# organic compounds

# N-(Diphenylcarbamothioyl)-3-methyl-benzamide

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The synthesis of the title compound, $C_{21}H_{18}N_2OS$, involves the reaction of 3-methylbenzoyl chloride with potassium thiocyanate in dry acetone followed by condensation of the 3-methylbenzoyl isothiocyanate with diphenylamine. The carbonyl [C—O = 1.215 (2) Å] and thiocarbonyl [C—S = 1.6721 (17) Å] distances indicate that these correspond to double bonds. The short C—N bonds at the center of the molecule reveal the effects of resonance in this part of the molecule. The conformation of the molecule with respect to the thiocarbonyl and carbonyl groups is twisted. The 3-methylphenyl and two phenyl rings are also twisted, with dihedral angles of 75.67 (9) and 14.91 (9)°. The phenyl rings are rotated out of the mean plane of the N—C—S—N atoms by 66.87 (8) and 78.40 (9)°. Pairs of molecules are linked into centrosymmetric dimers *via* intermolecular N—H···S interactions and a C—H···O link also occurs. The dimers are stacked along the *a* axis.

## Related literature

For synthesis, see: Özer *et al.* (2009); Mansuroğlu *et al.* (2008); Uğur *et al.* (2006); Arslan *et al.* (2003*a*, and references therein). For general background, see: Koch (2001); El Aamrani *et al.* (1998, 1999). For related compounds, see: Arslan *et al.* (2003*b*,*c*, 2004); Khawar Rauf *et al.* (2009*a*,*b*,*c*,*d*).
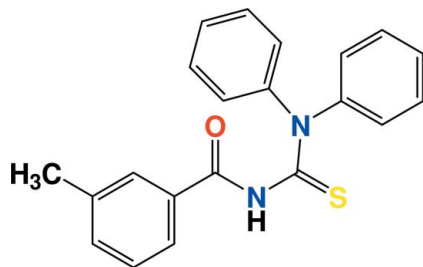


## Experimental

### Crystal data

$C_{21}H_{18}N_2OS$
$M_r = 346.43$
Orthorhombic, $Pbca$
$a = 10.6928 (12)$ Å
$b = 16.7647 (17)$ Å
$c = 19.865 (2)$ Å

$V = 3561.0 (6)$ Å$^3$
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.19$ mm$^{-1}$
$T = 120 (2)$ K
$0.41 \times 0.23 \times 0.08$ mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.925$, $T_{max} = 0.985$

29957 measured reflections
4241 independent reflections
2955 reflections with $I > 2\sigma(I)$
$R_{int} = 0.098$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.101$
$S = 0.95$
4241 reflections
231 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.38$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···S1[i] | 0.899 (14) | 2.525 (14) | 3.4123 (15) | 169.3 (13) |
| C5—H5B···O1[ii] | 0.98 | 2.59 | 3.461 (2) | 148 |

Symmetry codes: (i) $-x + 1, -y + 2, -z + 1$; (ii) $-x + 2, -y + 2, -z + 1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2712).

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# supporting information

## *N*-(Diphenylcarbamothioyl)-3-methylbenzamide

**Gün Binzet, Ulrich Flörke, Nevzat Külcü and Hakan Arslan**

### S1. Comment

Thioureas are basic and excellent ligands for the transition group metals, so many derivatives have been prepared for use in the liquid–liquid extraction and separation of some transition metal ions (Koch, 2001). Especially, *N*-benzoylthiourea derivatives have been successfully used in the extraction of copper(II) and gold(III) ions (El Aamrani *et al.*, 1998, 1999).

Recently, we have focused on the synthesis, characterization, crystal structure, thermal behavior and antimicrobial activity of new thiourea derivatives (Özer *et al.*, 2009; Mansuroğlu *et al.*, 2008; Uğur *et al.*, 2006; Arslan *et al.*, 2003*c*, and references therein). In the present study, we have synthesized and characterized a new thiourea derivative, *N*-(diphenylcarbamothioyl)-3-methylbenzamide, (I). The molecular structure of the title compound is depicted in Fig. 1.

The carbonyl (C1—O1 = 1.215 (2) Å) and thiocarbonyl (C9—S1 = 1.6721 (17) Å) distances indicates that these correspond to double bonds and are comparable to those observed for *N*′-(4-chlorobenzoyl)-*N*,*N*-diphenylthiourea [1.213 (3) Å for C—O and 1.664 (2) Å for C—S] (Arslan *et al.*, 2003*a*), 1-(4-chloro-benzoyl)-3-naphthalen-1-yl-thiourea [1.224 (2) Å for C—O and 1.6696 (17) Å for C—S] (Arslan *et al.*, 2003*b*), 1-(2-chloro-benzoyl)-3-*p*-tolyl-thiourea [1.216 (2) Å for C—O and 1.666 (2) Å for C—S] (Arslan *et al.*, 2004), 1-(4-chlorobenzoyl)-3-(2,4,6-trichlorophenyl)thiourea [1.229 (3) Å for C—O and 1.666 (2) Å for C—S] (Khawar Rauf *et al.*, 2009*b*).

The C—N bond distances [C1—N1 = 1.395 (2) Å, C9—N1 = 1.396 (2) Å and C9—N2 = 1.344 (2) Å] observed in the title compound are consistent with those reported for the other thiourea derivatives (1-(3-chlorophenyl)-3-(2,6-dichloro-benzoyl)thiourea (Khawar Rauf *et al.*, 2009*d*), 1-(3-chlorobenzoyl)-3-(2,3-dimethylphenyl)thiourea (Khawar Rauf *et al.*, 2009*c*), 1-(2,6-dichlorobenzoyl)-3-(2,3,5,6-tetrachlorophenyl)thiourea (Khawar Rauf *et al.*, 2009*a*), suggesting a partial double-bond character.

The conformation of the title molecule with respect to the thiocarbonyl and carbonyl moieties is twisted, as reflected by the C9—N1—C1—O1 and C1—N1—C9—N2 torsion angles of 0.1 (3)° and -56.1 (2)°, respectively. The 3-methyl-phenyl and phenyl rings (C10–C15 and C16–C21 rings) are also twisted with dihedral angles of 75.67 (9)° and 14.91 (9)°, respectively.

The atom N2 is *sp*²-hybridized, because of the sum of the angles around atom N2 is 359.91 (13)°. The phenyl rings are rotated out of the mean plane of the N1–C9–S1–N2 atoms by 66.87 (8)° (C10–C15 ring) and 78.40 (9)° (C16–C21 ring). In addition, the dihedral angle between C10–C15 ring and C16–C21 ring is 87.81 (9)°.

As can be seen from the packing diagram (Fig. 2), intermolecular N—H⋯S hydrogen bond (Table 1) links the molecules into dimers, which are stacked along the *a* axis. The C—H⋯O intermolecular contact is also listed in Table 1.

### S2. Experimental

The title compound was prepared with a procedure similar to that reported in the literature (Arslan *et al.*, 2003*c*). A solution of 3-methylbenzoyl chloride (0.01 mol) in acetone (50 ml) was added dropwise to a suspension of potassium thiocyanate (0.01 mol) in acetone (30 ml). The reaction mixture was heated under reflux for 30 min, and then cooled to

room temperature. A solution of diphenylamine (0.01 mol) in acetone (10 ml) was added and the resulting mixture was stirred for 2 h. Hydrochloric acid (0.1 N, 300 ml) was added to the solution, which was then filtered. The solid product was washed with water and purifed by recrystalization from an ethanol:dichloromethane mixture (1:2). Analysis calculated for $C_{21}H_{18}N_2OS$: C 72.8, H 5.2, N 8.1%. Found: C 72.6, H 5.2, N 8.0%.

## S3. Refinement

H atoms bound to C atoms were placed geometrically and allowed to ride during subsequent refinement with C—H = 0.95 and 0.98 Å and $U_{iso}(H) = 1.2$ or $1.5 U_{eq}(C)$. The nitrogen-bound H atom was located in a difference Fourier map and refined freely.
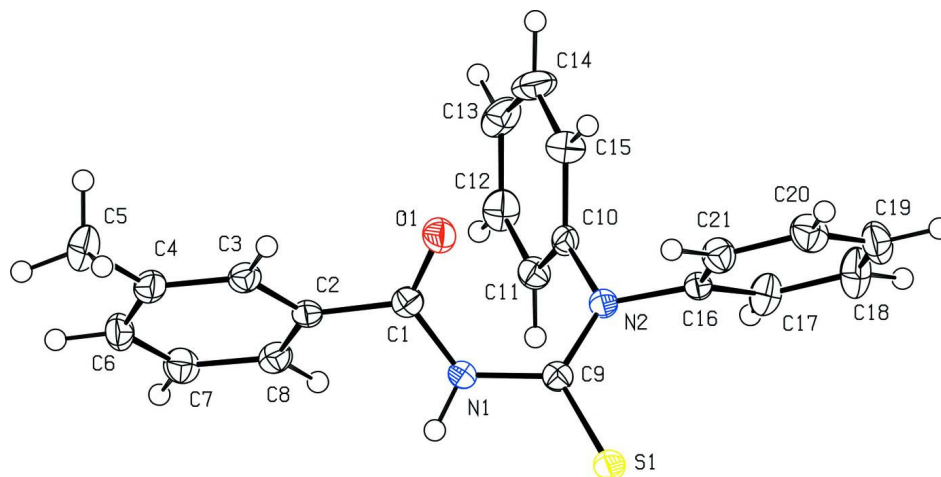


**Figure 1**

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.
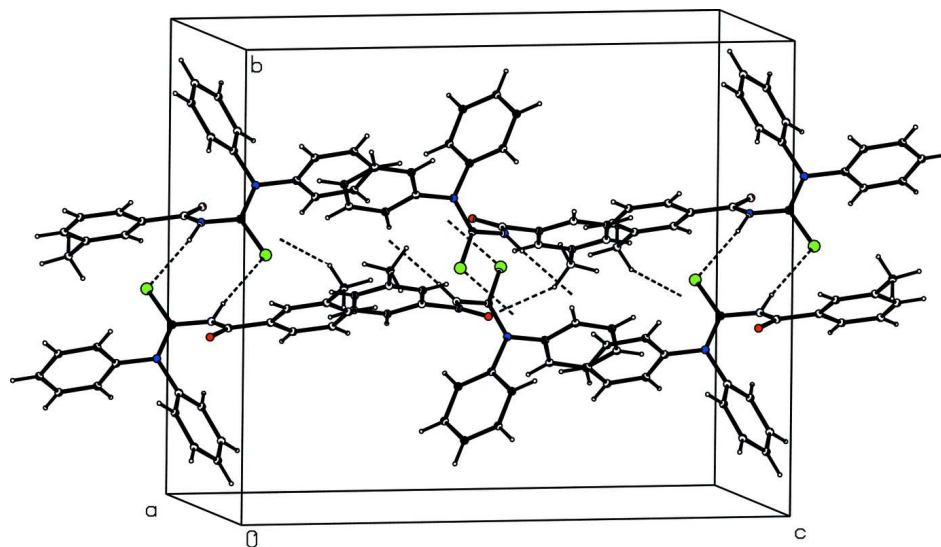


**Figure 2**

A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

## *N*-(Diphenylcarbamothioyl)-3-methylbenzamide

*Crystal data*

| | |
|---|---|
| $C_{21}H_{18}N_2OS$ | $F(000) = 1456$ |
| $M_r = 346.43$ | $D_x = 1.292$ Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo *Kα* radiation, $λ = 0.71073$ Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 801 reflections |
| $a = 10.6928$ (12) Å | $θ = 2.5–25.9°$ |
| $b = 16.7647$ (17) Å | $μ = 0.19$ mm$^{-1}$ |
| $c = 19.865$ (2) Å | $T = 120$ K |
| $V = 3561.0$ (6) Å$^3$ | Prism, colourless |
| $Z = 8$ | $0.41 × 0.23 × 0.08$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 29957 measured reflections |
| Radiation source: sealed tube | 4241 independent reflections |
| Graphite monochromator | 2955 reflections with $I > 2σ(I)$ |
| *φ* and *ω* scans | $R_{int} = 0.098$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2002) | $θ_{max} = 27.9°$, $θ_{min} = 2.1°$ |
| | $h = −14→14$ |
| | $k = −22→19$ |
| $T_{min} = 0.925$, $T_{max} = 0.985$ | $l = −26→26$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2σ(F^2)] = 0.047$ | Hydrogen site location: difference Fourier map |
| $wR(F^2) = 0.101$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 0.95$ | |
| 4241 reflections | $w = 1/[σ^2(F_o^2) + (0.0429P)^2]$ |
| 231 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1 restraint | $(Δ/σ)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $Δρ_{max} = 0.28$ e Å$^{-3}$ |
| | $Δρ_{min} = −0.38$ e Å$^{-3}$ |

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2sigma(F^2)$ 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 | 0.49155 (4) | 0.96594 (3) | 0.60687 (2) | 0.02357 (13) |
| O1 | 0.83581 (11) | 0.88560 (8) | 0.53805 (6) | 0.0282 (3) |
| N1 | 0.63123 (14) | 0.90441 (8) | 0.50788 (7) | 0.0194 (3) |
| H1 | 0.5892 (15) | 0.9383 (8) | 0.4811 (7) | 0.029 (5)* |
| N2 | 0.61147 (13) | 0.82693 (8) | 0.60385 (7) | 0.0198 (3) |

| | | | |
|---|---|---|---|
| C1 | 0.75893 (17) | 0.89869 (10) | 0.49441 (8) | 0.0204 (4) |
| C2 | 0.79282 (16) | 0.91117 (10) | 0.42236 (8) | 0.0201 (4) |
| C3 | 0.90683 (17) | 0.94747 (10) | 0.40766 (9) | 0.0226 (4) |
| H3A | 0.9603 | 0.9633 | 0.4434 | 0.027* |
| C4 | 0.94370 (18) | 0.96101 (10) | 0.34132 (9) | 0.0252 (4) |
| C5 | 1.0663 (2) | 1.00079 (12) | 0.32634 (10) | 0.0360 (5) |
| H5A | 1.1349 | 0.9638 | 0.3362 | 0.054* |
| H5B | 1.0750 | 1.0486 | 0.3543 | 0.054* |
| H5C | 1.0692 | 1.0159 | 0.2787 | 0.054* |
| C6 | 0.86364 (18) | 0.93615 (11) | 0.29006 (9) | 0.0283 (4) |
| H6A | 0.8863 | 0.9456 | 0.2445 | 0.034* |
| C7 | 0.75192 (18) | 0.89805 (11) | 0.30421 (8) | 0.0280 (4) |
| H7A | 0.7001 | 0.8801 | 0.2685 | 0.034* |
| C8 | 0.71531 (17) | 0.88605 (10) | 0.37039 (9) | 0.0237 (4) |
| H8A | 0.6378 | 0.8608 | 0.3801 | 0.028* |
| C9 | 0.58249 (16) | 0.89564 (10) | 0.57267 (8) | 0.0190 (4) |
| C10 | 0.65779 (16) | 0.75696 (10) | 0.56929 (8) | 0.0200 (4) |
| C11 | 0.58848 (18) | 0.72236 (10) | 0.51815 (8) | 0.0241 (4) |
| H11A | 0.5118 | 0.7455 | 0.5040 | 0.029* |
| C12 | 0.63308 (19) | 0.65307 (10) | 0.48777 (9) | 0.0307 (5) |
| H12A | 0.5872 | 0.6291 | 0.4522 | 0.037* |
| C13 | 0.7437 (2) | 0.61921 (11) | 0.50926 (10) | 0.0346 (5) |
| H13A | 0.7741 | 0.5723 | 0.4881 | 0.042* |
| C14 | 0.81025 (19) | 0.65320 (11) | 0.56141 (11) | 0.0360 (5) |
| H14A | 0.8852 | 0.6289 | 0.5768 | 0.043* |
| C15 | 0.76792 (18) | 0.72271 (11) | 0.59126 (9) | 0.0287 (4) |
| H15A | 0.8143 | 0.7467 | 0.6267 | 0.034* |
| C16 | 0.59063 (16) | 0.81692 (10) | 0.67524 (8) | 0.0191 (4) |
| C17 | 0.50108 (18) | 0.76351 (11) | 0.69656 (9) | 0.0285 (4) |
| H17A | 0.4504 | 0.7359 | 0.6649 | 0.034* |
| C18 | 0.4862 (2) | 0.75071 (12) | 0.76492 (9) | 0.0371 (5) |
| H18A | 0.4246 | 0.7143 | 0.7803 | 0.045* |
| C19 | 0.5600 (2) | 0.79033 (11) | 0.81074 (9) | 0.0342 (5) |
| H19A | 0.5486 | 0.7814 | 0.8576 | 0.041* |
| C20 | 0.65082 (19) | 0.84305 (12) | 0.78887 (9) | 0.0317 (5) |
| H20A | 0.7026 | 0.8697 | 0.8206 | 0.038* |
| C21 | 0.66613 (18) | 0.85692 (11) | 0.72048 (9) | 0.0258 (4) |
| H21A | 0.7277 | 0.8934 | 0.7050 | 0.031* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0282 (3) | 0.0204 (2) | 0.0221 (2) | 0.00679 (19) | 0.0028 (2) | 0.00050 (19) |
| O1 | 0.0228 (7) | 0.0371 (8) | 0.0248 (7) | −0.0013 (6) | −0.0014 (6) | 0.0050 (6) |
| N1 | 0.0203 (8) | 0.0184 (8) | 0.0197 (7) | 0.0027 (6) | 0.0013 (6) | 0.0037 (6) |
| N2 | 0.0226 (8) | 0.0181 (7) | 0.0186 (7) | 0.0038 (6) | 0.0025 (6) | 0.0010 (6) |
| C1 | 0.0207 (9) | 0.0157 (9) | 0.0246 (9) | −0.0010 (7) | 0.0012 (8) | −0.0003 (7) |
| C2 | 0.0237 (10) | 0.0155 (9) | 0.0210 (9) | 0.0059 (7) | 0.0022 (7) | 0.0008 (7) |

| C3 | 0.0225 (10) | 0.0206 (9) | 0.0248 (9) | 0.0027 (8) | 0.0010 (8) | −0.0007 (7) |
| C4 | 0.0290 (10) | 0.0188 (9) | 0.0278 (10) | 0.0045 (8) | 0.0068 (8) | 0.0008 (8) |
| C5 | 0.0366 (12) | 0.0390 (12) | 0.0324 (11) | −0.0055 (10) | 0.0126 (9) | −0.0003 (9) |
| C6 | 0.0384 (12) | 0.0260 (10) | 0.0204 (9) | 0.0050 (9) | 0.0079 (8) | 0.0023 (8) |
| C7 | 0.0320 (11) | 0.0291 (10) | 0.0231 (9) | 0.0031 (9) | −0.0026 (8) | −0.0032 (8) |
| C8 | 0.0221 (10) | 0.0218 (10) | 0.0271 (9) | 0.0018 (8) | 0.0018 (8) | −0.0010 (8) |
| C9 | 0.0177 (9) | 0.0196 (9) | 0.0196 (9) | −0.0018 (7) | −0.0022 (7) | −0.0003 (7) |
| C10 | 0.0228 (10) | 0.0163 (9) | 0.0208 (9) | 0.0013 (7) | 0.0055 (7) | 0.0032 (7) |
| C11 | 0.0263 (11) | 0.0214 (9) | 0.0247 (9) | 0.0010 (8) | 0.0013 (8) | 0.0025 (7) |
| C12 | 0.0389 (12) | 0.0223 (10) | 0.0310 (10) | −0.0028 (9) | 0.0046 (9) | −0.0046 (8) |
| C13 | 0.0370 (12) | 0.0186 (10) | 0.0482 (12) | 0.0033 (9) | 0.0164 (10) | −0.0037 (9) |
| C14 | 0.0250 (11) | 0.0288 (11) | 0.0542 (13) | 0.0104 (9) | 0.0037 (10) | 0.0050 (10) |
| C15 | 0.0264 (11) | 0.0275 (11) | 0.0322 (10) | 0.0034 (8) | −0.0042 (8) | 0.0007 (8) |
| C16 | 0.0222 (10) | 0.0173 (8) | 0.0178 (8) | 0.0045 (7) | 0.0025 (7) | 0.0013 (7) |
| C17 | 0.0304 (11) | 0.0289 (10) | 0.0263 (10) | −0.0072 (9) | 0.0060 (8) | −0.0042 (8) |
| C18 | 0.0515 (14) | 0.0301 (11) | 0.0297 (11) | −0.0106 (10) | 0.0151 (10) | −0.0013 (9) |
| C19 | 0.0529 (14) | 0.0291 (11) | 0.0206 (10) | 0.0054 (10) | 0.0078 (9) | 0.0029 (8) |
| C20 | 0.0378 (13) | 0.0328 (11) | 0.0243 (10) | 0.0024 (9) | −0.0047 (9) | −0.0039 (8) |
| C21 | 0.0256 (11) | 0.0259 (10) | 0.0259 (9) | −0.0025 (8) | 0.0002 (8) | 0.0013 (8) |

*Geometric parameters (Å, º)*

| S1—C9 | 1.6721 (17) | C10—C15 | 1.381 (2) |
|---|---|---|---|
| O1—C1 | 1.215 (2) | C10—C11 | 1.385 (2) |
| N1—C1 | 1.395 (2) | C11—C12 | 1.393 (2) |
| N1—C9 | 1.396 (2) | C11—H11A | 0.9500 |
| N1—H1 | 0.899 (14) | C12—C13 | 1.379 (3) |
| N2—C9 | 1.344 (2) | C12—H12A | 0.9500 |
| N2—C16 | 1.445 (2) | C13—C14 | 1.380 (3) |
| N2—C10 | 1.447 (2) | C13—H13A | 0.9500 |
| C1—C2 | 1.491 (2) | C14—C15 | 1.384 (3) |
| C2—C8 | 1.389 (2) | C14—H14A | 0.9500 |
| C2—C3 | 1.393 (2) | C15—H15A | 0.9500 |
| C3—C4 | 1.394 (2) | C16—C17 | 1.378 (2) |
| C3—H3A | 0.9500 | C16—C21 | 1.382 (2) |
| C4—C6 | 1.394 (3) | C17—C18 | 1.384 (2) |
| C4—C5 | 1.501 (3) | C17—H17A | 0.9500 |
| C5—H5A | 0.9800 | C18—C19 | 1.375 (3) |
| C5—H5B | 0.9800 | C18—H18A | 0.9500 |
| C5—H5C | 0.9800 | C19—C20 | 1.383 (3) |
| C6—C7 | 1.383 (3) | C19—H19A | 0.9500 |
| C6—H6A | 0.9500 | C20—C21 | 1.388 (2) |
| C7—C8 | 1.386 (2) | C20—H20A | 0.9500 |
| C7—H7A | 0.9500 | C21—H21A | 0.9500 |
| C8—H8A | 0.9500 | | |
| | | | |
| C1—N1—C9 | 122.34 (14) | C15—C10—C11 | 120.93 (16) |
| C1—N1—H1 | 114.8 (12) | C15—C10—N2 | 118.63 (15) |

| | | | |
|---|---|---|---|
| C9—N1—H1 | 115.1 (12) | C11—C10—N2 | 120.30 (15) |
| C9—N2—C16 | 121.07 (14) | C10—C11—C12 | 118.94 (18) |
| C9—N2—C10 | 123.73 (14) | C10—C11—H11A | 120.5 |
| C16—N2—C10 | 115.11 (13) | C12—C11—H11A | 120.5 |
| O1—C1—N1 | 122.56 (16) | C13—C12—C11 | 120.17 (18) |
| O1—C1—C2 | 123.08 (16) | C13—C12—H12A | 119.9 |
| N1—C1—C2 | 114.35 (15) | C11—C12—H12A | 119.9 |
| C8—C2—C3 | 119.91 (16) | C12—C13—C14 | 120.30 (18) |
| C8—C2—C1 | 121.72 (16) | C12—C13—H13A | 119.9 |
| C3—C2—C1 | 118.36 (16) | C14—C13—H13A | 119.9 |
| C2—C3—C4 | 121.10 (17) | C13—C14—C15 | 120.05 (19) |
| C2—C3—H3A | 119.5 | C13—C14—H14A | 120.0 |
| C4—C3—H3A | 119.5 | C15—C14—H14A | 120.0 |
| C6—C4—C3 | 117.92 (17) | C10—C15—C14 | 119.59 (18) |
| C6—C4—C5 | 121.64 (17) | C10—C15—H15A | 120.2 |
| C3—C4—C5 | 120.44 (17) | C14—C15—H15A | 120.2 |
| C4—C5—H5A | 109.5 | C17—C16—C21 | 121.43 (16) |
| C4—C5—H5B | 109.5 | C17—C16—N2 | 118.97 (15) |
| H5A—C5—H5B | 109.5 | C21—C16—N2 | 119.46 (16) |
| C4—C5—H5C | 109.5 | C16—C17—C18 | 118.82 (18) |
| H5A—C5—H5C | 109.5 | C16—C17—H17A | 120.6 |
| H5B—C5—H5C | 109.5 | C18—C17—H17A | 120.6 |
| C7—C6—C4 | 121.31 (17) | C19—C18—C17 | 120.57 (18) |
| C7—C6—H6A | 119.3 | C19—C18—H18A | 119.7 |
| C4—C6—H6A | 119.3 | C17—C18—H18A | 119.7 |
| C6—C7—C8 | 120.24 (17) | C18—C19—C20 | 120.24 (17) |
| C6—C7—H7A | 119.9 | C18—C19—H19A | 119.9 |
| C8—C7—H7A | 119.9 | C20—C19—H19A | 119.9 |
| C7—C8—C2 | 119.48 (17) | C19—C20—C21 | 119.82 (18) |
| C7—C8—H8A | 120.3 | C19—C20—H20A | 120.1 |
| C2—C8—H8A | 120.3 | C21—C20—H20A | 120.1 |
| N2—C9—N1 | 115.40 (15) | C16—C21—C20 | 119.11 (18) |
| N2—C9—S1 | 123.43 (13) | C16—C21—H21A | 120.4 |
| N1—C9—S1 | 121.15 (12) | C20—C21—H21A | 120.4 |
| | | | |
| C9—N1—C1—O1 | 0.1 (2) | C16—N2—C10—C15 | −57.2 (2) |
| C9—N1—C1—C2 | −179.01 (15) | C9—N2—C10—C11 | −57.8 (2) |
| O1—C1—C2—C8 | 146.21 (18) | C16—N2—C10—C11 | 118.63 (17) |
| N1—C1—C2—C8 | −34.7 (2) | C15—C10—C11—C12 | −1.6 (3) |
| O1—C1—C2—C3 | −32.3 (2) | N2—C10—C11—C12 | −177.31 (15) |
| N1—C1—C2—C3 | 146.80 (15) | C10—C11—C12—C13 | 0.9 (3) |
| C8—C2—C3—C4 | 1.6 (3) | C11—C12—C13—C14 | 0.7 (3) |
| C1—C2—C3—C4 | −179.81 (15) | C12—C13—C14—C15 | −1.7 (3) |
| C2—C3—C4—C6 | −0.8 (3) | C11—C10—C15—C14 | 0.6 (3) |
| C2—C3—C4—C5 | 179.50 (16) | N2—C10—C15—C14 | 176.38 (16) |
| C3—C4—C6—C7 | −1.0 (3) | C13—C14—C15—C10 | 1.1 (3) |
| C5—C4—C6—C7 | 178.66 (17) | C9—N2—C16—C17 | 112.65 (19) |
| C4—C6—C7—C8 | 2.1 (3) | C10—N2—C16—C17 | −63.9 (2) |

| C6—C7—C8—C2 | −1.2 (3) | C9—N2—C16—C21 | −71.6 (2) |
|---|---|---|---|
| C3—C2—C8—C7 | −0.6 (3) | C10—N2—C16—C21 | 111.85 (18) |
| C1—C2—C8—C7 | −179.09 (16) | C21—C16—C17—C18 | 0.7 (3) |
| C16—N2—C9—N1 | 166.64 (14) | N2—C16—C17—C18 | 176.37 (16) |
| C10—N2—C9—N1 | −17.1 (2) | C16—C17—C18—C19 | −0.3 (3) |
| C16—N2—C9—S1 | −14.9 (2) | C17—C18—C19—C20 | −0.5 (3) |
| C10—N2—C9—S1 | 161.33 (13) | C18—C19—C20—C21 | 1.0 (3) |
| C1—N1—C9—N2 | −56.1 (2) | C17—C16—C21—C20 | −0.2 (3) |
| C1—N1—C9—S1 | 125.41 (15) | N2—C16—C21—C20 | −175.88 (16) |
| C9—N2—C10—C15 | 126.34 (18) | C19—C20—C21—C16 | −0.6 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| N1—H1⋯S1[i] | 0.90 (1) | 2.53 (1) | 3.4123 (15) | 169 (1) |
| C5—H5*B*⋯O1[ii] | 0.98 | 2.59 | 3.461 (2) | 148 |

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