

2-Anilino-4,5-dimethyl-1,3-thiazole

Juliana Jumal and Bohari M.
Yamin*School of Chemical Sciences and Food
Technology, Universiti Kebangsaan Malaysia,
43600 Bangi, Selangor, MalaysiaCorrespondence e-mail:
bohari@pkrisc.cc.ukm.my

Key indicators

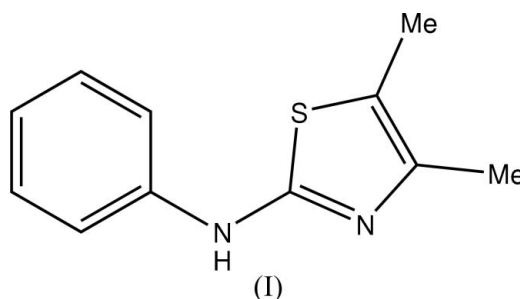
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.044
 wR factor = 0.120
Data-to-parameter ratio = 17.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_{11}\text{H}_{12}\text{N}_2\text{S}$, the whole molecule is essentially planar except for methyl H atoms, with a maximum deviation of 0.160 (2) Å for the aniline N atom. The molecule is stabilized by π - π interactions and intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, which produce dimers located about the crystallographic twofold rotation axes.

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Comment

The 1,3-thiazole ring is well known as the central structural group of a number of biologically active natural products (Crews *et al.*, 1988) and pharmacologically active compounds (Metzger, 1984). The title compound, (I), is analogous to 5-acetyl-4-methyl-2-phenylamino-1,3-thiazole, (II) (Kasim & Yamin, 2005), except that the substituents at the 4- and 5-positions of the thiazole ring are methyl groups. In contrast to (II), the whole molecule is essentially planar except for methyl H atoms, with a maximum deviation of 0.160 (2) Å for atom N1. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable to those in (II). In the structure, the molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds (Table 2), forming dimers located about the crystallographic twofold rotation axes (Fig. 2). In addition, there is π - π stacking between the 1,3-thiazole ring and its symmetry equivalent at $(1-x, 1-y, 1-z)$. The distance between the ring centroids is 3.7629 (15) Å.



Experimental

A solution of aniline (1.86 g, 0.02 mol) in acetone (40 ml) was added dropwise to an acetone solution (40 ml) containing an equimolar amount of 3-chlorobutan-2-one (2.14 g, 0.02 mol) and ammonium thiocyanate (1.52 g, 0.02 mol) in a two-necked round-bottomed flask. The mixture was refluxed for 3 h. The resulting solution was poured into a beaker containing ice cubes. The resulting white precipitate was filtered off and washed with distilled water and ethanol, then dried. Recrystallization from acetone yielded single crystals suitable for X-ray analysis.

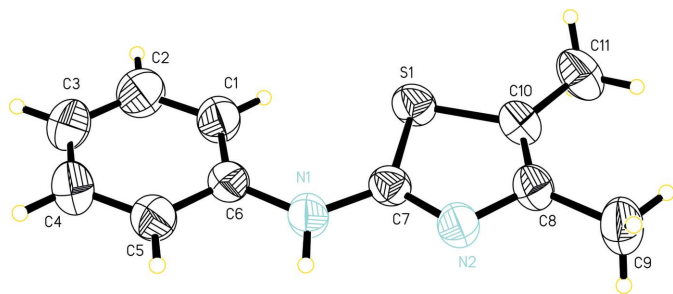


Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids

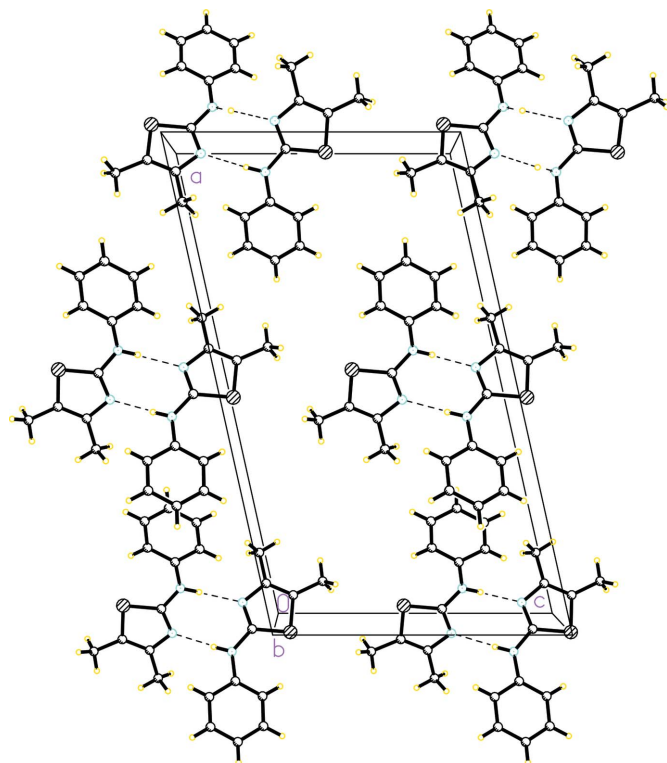


Figure 2
Packing diagram of the title compound, (I), viewed down the *b* axis. The dashed lines denote N—H...N hydrogen bonds.

Crystal data

C₁₁H₁₂N₂S
M_r = 204.29
 Monoclinic, *C*2/*c*
a = 21.697 (6) Å
b = 7.860 (2) Å
c = 12.617 (3) Å
 β = 102.535 (5)°
V = 2100.6 (9) Å³

Z = 8
D_x = 1.292 Mg m⁻³
 Mo *K*α radiation
 μ = 0.27 mm⁻¹
T = 298 (2) K
 Block, colourless
 0.49 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
T_{min} = 0.879, *T_{max}* = 0.958

6033 measured reflections
 2276 independent reflections
 1694 reflections with *I* > 2σ(*I*)
R_{int} = 0.025
 θ_{max} = 27.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.044
wR(*F*²) = 0.120
S = 1.02
 2276 reflections
 129 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0623*P*)² + 0.7325*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.16 e Å⁻³
 Δρ_{min} = -0.16 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...N2 ⁱ	0.86	2.09	2.946 (2)	173

Symmetry code: (i) -*x* + 1, *y*, -*z* + ½.

After their location in a difference map, all H atoms were positioned geometrically at idealized positions and allowed to ride on the parent atoms, with C—H = 0.93–0.96 Å, N—H = 0.86 Å, and *U*_{iso}(H) = 1.5*U*_{eq}(methyl C) and 1.2*U*_{eq}(C,N).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bruker (2000). SADABS (Version 2.01), SMART (Version 5.630a) and SAINT (6.36a). Bruker AXS Inc., Madison, Wisconsin, USA.
 Crews, P., Kakou, Y. & Quinoa, E. (1988). *J. Amer. Chem. Soc.* **110**, 4365–4369.
 Kasim, N. A. M. & Yamin, B. M. (2005). *Acta Cryst.* **E61**, o3794–o3795.
 Metzger, J. V. (1984). *Thiazoles and their Benzo Derivatives*, edited by K. T. Potts, Vol. 6. New York: Pergamon Press.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97, University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.