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# 4-(Naphthalene-2-carboxamido)pyridin-1-ium thiocyanate–*N*-(pyridin-4-yl)-naphthalene-2-carboxamide (1/1)

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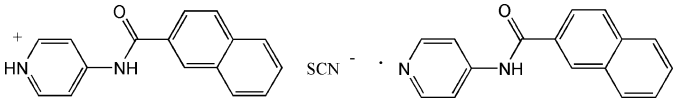
Key indicators: single-crystal X-ray study; $T$ = 123 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.137; data-to-parameter ratio = 25.2.

The asymmetric unit of the title compound, $C_{16}H_{13}N_2O^+\cdot NCS^-\cdot C_{16}H_{12}N_2O$, contains two *N*-(pyridin-4-yl)naphthalene-2-carboxamide molecules, both are partially protonated in the pyridine moiety, *i.e.* the H atom attached to the pyridine N atom is partially occupied with an occupancy factor of 0.61 (3) and 0.39 (3), respectively. In the crystal, protonated and neutral *N*-(pyridin-4-yl)naphthalene-2-carboxamide molecules are linked by N—H···N hydrogen bonding; the thiocyanate counter-ion links with both protonated and neutral *N*-(pyridin-4-yl)naphthalene-2-carboxamide molecules *via* N—H···S and N—H···N hydrogen bonding. The dihedral angles between the pyridine ring and naphthalene ring systems are 11.33 (6) and 9.51 (6)°, respectively. $\pi$–$\pi$ stacking is observed in the crystal structure, the shortest centroid–centroid distance being 3.5929 (8) Å. The crystal structure was determined from a nonmerohedral twin {ratio of the twin components = 0.357 (1):0.643 (1) and twin law [$\bar{1}$00 0$\bar{1}$0 $\bar{1}$01]}.

## Related literature

For background to the biological activity of *N*-substituted benzamides and their applications in synthesis, see: Saeed *et al.* (2011); Priya *et al.* (2005). For related structures and use in molecular recognition, see: Toda *et al.* (1987); Saeed *et al.* (2011, 2012). For bond lengths, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_{16}H_{13}N_2O^+\cdot NCS^-\cdot C_{16}H_{12}N_2O$
$M_r$ = 555.64
Triclinic, $P\bar{1}$
$a$ = 8.2667 (4) Å
$b$ = 12.9008 (7) Å
$c$ = 13.5579 (8) Å
$\alpha$ = 84.047 (5)°
$\beta$ = 77.566 (5)°
$\gamma$ = 71.684 (5)°
$V$ = 1339.45 (13) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 1.41 mm$^{-1}$
$T$ = 123 K
0.38 × 0.35 × 0.29 mm

### Data collection

Xcalibur (Ruby, Gemini) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2011)
$T_{min}$ = 0.904, $T_{max}$ = 1.000
9408 measured reflections
9408 independent reflections
8399 reflections with $I > 2\sigma(I)$

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
$S$ = 1.02
9408 reflections
374 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1$A$—H1$AA$···N1$S$ | 0.88 | 2.24 | 3.0576 (17) | 154 |
| N1$B$—H1$BA$···S1$S^{i}$ | 0.88 | 2.69 | 3.5345 (11) | 162 |
| N2$A$—H2$AA$···N2$B$ | 0.88 | 1.77 | 2.6492 (15) | 173 |
| N2$B$—H2$BB$···N2$A$ | 0.88 | 1.77 | 2.6492 (15) | 177 |

Symmetry code: (i) $x, y-1, z$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5617).

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# supporting information

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## 4-(Naphthalene-2-carboxamido)pyridin-1-ium thiocyanate–*N*-(pyridin-4-yl)naphthalene-2-carboxamide (1/1)

**Sohail Saeed, Naghmana Rashid, Ray J. Butcher, Sema Öztürk Yildirim and Rizwan Hussain**

### S1. Comment

Amides are known to play a pivital role in molecular recognition, being important components in supramolecular chemical anion sensors technology (Saeed *et al.*, 2011, 2012). Moreover, amides have also been reported as antimicrobial agents (Priya *et al.*, 2005). A compound with the same basic skeleton as the title compound has been used in host–guest chemistry to form numerous highly crystalline adducts with a variety of common organic solvents (Toda *et al.*, 1987). The structure of the title compound has been determined to explore the effect of substituents on the structure of the title compound.

The crystal structure of the title compound (Fig. 1), $[(C_{16}H_{13}N_2O^+)(NCS^-)]$ $C_{16}H_{12}N_2O$, there are two independent molecules (A and B) in the asymmetric unit of which one is protonated (A). In both A and B the naphthyl and pyridine rings are planar with a mean deviation from the least- squares plane defined by naphthyl ring carbon atoms (C1—C10) and C1A of -0.018 (1) Å and 0.0268 (1) Å for the C1B. All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the naphthyl and pyridine ring systems is 11.33 (6) and 9.51 (6)° in molecules A and B, respectively. In the crystal, N—H···N and N—H···S intra- and intermolecular hydrogen bonds are observed (Table 1) as well as weak $\pi$ - $\pi$ stacking interactions [*Cg*1···*Cg*4 (*x*,1 + *y*,*z*] = 3.615 (1) Å, *Cg*2···*Cg*3 (*x*,-1 + *y*,*z*] = 3.593 (1) Å, *Cg*2···*Cg*5 (*x*,-1 + *y*,*z*] = 3.735 (1) Å and *Cg*3···*Cg*4 (1 + *x*,1 + *y*,*y*] = 3.686 (1) Å where *Cg*1(N2A/C12A—C16A), *Cg*2(N2B/C12B—C16B), *Cg*3(C1A—C3A/C8A—C10A), *Cg*4(C1B—C3B/C8B—C10B) and *Cg*5(C3A—C8A) are the centroids of the pyridinium and naphthalene rings](Fig. 2).

### S2. Experimental

A solution of naphthalene 2-carbonyl chloride (0.01 mol) in anhydrous acetone (80 ml) was added dropwise to a suspension of dry sodium thiocyanate (0.01 mol) in acetone (50 ml) and the reaction mixture was refluxed for 45 min. After cooling to room temperature, a solution of 4-aminopyridine (0.01 mol) in anhydrous acetone (25 ml) was added and the resulting mixture refluxed for 2 h. Hydrochloric acid (0.1 N, 400 ml) was added, and the solution was filtered.

### S3. Refinement

The crystal structure was solved from non-merohedral twin with the twin law in the reciprocal matrix of -1, 0, 0: 0, -1, 0: -1, 0, 1 and the twin component ratio of 0.357 (1)/0.643 (1). In the refinement the HKLF 5 reflection file format in *SHELXL* was used.

H atoms attached to C atoms were positioned with idealized geometry and were refined isotropic with $U_{eq}$(H) set to 1.2 times of the $U_{eq}$(C) using a riding model with C—H = 0.95 Å. H atoms attached to N atoms were positioned with idealized geometry and were refined isotropically with $U_{eq}$(H) set to 1.2 times of $U_{eq}$(N) using a riding model with N—H = 0.88 Å. The H atom attached to the pyridine-N atom is disordered over two sites in a ratio of 0.61 (3):0.39 (3).

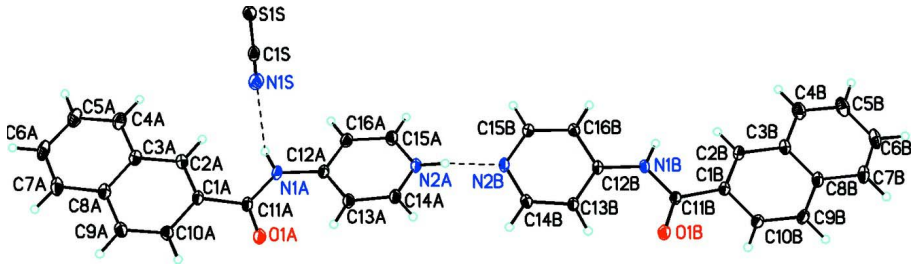


**Figure 1**

The molecular structure of the title compound with labeling and displacement ellipsoids drawn at the 30% probability level. The dashed line denotes the hydrogen bond between the neutral and protonated moieties.

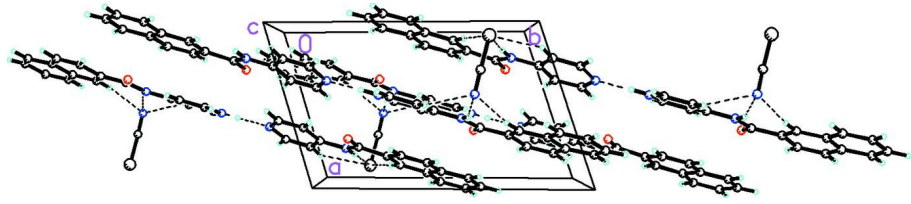


**Figure 2**

The molecular packing of the title compound, viewed down *c* axis. The dashed lines denote hydrogen bonds.

### 4-(Naphthalene-2-carboxamido)pyridin-1-ium thiocyanate– *N*-(pyridin-4-yl)naphthalene-2-carboxamide (1/1)

*Crystal data*

$C_{16}H_{13}N_2O^+·NCS^-·C_{16}H_{12}N_2O$
$M_r = 555.64$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.2667\ (4)$ Å
$b = 12.9008\ (7)$ Å
$c = 13.5579\ (8)$ Å
$\alpha = 84.047\ (5)°$
$\beta = 77.566\ (5)°$
$\gamma = 71.684\ (5)°$
$V = 1339.45\ (13)$ Å$^3$
$Z = 2$
$F(000) = 580$
$D_x = 1.378$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 3931 reflections
$\theta = 3.3–75.5°$
$\mu = 1.41$ mm$^{-1}$
$T = 123$ K
Prism, colorless
0.38 × 0.35 × 0.29 mm

*Data collection*

Xcalibur (Ruby, Gemini) diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan [*CrysAlis RED* (Agilent, 2011) based on expressions derived from Clark & Reid (1995)]
$T_{min} = 0.904$, $T_{max} = 1.000$
9408 measured reflections
9408 independent reflections
8399 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 76.0°$, $\theta_{min} = 3.3°$
$h = -10\rightarrow10$
$k = -16\rightarrow16$
$l = -12\rightarrow16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.137$
$S = 1.02$
9408 reflections
374 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
$w = 1/[\sigma^2(F_o^2) + (0.0899P)^2 + 0.2899P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ e Å$^{-3}$

*Special details*

**Experimental**. CrysAlis RED, (Agilent, 2011) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm (Clark & Reid, 1995).

**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) |
|---|---|---|---|---|---|
| S1S | 0.08493 (5) | 0.80651 (3) | 0.97576 (3) | 0.03396 (11) | |
| N1S | 0.4395 (2) | 0.68666 (11) | 0.95127 (10) | 0.0399 (3) | |
| C1S | 0.2927 (2) | 0.73545 (11) | 0.96208 (10) | 0.0315 (3) | |
| O1A | 0.66272 (14) | 0.64980 (8) | 0.56439 (7) | 0.0319 (2) | |
| N1A | 0.58653 (14) | 0.61521 (8) | 0.73364 (8) | 0.0240 (2) | |
| H1AA | 0.5779 | 0.6394 | 0.7935 | 0.034 (4)* | |
| N2A | 0.44101 (14) | 0.33301 (8) | 0.74169 (8) | 0.0245 (2) | |
| H2AA | 0.4093 | 0.2735 | 0.7445 | 0.029* | 0.61 (3) |
| C1A | 0.68983 (17) | 0.77349 (10) | 0.67251 (10) | 0.0245 (3) | |
| C2A | 0.66122 (17) | 0.81223 (11) | 0.76696 (10) | 0.0263 (3) | |
| H2AB | 0.6133 | 0.7745 | 0.8239 | 0.032* | |
| C3A | 0.70214 (18) | 0.90814 (11) | 0.78109 (11) | 0.0276 (3) | |
| C4A | 0.6715 (2) | 0.94936 (12) | 0.87865 (12) | 0.0358 (3) | |
| H4AA | 0.6243 | 0.9120 | 0.9362 | 0.043* | |
| C5A | 0.7098 (2) | 1.04289 (13) | 0.89019 (14) | 0.0412 (4) | |
| H5AA | 0.6893 | 1.0700 | 0.9557 | 0.049* | |
| C6A | 0.7797 (2) | 1.09912 (12) | 0.80502 (14) | 0.0401 (4) | |
| H6AA | 0.8058 | 1.1638 | 0.8138 | 0.048* | |
| C7A | 0.81019 (19) | 1.06170 (12) | 0.71064 (13) | 0.0356 (3) | |
| H7AA | 0.8561 | 1.1009 | 0.6541 | 0.043* | |
| C8A | 0.77348 (17) | 0.96363 (11) | 0.69583 (11) | 0.0277 (3) | |
| C9A | 0.80472 (18) | 0.92153 (11) | 0.59920 (11) | 0.0295 (3) | |
| H9AA | 0.8545 | 0.9577 | 0.5416 | 0.035* | |
| C10A | 0.76400 (17) | 0.82912 (10) | 0.58763 (10) | 0.0268 (3) | |
| H10A | 0.7856 | 0.8019 | 0.5221 | 0.032* | |
| C11A | 0.64576 (16) | 0.67468 (10) | 0.65080 (10) | 0.0239 (3) | |
| C12A | 0.53898 (16) | 0.52088 (10) | 0.73240 (10) | 0.0218 (2) | |
| C13A | 0.53009 (17) | 0.47683 (10) | 0.64435 (10) | 0.0242 (3) | |

| | | | | | |
|---|---|---|---|---|---|
| H13A | 0.5583 | 0.5106 | 0.5800 | 0.029* | |
| C14A | 0.47968 (17) | 0.38366 (10) | 0.65253 (10) | 0.0256 (3) | |
| H14A | 0.4721 | 0.3544 | 0.5928 | 0.031* | |
| C15A | 0.45085 (19) | 0.37358 (11) | 0.82714 (10) | 0.0281 (3) | |
| H15A | 0.4240 | 0.3371 | 0.8902 | 0.034* | |
| C16A | 0.49897 (18) | 0.46677 (11) | 0.82490 (10) | 0.0273 (3) | |
| H16A | 0.5050 | 0.4942 | 0.8859 | 0.033* | |
| O1B | 0.27783 (14) | −0.15477 (8) | 0.54771 (7) | 0.0330 (2) | |
| N1B | 0.21021 (14) | −0.12707 (8) | 0.71690 (8) | 0.0225 (2) | |
| H1BA | 0.1676 | −0.1533 | 0.7758 | 0.034 (4)* | |
| N2B | 0.35165 (14) | 0.15425 (8) | 0.73472 (8) | 0.0242 (2) | |
| H2BB | 0.3810 | 0.2132 | 0.7397 | 0.029* | 0.39 (3) |
| C1B | 0.16169 (16) | −0.28346 (10) | 0.65252 (10) | 0.0221 (2) | |
| C2B | 0.10170 (17) | −0.32263 (10) | 0.74599 (10) | 0.0242 (3) | |
| H2BA | 0.0943 | −0.2846 | 0.8040 | 0.029* | |
| C3B | 0.05008 (17) | −0.41902 (10) | 0.75834 (10) | 0.0255 (3) | |
| C4B | −0.0151 (2) | −0.45925 (13) | 0.85460 (12) | 0.0357 (3) | |
| H4BA | −0.0282 | −0.4202 | 0.9129 | 0.043* | |
| C5B | −0.0597 (2) | −0.55389 (13) | 0.86471 (13) | 0.0401 (4) | |
| H5BA | −0.1037 | −0.5800 | 0.9298 | 0.048* | |
| C6B | −0.0404 (2) | −0.61291 (12) | 0.77879 (14) | 0.0376 (4) | |
| H6BA | −0.0690 | −0.6795 | 0.7867 | 0.045* | |
| C7B | 0.01863 (19) | −0.57544 (11) | 0.68477 (12) | 0.0322 (3) | |
| H7BA | 0.0299 | −0.6157 | 0.6275 | 0.039* | |
| C8B | 0.06374 (16) | −0.47632 (10) | 0.67138 (11) | 0.0257 (3) | |
| C9B | 0.12318 (18) | −0.43331 (11) | 0.57536 (11) | 0.0277 (3) | |
| H9BA | 0.1309 | −0.4701 | 0.5166 | 0.033* | |
| C10B | 0.16984 (17) | −0.33960 (10) | 0.56551 (10) | 0.0255 (3) | |
| H10B | 0.2081 | −0.3116 | 0.5001 | 0.031* | |
| C11B | 0.22166 (16) | −0.18353 (10) | 0.63304 (9) | 0.0221 (2) | |
| C12B | 0.25886 (16) | −0.03295 (10) | 0.71828 (10) | 0.0212 (2) | |
| C13B | 0.34762 (17) | 0.01249 (10) | 0.63456 (10) | 0.0239 (3) | |
| H13B | 0.3773 | −0.0193 | 0.5705 | 0.029* | |
| C14B | 0.39169 (17) | 0.10469 (11) | 0.64657 (10) | 0.0259 (3) | |
| H14B | 0.4536 | 0.1345 | 0.5894 | 0.031* | |
| C15B | 0.26547 (18) | 0.11074 (11) | 0.81480 (10) | 0.0273 (3) | |
| H15B | 0.2363 | 0.1450 | 0.8777 | 0.033* | |
| C16B | 0.21700 (18) | 0.01874 (11) | 0.81046 (10) | 0.0267 (3) | |
| H16B | 0.1560 | −0.0094 | 0.8692 | 0.032* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1S | 0.0457 (2) | 0.03312 (19) | 0.02666 (18) | −0.02096 (15) | 0.00028 (14) | −0.00364 (13) |
| N1S | 0.0512 (9) | 0.0364 (7) | 0.0346 (7) | −0.0163 (6) | −0.0070 (6) | −0.0049 (5) |
| C1S | 0.0538 (10) | 0.0279 (6) | 0.0200 (6) | −0.0233 (7) | −0.0048 (6) | −0.0025 (5) |
| O1A | 0.0463 (6) | 0.0275 (5) | 0.0268 (5) | −0.0190 (4) | −0.0050 (4) | −0.0023 (4) |
| N1A | 0.0306 (6) | 0.0186 (5) | 0.0273 (5) | −0.0111 (4) | −0.0076 (4) | −0.0040 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2A | 0.0276 (5) | 0.0188 (5) | 0.0303 (6) | −0.0107 (4) | −0.0064 (4) | −0.0020 (4) |
| C1A | 0.0235 (6) | 0.0179 (6) | 0.0335 (7) | −0.0069 (5) | −0.0073 (5) | −0.0014 (5) |
| C2A | 0.0284 (6) | 0.0225 (6) | 0.0302 (7) | −0.0107 (5) | −0.0066 (5) | 0.0005 (5) |
| C3A | 0.0281 (6) | 0.0230 (6) | 0.0340 (7) | −0.0076 (5) | −0.0105 (5) | −0.0018 (5) |
| C4A | 0.0432 (8) | 0.0308 (7) | 0.0394 (8) | −0.0149 (6) | −0.0129 (7) | −0.0057 (6) |
| C5A | 0.0468 (9) | 0.0340 (8) | 0.0512 (10) | −0.0151 (7) | −0.0186 (7) | −0.0111 (7) |
| C6A | 0.0384 (8) | 0.0248 (7) | 0.0664 (11) | −0.0117 (6) | −0.0223 (7) | −0.0106 (7) |
| C7A | 0.0323 (7) | 0.0240 (6) | 0.0549 (9) | −0.0119 (6) | −0.0134 (7) | 0.0001 (6) |
| C8A | 0.0238 (6) | 0.0228 (6) | 0.0384 (7) | −0.0070 (5) | −0.0103 (5) | 0.0000 (5) |
| C9A | 0.0298 (7) | 0.0243 (6) | 0.0349 (7) | −0.0106 (5) | −0.0061 (6) | 0.0038 (5) |
| C10A | 0.0282 (6) | 0.0224 (6) | 0.0294 (7) | −0.0078 (5) | −0.0053 (5) | −0.0002 (5) |
| C11A | 0.0233 (6) | 0.0163 (5) | 0.0332 (7) | −0.0068 (5) | −0.0068 (5) | −0.0010 (5) |
| C12A | 0.0216 (6) | 0.0171 (5) | 0.0280 (6) | −0.0064 (4) | −0.0059 (5) | −0.0022 (5) |
| C13A | 0.0281 (6) | 0.0229 (6) | 0.0255 (6) | −0.0119 (5) | −0.0065 (5) | −0.0017 (5) |
| C14A | 0.0303 (6) | 0.0230 (6) | 0.0276 (6) | −0.0121 (5) | −0.0067 (5) | −0.0038 (5) |
| C15A | 0.0361 (7) | 0.0245 (6) | 0.0266 (6) | −0.0132 (5) | −0.0073 (5) | 0.0010 (5) |
| C16A | 0.0363 (7) | 0.0251 (6) | 0.0235 (6) | −0.0122 (5) | −0.0063 (5) | −0.0032 (5) |
| O1B | 0.0489 (6) | 0.0308 (5) | 0.0251 (5) | −0.0232 (4) | −0.0024 (4) | −0.0017 (4) |
| N1B | 0.0290 (5) | 0.0188 (5) | 0.0229 (5) | −0.0128 (4) | −0.0041 (4) | 0.0003 (4) |
| N2B | 0.0276 (5) | 0.0176 (5) | 0.0303 (6) | −0.0109 (4) | −0.0051 (4) | −0.0017 (4) |
| C1B | 0.0224 (6) | 0.0181 (6) | 0.0275 (6) | −0.0068 (4) | −0.0072 (5) | −0.0018 (5) |
| C2B | 0.0292 (6) | 0.0216 (6) | 0.0259 (6) | −0.0110 (5) | −0.0085 (5) | −0.0018 (5) |
| C3B | 0.0277 (6) | 0.0217 (6) | 0.0308 (7) | −0.0109 (5) | −0.0098 (5) | 0.0025 (5) |
| C4B | 0.0457 (8) | 0.0344 (7) | 0.0343 (8) | −0.0219 (6) | −0.0118 (6) | 0.0063 (6) |
| C5B | 0.0481 (9) | 0.0370 (8) | 0.0442 (9) | −0.0261 (7) | −0.0158 (7) | 0.0148 (7) |
| C6B | 0.0366 (8) | 0.0232 (6) | 0.0605 (10) | −0.0167 (6) | −0.0187 (7) | 0.0096 (6) |
| C7B | 0.0310 (7) | 0.0212 (6) | 0.0495 (8) | −0.0102 (5) | −0.0143 (6) | −0.0027 (6) |
| C8B | 0.0229 (6) | 0.0186 (6) | 0.0376 (7) | −0.0064 (5) | −0.0098 (5) | −0.0014 (5) |
| C9B | 0.0297 (7) | 0.0238 (6) | 0.0317 (7) | −0.0083 (5) | −0.0065 (5) | −0.0079 (5) |
| C10B | 0.0288 (6) | 0.0232 (6) | 0.0258 (6) | −0.0088 (5) | −0.0049 (5) | −0.0040 (5) |
| C11B | 0.0249 (6) | 0.0194 (6) | 0.0238 (6) | −0.0088 (5) | −0.0052 (5) | −0.0011 (5) |
| C12B | 0.0225 (6) | 0.0175 (5) | 0.0259 (6) | −0.0077 (4) | −0.0072 (5) | −0.0002 (4) |
| C13B | 0.0290 (6) | 0.0223 (6) | 0.0234 (6) | −0.0124 (5) | −0.0041 (5) | −0.0014 (5) |
| C14B | 0.0287 (6) | 0.0237 (6) | 0.0276 (6) | −0.0128 (5) | −0.0040 (5) | 0.0007 (5) |
| C15B | 0.0320 (7) | 0.0244 (6) | 0.0280 (7) | −0.0133 (5) | −0.0015 (5) | −0.0059 (5) |
| C16B | 0.0314 (7) | 0.0255 (6) | 0.0252 (6) | −0.0145 (5) | −0.0007 (5) | −0.0013 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1S—C1S | 1.6530 (17) | C16A—H16A | 0.9500 |
| N1S—C1S | 1.163 (2) | O1B—C11B | 1.2163 (16) |
| O1A—C11A | 1.2141 (17) | N1B—C11B | 1.3836 (16) |
| N1A—C11A | 1.3822 (17) | N1B—C12B | 1.3969 (15) |
| N1A—C12A | 1.3941 (15) | N1B—H1BA | 0.8800 |
| N1A—H1AA | 0.8800 | N2B—C15B | 1.3392 (17) |
| N2A—C14A | 1.3384 (17) | N2B—C14B | 1.3424 (17) |
| N2A—C15A | 1.3476 (17) | N2B—H2BB | 0.8800 |
| N2A—H2AA | 0.8800 | C1B—C2B | 1.3642 (18) |

| | | | |
|---|---|---|---|
| C1A—C2A | 1.3688 (19) | C1B—C10B | 1.4258 (17) |
| C1A—C10A | 1.4201 (19) | C1B—C11B | 1.5007 (17) |
| C1A—C11A | 1.5035 (17) | C2B—C3B | 1.4199 (17) |
| C2A—C3A | 1.4220 (18) | C2B—H2BA | 0.9500 |
| C2A—H2AB | 0.9500 | C3B—C4B | 1.413 (2) |
| C3A—C8A | 1.413 (2) | C3B—C8B | 1.4213 (19) |
| C3A—C4A | 1.419 (2) | C4B—C5B | 1.368 (2) |
| C4A—C5A | 1.372 (2) | C4B—H4BA | 0.9500 |
| C4A—H4AA | 0.9500 | C5B—C6B | 1.411 (2) |
| C5A—C6A | 1.413 (3) | C5B—H5BA | 0.9500 |
| C5A—H5AA | 0.9500 | C6B—C7B | 1.360 (2) |
| C6A—C7A | 1.359 (2) | C6B—H6BA | 0.9500 |
| C6A—H6AA | 0.9500 | C7B—C8B | 1.4247 (18) |
| C7A—C8A | 1.4330 (19) | C7B—H7BA | 0.9500 |
| C7A—H7AA | 0.9500 | C8B—C9B | 1.411 (2) |
| C8A—C9A | 1.411 (2) | C9B—C10B | 1.3659 (18) |
| C9A—C10A | 1.3685 (19) | C9B—H9BA | 0.9500 |
| C9A—H9AA | 0.9500 | C10B—H10B | 0.9500 |
| C10A—H10A | 0.9500 | C12B—C13B | 1.3943 (17) |
| C12A—C16A | 1.3981 (18) | C12B—C16B | 1.4029 (18) |
| C12A—C13A | 1.3993 (17) | C13B—C14B | 1.3828 (17) |
| C13A—C14A | 1.3777 (17) | C13B—H13B | 0.9500 |
| C13A—H13A | 0.9500 | C14B—H14B | 0.9500 |
| C14A—H14A | 0.9500 | C15B—C16B | 1.3770 (18) |
| C15A—C16A | 1.3757 (18) | C15B—H15B | 0.9500 |
| C15A—H15A | 0.9500 | C16B—H16B | 0.9500 |

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|---|---|---|---|
| N1S—C1S—S1S | 178.91 (14) | C11B—N1B—C12B | 127.11 (11) |
| C11A—N1A—C12A | 126.84 (11) | C11B—N1B—H1BA | 116.4 |
| C11A—N1A—H1AA | 116.6 | C12B—N1B—H1BA | 116.4 |
| C12A—N1A—H1AA | 116.6 | C15B—N2B—C14B | 117.55 (11) |
| C14A—N2A—C15A | 119.63 (11) | C15B—N2B—H2BB | 121.2 |
| C14A—N2A—H2AA | 120.2 | C14B—N2B—H2BB | 121.2 |
| C15A—N2A—H2AA | 120.2 | C2B—C1B—C10B | 119.21 (11) |
| C2A—C1A—C10A | 119.38 (12) | C2B—C1B—C11B | 124.68 (11) |
| C2A—C1A—C11A | 124.32 (12) | C10B—C1B—C11B | 116.11 (11) |
| C10A—C1A—C11A | 116.29 (12) | C1B—C2B—C3B | 121.33 (12) |
| C1A—C2A—C3A | 121.01 (12) | C1B—C2B—H2BA | 119.3 |
| C1A—C2A—H2AB | 119.5 | C3B—C2B—H2BA | 119.3 |
| C3A—C2A—H2AB | 119.5 | C4B—C3B—C2B | 121.82 (12) |
| C8A—C3A—C4A | 119.61 (13) | C4B—C3B—C8B | 119.12 (12) |
| C8A—C3A—C2A | 119.03 (12) | C2B—C3B—C8B | 119.06 (12) |
| C4A—C3A—C2A | 121.36 (13) | C5B—C4B—C3B | 120.68 (15) |
| C5A—C4A—C3A | 120.27 (15) | C5B—C4B—H4BA | 119.7 |
| C5A—C4A—H4AA | 119.9 | C3B—C4B—H4BA | 119.7 |
| C3A—C4A—H4AA | 119.9 | C4B—C5B—C6B | 120.25 (15) |
| C4A—C5A—C6A | 120.28 (15) | C4B—C5B—H5BA | 119.9 |
| C4A—C5A—H5AA | 119.9 | C6B—C5B—H5BA | 119.9 |

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|---|---|---|---|
| C6A—C5A—H5AA | 119.9 | C7B—C6B—C5B | 120.65 (13) |
| C7A—C6A—C5A | 120.78 (13) | C7B—C6B—H6BA | 119.7 |
| C7A—C6A—H6AA | 119.6 | C5B—C6B—H6BA | 119.7 |
| C5A—C6A—H6AA | 119.6 | C6B—C7B—C8B | 120.55 (14) |
| C6A—C7A—C8A | 120.49 (15) | C6B—C7B—H7BA | 119.7 |
| C6A—C7A—H7AA | 119.8 | C8B—C7B—H7BA | 119.7 |
| C8A—C7A—H7AA | 119.8 | C9B—C8B—C3B | 118.65 (12) |
| C9A—C8A—C3A | 119.19 (12) | C9B—C8B—C7B | 122.67 (13) |
| C9A—C8A—C7A | 122.24 (14) | C3B—C8B—C7B | 118.68 (13) |
| C3A—C8A—C7A | 118.57 (13) | C10B—C9B—C8B | 121.09 (12) |
| C10A—C9A—C8A | 120.63 (13) | C10B—C9B—H9BA | 119.5 |
| C10A—C9A—H9AA | 119.7 | C8B—C9B—H9BA | 119.5 |
| C8A—C9A—H9AA | 119.7 | C9B—C10B—C1B | 120.62 (12) |
| C9A—C10A—C1A | 120.75 (13) | C9B—C10B—H10B | 119.7 |
| C9A—C10A—H10A | 119.6 | C1B—C10B—H10B | 119.7 |
| C1A—C10A—H10A | 119.6 | O1B—C11B—N1B | 122.34 (11) |
| O1A—C11A—N1A | 122.82 (11) | O1B—C11B—C1B | 121.19 (11) |
| O1A—C11A—C1A | 120.67 (12) | N1B—C11B—C1B | 116.47 (11) |
| N1A—C11A—C1A | 116.51 (11) | C13B—C12B—N1B | 124.48 (11) |
| N1A—C12A—C16A | 117.80 (11) | C13B—C12B—C16B | 117.79 (11) |
| N1A—C12A—C13A | 124.03 (12) | N1B—C12B—C16B | 117.72 (11) |
| C16A—C12A—C13A | 118.16 (11) | C14B—C13B—C12B | 118.76 (12) |
| C14A—C13A—C12A | 118.88 (12) | C14B—C13B—H13B | 120.6 |
| C14A—C13A—H13A | 120.6 | C12B—C13B—H13B | 120.6 |
| C12A—C13A—H13A | 120.6 | N2B—C14B—C13B | 123.50 (12) |
| N2A—C14A—C13A | 122.28 (12) | N2B—C14B—H14B | 118.3 |
| N2A—C14A—H14A | 118.9 | C13B—C14B—H14B | 118.3 |
| C13A—C14A—H14A | 118.9 | N2B—C15B—C16B | 123.19 (12) |
| N2A—C15A—C16A | 121.34 (12) | N2B—C15B—H15B | 118.4 |
| N2A—C15A—H15A | 119.3 | C16B—C15B—H15B | 118.4 |
| C16A—C15A—H15A | 119.3 | C15B—C16B—C12B | 119.21 (12) |
| C15A—C16A—C12A | 119.70 (12) | C15B—C16B—H16B | 120.4 |
| C15A—C16A—H16A | 120.1 | C12B—C16B—H16B | 120.4 |
| C12A—C16A—H16A | 120.1 | | |

| | | | |
|---|---|---|---|
| C10A—C1A—C2A—C3A | 1.0 (2) | C10B—C1B—C2B—C3B | 1.06 (19) |
| C11A—C1A—C2A—C3A | −178.25 (12) | C11B—C1B—C2B—C3B | −178.88 (11) |
| C1A—C2A—C3A—C8A | 0.0 (2) | C1B—C2B—C3B—C4B | −178.85 (13) |
| C1A—C2A—C3A—C4A | 179.46 (13) | C1B—C2B—C3B—C8B | 0.88 (19) |
| C8A—C3A—C4A—C5A | 0.4 (2) | C2B—C3B—C4B—C5B | −178.25 (14) |
| C2A—C3A—C4A—C5A | −179.09 (14) | C8B—C3B—C4B—C5B | 2.0 (2) |
| C3A—C4A—C5A—C6A | 0.1 (2) | C3B—C4B—C5B—C6B | 0.2 (2) |
| C4A—C5A—C6A—C7A | 0.0 (2) | C4B—C5B—C6B—C7B | −1.5 (2) |
| C5A—C6A—C7A—C8A | −0.6 (2) | C5B—C6B—C7B—C8B | 0.6 (2) |
| C4A—C3A—C8A—C9A | 179.51 (13) | C4B—C3B—C8B—C9B | 177.77 (13) |
| C2A—C3A—C8A—C9A | −0.98 (19) | C2B—C3B—C8B—C9B | −1.97 (18) |
| C4A—C3A—C8A—C7A | −1.1 (2) | C4B—C3B—C8B—C7B | −2.89 (19) |
| C2A—C3A—C8A—C7A | 178.46 (12) | C2B—C3B—C8B—C7B | 177.37 (12) |

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|---|---|---|---|
| C6A—C7A—C8A—C9A | −179.41 (13) | C6B—C7B—C8B—C9B | −179.07 (13) |
| C6A—C7A—C8A—C3A | 1.2 (2) | C6B—C7B—C8B—C3B | 1.6 (2) |
| C3A—C8A—C9A—C10A | 1.1 (2) | C3B—C8B—C9B—C10B | 1.13 (19) |
| C7A—C8A—C9A—C10A | −178.35 (13) | C7B—C8B—C9B—C10B | −178.18 (13) |
| C8A—C9A—C10A—C1A | −0.1 (2) | C8B—C9B—C10B—C1B | 0.8 (2) |
| C2A—C1A—C10A—C9A | −0.9 (2) | C2B—C1B—C10B—C9B | −1.93 (19) |
| C11A—C1A—C10A—C9A | 178.39 (12) | C11B—C1B—C10B—C9B | 178.01 (12) |
| C12A—N1A—C11A—O1A | 0.2 (2) | C12B—N1B—C11B—O1B | −0.2 (2) |
| C12A—N1A—C11A—C1A | −179.59 (11) | C12B—N1B—C11B—C1B | 179.44 (11) |
| C2A—C1A—C11A—O1A | 174.42 (13) | C2B—C1B—C11B—O1B | 178.11 (13) |
| C10A—C1A—C11A—O1A | −4.83 (18) | C10B—C1B—C11B—O1B | −1.83 (18) |
| C2A—C1A—C11A—N1A | −5.75 (19) | C2B—C1B—C11B—N1B | −1.58 (18) |
| C10A—C1A—C11A—N1A | 175.00 (11) | C10B—C1B—C11B—N1B | 178.48 (11) |
| C11A—N1A—C12A—C16A | 173.68 (12) | C11B—N1B—C12B—C13B | −8.2 (2) |
| C11A—N1A—C12A—C13A | −5.9 (2) | C11B—N1B—C12B—C16B | 172.51 (12) |
| N1A—C12A—C13A—C14A | −179.35 (12) | N1B—C12B—C13B—C14B | −178.54 (12) |
| C16A—C12A—C13A—C14A | 1.12 (18) | C16B—C12B—C13B—C14B | 0.80 (18) |
| C15A—N2A—C14A—C13A | 0.1 (2) | C15B—N2B—C14B—C13B | 0.5 (2) |
| C12A—C13A—C14A—N2A | −0.9 (2) | C12B—C13B—C14B—N2B | −0.9 (2) |
| C14A—N2A—C15A—C16A | 0.4 (2) | C14B—N2B—C15B—C16B | 0.0 (2) |
| N2A—C15A—C16A—C12A | −0.1 (2) | N2B—C15B—C16B—C12B | 0.0 (2) |
| N1A—C12A—C16A—C15A | 179.78 (12) | C13B—C12B—C16B—C15B | −0.39 (19) |
| C13A—C12A—C16A—C15A | −0.65 (19) | N1B—C12B—C16B—C15B | 179.00 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H1*AA*···N1*S* | 0.88 | 2.24 | 3.0576 (17) | 154 |
| N1*B*—H1*BA*···S1*S*$^{i}$ | 0.88 | 2.69 | 3.5345 (11) | 162 |
| N2*A*—H2*AA*···N2*B* | 0.88 | 1.77 | 2.6492 (15) | 173 |
| N2*B*—H2*BB*···N2*A* | 0.88 | 1.77 | 2.6492 (15) | 177 |

Symmetry code: (i) *x*, *y*−1, *z*.