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# 5*H*-1-Benzothiopyrano[2,3-*b*]pyridin-5-one

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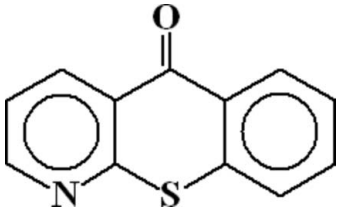
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.071; $wR$ factor = 0.180; data-to-parameter ratio = 14.3.

Molecules of the title compound, $C_{12}H_7NOS$, with one half-molecule in the asymmetric unit, are disordered about a crystallographic centre of inversion. Refinement showed that the C=O group is disordered with the S atom and the N atom is disordered over four positions. Adjacent molecules are connected through C—H···O hydrogen bonds and $\pi\cdots\pi$ interactions (centroid–centroid distances of 3.635 and 3.858 Å).

## Related literature

For related literature, see: Hidetoshi (1997); Khan *et al.* (2008); Mann & Reid (1952).




## Experimental

### Crystal data

$C_{12}H_7NOS$
$M_r$ = 213.26
Monoclinic, $P2_1/c$
$a$ = 7.7308 (18) Å
$b$ = 3.8585 (9) Å
$c$ = 15.771 (3) Å
$\beta$ = 99.333 (9)°
$V$ = 464.20 (18) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.31 mm$^{-1}$
$T$ = 296 (2) K
0.25 × 0.06 × 0.04 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.977, $T_{max}$ = 0.987
5384 measured reflections
1189 independent reflections
822 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.179
$S$ = 1.17
1189 reflections
83 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 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···$A$ |
|---|---|---|---|---|
| C5—H5···O1$^{i}$ | 0.93 | 2.53 | 3.286 (7) | 139 |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2756).

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# supporting information

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## 5*H*-1-Benzothiopyrano[2,3-*b*]pyridin-5-one

**Muhammad Naeem Khan, M. Nawaz Tahir, Misbahul Ain Khan, Islam Ullah Khan and Muhammad Nadeem Arshad**

### S1. Comment

In continuation of our studies of pyridine containing heterocyclic compounds (Khan, *et al.*, 2008), the title compound has been synthesized. As the molecule is located on a centre of inversion the thio (S1) and carbonyl group (C6=O1) are disordered over two sites with 50% occupancy. For the N atom four different positions were found with an occupancy factor of 0.25. Adjacent molecules are linked to each other through intermolecular H-bonding of C—H···O type (Table 1). In addition, there are $\pi\cdots\pi$-interactions between the the adjacent molecules. The centroid of the ring composed by C1, C2, C3A, C4, C5, and N1B is at 3.635Å from the centroid of the central ring and at 3.858Å from the centroid of its symmetry equivalent (symmetry operator for both centroids: *x*, *y*-1, *z*)

### S2. Experimental

A mixture of 2-chloronicotinic acid (1.57 g, 10 mmol) and thiophenol (2 ml) was heated under reflux for two hours to produce 2-(phenylsulfanyl) pyridine-3-carboxylic acid (Mann & Reid, 1952). The pollyphosforic acid (PPA) (Hidetoshi, 1997) was used to obtain 5*H*-benzothiopyrano[2,3-*b*]pyridin-5-one after cyclization. Crystals suitable for X-ray diffraction were obtained by cooling the saturated solution of the title compound in chloroform.

### S3. Refinement

For the molecule is disordered, during refinement EXYZ and EADP were used for N1A, C3B and N1B, C3A. The occupancy factors for N1A and N1B refined to 0.231 (4) and 0.269 (4), respectively. Thus, they were fixed to 0.25 whereas for C3A and C3B the site occupation factors were fixed to 0.75.

The H atoms were positioned geometrically, with C—H = 0.93 Å, $U_{iso}$(H) = 1.2$U_{eq}$(C) and constrained to ride on their parent atoms.

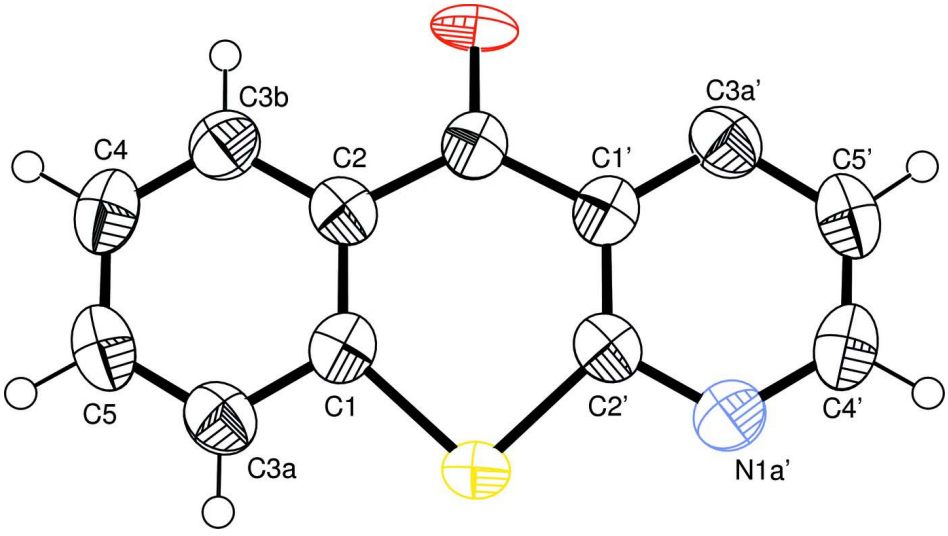


**Figure 1**

*ORTEP-3 for Windows* (Farrugia, 1997) drawing of the title compound. The symmetry related atoms are shown by putting ′ on the names. The displacement ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii.

### 5*H*-1-Benzothiopyrano[2,3-*b*]pyridin-5-one

*Crystal data*

$C_{12}H_7NOS$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7308\ (18)$ Å
$b = 3.8585\ (9)$ Å
$c = 15.771\ (3)$ Å
$\beta = 99.333\ (9)°$
$V = 464.20\ (18)$ Å$^3$
$Z = 2$
$F(000) = 220$
$D_x = 1.525$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1189 reflections
$\theta = 2.6–28.7°$
$\mu = 0.31$ mm$^{-1}$
$T = 296$ K
Needle, light yellow
0.25 × 0.06 × 0.04 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.5 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.977$, $T_{max} = 0.987$
5384 measured reflections
1189 independent reflections
822 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 28.7°$, $\theta_{min} = 2.6°$
$h = -10\rightarrow10$
$k = -5\rightarrow3$
$l = -21\rightarrow21$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.179$
$S = 1.17$
1189 reflections
83 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.435P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.62$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

*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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C6 | 0.3852 (15) | 0.001 (3) | 0.0576 (7) | 0.044 (2) | 0.50 |
| O1 | 0.2856 (9) | −0.006 (2) | 0.1093 (4) | 0.083 (3) | 0.50 |
| C3B | 0.1527 (4) | −0.2668 (8) | −0.05322 (19) | 0.0508 (7) | 0.75 |
| H3B | 0.0739 | −0.2649 | −0.0145 | 0.061* | 0.75 |
| N1B | 0.3839 (4) | −0.2689 (8) | −0.17028 (19) | 0.0530 (8) | 0.25 |
| S1 | 0.6613 (4) | −0.0009 (10) | −0.08029 (19) | 0.0481 (6) | 0.50 |
| C3A | 0.3839 (4) | −0.2689 (8) | −0.17028 (19) | 0.0530 (8) | 0.75 |
| H3A | 0.4605 | −0.2676 | −0.2100 | 0.064* | 0.75 |
| N1A | 0.1527 (4) | −0.2668 (8) | −0.05322 (19) | 0.0508 (7) | 0.25 |
| C1 | 0.4368 (4) | −0.1412 (8) | −0.08724 (19) | 0.0444 (7) | |
| C2 | 0.3204 (4) | −0.1406 (8) | −0.02885 (17) | 0.0437 (7) | |
| C4 | 0.1027 (4) | −0.3937 (9) | −0.1335 (2) | 0.0577 (9) | |
| H4 | −0.0102 | −0.4803 | −0.1495 | 0.069* | |
| C5 | 0.2178 (5) | −0.3955 (9) | −0.1919 (2) | 0.0570 (9) | |
| H5 | 0.1816 | −0.4842 | −0.2467 | 0.068* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C6 | 0.045 (6) | 0.038 (3) | 0.047 (6) | 0.002 (4) | 0.002 (3) | 0.008 (4) |
| O1 | 0.073 (4) | 0.134 (6) | 0.053 (4) | −0.023 (4) | 0.047 (3) | −0.015 (4) |
| C3B | 0.0496 (16) | 0.0468 (16) | 0.0563 (17) | −0.0002 (13) | 0.0099 (13) | 0.0016 (13) |
| N1B | 0.0632 (19) | 0.0482 (17) | 0.0476 (15) | 0.0023 (14) | 0.0090 (13) | −0.0010 (13) |
| S1 | 0.0438 (17) | 0.0620 (11) | 0.0411 (16) | −0.0040 (13) | 0.0145 (8) | −0.0032 (13) |
| C3A | 0.0632 (19) | 0.0482 (17) | 0.0476 (15) | 0.0023 (14) | 0.0090 (13) | −0.0010 (13) |
| N1A | 0.0496 (16) | 0.0468 (16) | 0.0563 (17) | −0.0002 (13) | 0.0099 (13) | 0.0016 (13) |
| C1 | 0.0434 (14) | 0.0371 (14) | 0.0512 (16) | 0.0028 (12) | 0.0031 (12) | 0.0058 (12) |
| C2 | 0.0522 (16) | 0.0361 (14) | 0.0420 (14) | 0.0059 (12) | 0.0049 (12) | 0.0037 (11) |
| C4 | 0.0501 (18) | 0.0507 (19) | 0.066 (2) | −0.0058 (14) | −0.0082 (15) | 0.0034 (15) |
| C5 | 0.069 (2) | 0.0495 (18) | 0.0474 (17) | 0.0000 (16) | −0.0075 (15) | −0.0048 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C6—O1 | 1.209 (9) | S1—C2$^{i}$ | 1.791 (4) |
| C6—C2 | 1.480 (13) | S1—C1 | 1.803 (4) |
| C6—C1$^{i}$ | 1.481 (13) | C1—C2 | 1.388 (4) |
| C3B—C4 | 1.355 (4) | C1—C6$^{i}$ | 1.481 (13) |
| C3B—C2 | 1.380 (4) | C2—S1$^{i}$ | 1.791 (4) |
| C3B—H3B | 0.9300 | C4—C5 | 1.380 (5) |
| N1B—C5 | 1.364 (5) | C4—H4 | 0.9300 |
| N1B—C1 | 1.398 (4) | C5—H5 | 0.9300 |
| | | | |
| O1—C6—C2 | 117.2 (11) | C6$^{i}$—C1—S1 | 15.5 (4) |
| O1—C6—C1$^{i}$ | 117.1 (11) | C3B—C2—C1 | 119.7 (3) |
| C2—C6—C1$^{i}$ | 125.7 (7) | C3B—C2—C6 | 123.3 (5) |
| C4—C3B—C2 | 120.0 (3) | C1—C2—C6 | 117.0 (5) |
| C4—C3B—H3B | 120.0 | C3B—C2—S1$^{i}$ | 107.4 (2) |
| C2—C3B—H3B | 120.0 | C1—C2—S1$^{i}$ | 132.9 (3) |
| C5—N1B—C1 | 118.7 (3) | C6—C2—S1$^{i}$ | 15.9 (4) |
| C2$^{i}$—S1—C1 | 94.30 (18) | C3B—C4—C5 | 120.6 (3) |
| C2—C1—N1B | 120.0 (3) | C3B—C4—H4 | 119.7 |
| C2—C1—C6$^{i}$ | 117.3 (5) | C5—C4—H4 | 119.7 |
| N1B—C1—C6$^{i}$ | 122.7 (5) | N1B—C5—C4 | 120.9 (3) |
| C2—C1—S1 | 132.8 (3) | N1B—C5—H5 | 119.6 |
| N1B—C1—S1 | 107.2 (2) | C4—C5—H5 | 119.6 |
| | | | |
| C5—N1B—C1—C2 | −0.8 (5) | S1—C1—C2—C6 | 0.2 (6) |
| C5—N1B—C1—C6$^{i}$ | 179.5 (6) | N1B—C1—C2—S1$^{i}$ | −179.5 (3) |
| C5—N1B—C1—S1 | 179.4 (3) | C6$^{i}$—C1—C2—S1$^{i}$ | 0.2 (6) |
| C2$^{i}$—S1—C1—C2 | −0.2 (4) | S1—C1—C2—S1$^{i}$ | 0.2 (6) |
| C2$^{i}$—S1—C1—N1B | 179.6 (2) | O1—C6—C2—C3B | 2.2 (15) |
| C2$^{i}$—S1—C1—C6$^{i}$ | 0 (2) | C1$^{i}$—C6—C2—C3B | −179.7 (7) |
| C4—C3B—C2—C1 | 0.6 (5) | O1—C6—C2—C1 | −178.2 (10) |
| C4—C3B—C2—C6 | −179.8 (6) | C1$^{i}$—C6—C2—C1 | −0.1 (12) |
| C4—C3B—C2—S1$^{i}$ | −179.7 (3) | O1—C6—C2—S1$^{i}$ | 2.0 (11) |
| N1B—C1—C2—C3B | 0.1 (4) | C1$^{i}$—C6—C2—S1$^{i}$ | −180 (3) |
| C6$^{i}$—C1—C2—C3B | 179.7 (6) | C2—C3B—C4—C5 | −0.5 (5) |
| S1—C1—C2—C3B | 179.8 (3) | C1—N1B—C5—C4 | 0.9 (5) |
| N1B—C1—C2—C6 | −179.6 (6) | C3B—C4—C5—N1B | −0.2 (5) |
| C6$^{i}$—C1—C2—C6 | 0.1 (11) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5···O1$^{ii}$ | 0.93 | 2.53 | 3.286 (7) | 139 |

Symmetry code: (ii) *x*, −*y*−1/2, *z*−1/2.