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# *N*-(*m*-Tolyl)thioacetamide

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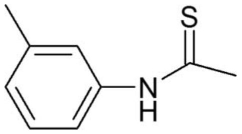
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.039; $wR$ factor = 0.086; data-to-parameter ratio = 28.2.

The crystal structure of the title compound, $C_9H_{11}NS$, is stabilized by weak intermolecular N—H···S hydrogen bonds.

## Related literature

For related literature, see: Desiraju & Steiner (1999); Flakus & Miros (2001); Flakus & Pyzik (2006); Flakus *et al.* (2002, 2003, 2007); Hopkins & Hunter (1942).



## Experimental

### *Crystal data*

$C_9H_{11}NS$
$M_r$ = 165.25
Monoclinic, $P2_1/c$
$a$ = 16.023 (3) Å
$b$ = 6.9877 (14) Å
$c$ = 8.0670 (16) Å
$\beta$ = 103.35 (3)°

$V$ = 878.8 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 100 (2) K
0.6 × 0.04 × 0.03 mm

### *Data collection*

Oxford Diffraction KM-4 Sapphire3 CCD diffractometer
Absorption correction: none
8311 measured reflections

2963 independent reflections
1480 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.046

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.086
$S$ = 0.81
2963 reflections
105 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.27 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···S1$^{\rm i}$ | 0.808 (15) | 2.520 (16) | 3.3233 (14) | 173.0 (15) |

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2046).

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# supplementary materials

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# *N*-(*m*-Tolyl)thioacetamide

**W. Smiszek-Lindert, M. Nowak and J. Kusz**

## Comment

The objective of our research were measurements and a theoretical interpretation of the polarized IR–spectra of the hydrogen bond in *N*–(*m*–tolyl)thioacetamide crystals. The spectral studies were preceded by determination of the crystal *X*–ray structure of this compound. We interpret spectroscopic effects, which are observed in the frequency ranges of the proton and deuteron stretching vibrations for the associated molecules, see: Flakus & Miros, 2001; Flakus & Pyzik, 2006; Flakus *et al.* (2002, 2003, 2007).

The structure of *N*–(*m*–tolyl)thioacetamide with the atomic numbering scheme is presented on Fig. 1. The molecules of (I) form an intermolecular N—H···S hydrogen bonds. These intermolecular hydrogen bonds link the molecules of *N*–(*m*–tolyl)thioacetamide giving infinite zigzag chains parallel to the *c* axis (Fig. 2). The values of the H···S (2.520 (16) Å) and N···S (3.3233 (14) Å) distances and N—H···S (173.0 (15)°) angle characterize this bond as a weak hydrogen bond: Desiraju & Steiner, (1999). The strength of the hydrogen bond in (I) was investigated by IR. The band of the isolated N—H stretching vibration, $\nu_{N—H}$, is located in the 3200–2850 $cm^{-1}$ frequency range.

For related literature, see: Flakus & Miros (2001); Flakus & Pyzik, 2006; Flakus *et al.* (2002, 2003, 2007); Hopkins & Hunter (1942). For discussion of hydrogen bonding, see: Desiraju & Steiner (1999).

## Experimental

Phosphorus pentasulfide (0.31 g, 0.1 mol) was added small portions to *m*–acetotoluidide (1.03 g, 0.5 mol) in toluene (3.45 ml) at 343–353 K with stirring. The reaction mixture was then brought to reflux for 2.5 h. After heating the hot reaction mixture was decanted and the solution was concentrated to give a yellow precipitate. The precipitate was dissolved in ethanol and the solution was left for crystallization at room temperature. After one month, the deposited yellow crystals were collected and recrystallized from acetone and petroleum ether, giving needle–shaped crystals of quality suitable for *X*–ray measurement. Yeld: 0.78 g (69.57%). *M*.p. 316–317 K [literature m.p. 315–316 K, see: Hopkins & Hunter (1942)].

The IR–spectra of (I) crystals were measured by a transmission method, with the help of the FT–IR Nicolet Magna spectrometer, for two different, mutually perpendicular polarizations of IR–beam. The spectra were measured for the $\nu_{N—H}$ and $\nu_{N—D}$ band frequency ranges at temperatures of 298 and 77 K.

## Refinement

The amide H atom was located in a difference Fourier map and refined freely; The hydrogen atoms attached on C atoms were introduced in geometrically idealized positions and refined with an appropriate riding model, with C—H = 0.95Å (for aromatic C) or 0.98Å (for methyl C). Their isotropic displacement parameters were constrained with $U_{iso}$(H) values of $1.2U_{eq}$ (C) for H atoms in CH groups and $1.5U_{eq}$(C) for the methyl H atoms.

### Figures

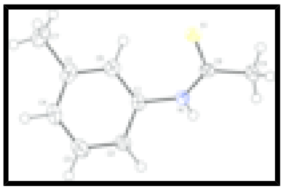

Fig. 1. The conformation of (I) molecule with the atom numbering scheme. Displacement ellipsoids are presented with 50% probability level. H atoms are depicted as small circles of arbitrary radii.

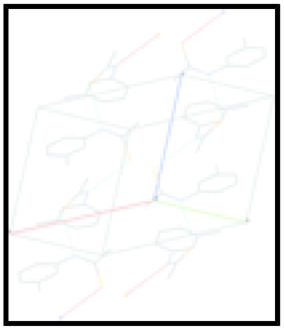

Fig. 2. The crystal packing of the (I) in the unit cell. The intermolecular N—H···S interactions are represented by dashed lines.

## *N*-(*m*-Tolyl)thioacetamide

### *Crystal data*

| | |
|---|---|
| $C_9H_{11}NS$ | $F_{000} = 352$ |
| $M_r = 165.25$ | $D_x = 1.249$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 1296 reflections |
| $a = 16.023$ (3) Å | $\theta = 3.2$–$32.9$° |
| $b = 6.9877$ (14) Å | $\mu = 0.30$ mm$^{-1}$ |
| $c = 8.0670$ (16) Å | $T = 100$ (2) K |
| $\beta = 103.35$ (3)° | Needle, yellow |
| $V = 878.8$ (3) Å$^3$ | 0.6 × 0.04 × 0.03 mm |
| $Z = 4$ | |

### *Data collection*

| | |
|---|---|
| Oxford Diffraction KM-4 CCD Sapphire3 diffractometer | 1480 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine–focus sealed tube | $R_{int} = 0.046$ |
| Monochromator: graphite | $\theta_{max} = 32.9$° |
| $T = 100(2)$ K | $\theta_{min} = 3.2$° |
| $\theta$ scans | $h = -24 \rightarrow 23$ |
| Absorption correction: none | $k = -10 \rightarrow 10$ |
| 8311 measured reflections | $l = -5 \rightarrow 12$ |
| 2963 independent reflections | |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.086$ | $w = 1/[\sigma^2(F_o^2) + (0.0405P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.81$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 2963 reflections | $\Delta\rho_{max} = 0.34$ e Å$^{-3}$ |
| 105 parameters | $\Delta\rho_{min} = -0.27$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

### *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 $F^2 > 2\sigma(F^2)$ 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* | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.37438 (3) | 0.45335 (6) | 0.42670 (5) | 0.02406 (12) |
| N1 | 0.33040 (8) | 0.74590 (18) | 0.21414 (15) | 0.0190 (3) |
| H1 | 0.3414 (10) | 0.810 (2) | 0.139 (2) | 0.023* |
| C1 | 0.26269 (9) | 0.8204 (2) | 0.28248 (17) | 0.0202 (3) |
| C2 | 0.26115 (10) | 1.0164 (2) | 0.30967 (19) | 0.0253 (4) |
| H2 | 0.3041 | 1.0971 | 0.2836 | 0.030* |
| C3 | 0.19587 (11) | 1.0919 (2) | 0.3755 (2) | 0.0328 (4) |
| H3 | 0.1945 | 1.2253 | 0.3972 | 0.039* |
| C4 | 0.13338 (11) | 0.9758 (3) | 0.4094 (2) | 0.0307 (4) |
| H4 | 0.0891 | 1.0304 | 0.4547 | 0.037* |
| C5 | 0.13278 (10) | 0.7807 (2) | 0.37947 (19) | 0.0270 (4) |
| C6 | 0.19927 (9) | 0.7042 (2) | 0.31496 (18) | 0.0229 (3) |
| H6 | 0.2006 | 0.5707 | 0.2934 | 0.027* |
| C7 | 0.06452 (11) | 0.6518 (3) | 0.4158 (2) | 0.0386 (5) |
| H7A | 0.0825 | 0.6024 | 0.5323 | 0.058* |
| H7B | 0.0554 | 0.5447 | 0.3352 | 0.058* |
| H7C | 0.0110 | 0.7239 | 0.4037 | 0.058* |

| | | | | |
|---|---|---|---|---|
| C8 | 0.37891 (9) | 0.5926 (2) | 0.26284 (18) | 0.0185 (3) |
| C9 | 0.44358 (10) | 0.5519 (2) | 0.15960 (18) | 0.0231 (3) |
| H9A | 0.4288 | 0.4317 | 0.0971 | 0.035* |
| H9B | 0.5007 | 0.5408 | 0.2356 | 0.035* |
| H9C | 0.4435 | 0.6566 | 0.0787 | 0.035* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0297 (2) | 0.0214 (2) | 0.0216 (2) | 0.00168 (18) | 0.00700 (16) | 0.00134 (18) |
| N1 | 0.0233 (7) | 0.0201 (7) | 0.0145 (6) | 0.0011 (6) | 0.0062 (5) | 0.0021 (5) |
| C1 | 0.0202 (8) | 0.0263 (8) | 0.0131 (7) | 0.0035 (7) | 0.0021 (6) | 0.0017 (6) |
| C2 | 0.0288 (9) | 0.0238 (9) | 0.0238 (8) | 0.0023 (7) | 0.0073 (7) | 0.0015 (7) |
| C3 | 0.0403 (11) | 0.0283 (10) | 0.0309 (9) | 0.0083 (8) | 0.0104 (8) | −0.0001 (7) |
| C4 | 0.0268 (9) | 0.0414 (11) | 0.0247 (8) | 0.0111 (8) | 0.0076 (7) | 0.0003 (8) |
| C5 | 0.0197 (9) | 0.0424 (11) | 0.0173 (7) | −0.0013 (7) | 0.0008 (7) | 0.0047 (7) |
| C6 | 0.0219 (8) | 0.0270 (9) | 0.0171 (8) | 0.0004 (7) | −0.0008 (6) | 0.0018 (6) |
| C7 | 0.0273 (10) | 0.0553 (13) | 0.0333 (10) | −0.0071 (9) | 0.0069 (8) | 0.0004 (9) |
| C8 | 0.0193 (8) | 0.0178 (8) | 0.0163 (7) | −0.0024 (6) | −0.0002 (6) | −0.0042 (6) |
| C9 | 0.0242 (8) | 0.0230 (8) | 0.0222 (8) | 0.0034 (7) | 0.0053 (6) | −0.0022 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C8 | 1.6565 (15) | C4—H4 | 0.9500 |
| N1—C8 | 1.3283 (18) | C5—C6 | 1.396 (2) |
| N1—C1 | 1.4241 (18) | C5—C7 | 1.497 (2) |
| N1—H1 | 0.808 (15) | C6—H6 | 0.9500 |
| C1—C6 | 1.372 (2) | C7—H7A | 0.9800 |
| C1—C2 | 1.389 (2) | C7—H7B | 0.9800 |
| C2—C3 | 1.382 (2) | C7—H7C | 0.9800 |
| C2—H2 | 0.9500 | C8—C9 | 1.499 (2) |
| C3—C4 | 1.364 (2) | C9—H9A | 0.9800 |
| C3—H3 | 0.9500 | C9—H9B | 0.9800 |
| C4—C5 | 1.384 (2) | C9—H9C | 0.9800 |
| C8—N1—C1 | 128.77 (13) | C1—C6—C5 | 120.47 (15) |
| C8—N1—H1 | 117.2 (11) | C1—C6—H6 | 119.8 |
| C1—N1—H1 | 113.9 (11) | C5—C6—H6 | 119.8 |
| C6—C1—C2 | 120.84 (14) | C5—C7—H7A | 109.5 |
| C6—C1—N1 | 121.43 (14) | C5—C7—H7B | 109.5 |
| C2—C1—N1 | 117.69 (13) | H7A—C7—H7B | 109.5 |
| C3—C2—C1 | 118.66 (15) | C5—C7—H7C | 109.5 |
| C3—C2—H2 | 120.7 | H7A—C7—H7C | 109.5 |
| C1—C2—H2 | 120.7 | H7B—C7—H7C | 109.5 |
| C4—C3—C2 | 120.38 (16) | N1—C8—C9 | 114.90 (12) |
| C4—C3—H3 | 119.8 | N1—C8—S1 | 125.40 (11) |
| C2—C3—H3 | 119.8 | C9—C8—S1 | 119.68 (11) |
| C3—C4—C5 | 121.77 (15) | C8—C9—H9A | 109.5 |
| C3—C4—H4 | 119.1 | C8—C9—H9B | 109.5 |

| | | | |
|---|---|---|---|
| C5—C4—H4 | 119.1 | H9A—C9—H9B | 109.5 |
| C4—C5—C6 | 117.85 (15) | C8—C9—H9C | 109.5 |
| C4—C5—C7 | 122.30 (15) | H9A—C9—H9C | 109.5 |
| C6—C5—C7 | 119.85 (16) | H9B—C9—H9C | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···S1$^{i}$ | 0.808 (15) | 2.520 (16) | 3.3233 (14) | 173.0 (15) |

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

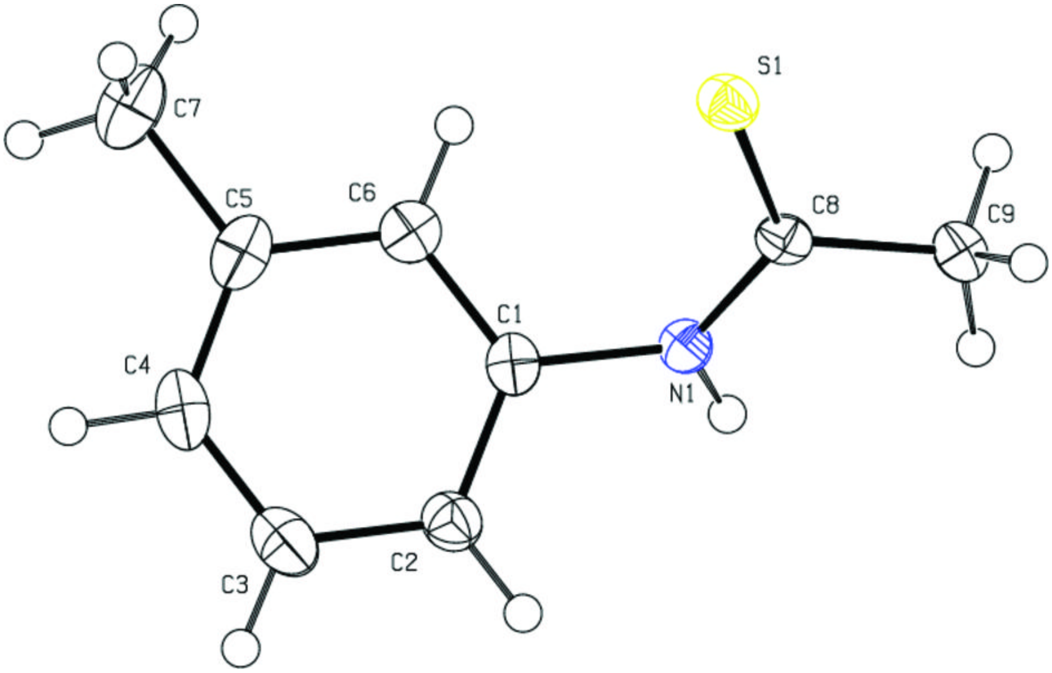

**Fig. 2**