.0009 (7)	−0.0007 (7)	0.0027 (7)
C5	0.0138 (9)	0.0110 (8)	0.0162 (10)	0.0023 (7)	0.0008 (7)	0.0009 (7)

Geometric parameters (Å, º)

S1—C3	1.732 (2)	N3—N4	1.370 (2)
S1—C4	1.765 (2)	N3—C2	1.372 (3)
Cl1—C5	1.772 (2)	N4—C4	1.289 (3)
Cl2—C5	1.778 (2)	C1—C2	1.483 (3)
Cl3—C5	1.763 (2)	C1—H1A	0.9800
N1—C2	1.313 (3)	C1—H1B	0.9800
N1—N2	1.415 (2)	C1—H1C	0.9800
N2—C3	1.304 (3)	C4—C5	1.501 (3)
N3—C3	1.367 (3)		
Cg1···Cg1^{i}	3.340 (1)	Cg1···Cg2^{i}	3.682 (1)
C3—S1—C4	86.65 (9)	N1—C2—C1	126.9 (2)
C2—N1—N2	108.78 (17)	N3—C2—C1	124.63 (18)
C3—N2—N1	105.63 (17)	N2—C3—N3	111.08 (18)
C3—N3—N4	118.75 (16)	N2—C3—S1	139.59 (16)
C3—N3—C2	106.06 (17)	N3—C3—S1	109.33 (14)
N4—N3—C2	135.19 (17)	N4—C4—C5	121.67 (18)
C4—N4—N3	106.78 (16)	N4—C4—S1	118.48 (15)
C2—C1—H1A	109.5	C5—C4—S1	119.77 (15)
C2—C1—H1B	109.5	C4—C5—Cl3	111.82 (14)
H1A—C1—H1B	109.5	C4—C5—Cl1	108.66 (13)
C2—C1—H1C	109.5	Cl3—C5—Cl1	109.30 (11)
H1A—C1—H1C	109.5	C4—C5—Cl2	108.97 (14)
H1B—C1—H1C	109.5	Cl3—C5—Cl2	109.20 (10)
N1—C2—N3	108.44 (17)	Cl1—C5—Cl2	108.84 (10)
C2—N1—N2—C3	−0.4 (2)	C2—N3—C3—S1	178.46 (13)
C3—N3—N4—C4	0.9 (2)	C4—S1—C3—N2	179.8 (3)
C2—N3—N4—C4	−178.6 (2)	C4—S1—C3—N3	0.80 (14)
N2—N1—C2—N3	−0.2 (2)	N3—N4—C4—C5	−176.81 (17)
N2—N1—C2—C1	−179.41 (19)	N3—N4—C4—S1	−0.2 (2)
C3—N3—C2—N1	0.6 (2)	C3—S1—C4—N4	−0.36 (16)
N4—N3—C2—N1	−179.8 (2)	C3—S1—C4—C5	176.31 (16)
C3—N3—C2—C1	179.86 (19)	N4—C4—C5—Cl3	−25.8 (2)
N4—N3—C2—C1	−0.6 (3)	S1—C4—C5—Cl3	157.64 (11)
N1—N2—C3—N3	0.7 (2)	N4—C4—C5—Cl1	94.93 (19)
N1—N2—C3—S1	−178.24 (19)	S1—C4—C5—Cl1	−81.63 (16)
N4—N3—C3—N2	179.50 (16)	N4—C4—C5—Cl2	−146.61 (16)

C2—N3—C3—N2	−0.8 (2)	S1—C4—C5—Cl2	36.83 (18)
N4—N3—C3—S1	−1.2 (2)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.