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5-*m*-Tolyl-1,3,4-thiadiazol-2-ylamine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.150
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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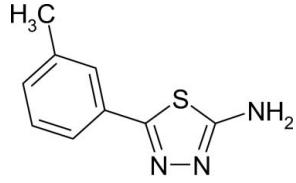
The asymmetric unit of the title compound, $C_9H_9N_3S$, contains four molecules which are interconnected through N—H···N hydrogen bonding, resulting in the formation of a tetrameric structure.

Comment

1,3,4-Thiadiazole derivatives represent an interesting class of compounds possessing a broad spectrum of biological activities (Nakagawa *et al.*, 1996; Wang *et al.*, 1999). These compounds are known to exhibit diverse biological effects, such as insecticidal and fungicidal activities (Wang *et al.*, 1999).



(I)

The asymetric unit of the title compound, (I), is built up from four independent molecules (*A*, *B*, *C* and *D*) interconnected through N—H···N hydrogen bonding, resulting in the formation of a pseudo-tetrameric structure (Fig. 1). The four molecules are roughly planar. The thiadiazole and tolyl rings are only slightly twisted with dihedral angles of 5.7 (*A*), 10.8 (*B*), 16.5 (*C*) and 3.4° (*D*).

Experimental

3-Methylbenzoic acid (5 mmol) and thiosemicarbazide (5 mmol) were added to toluene (50 ml) and heated under reflux for 4 h. The reaction mixture was left to cool to room temperature and then filtered; the filter cake was crystallized from acetone to give pure compound (I) (m.p. 513–514 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

Crystal data

$C_9H_9N_3S$
M_r = 191.26
Triclinic, $P\overline{1}$
a = 10.793 (2) Å
b = 10.820 (2) Å
c = 17.607 (4) Å
α = 73.85 (3)°
β = 78.97 (3)°
γ = 73.51 (3)°
V = 1879.6 (8) Å^3
Z = 8
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.924, T_{max} = 0.943
7785 measured reflections
7370 independent reflections
4158 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.150
S = 1.02
7370 reflections
473 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.39P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3*C*—H3*C*1···N2*B*	0.86	2.17	2.994 (5)	161
N3*B*—H3*B*1···N2*C*	0.86	2.14	2.959 (5)	161
N3*A*—H3*A*2···N1*B*	0.86	2.42	3.187 (4)	149
N3*A*—H3*A*2···N2*B*	0.86	2.33	3.169 (5)	164
N3*D*—H3*D*2···N1*C*	0.86	2.22	3.064 (4)	169
N3*D*—H3*D*1···N2*D*^{i}	0.86	2.22	3.066 (4)	169
N3*C*—H3*C*2···N1*D*^{ii}	0.86	2.18	3.004 (4)	161
N3*A*—H3*A*1···N2*A*^{iii}	0.86	2.21	3.014 (5)	155

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x+1, y, z$; (iii) $-x+2, -y+1, -z$.

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(aromatic C, N) or 1.5U_{eq}(methyl C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

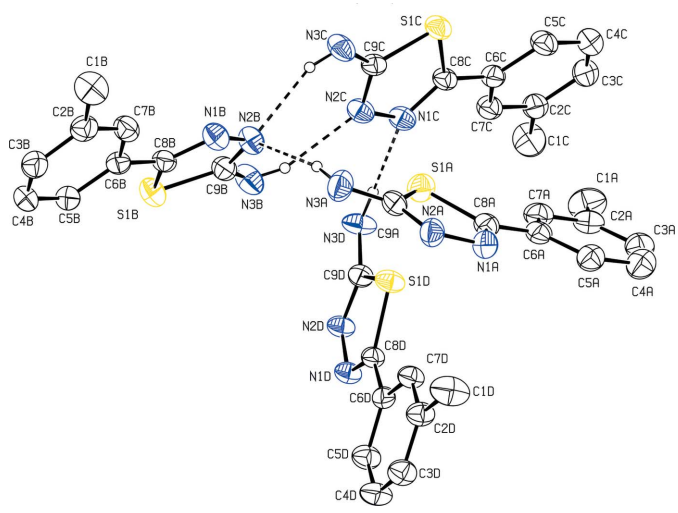


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radius. N—H···N bonds are represented as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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