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# 3-[3-Methyl-4-(4-nitrobenzylidene-amino)-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-yl]-1,3-diphenylpropan-1-one dichloromethane monosolvate

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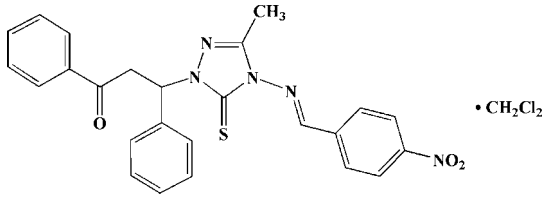
Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.004 Å; disorder in solvent or counterion; $R$ factor = 0.052; $wR$ factor = 0.159; data-to-parameter ratio = 15.7.

In the title compound, $C_{25}H_{21}N_5O_3S\cdot CH_2Cl_2$, the dichloromethane solvent molecule is disordered over four positions, with an occupancy ratio of 0.271 (3):0.3884 (18):0.298 (2):-0.0424 (15). The 1,2,4-triazole ring makes dihedral angles of 47.3 (2)/87.3 (2) and 3.6 (2)° with the phenyl and nitrophenyl rings, respectively. An intramolecular C—H···S hydrogen bond results in the formation of an almost planar six-membered ring [r.m.s. derivation = 0.0051 (2) Å]. Intermolecular C—H···O hydrogen bonding consolidates the structure.

## Related literature

For crystal structures related to 1,2,4-triazole-5(4*H*)-thione, see: Al-Tamimi *et al.* (2010); Fun *et al.* (2009); Gao *et al.* (2011); Tan *et al.* (2010); Wang *et al.* (2011); Zhao *et al.* (2010).




## Experimental

### Crystal data

$C_{25}H_{21}N_5O_3S\cdot CH_2Cl_2$ $M_r$ = 556.45
Triclinic, $P\overline{1}$
$a$ = 8.9880 (13) Å
$b$ = 11.4440 (15) Å
$c$ = 14.8604 (18) Å
$\alpha$ = 70.212 (11)°
$\beta$ = 88.973 (13)°
$\gamma$ = 67.020 (9)°
$V$ = 1312.6 (3) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.37 mm$^{-1}$
$T$ = 113 K
0.26 × 0.24 × 0.20 mm

### Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.911, $T_{max}$ = 0.931
16932 measured reflections
6203 independent reflections
4100 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.159
$S$ = 1.07
6203 reflections
396 parameters
91 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C19—H19···S1 | 0.95 | 2.43 | 3.199 (3) | 137 |
| C18—H18$B$···O1$^{i}$ | 0.98 | 2.55 | 3.443 (4) | 152 |
| C22—H22···O1$^{ii}$ | 0.95 | 2.34 | 3.202 (3) | 151 |

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *CrystalStructure* (Rigaku/MSC, 2007).

The authors gratefully acknowledge support of this project by the Key Laboratory Project of Liaoning Province (grant No. 2008S127) and by the Doctor Starting Foundation of Liaoning Province (grant No. 20071103).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZK2024).

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# supporting information

*Acta Cryst.* (2011). E**67**, o3135 [doi:10.1107/S1600536811043777]

# 3-[3-Methyl-4-(4-nitrobenzylideneamino)-5-sulfanylidene-4,5-dihydro-1*H*-1,2,4-triazol-1-yl]-1,3-diphenylpropan-1-one dichloromethane monosolvate

**Wei Wang, Wei-Min Jia, Chao Xu, Wen-Peng Wu and Qing-Lei Liu**

## S1. Comment

In continuation of our structural studies of derivatives of Mannich bases synthesized from amino heterocycles and aromatic aldehydes in our group (Wang *et al.*, 2011), we present here the crystal structure of the title compound named 3-[4-(4-Nitrobenzylideneamino)-5-thioxo-3-(4-tolyl)-4,5- dihydro-1*H*-1,2,4-triazol-1-yl]-1,3-diphenylpropan-1-one.

The bond lengths and angles in title compound are found to have normal values comparable with those reported in related 1,2,4-triazole- 5(4*H*)-thione derivatives (Al-Tamimi *et al.*, 2010; Fun *et al.*, 2009; Tan *et al.*, 2010; Wang *et al.*, 2011). An intramolecular C—H···S hydrogen bond results in the formation of a planar [an r.m.s. deviation of 0.0051 (2) Å] six-membered ring (Table 1) and the maxmium deviation of 0.0088 (2) Å for atom N4. The 1,2,4-triazole ring is almost planar with an r.m.s. deviation of 0.0039 (2) Å and the maxmium deviation of 0.0061 (2) Å for atom N1. The 1,2,4-triazole ring mean plane forms the dihedral angles of 47.3 (2), 87.3 (2) and 3.6 (2)° with two phenyl rings (C1–C6 and C10–C15) and nitrophenyl ring, respectively. Two C atoms in the 1,2,4-triazole ring show distorted C*sp*$^2$ hybridization states with the bond angles of 101.95 (16)° (N1—C16—N3), 130.44 (15)° (N3—C16—S1), 110.44 (18)° (N2—C17—N3) and 25.88 (19)° (N3—C17—C18), which are similar to those of similar reported triazole derivatives (Zhao *et al.*, 2010; Gao *et al.*, 2011).

In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 1) are observed and consolidate the crystal structure.

## S2. Experimental

The title compound was synthesized with the reaction of 4-nitrobenzaldehyde (2.0 mmol) and 3-(4-amino-3-methyl-5-thioxo-4,5-dihydro-1*H*-1,2,4-triazol-1-yl)-1,3-diphenylpropan-1-one (2.0 mmol) by refluxing in ethanol. The reaction progress was monitored *via* TLC. The resulting precipitate was filtered off, washed with cold ethanol, dried and purified to give the target product as a colorless solid in 74% yield. Crystals of title compound suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in dichloromethanle–ethanol (1:1).

## S3. Refinement

All H atoms were positioned geometrically and refined as riding (C—H = 0.95–1.00 Å) on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C). One molecule of solvent dichloromethane is present in the asymmetric unit. This was refined as disordered over four positions with occupancies of 0.271 (3):0.3884 (18):0.298 (2):0.0424 (15).

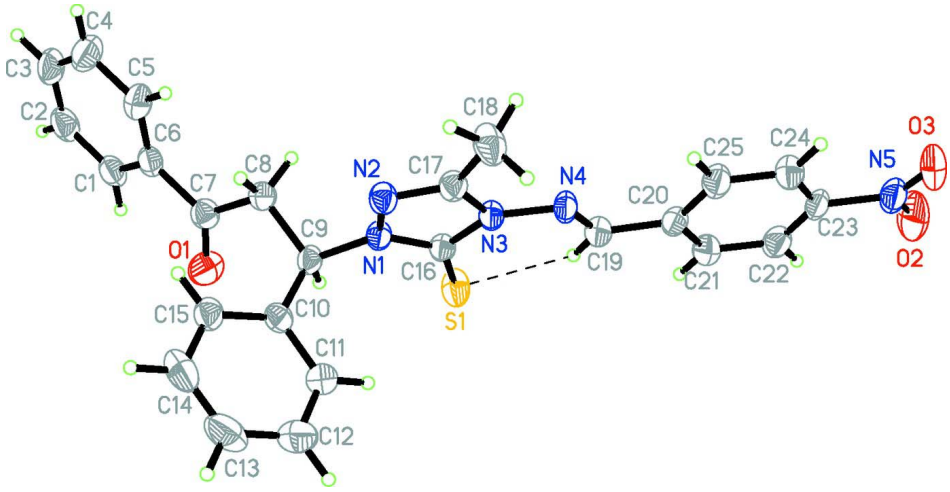


**Figure 1**

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 60% probability level. The disordered dichloromethane molecule is omitted.

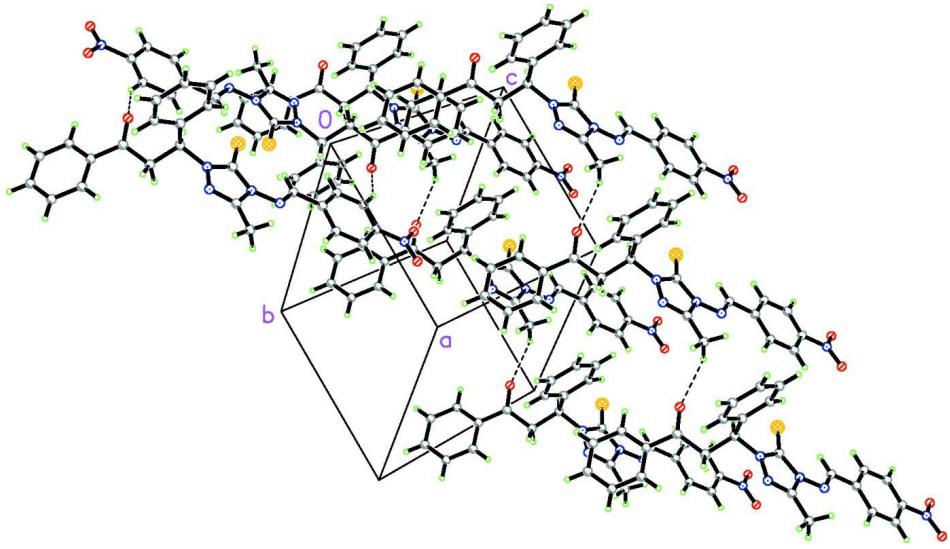


**Figure 2**

Packing diagram of the crystal structure. The disordered dichloromethane molecule is omitted.

### 3-[3-Methyl-4-(4-nitrobenzylideneamino)-5-sulfanylidene-4,5-dihydro-1*H*- 1,2,4-triazol-1-yl]-1,3-diphenylpropan-1-one dichloromethane monosolvate

*Crystal data*

$C_{25}H_{21}N_5O_3S\cdot CH_2Cl_2$
$M_r$ = 556.45
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 8.9880 (13) Å
$b$ = 11.4440 (15) Å
$c$ = 14.8604 (18) Å
$\alpha$ = 70.212 (11)°
$\beta$ = 88.973 (13)°
$\gamma$ = 67.020 (9)°
$V$ = 1312.6 (3) Å$^3$
$Z$ = 2
$F(000)$ = 576
$D_x$ = 1.408 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4632 reflections
$\theta$ = 2.1–27.9°
$\mu$ = 0.37 mm$^{-1}$

$T$ = 113 K
Prism, colourless

0.26 × 0.24 × 0.20 mm

*Data collection*

Rigaku Saturn CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.911, $T_{max}$ = 0.931

16932 measured reflections
6203 independent reflections
4100 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035
$\theta_{max}$ = 27.9°, $\theta_{min}$ = 2.1°
$h$ = −11→11
$k$ = −15→15
$l$ = −19→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.159
$S$ = 1.07
6203 reflections
396 parameters
91 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
$w = 1/[\sigma^2(F_o^2) + (0.0904P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.48389 (7) | 0.55051 (6) | 0.09792 (4) | 0.03327 (18) | |
| O1 | 0.91577 (19) | 0.35679 (16) | 0.36980 (12) | 0.0382 (4) | |
| O2 | −0.3675 (2) | 0.7421 (2) | −0.32320 (14) | 0.0570 (5) | |
| O3 | −0.5429 (2) | 0.7897 (2) | −0.22653 (14) | 0.0565 (5) | |
| N1 | 0.4215 (2) | 0.54864 (17) | 0.27759 (11) | 0.0220 (4) | |
| N2 | 0.2982 (2) | 0.57257 (18) | 0.33400 (12) | 0.0252 (4) | |
| N4 | 0.0767 (2) | 0.63610 (17) | 0.12001 (12) | 0.0253 (4) | |
| N5 | −0.4037 (3) | 0.75374 (19) | −0.24612 (15) | 0.0387 (5) | |
| C1 | 1.0752 (3) | 0.1110 (2) | 0.52278 (17) | 0.0316 (5) | |
| H1 | 1.1363 | 0.1371 | 0.4730 | 0.038* | |
| C2 | 1.1508 (3) | −0.0061 (2) | 0.60281 (18) | 0.0376 (6) | |
| H2 | 1.2634 | −0.0614 | 0.6072 | 0.045* | |
| C3 | 1.0636 (3) | −0.0431 (2) | 0.67630 (18) | 0.0390 (6) | |
| H3 | 1.1169 | −0.1228 | 0.7316 | 0.047* | |

| | | | | | |
|---|---|---|---|---|---|
| C4 | 0.8977 (3) | 0.0356 (2) | 0.67000 (17) | 0.0401 (6) | |
| H4 | 0.8377 | 0.0097 | 0.7206 | 0.048* | |
| C5 | 0.8204 (3) | 0.1531 (2) | 0.58855 (16) | 0.0320 (5) | |
| H5 | 0.7072 | 0.2070 | 0.5835 | 0.038* | |
| C6 | 0.9085 (2) | 0.1913 (2) | 0.51510 (15) | 0.0262 (4) | |
| C7 | 0.8314 (3) | 0.3141 (2) | 0.42531 (15) | 0.0270 (5) | |
| C8 | 0.6478 (2) | 0.3800 (2) | 0.40255 (15) | 0.0270 (5) | |
| H8A | 0.5998 | 0.3976 | 0.4596 | 0.032* | |
| H8B | 0.6098 | 0.3167 | 0.3888 | 0.032* | |
| C9 | 0.5887 (2) | 0.5135 (2) | 0.31618 (14) | 0.0232 (4) | |
| H9 | 0.6596 | 0.4983 | 0.2650 | 0.028* | |
| C10 | 0.5934 (2) | 0.6341 (2) | 0.33558 (15) | 0.0246 (4) | |
| C11 | 0.5370 (3) | 0.7600 (2) | 0.25927 (17) | 0.0331 (5) | |
| H11 | 0.5027 | 0.7664 | 0.1970 | 0.040* | |
| C12 | 0.5308 (3) | 0.8754 (3) | 0.2736 (2) | 0.0440 (6) | |
| H12 | 0.4908 | 0.9606 | 0.2217 | 0.053* | |
| C13 | 0.5833 (3) | 0.8658 (3) | 0.3641 (2) | 0.0469 (7) | |
| H13 | 0.5785 | 0.9446 | 0.3743 | 0.056* | |
| C14 | 0.6420 (3) | 0.7426 (3) | 0.43870 (19) | 0.0406 (6) | |
| H14 | 0.6787 | 0.7362 | 0.5004 | 0.049* | |
| C15 | 0.6480 (3) | 0.6268 (2) | 0.42456 (16) | 0.0284 (5) | |
| H15 | 0.6902 | 0.5417 | 0.4766 | 0.034* | |
| C16 | 0.3708 (2) | 0.56787 (19) | 0.18648 (14) | 0.0228 (4) | |
| N3 | 0.2032 (2) | 0.60395 (16) | 0.18804 (12) | 0.0223 (4) | |
| C17 | 0.1673 (2) | 0.6053 (2) | 0.27919 (14) | 0.0249 (4) | |
| C18 | 0.0009 (3) | 0.6404 (3) | 0.30726 (17) | 0.0363 (5) | |
| H18A | 0.0046 | 0.6354 | 0.3744 | 0.054* | |
| H18B | −0.0412 | 0.5762 | 0.3008 | 0.054* | |
| H18C | −0.0707 | 0.7329 | 0.2652 | 0.054* | |
| C19 | 0.1070 (3) | 0.6337 (2) | 0.03578 (15) | 0.0275 (5) | |
| H19 | 0.2148 | 0.6118 | 0.0195 | 0.033* | |
| C20 | −0.0282 (3) | 0.6655 (2) | −0.03495 (15) | 0.0264 (5) | |
| C21 | 0.0061 (3) | 0.6678 (2) | −0.12770 (16) | 0.0303 (5) | |
| H21 | 0.1140 | 0.6493 | −0.1430 | 0.036* | |
| C22 | −0.1174 (3) | 0.6972 (2) | −0.19751 (16) | 0.0318 (5) | |
| H22 | −0.0955 | 0.6998 | −0.2608 | 0.038* | |
| C23 | −0.2716 (3) | 0.7224 (2) | −0.17278 (15) | 0.0297 (5) | |
| C24 | −0.3097 (3) | 0.7184 (2) | −0.08119 (16) | 0.0324 (5) | |
| H24 | −0.4176 | 0.7352 | −0.0662 | 0.039* | |
| C25 | −0.1863 (3) | 0.6893 (2) | −0.01263 (16) | 0.0311 (5) | |
| H25 | −0.2093 | 0.6855 | 0.0507 | 0.037* | |
| Cl1 | 0.8120 (5) | 1.0389 (4) | −0.0808 (3) | 0.0362 (8) | 0.271 (3) |
| Cl2 | 0.7811 (5) | 1.0728 (4) | 0.0934 (3) | 0.0640 (12) | 0.271 (3) |
| C26 | 0.6696 (14) | 1.0852 (19) | −0.0030 (9) | 0.094 (3) | 0.271 (3) |
| H26A | 0.5868 | 1.1794 | −0.0348 | 0.112* | 0.271 (3) |
| H26B | 0.6137 | 1.0229 | 0.0170 | 0.112* | 0.271 (3) |
| C27 | 0.7212 (11) | 0.9953 (13) | 0.0241 (7) | 0.094 (3) | 0.3884 (18) |
| H27A | 0.7977 | 0.9021 | 0.0305 | 0.112* | 0.3884 (18) |

| | | | | | |
|---|---|---|---|---|---|
| H27B | 0.7413 | 1.0585 | −0.0343 | 0.112* | 0.3884 (18) |
| Cl3 | 0.5337 (2) | 1.0078 (2) | 0.00310 (16) | 0.0435 (6) | 0.3884 (18) |
| Cl4 | 0.7722 (3) | 1.0250 (3) | 0.11800 (18) | 0.0621 (7) | 0.3884 (18) |
| C28 | 0.9267 (13) | 0.9766 (13) | 0.0362 (7) | 0.079 (4) | 0.298 (2) |
| H28A | 0.9329 | 0.8830 | 0.0550 | 0.095* | 0.298 (2) |
| H28B | 0.8410 | 1.0225 | 0.0705 | 0.095* | 0.298 (2) |
| Cl6 | 1.1012 (7) | 0.9588 (6) | 0.0885 (4) | 0.0887 (19) | 0.298 (2) |
| Cl5 | 0.8513 (7) | 1.0491 (5) | −0.0756 (4) | 0.0586 (13) | 0.298 (2) |
| Cl7 | 1.0305 (17) | 0.9999 (14) | −0.0990 (9) | 0.0362 (8) | 0.0424 (15) |
| Cl8 | 1.176 (3) | 1.000 (2) | 0.0685 (12) | 0.0362 (8) | 0.0424 (15) |
| C29 | 1.185 (7) | 0.908 (4) | −0.004 (4) | 0.079 (4) | 0.0424 (15) |
| H29A | 1.2915 | 0.8843 | −0.0290 | 0.095* | 0.0424 (15) |
| H29B | 1.1758 | 0.8219 | 0.0345 | 0.095* | 0.0424 (15) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0257 (3) | 0.0509 (4) | 0.0245 (3) | −0.0139 (3) | 0.0057 (2) | −0.0174 (3) |
| O1 | 0.0242 (8) | 0.0376 (9) | 0.0401 (9) | −0.0080 (7) | 0.0043 (7) | −0.0043 (8) |
| O2 | 0.0592 (13) | 0.0715 (13) | 0.0390 (11) | −0.0221 (11) | −0.0150 (9) | −0.0229 (10) |
| O3 | 0.0368 (11) | 0.0656 (13) | 0.0511 (12) | −0.0185 (10) | −0.0200 (9) | −0.0036 (10) |
| N1 | 0.0193 (8) | 0.0270 (9) | 0.0185 (8) | −0.0089 (7) | 0.0019 (6) | −0.0073 (7) |
| N2 | 0.0218 (8) | 0.0323 (9) | 0.0213 (8) | −0.0107 (7) | 0.0031 (7) | −0.0098 (8) |
| N4 | 0.0229 (9) | 0.0259 (9) | 0.0234 (9) | −0.0079 (7) | −0.0056 (7) | −0.0065 (7) |
| N5 | 0.0428 (13) | 0.0293 (10) | 0.0365 (12) | −0.0141 (9) | −0.0164 (10) | −0.0029 (9) |
| C1 | 0.0258 (11) | 0.0290 (11) | 0.0397 (13) | −0.0120 (9) | −0.0047 (10) | −0.0106 (10) |
| C2 | 0.0275 (12) | 0.0291 (12) | 0.0502 (15) | −0.0093 (10) | −0.0122 (11) | −0.0088 (11) |
| C3 | 0.0400 (14) | 0.0278 (12) | 0.0373 (13) | −0.0088 (11) | −0.0169 (11) | −0.0024 (10) |
| C4 | 0.0469 (15) | 0.0356 (13) | 0.0289 (12) | −0.0140 (12) | −0.0045 (11) | −0.0039 (11) |
| C5 | 0.0299 (12) | 0.0309 (11) | 0.0284 (11) | −0.0067 (10) | −0.0033 (9) | −0.0090 (10) |
| C6 | 0.0241 (10) | 0.0240 (10) | 0.0291 (11) | −0.0086 (9) | −0.0046 (9) | −0.0092 (9) |
| C7 | 0.0244 (10) | 0.0265 (10) | 0.0292 (11) | −0.0091 (9) | 0.0017 (9) | −0.0102 (9) |
| C8 | 0.0232 (10) | 0.0259 (10) | 0.0277 (11) | −0.0104 (9) | −0.0010 (8) | −0.0043 (9) |
| C9 | 0.0170 (9) | 0.0270 (10) | 0.0213 (10) | −0.0075 (8) | −0.0002 (8) | −0.0050 (8) |
| C10 | 0.0185 (9) | 0.0289 (11) | 0.0269 (10) | −0.0110 (8) | 0.0031 (8) | −0.0090 (9) |
| C11 | 0.0350 (12) | 0.0322 (12) | 0.0311 (12) | −0.0174 (10) | −0.0005 (10) | −0.0058 (10) |
| C12 | 0.0467 (15) | 0.0329 (13) | 0.0494 (16) | −0.0211 (12) | 0.0000 (12) | −0.0055 (12) |
| C13 | 0.0467 (15) | 0.0402 (14) | 0.0666 (19) | −0.0257 (12) | 0.0034 (14) | −0.0248 (14) |
| C14 | 0.0380 (13) | 0.0565 (16) | 0.0431 (14) | −0.0270 (12) | 0.0052 (11) | −0.0275 (13) |
| C15 | 0.0243 (10) | 0.0351 (12) | 0.0259 (11) | −0.0135 (9) | 0.0025 (9) | −0.0095 (9) |
| C16 | 0.0223 (10) | 0.0215 (10) | 0.0231 (10) | −0.0084 (8) | −0.0002 (8) | −0.0068 (8) |
| N3 | 0.0202 (8) | 0.0246 (9) | 0.0206 (8) | −0.0082 (7) | 0.0001 (7) | −0.0073 (7) |
| C17 | 0.0244 (10) | 0.0293 (11) | 0.0211 (10) | −0.0116 (9) | 0.0029 (8) | −0.0083 (9) |
| C18 | 0.0237 (11) | 0.0567 (15) | 0.0323 (12) | −0.0167 (11) | 0.0073 (9) | −0.0204 (12) |
| C19 | 0.0260 (11) | 0.0267 (11) | 0.0271 (11) | −0.0102 (9) | −0.0011 (9) | −0.0068 (9) |
| C20 | 0.0283 (11) | 0.0227 (10) | 0.0242 (10) | −0.0095 (9) | −0.0038 (9) | −0.0043 (9) |
| C21 | 0.0310 (11) | 0.0334 (11) | 0.0263 (11) | −0.0145 (10) | 0.0004 (9) | −0.0088 (9) |
| C22 | 0.0378 (13) | 0.0306 (11) | 0.0243 (11) | −0.0139 (10) | −0.0026 (9) | −0.0063 (9) |

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|---|---|---|---|---|---|---|
| C23 | 0.0349 (12) | 0.0217 (10) | 0.0256 (11) | −0.0105 (9) | −0.0122 (9) | −0.0007 (9) |
| C24 | 0.0265 (11) | 0.0318 (12) | 0.0346 (12) | −0.0101 (10) | −0.0023 (9) | −0.0088 (10) |
| C25 | 0.0299 (11) | 0.0339 (12) | 0.0254 (11) | −0.0113 (10) | −0.0012 (9) | −0.0078 (10) |
| Cl1 | 0.0368 (10) | 0.0373 (10) | 0.0345 (9) | −0.0143 (6) | 0.0046 (6) | −0.0140 (6) |
| Cl2 | 0.0677 (15) | 0.0600 (14) | 0.0678 (15) | −0.0239 (10) | 0.0039 (9) | −0.0294 (10) |
| C26 | 0.094 (3) | 0.093 (3) | 0.093 (3) | −0.0377 (14) | 0.0128 (9) | −0.0339 (13) |
| C27 | 0.094 (3) | 0.093 (3) | 0.093 (3) | −0.0377 (14) | 0.0128 (9) | −0.0339 (13) |
| Cl3 | 0.0459 (10) | 0.0446 (8) | 0.0421 (7) | −0.0168 (7) | 0.0075 (8) | −0.0201 (6) |
| Cl4 | 0.0617 (10) | 0.0766 (11) | 0.0486 (10) | −0.0279 (8) | 0.0057 (7) | −0.0234 (8) |
| C28 | 0.079 (4) | 0.079 (4) | 0.079 (4) | −0.0324 (16) | 0.0104 (9) | −0.0286 (15) |
| Cl6 | 0.089 (2) | 0.088 (2) | 0.090 (2) | −0.0372 (12) | 0.0078 (10) | −0.0314 (11) |
| Cl5 | 0.0574 (16) | 0.0577 (15) | 0.0607 (15) | −0.0229 (10) | 0.0094 (10) | −0.0221 (10) |
| Cl7 | 0.0368 (10) | 0.0373 (10) | 0.0345 (9) | −0.0143 (6) | 0.0046 (6) | −0.0140 (6) |
| Cl8 | 0.0368 (10) | 0.0373 (10) | 0.0345 (9) | −0.0143 (6) | 0.0046 (6) | −0.0140 (6) |
| C29 | 0.079 (4) | 0.079 (4) | 0.079 (4) | −0.0324 (16) | 0.0104 (9) | −0.0286 (15) |

*Geometric parameters (Å, º)*

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| S1—C16 | 1.666 (2) | C14—C15 | 1.391 (3) |
| O1—C7 | 1.220 (3) | C14—H14 | 0.9500 |
| O2—N5 | 1.221 (3) | C15—H15 | 0.9500 |
| O3—N5 | 1.219 (3) | C16—N3 | 1.399 (2) |
| N1—C16 | 1.353 (2) | N3—C17 | 1.391 (2) |
| N1—N2 | 1.375 (2) | C17—C18 | 1.481 (3) |
| N1—C9 | 1.467 (2) | C18—H18A | 0.9800 |
| N2—C17 | 1.293 (3) | C18—H18B | 0.9800 |
| N4—C19 | 1.283 (3) | C18—H18C | 0.9800 |
| N4—N3 | 1.383 (2) | C19—C20 | 1.469 (3) |
| N5—C23 | 1.475 (3) | C19—H19 | 0.9500 |
| C1—C2 | 1.383 (3) | C20—C25 | 1.394 (3) |
| C1—C6 | 1.401 (3) | C20—C21 | 1.401 (3) |
| C1—H1 | 0.9500 | C21—C22 | 1.391 (3) |
| C2—C3 | 1.379 (4) | C21—H21 | 0.9500 |
| C2—H2 | 0.9500 | C22—C23 | 1.371 (3) |
| C3—C4 | 1.394 (4) | C22—H22 | 0.9500 |
| C3—H3 | 0.9500 | C23—C24 | 1.388 (3) |
| C4—C5 | 1.398 (3) | C24—C25 | 1.379 (3) |
| C4—H4 | 0.9500 | C24—H24 | 0.9500 |
| C5—C6 | 1.388 (3) | C25—H25 | 0.9500 |
| C5—H5 | 0.9500 | Cl1—C26 | 1.757 (9) |
| C6—C7 | 1.496 (3) | Cl2—C26 | 1.696 (9) |
| C7—C8 | 1.510 (3) | C26—H26A | 0.9900 |
| C8—C9 | 1.525 (3) | C26—H26B | 0.9900 |
| C8—H8A | 0.9900 | C27—Cl4 | 1.656 (8) |
| C8—H8B | 0.9900 | C27—Cl3 | 1.661 (8) |
| C9—C10 | 1.518 (3) | C27—H27A | 0.9900 |
| C9—H9 | 1.0000 | C27—H27B | 0.9900 |
| C10—C15 | 1.383 (3) | C28—Cl5 | 1.606 (8) |

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| C10—C11 | 1.401 (3) | C28—Cl6 | 1.671 (8) |
| C11—C12 | 1.388 (3) | C28—H28A | 0.9900 |
| C11—H11 | 0.9500 | C28—H28B | 0.9900 |
| C12—C13 | 1.386 (4) | Cl7—C29 | 1.718 (11) |
| C12—H12 | 0.9500 | Cl8—C29 | 1.723 (11) |
| C13—C14 | 1.370 (4) | C29—H29A | 0.9900 |
| C13—H13 | 0.9500 | C29—H29B | 0.9900 |

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| C16—N1—N2 | 113.97 (16) | N1—C16—S1 | 127.61 (15) |
| C16—N1—C9 | 125.89 (17) | N3—C16—S1 | 130.44 (15) |
| N2—N1—C9 | 120.04 (16) | N4—N3—C17 | 118.17 (16) |
| C17—N2—N1 | 105.18 (16) | N4—N3—C16 | 133.38 (17) |
| C19—N4—N3 | 119.39 (18) | C17—N3—C16 | 108.45 (16) |
| O3—N5—O2 | 123.6 (2) | N2—C17—N3 | 110.44 (18) |
| O3—N5—C23 | 118.3 (2) | N2—C17—C18 | 125.88 (19) |
| O2—N5—C23 | 118.2 (2) | N3—C17—C18 | 123.68 (18) |
| C2—C1—C6 | 120.0 (2) | C17—C18—H18A | 109.5 |
| C2—C1—H1 | 120.0 | C17—C18—H18B | 109.5 |
| C6—C1—H1 | 120.0 | H18A—C18—H18B | 109.5 |
| C3—C2—C1 | 120.3 (2) | C17—C18—H18C | 109.5 |
| C3—C2—H2 | 119.9 | H18A—C18—H18C | 109.5 |
| C1—C2—H2 | 119.9 | H18B—C18—H18C | 109.5 |
| C2—C3—C4 | 120.5 (2) | N4—C19—C20 | 118.7 (2) |
| C2—C3—H3 | 119.8 | N4—C19—H19 | 120.7 |
| C4—C3—H3 | 119.8 | C20—C19—H19 | 120.7 |
| C3—C4—C5 | 119.3 (2) | C25—C20—C21 | 119.3 (2) |
| C3—C4—H4 | 120.3 | C25—C20—C19 | 122.8 (2) |
| C5—C4—H4 | 120.3 | C21—C20—C19 | 117.8 (2) |
| C6—C5—C4 | 120.2 (2) | C22—C21—C20 | 120.2 (2) |
| C6—C5—H5 | 119.9 | C22—C21—H21 | 119.9 |
| C4—C5—H5 | 119.9 | C20—C21—H21 | 119.9 |
| C5—C6—C1 | 119.6 (2) | C23—C22—C21 | 118.4 (2) |
| C5—C6—C7 | 122.49 (19) | C23—C22—H22 | 120.8 |
| C1—C6—C7 | 117.9 (2) | C21—C22—H22 | 120.8 |
| O1—C7—C6 | 120.42 (19) | C22—C23—C24 | 123.1 (2) |
| O1—C7—C8 | 120.74 (19) | C22—C23—N5 | 118.9 (2) |
| C6—C7—C8 | 118.78 (18) | C24—C23—N5 | 118.0 (2) |
| C7—C8—C9 | 111.96 (17) | C25—C24—C23 | 118.0 (2) |
| C7—C8—H8A | 109.2 | C25—C24—H24 | 121.0 |
| C9—C8—H8A | 109.2 | C23—C24—H24 | 121.0 |
| C7—C8—H8B | 109.2 | C24—C25—C20 | 120.9 (2) |
| C9—C8—H8B | 109.2 | C24—C25—H25 | 119.5 |
| H8A—C8—H8B | 107.9 | C20—C25—H25 | 119.5 |
| N1—C9—C10 | 108.68 (16) | Cl2—C26—Cl1 | 104.9 (6) |
| N1—C9—C8 | 109.28 (16) | Cl2—C26—H26A | 110.8 |
| C10—C9—C8 | 115.60 (17) | Cl1—C26—H26A | 110.8 |
| N1—C9—H9 | 107.7 | Cl2—C26—H26B | 110.8 |
| C10—C9—H9 | 107.7 | Cl1—C26—H26B | 110.8 |

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| C8—C9—H9 | 107.7 | H26A—C26—H26B | 108.8 |
| C15—C10—C11 | 118.4 (2) | Cl4—C27—Cl3 | 119.9 (6) |
| C15—C10—C9 | 123.63 (18) | Cl4—C27—H27A | 107.4 |
| C11—C10—C9 | 118.01 (19) | Cl3—C27—H27A | 107.3 |
| C12—C11—C10 | 120.7 (2) | Cl4—C27—H27B | 107.4 |
| C12—C11—H11 | 119.6 | Cl3—C27—H27B | 107.4 |
| C10—C11—H11 | 119.6 | H27A—C27—H27B | 106.9 |
| C13—C12—C11 | 119.7 (2) | Cl5—C28—Cl6 | 128.9 (7) |
| C13—C12—H12 | 120.1 | Cl5—C28—H28A | 105.1 |
| C11—C12—H12 | 120.1 | Cl6—C28—H28A | 105.1 |
| C14—C13—C12 | 120.1 (2) | Cl5—C28—H28B | 105.1 |
| C14—C13—H13 | 120.0 | Cl6—C28—H28B | 105.1 |
| C12—C13—H13 | 120.0 | H28A—C28—H28B | 105.9 |
| C13—C14—C15 | 120.3 (2) | Cl7—C29—Cl8 | 110.7 (10) |
| C13—C14—H14 | 119.8 | Cl7—C29—H29A | 109.5 |
| C15—C14—H14 | 119.8 | Cl8—C29—H29A | 109.5 |
| C10—C15—C14 | 120.8 (2) | Cl7—C29—H29B | 109.5 |
| C10—C15—H15 | 119.6 | Cl8—C29—H29B | 109.5 |
| C14—C15—H15 | 119.6 | H29A—C29—H29B | 108.1 |
| N1—C16—N3 | 101.95 (16) | | |

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|---|---|---|---|
| C16—N1—N2—C17 | 1.0 (2) | N2—N1—C16—N3 | −1.2 (2) |
| C9—N1—N2—C17 | 177.73 (17) | C9—N1—C16—N3 | −177.65 (17) |
| C6—C1—C2—C3 | −1.3 (3) | N2—N1—C16—S1 | 179.26 (15) |
| C1—C2—C3—C4 | 1.2 (4) | C9—N1—C16—S1 | 2.8 (3) |
| C2—C3—C4—C5 | −0.3 (4) | C19—N4—N3—C17 | −179.04 (18) |
| C3—C4—C5—C6 | −0.4 (4) | C19—N4—N3—C16 | 1.0 (3) |
| C4—C5—C6—C1 | 0.3 (3) | N1—C16—N3—N4 | −179.15 (19) |
| C4—C5—C6—C7 | 178.5 (2) | S1—C16—N3—N4 | 0.4 (3) |
| C2—C1—C6—C5 | 0.5 (3) | N1—C16—N3—C17 | 0.9 (2) |
| C2—C1—C6—C7 | −177.69 (19) | S1—C16—N3—C17 | −179.57 (16) |
| C5—C6—C7—O1 | 168.2 (2) | N1—N2—C17—N3 | −0.4 (2) |
| C1—C6—C7—O1 | −13.7 (3) | N1—N2—C17—C18 | 179.9 (2) |
| C5—C6—C7—C8 | −14.6 (3) | N4—N3—C17—N2 | 179.70 (17) |
| C1—C6—C7—C8 | 163.60 (19) | C16—N3—C17—N2 | −0.3 (2) |
| O1—C7—C8—C9 | −10.1 (3) | N4—N3—C17—C18 | −0.5 (3) |
| C6—C7—C8—C9 | 172.62 (18) | C16—N3—C17—C18 | 179.4 (2) |
| C16—N1—C9—C10 | 111.5 (2) | N3—N4—C19—C20 | 178.82 (17) |
| N2—N1—C9—C10 | −64.8 (2) | N4—C19—C20—C25 | −4.4 (3) |
| C16—N1—C9—C8 | −121.6 (2) | N4—C19—C20—C21 | 177.44 (19) |
| N2—N1—C9—C8 | 62.1 (2) | C25—C20—C21—C22 | 1.7 (3) |
| C7—C8—C9—N1 | 161.13 (17) | C19—C20—C21—C22 | 179.93 (19) |
| C7—C8—C9—C10 | −75.9 (2) | C20—C21—C22—C23 | −0.5 (3) |
| N1—C9—C10—C15 | 123.7 (2) | C21—C22—C23—C24 | −0.7 (3) |
| C8—C9—C10—C15 | 0.4 (3) | C21—C22—C23—N5 | 179.95 (19) |
| N1—C9—C10—C11 | −55.4 (2) | O3—N5—C23—C22 | −172.5 (2) |
| C8—C9—C10—C11 | −178.63 (18) | O2—N5—C23—C22 | 7.6 (3) |
| C15—C10—C11—C12 | −2.3 (3) | O3—N5—C23—C24 | 8.2 (3) |

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| C9—C10—C11—C12 | 176.8 (2) | O2—N5—C23—C24 | −171.8 (2) |
| C10—C11—C12—C13 | 1.0 (4) | C22—C23—C24—C25 | 0.8 (3) |
| C11—C12—C13—C14 | 0.4 (4) | N5—C23—C24—C25 | −179.88 (19) |
| C12—C13—C14—C15 | −0.6 (4) | C23—C24—C25—C20 | 0.4 (3) |
| C11—C10—C15—C14 | 2.1 (3) | C21—C20—C25—C24 | −1.6 (3) |
| C9—C10—C15—C14 | −176.9 (2) | C19—C20—C25—C24 | −179.8 (2) |
| C13—C14—C15—C10 | −0.8 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C19—H19···S1 | 0.95 | 2.43 | 3.199 (3) | 137 |
| C18—H18*B*···O1$^{i}$ | 0.98 | 2.55 | 3.443 (4) | 152 |
| C22—H22···O1$^{ii}$ | 0.95 | 2.34 | 3.202 (3) | 151 |

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