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Redetermination of 4-(phenyl)thiosemicarbazide

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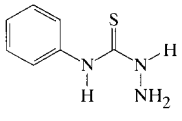
The structure of the title compound, $C_7H_9N_3S$, comprises twisted molecules that associate *via* N—H···N and N—H···S hydrogen-bonding interactions. The dihedral angle between the phenyl ring and the five-membered thiosemicarbazide plane is 67.56 (5)°. This structure was previously determined using data from visual estimation of photographic intensity (measurements at room temperature, with R = 0.112).

Comment

The structure of the title compound, (I), was initially determined using data from visual estimation of photographic intensity measurements at room temperature, with refinement to R = 0.112 (Kalman *et al.*, 1972). However, reported here is a redetermination of the structure using data collected by a modern area detector at 150 K, giving R = 0.040. The previously published value for the dihedral angle between the phenyl ring and the five-membered thiosemicarbazide was 67.5°, which can now be updated to 67.56 (5)°.



(I)

Experimental

Crystals of (I) were obtained from Spa Contract Synthesis.

Crystal data

$C_7H_9N_3S$
M_r = 167.23
Monoclinic, $P2_1/c$
a = 11.9231 (7) Å
b = 5.4888 (3) Å
c = 12.3788 (8) Å
β = 91.527 (3)°
V = 809.82 (8) Å^3
Z = 4
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6169 reflections
θ = 1.00–27.48°
μ = 0.334 mm^{-1}
T = 150 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.878, T_{max} = 0.936
4691 measured reflections
1803 independent reflections
1483 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.47°
$h = -15 \rightarrow 15$
$k = -6 \rightarrow 7$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.132
S = 1.130
1803 reflections
116 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.3297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N1	0.83 (2)	2.19 (2)	2.6317 (17)	113 (2)
N2—H2···S3^{i}	0.89 (2)	2.43 (2)	3.3097 (17)	168 (2)
N1—H11···S3^{ii}	0.87 (3)	2.83 (3)	3.5100 (18)	137 (2)
N1—H12···N2^{iii}	0.92 (3)	2.56 (3)	3.296 (2)	137 (2)

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

All H atoms were included in the refinement, at calculated positions, as riding models with C—H set to 0.95 Å, except for the amine H atoms, which were located on difference syntheses and both positional and displacement parameters were refined.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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