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2-Amino-5-phenyl-1,3,4-thiadiazole at 110 K

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.078
Data-to-parameter ratio = 18.6

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

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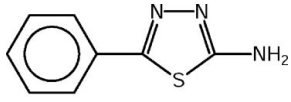
The crystal structure of the title compound, $C_8H_7N_3S$, previously determined at room temperature [Ishankhodzhaeva, Kadyrova, Surazhskaya, Parpiev & Koz'min (2001). *Zh. Org. Khim*, **37**, 759–761], has been redetermined at 110 K. Comparison of the two structures reveals significant differences in the unit-cell parameters, while the molecular geometry and hydrogen-bonding network are essentially the same in both structures.

Comment

The 1,3,4-thiadiazole ring is associated with diverse biological activities, which can be explained by the presence of the toxiphoric —N=C—S— linkage, the importance of which in many pesticides has been reported (Abdel-Rahma *et al.*, 1983; Foerster *et al.*, 1981; Tiwari *et al.*, 1989). Various 2-amino-substituted 1,3,4-thiadiazoles and their Schiff bases also exhibit diverse biological and pharmacological activities (Singh & Yadav, 1976; Russo & Santagari, 1985; Masahito & Kazuyuki, 1977; Ramalingam *et al.*, 1989; Ludwing *et al.*, 1987; Öztürk *et al.*, 2004).



(I)

The crystal and molecular structure of the title compound, (I), has been previously determined at room temperature (Ishankhodzhaeva *et al.*, 2001) (Cambridge Structural Database, Version 5.24.1; refcode NOTSAQ; Allen, 2002). The monoclinic unit-cell dimensions of NOTSAQ are a = 11.085 (3) Å, b = 7.544 (3) Å, c = 11.180 (3) Å and β = 115.22 (2)°.

We report here the crystal structure of the title compound, (I) (Fig. 1), at 110 K. The bond lengths and angles (Table 1) are essentially the same as in NOTSAQ. The geometric parameters of the 1,3,4-thiadiazole ring in (I) and in NOTSAQ agree with those in 3,4-diphenyl-1,2,5-thiadiazole (Mellini & Merlino, 1976). The hydrogen-bonding geometry in (I) (Table 2) is close to that in NOTSAQ.

The most significant differences between (I) and NOTSAQ are associated with the unit-cell dimensions. The lowering of the temperature to 110 K resulted in an unexpected increase of the unit-cell parameter b by more than 5%.

Experimental

Compound (I) was synthesized by the ring-closure reaction of benzoic acid with thiosemicarbazide. A mixture of benzoic acid (0.01 mol), thiosemicarbazide (0.013 mol) and phosphorus oxy-

chloride (0.013 mol) was warmed at 333 K for 1 h and the temperature was then raised to 368 K for an additional 2 h. The contents were then poured on to crushed ice, cooled to 283 K, the pH adjusted to 9–10 with 10 *M* NaOH, and the resulting solid crystallized from DMF (yield 73%, m.p. 495–496 K). IR (KBr): ν (cm^{-1}) 3420, 1620, 1211, 1074, 982, 876. ^{1}H NMR (DMSO-d_6): δ 7.85–7.38 (*m*, 5H, Ar–H), 7.26 (*s*, 2H, NH_2). ^{13}C NMR (DMSO, TMS): δ 170.25, 162.93, 131.46, 127.12, 125.39. Calculated for $C_8H_7N_3S$: C 54.22, H 3.98, N 23.71, S 18.09%; found: C 54.17, H 3.91, N 23.68, S 17.98%.

Crystal data

$C_8H_7N_3S$
M_r = 177.24
Monoclinic, $P2_1/c$
a = 10.604 (5) Å
b = 7.922 (5) Å
c = 11.116 (5) Å
β = 117.965 (5)°
V = 824.8 (7) Å^3
Z = 4
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 73 reflections
θ = 6–20°
μ = 0.33 mm^{-1}
T = 100 K
Plate, colourless
0.37 × 0.28 × 0.07 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.887, T_{max} = 0.977
23 236 measured reflections
2176 independent reflections
1699 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 29.0°
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.078
S = 1.04
2176 reflections
117 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 0.3514P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.749 (2)	N2—N3	1.384 (2)
S1—C2	1.7518 (18)	N2—C1	1.320 (2)
N1—C1	1.337 (2)	N3—C2	1.300 (2)
C1—S1—C2	87.01 (7)	S1—C1—N1	122.04 (12)
N3—N2—C1	112.21 (13)	S1—C2—N3	113.30 (11)
N2—N3—C2	113.94 (12)	S1—C2—C3	122.48 (12)
N1—C1—N2	124.42 (15)	N3—C2—C3	124.18 (13)
S1—C1—N2	113.55 (11)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N3^{i}	0.86 (2)	2.13 (2)	2.982 (3)	169.9 (17)
N1—H1B···N2^{ii}	0.85 (2)	2.12 (2)	2.972 (3)	173.9 (18)

Symmetry codes: (i) $x, -\frac{1}{2}-y, z-\frac{1}{2}$; (ii) $-x, -1-y, 1-z$.

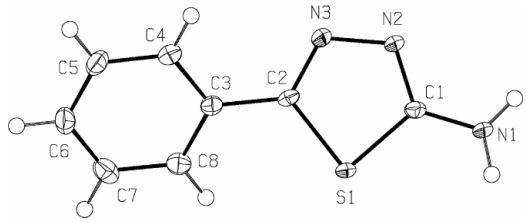


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

The amine H atoms were located in a difference synthesis and both positional and displacement parameters were refined. All other H atoms were included in the refinement at calculated positions in the riding-model approximation, with C—H at 0.93 Å (Ar–H), and the isotropic displacement parameters were set equal to $1.2U_{eq}$(C).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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