Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3-Dimethyl-1-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl]-butan-2-one

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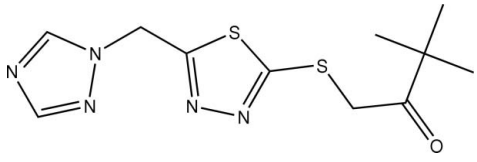
Received 7 November 2007; accepted 24 December 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.144; data-to-parameter ratio = 14.7.

In the molecule of the title compound, $C_{11}H_{15}N_5OS_2$, the thiadiazole and triazole rings are not coplanar, the dihedral angle formed by their mean planes being 59.9 (2)°. The exocyclic S atom, and the methylene, carbonyl, *tert*-butyl and one methyl carbon form an approximately planar zigzag chain, which makes a dihedral angle of 74.6 (1)° with the thiadiazole ring.

Related literature

For the structure of the related compound, 1-(2,4-dichlorophenyl)-2-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl] ethanone, see: Wei *et al.* (2007). For the synthesis of the starting material 5-(1*H*-1,2,4-triazol-1-yl)methyl)-1,3,4-thiadiazole-2(3*H*)-thione, see: Hu *et al.* (2006), Xu *et al.* (2005, 2006).



Experimental

Crystal data

$C_{11}H_{15}N_5OS_2$
M_r = 297.40
Triclinic, $P\overline{1}$
a = 8.9723 (8) Å
b = 10.1103 (8) Å
c = 10.1734 (8) Å
α = 60.728 (1)°
β = 80.340 (1)°
γ = 65.416 (1)°
V = 731.3 (1) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 293 (2) K
0.41 × 0.22 × 0.18 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.865, T_{max} = 0.938
3810 measured reflections
2529 independent reflections
2246 reflections with $I > 2\sigma(I)$
R_{int} = 0.010

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.143
S = 1.05
2529 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

This project was supported by the Natural Science Foundation of Shandong Province (grant Nos. Z2006B01 and Y2006B07).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2060).

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  doi:10.1107/S1600536807068286

supporting information

Acta Cryst. (2008). E**64**, o412 [doi:10.1107/S1600536807068286]

3,3-Dimethyl-1-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl]butan-2-one

Qing-Li Wei, Fu-Jin He, Fang Li and Sai Bi

S1. Comment

Recently, we have reported the structure of 1-(2,4-dichlorophenyl)-2-[5-(1*H*-1,2,4- triazol-1-ylmethyl)-1,3,4-thiadiazol-2-ylsulfanyl] ethanone (Wei *et al.*, 2007). As part of our ongoing investigation of biological properties of 1,2,4-triazole and 1,3,4-thiadiazole derivatives, the title compound, (I), was synthesized; its crystal structure is reported here.

The bond lengths and angles are comparable with those of the above mentioned related compound, reported by Wei *et al.* (2007). The whole molecule is non-planar with a dihedral angle of 59.9 (2)° between the thiadiazole (C1/C2/N1/N2/S1) and triazole (N3—N5/C4/C5) rings. The S2—C6—C7—C8—C10 atoms form approximately planar zigzag chain, which makes a dihedral angle of 74.6 (1)° with the thiadiazole ring.

S2. Experimental

8 mmol of 5-((1*H*-1,2,4-triazol-1-yl)methyl)-1,3,4-thiadiazole-2(3*H*)-thione (Hu *et al.*, 2006; Xu *et al.*, 2005; Xu *et al.*, 2006) was refluxed for 4 h with 8 mmol of 1-bromo-3,3-dimethylbutan-2-one in 50 ml of acetone in the presence of 8 mmol of triethylamine. The solid that precipitated was recrystallized from ethanol (1.21 g, yield 50.86%). Single crystals suitable for X-ray measurements were obtained by slow evaporation of ethylacetate solution at room temperature.

S3. Refinement

After the H atoms were located in the difference map, they were fixed geometrically in the idealized positions and allowed to ride on the parent C atoms, with C—H distances of 0.96 Å (methyl), 0.97 Å (CH2) or 0.93 Å (CH), and with U_{iso}(H) values of 1.2U_{eq}(C) and 1.5 U_{eq}(C) (for methyl H atoms).

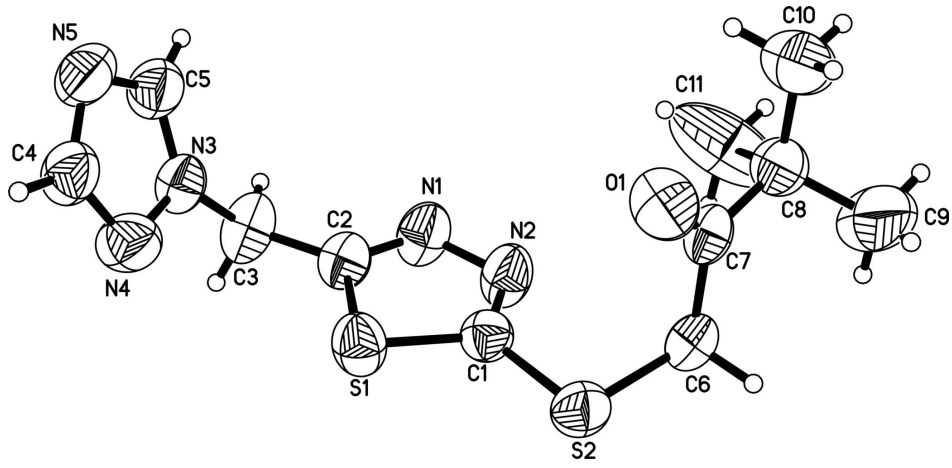

Figure 1

The structure of the title compound (I), showing 50% probability displacement ellipsoids and the atom numbering scheme.

3,3-Dimethyl-1-[5-(1*H*-1,2,4-triazol-1-ylmethyl)-1,3,4-thiadiazol-2-*ψ*lsulfanyl]butan-2-one

Crystal data

$C_{11}H_{15}N_5OS_2$
M_r = 297.40
Triclinic, $P\overline{1}$
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c = 10.1734 (8) Å
α = 60.728 (1)°
β = 80.340 (1)°
γ = 65.416 (1)°
V = 731.3 (1) Å^3
Z = 2
F(000) = 312
D_x = 1.351 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2210 reflections
θ = 2.5–25.6°
μ = 0.36 mm^{-1}
T = 293 K
Block, colourless
0.41 × 0.22 × 0.18 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.33 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.865, T_{max} = 0.938
3810 measured reflections
2529 independent reflections
2246 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −9→10
k = −9→12
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.143
S = 1.05
2529 reflections
172 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

$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2 + 0.357P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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}
N1	0.5556 (3)	0.4597 (3)	0.7067 (3)	0.0680 (7)
N2	0.6886 (3)	0.4050 (3)	0.7986 (3)	0.0670 (6)
N3	0.2627 (3)	0.8466 (3)	0.4764 (2)	0.0546 (5)
N4	0.2154 (4)	0.9710 (3)	0.5103 (3)	0.0718 (7)
N5	0.2371 (4)	1.0629 (3)	0.2618 (3)	0.0762 (7)
O1	1.0413 (3)	0.4947 (2)	0.7133 (2)	0.0746 (6)
S1	0.49906 (8)	0.68646 (9)	0.77341 (9)	0.0627 (3)
S2	0.81659 (9)	0.48127 (9)	0.96116 (8)	0.0609 (2)
C1	0.6733 (3)	0.5099 (3)	0.8425 (3)	0.0508 (6)
C2	0.4489 (3)	0.6004 (3)	0.6854 (3)	0.0548 (6)
C3	0.2921 (4)	0.6795 (4)	0.5949 (4)	0.0698 (8)
H3A	0.2954	0.6133	0.5496	0.084*
H3B	0.2013	0.6817	0.6618	0.084*
C4	0.2003 (4)	1.0979 (3)	0.3766 (3)	0.0657 (7)
H4	0.1665	1.2043	0.3630	0.079*
C5	0.2751 (4)	0.9038 (4)	0.3298 (3)	0.0695 (8)
H5	0.3066	0.8398	0.2807	0.083*
C6	0.9842 (3)	0.3090 (3)	0.9519 (3)	0.0543 (6)
H6A	1.0658	0.2611	1.0311	0.065*
H6B	0.9439	0.2261	0.9711	0.065*
C7	1.0658 (3)	0.3532 (3)	0.8017 (3)	0.0501 (6)
C8	1.1780 (4)	0.2127 (3)	0.7691 (3)	0.0629 (7)
C9	1.2994 (7)	0.0845 (7)	0.8962 (5)	0.199 (4)
H9A	1.3660	0.1311	0.9096	0.298*
H9B	1.3679	−0.0042	0.8744	0.298*
H9C	1.2430	0.0437	0.9868	0.298*
C10	1.2626 (5)	0.2749 (5)	0.6225 (4)	0.0910 (11)
H10A	1.3325	0.3208	0.6317	0.136*
H10B	1.1816	0.3581	0.5422	0.136*
H10C	1.3272	0.1853	0.6008	0.136*
C11	1.0686 (7)	0.1417 (8)	0.7445 (8)	0.154 (3)
H11A	1.1360	0.0522	0.7235	0.231*

H11B	0.9921	0.2261	0.6609	0.231*
H11C	1.0097	0.1028	0.8338	0.231*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0740 (15)	0.0541 (13)	0.0763 (16)	−0.0174 (12)	−0.0189 (12)	−0.0300 (12)
N2	0.0678 (14)	0.0555 (13)	0.0776 (16)	−0.0112 (11)	−0.0206 (12)	−0.0341 (12)
N3	0.0551 (12)	0.0475 (11)	0.0590 (13)	−0.0186 (9)	−0.0090 (10)	−0.0209 (10)
N4	0.0947 (18)	0.0605 (14)	0.0618 (14)	−0.0268 (13)	0.0001 (13)	−0.0315 (12)
N5	0.0926 (19)	0.0611 (15)	0.0560 (14)	−0.0159 (13)	−0.0140 (13)	−0.0200 (12)
O1	0.0788 (13)	0.0500 (11)	0.0763 (13)	−0.0282 (10)	0.0123 (11)	−0.0161 (10)
S1	0.0575 (4)	0.0554 (4)	0.0780 (5)	−0.0157 (3)	−0.0043 (3)	−0.0359 (4)
S2	0.0656 (4)	0.0655 (5)	0.0606 (4)	−0.0234 (3)	−0.0027 (3)	−0.0359 (4)
C1	0.0536 (13)	0.0487 (13)	0.0494 (13)	−0.0200 (11)	0.0021 (11)	−0.0222 (11)
C2	0.0572 (14)	0.0470 (14)	0.0568 (14)	−0.0238 (12)	−0.0020 (11)	−0.0175 (11)
C3	0.0673 (17)	0.0545 (15)	0.0793 (19)	−0.0301 (14)	−0.0174 (15)	−0.0138 (14)
C4	0.0773 (18)	0.0486 (15)	0.0634 (17)	−0.0160 (13)	−0.0089 (14)	−0.0239 (13)
C5	0.0795 (19)	0.0606 (17)	0.0645 (18)	−0.0124 (14)	−0.0127 (14)	−0.0336 (15)
C6	0.0585 (14)	0.0493 (13)	0.0493 (14)	−0.0200 (11)	−0.0091 (11)	−0.0161 (11)
C7	0.0472 (12)	0.0462 (13)	0.0537 (14)	−0.0198 (10)	−0.0097 (10)	−0.0162 (11)
C8	0.0658 (16)	0.0536 (15)	0.0588 (16)	−0.0192 (13)	0.0017 (13)	−0.0216 (13)
C9	0.181 (6)	0.165 (5)	0.085 (3)	0.104 (5)	−0.051 (3)	−0.063 (3)
C10	0.095 (3)	0.095 (3)	0.082 (2)	−0.037 (2)	0.021 (2)	−0.046 (2)
C11	0.170 (5)	0.186 (5)	0.237 (7)	−0.129 (5)	0.117 (5)	−0.176 (6)

Geometric parameters (Å, º)

N1—C2	1.274 (3)	C5—H5	0.9300
N1—N2	1.387 (3)	C6—C7	1.516 (4)
N2—C1	1.290 (3)	C6—H6A	0.9700
N3—C5	1.313 (4)	C6—H6B	0.9700
N3—N4	1.347 (3)	C7—C8	1.517 (4)
N3—C3	1.456 (3)	C8—C9	1.482 (5)
N4—C4	1.314 (4)	C8—C10	1.515 (4)
N5—C5	1.310 (4)	C8—C11	1.544 (5)
N5—C4	1.332 (4)	C9—H9A	0.9600
O1—C7	1.205 (3)	C9—H9B	0.9600
S1—C1	1.715 (3)	C9—H9C	0.9600
S1—C2	1.728 (3)	C10—H10A	0.9600
S2—C1	1.751 (3)	C10—H10B	0.9600
S2—C6	1.799 (3)	C10—H10C	0.9600
C2—C3	1.498 (4)	C11—H11A	0.9600
C3—H3A	0.9700	C11—H11B	0.9600
C3—H3B	0.9700	C11—H11C	0.9600
C4—H4	0.9300		
C2—N1—N2	113.0 (2)	C7—C6—H6B	108.8

C1—N2—N1	111.5 (2)	S2—C6—H6B	108.8
C5—N3—N4	109.3 (2)	H6A—C6—H6B	107.7
C5—N3—C3	130.1 (3)	O1—C7—C6	120.5 (2)
N4—N3—C3	120.6 (2)	O1—C7—C8	122.2 (2)
C4—N4—N3	102.1 (2)	C6—C7—C8	117.3 (2)
C5—N5—C4	102.3 (2)	C9—C8—C10	111.0 (4)
C1—S1—C2	86.46 (12)	C9—C8—C7	110.6 (3)
C1—S2—C6	98.80 (12)	C10—C8—C7	110.9 (2)
N2—C1—S1	114.8 (2)	C9—C8—C11	110.8 (5)
N2—C1—S2	124.2 (2)	C10—C8—C11	106.0 (3)
S1—C1—S2	121.01 (15)	C7—C8—C11	107.3 (3)
N1—C2—C3	121.9 (3)	C8—C9—H9A	109.5
N1—C2—S1	114.2 (2)	C8—C9—H9B	109.5
C3—C2—S1	123.9 (2)	H9A—C9—H9B	109.5
N3—C3—C2	112.9 (2)	C8—C9—H9C	109.5
N3—C3—H3A	109.0	H9A—C9—H9C	109.5
C2—C3—H3A	109.0	H9B—C9—H9C	109.5
N3—C3—H3B	109.0	C8—C10—H10A	109.5
C2—C3—H3B	109.0	C8—C10—H10B	109.5
H3A—C3—H3B	107.8	H10A—C10—H10B	109.5
N4—C4—N5	115.0 (3)	C8—C10—H10C	109.5
N4—C4—H4	122.5	H10A—C10—H10C	109.5
N5—C4—H4	122.5	H10B—C10—H10C	109.5
N5—C5—N3	111.1 (3)	C8—C11—H11A	109.5
N5—C5—H5	124.4	C8—C11—H11B	109.5
N3—C5—H5	124.4	H11A—C11—H11B	109.5
C7—C6—S2	113.74 (17)	C8—C11—H11C	109.5
C7—C6—H6A	108.8	H11A—C11—H11C	109.5
S2—C6—H6A	108.8	H11B—C11—H11C	109.5

C2—N1—N2—C1	0.3 (4)	S1—C2—C3—N3	53.9 (4)
C5—N3—N4—C4	0.9 (3)	N3—N4—C4—N5	−1.1 (4)
C3—N3—N4—C4	−179.3 (2)	C5—N5—C4—N4	0.9 (4)
N1—N2—C1—S1	−1.4 (3)	C4—N5—C5—N3	−0.3 (4)
N1—N2—C1—S2	179.3 (2)	N4—N3—C5—N5	−0.4 (4)
C2—S1—C1—N2	1.6 (2)	C3—N3—C5—N5	179.8 (3)
C2—S1—C1—S2	−179.13 (18)	C1—S2—C6—C7	71.9 (2)
C6—S2—C1—N2	13.2 (3)	S2—C6—C7—O1	15.0 (3)
C6—S2—C1—S1	−166.03 (16)	S2—C6—C7—C8	−165.12 (19)
N2—N1—C2—C3	−177.4 (3)	O1—C7—C8—C9	129.1 (4)
N2—N1—C2—S1	0.9 (3)	C6—C7—C8—C9	−50.8 (5)
C1—S1—C2—N1	−1.4 (2)	O1—C7—C8—C10	5.4 (4)
C1—S1—C2—C3	176.9 (2)	C6—C7—C8—C10	−174.5 (2)
C5—N3—C3—C2	105.8 (3)	O1—C7—C8—C11	−109.9 (4)
N4—N3—C3—C2	−74.1 (4)	C6—C7—C8—C11	70.2 (4)
N1—C2—C3—N3	−127.9 (3)		