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# Crystal structure of 2-[(*E*)-2-(2-chlorobenzylidene)hydrazin-1-yl]-4-phenyl-1,3-thiazole

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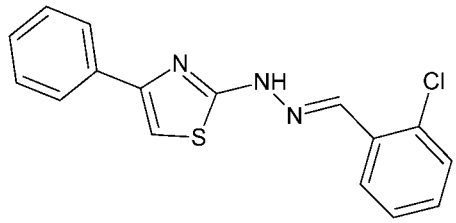
The asymmetric unit of the title compound, $C_{16}H_{12}ClN_3S$, contains two independent molecules whose conformations differ primarily in the orientations of the phenyl and chlorobenzene rings with respect to the thiazole ring. In the first molecule, the dihedral angles are 3.0 (1) and 9.2 (1)°, respectively, for the phenyl ring and the chlorobenzene ring, while in the second molecule, the corresponding angles are 18.6 (1) and 23.4 (1)°. In the crystal, the two independent molecules are associated *via* complementary N—H···N hydrogen bonds into a dimer. These dimers are associated through weak C—H···Cl and C—H···S interactions into supramolecular chains propagating along the *a*-axis direction.

**Keywords:** crystal structure; 1,3-thiazole; hydrogen bonding; hydrogen-bonded dimers.

**CCDC reference**: 1013753

## 1. Related literature

For pharmaceutical properties of thiazole derivatives, see: Siddiqui *et al.* (2011, 2009); Bakris *et al.* (2004); Little *et al.* (2005). For the synthesis of the title compound, see: Mohamed *et al.* (2013).




## 2. Experimental

### 2.1. Crystal data

$C_{16}H_{12}ClN_3S$
$M_r$ = 313.80
Orthorhombic, *Pbca*
*a* = 16.981 (4) Å
*b* = 8.1081 (17) Å
*c* = 41.660 (9) Å
*V* = 5736 (2) Å$^3$
*Z* = 16
Mo *Kα* radiation
$\mu$ = 0.41 mm$^{-1}$
*T* = 150 K
0.22 × 0.22 × 0.05 mm

### 2.2. Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min}$ = 0.80, $T_{max}$ = 0.98
99305 measured reflections
7415 independent reflections
5577 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.086

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.109
*S* = 1.04
7415 reflections
379 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···N6 | 0.91 | 2.02 | 2.901 (2) | 163 |
| N5—H5*A*···N3 | 0.91 | 2.05 | 2.946 (2) | 166 |
| C9—H9···S1$^{i}$ | 0.95 | 2.97 | 3.696 (2) | 134 |
| C25—H25···Cl1$^{ii}$ | 0.95 | 2.92 | 3.720 (2) | 143 |

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2013); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5803).

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# supporting information

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# Crystal structure of 2-[(*E*)-2-(2-chlorobenzylidene)hydrazin-1-yl]-4-phenyl-1,3-thiazole

**Joel T. Mague, Shaaban K. Mohamed, Mehmet Akkurt, Alaa A. Hassan and Mustafa R. Albayati**

## S1. Comment

Thiazole containing compounds have been reported to possess large number of biological properties (Siddiqui *et al.*, 2011; Siddiqui *et al.*, 2009). Sulfathiazol (antimicrobial drug), Ritonavir (antiretroviral drug), Abafungin (antifungal drug), Bleomycine and Tiazofurin (antineoplastic drug) are common drugs with thiazole-based structures. Alagebrium (formerly known as ALT-711) is also a thiazolium salt which was the first drug used for breaking the protein crosslinks caused by advanced glycation endproducts (AGEs). Through this effect Alagebrium is designed to reverse the stiffening of blood vessel walls that contributes to hypertension and cardiovascular disease (Bakris *et al.*, 2004; Little *et al.*, 2005). İn this context and as part of our study in synthesis of potential bioactive heterocyclic molecules, we report the synthesis and crystal structure of the title compound.

There are two independent molecules of the title compound in the asymmetric unit whose conformations differ primarily in the orientations of the phenyl rings with respect to the thiazole ring. For molecule 1, the dihedral angles are 3.0 (1) and 9.2 (1)°, respectively, for rings C1–C6 and C11–C16 while for molecule 2 the corresponding angles are 18.6 (1) and 23.4 (1)°.

In the crystal structure, the two independent molecules are associated *via* complementary N—H···N hydrogen bonds (Fig. 1 and Table 1). These pairs are associated into chains running along the *a* axis through weak C—H···Cl interactions (Table 1) and weak C—H···S interactions (Table 1).

## S2. Experimental

The title compound has been prepared according to our reported method (Mohamed *et al.*, 2013). Colourless crystals suitable for X-ray diffraction have been obtained by crystallization of the crude product (I) from ethanol.

## S3. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen were placed in locations derived from a difference map and, following initial independent refinement to verify their presence, their coordinates were adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

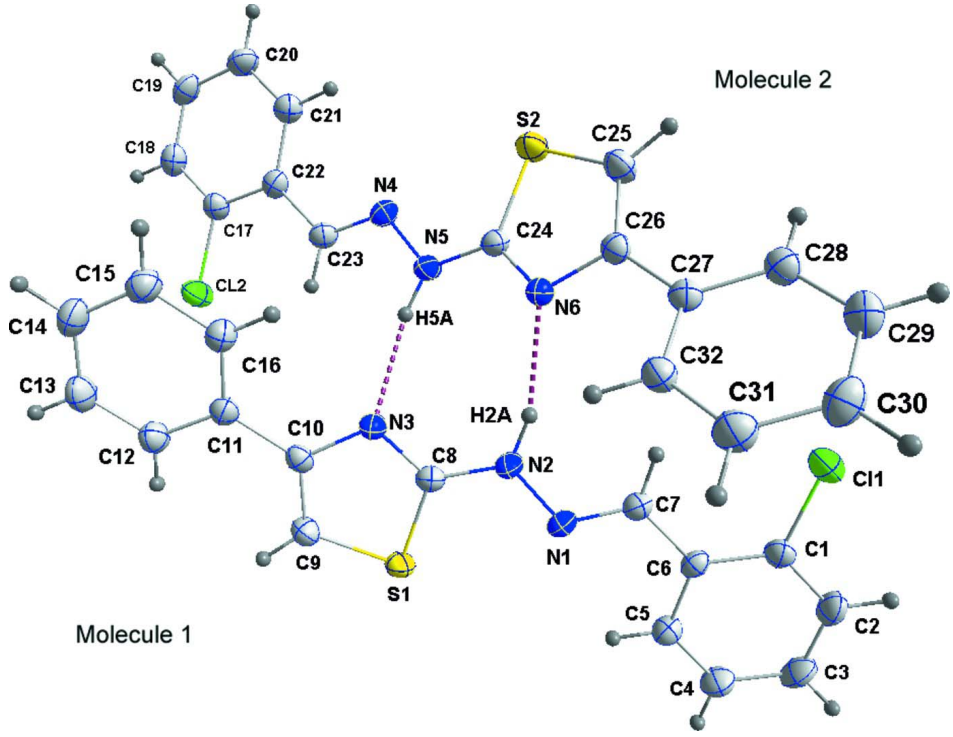


**Figure 1**

The asymmetric unit with the complementary N—H···N hydrogen bonds shown as dotted lines. Ellipsoids are drawn at the 50% probability level.

### 2-[(*E*)-2-[(2-chlorophenyl)methylidene]hydrazin-1-yl]-4-phenyl-1,3-thiazole

*Crystal data*

$C_{16}H_{12}ClN_3S$
$M_r = 313.80$
Orthorhombic, *Pbca*
$a = 16.981\ (4)$ Å
$b = 8.1081\ (17)$ Å
$c = 41.660\ (9)$ Å
$V = 5736\ (2)$ Å$^3$
$Z = 16$
$F(000) = 2592$
$D_x = 1.454$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9986 reflections
$\theta = 2.6$–$28.7°$
$\mu = 0.41$ mm$^{-1}$
$T = 150$ K
Plate, colourless
0.22 × 0.22 × 0.05 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.80$, $T_{max} = 0.98$
99305 measured reflections
7415 independent reflections
5577 reflections with $I > 2\sigma(I)$
$R_{int} = 0.086$
$\theta_{max} = 28.8°$, $\theta_{min} = 2.0°$
$h = -22 \rightarrow 22$
$k = -10 \rightarrow 10$
$l = -55 \rightarrow 56$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.109$
$S = 1.04$
7415 reflections
379 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
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 4.8434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.42$ e Å$^{-3}$
$\Delta\rho_{min} = -0.33$ e Å$^{-3}$

*Special details*

**Experimental**. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in $\omega$, collected at $\varphi$ = 0.00, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in $\varphi$, collected at $\omega$ = -30.00 and 210.00°. The scan time was 15 sec/frame.

**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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen were placed in locations derived from a difference map and, following initial independent refinement to verify their presence, their coordinates were adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.42874 (3) | 0.85456 (8) | 0.45306 (2) | 0.04262 (15) |
| S1 | 0.67429 (3) | 0.24168 (6) | 0.37657 (2) | 0.02704 (12) |
| N1 | 0.57267 (10) | 0.4806 (2) | 0.40738 (4) | 0.0248 (3) |
| N2 | 0.53242 (10) | 0.3466 (2) | 0.39636 (4) | 0.0266 (4) |
| H2A | 0.4789 | 0.3475 | 0.3972 | 0.032* |
| N3 | 0.54083 (9) | 0.0943 (2) | 0.36976 (4) | 0.0235 (3) |
| C1 | 0.52954 (12) | 0.8719 (3) | 0.44674 (5) | 0.0266 (4) |
| C2 | 0.56720 (14) | 1.0112 (3) | 0.45834 (5) | 0.0325 (5) |
| H2 | 0.5382 | 1.0943 | 0.4692 | 0.039* |
| C3 | 0.64723 (14) | 1.0279 (3) | 0.45392 (5) | 0.0354 (5) |
| H3 | 0.6737 | 1.1225 | 0.4620 | 0.042* |
| C4 | 0.68913 (13) | 0.9078 (3) | 0.43785 (5) | 0.0326 (5) |
| H4 | 0.7442 | 0.9205 | 0.4346 | 0.039* |
| C5 | 0.65150 (12) | 0.7700 (3) | 0.42652 (5) | 0.0268 (4) |
| H5 | 0.6811 | 0.6881 | 0.4156 | 0.032* |
| C6 | 0.57031 (11) | 0.7473 (2) | 0.43076 (4) | 0.0229 (4) |
| C7 | 0.53147 (11) | 0.5992 (2) | 0.41884 (5) | 0.0244 (4) |
| H7 | 0.4757 | 0.5905 | 0.4196 | 0.029* |
| C8 | 0.57334 (11) | 0.2272 (2) | 0.38144 (4) | 0.0221 (4) |

| | | | | |
|---|---|---|---|---|
| C9 | 0.67232 (11) | 0.0512 (2) | 0.35830 (5) | 0.0263 (4) |
| H9 | 0.7175 | −0.0043 | 0.3503 | 0.032* |
| C10 | 0.59793 (11) | −0.0091 (2) | 0.35689 (4) | 0.0226 (4) |
| C11 | 0.57380 (11) | −0.1716 (2) | 0.34490 (5) | 0.0239 (4) |
| C12 | 0.62690 (12) | −0.2731 (3) | 0.32880 (5) | 0.0295 (4) |
| H12 | 0.6792 | −0.2358 | 0.3252 | 0.035* |
| C13 | 0.60464 (13) | −0.4266 (3) | 0.31801 (5) | 0.0339 (5) |
| H13 | 0.6417 | −0.4947 | 0.3073 | 0.041* |
| C14 | 0.52845 (14) | −0.4821 (3) | 0.32276 (5) | 0.0336 (5) |
| H14 | 0.5131 | −0.5877 | 0.3151 | 0.040* |
| C15 | 0.47497 (13) | −0.3835 (3) | 0.33864 (5) | 0.0321 (5) |
| H15 | 0.4225 | −0.4210 | 0.3418 | 0.039* |
| C16 | 0.49763 (12) | −0.2292 (3) | 0.34997 (5) | 0.0276 (4) |
| H16 | 0.4608 | −0.1627 | 0.3613 | 0.033* |
| Cl2 | 0.51373 (3) | −0.18369 (8) | 0.25484 (2) | 0.03684 (14) |
| S2 | 0.23921 (3) | 0.26852 (7) | 0.35529 (2) | 0.03059 (13) |
| N4 | 0.35122 (10) | 0.0848 (2) | 0.31580 (4) | 0.0254 (4) |
| N5 | 0.38660 (10) | 0.1670 (2) | 0.34063 (4) | 0.0279 (4) |
| H5A | 0.4365 | 0.1386 | 0.3464 | 0.033* |
| N6 | 0.36410 (9) | 0.2951 (2) | 0.39001 (4) | 0.0242 (3) |
| C17 | 0.41198 (12) | −0.1953 (3) | 0.25184 (5) | 0.0255 (4) |
| C18 | 0.38063 (13) | −0.2967 (3) | 0.22837 (5) | 0.0301 (4) |
| H18 | 0.4145 | −0.3575 | 0.2146 | 0.036* |
| C19 | 0.30033 (13) | −0.3091 (3) | 0.22512 (5) | 0.0317 (5) |
| H19 | 0.2786 | −0.3785 | 0.2090 | 0.038* |
| C20 | 0.25097 (13) | −0.2208 (3) | 0.24522 (5) | 0.0327 (5) |
| H20 | 0.1955 | −0.2294 | 0.2429 | 0.039* |
| C21 | 0.28249 (12) | −0.1202 (3) | 0.26869 (5) | 0.0287 (4) |
| H21 | 0.2482 | −0.0608 | 0.2825 | 0.034* |
| C22 | 0.36410 (12) | −0.1042 (2) | 0.27246 (5) | 0.0245 (4) |
| C23 | 0.39660 (12) | −0.0014 (3) | 0.29797 (5) | 0.0258 (4) |
| H23 | 0.4519 | 0.0014 | 0.3014 | 0.031* |
| C24 | 0.33820 (11) | 0.2402 (2) | 0.36249 (5) | 0.0232 (4) |
| C25 | 0.23213 (12) | 0.3603 (3) | 0.39246 (5) | 0.0299 (5) |
| H25 | 0.1847 | 0.4030 | 0.4013 | 0.036* |
| C26 | 0.30284 (11) | 0.3639 (2) | 0.40748 (5) | 0.0236 (4) |
| C27 | 0.31902 (11) | 0.4215 (2) | 0.44032 (5) | 0.0233 (4) |
| C28 | 0.26964 (12) | 0.5346 (3) | 0.45548 (5) | 0.0281 (4) |
| H28 | 0.2254 | 0.5774 | 0.4443 | 0.034* |
| C29 | 0.28430 (13) | 0.5849 (3) | 0.48644 (5) | 0.0325 (5) |
| H29 | 0.2504 | 0.6627 | 0.4964 | 0.039* |
| C30 | 0.34853 (14) | 0.5223 (3) | 0.50318 (5) | 0.0353 (5) |
| H30 | 0.3586 | 0.5568 | 0.5246 | 0.042* |
| C31 | 0.39759 (13) | 0.4097 (3) | 0.48843 (5) | 0.0334 (5) |
| H31 | 0.4415 | 0.3667 | 0.4998 | 0.040* |
| C32 | 0.38344 (12) | 0.3590 (3) | 0.45732 (5) | 0.0269 (4) |
| H32 | 0.4176 | 0.2813 | 0.4474 | 0.032* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0279 (3) | 0.0448 (3) | 0.0552 (4) | 0.0053 (2) | 0.0023 (2) | −0.0128 (3) |
| S1 | 0.0191 (2) | 0.0260 (2) | 0.0360 (3) | −0.00266 (19) | −0.00276 (19) | 0.0003 (2) |
| N1 | 0.0269 (8) | 0.0228 (8) | 0.0246 (8) | −0.0049 (7) | −0.0031 (7) | −0.0022 (7) |
| N2 | 0.0205 (8) | 0.0251 (9) | 0.0341 (9) | −0.0045 (7) | 0.0005 (7) | −0.0074 (7) |
| N3 | 0.0202 (7) | 0.0236 (8) | 0.0266 (8) | −0.0010 (6) | 0.0007 (6) | −0.0024 (7) |
| C1 | 0.0280 (10) | 0.0266 (10) | 0.0253 (10) | 0.0033 (8) | −0.0039 (8) | −0.0001 (8) |
| C2 | 0.0422 (12) | 0.0242 (10) | 0.0311 (11) | 0.0037 (9) | −0.0050 (9) | −0.0056 (9) |
| C3 | 0.0445 (13) | 0.0260 (11) | 0.0356 (12) | −0.0073 (10) | −0.0116 (10) | −0.0035 (9) |
| C4 | 0.0326 (11) | 0.0320 (11) | 0.0333 (11) | −0.0066 (9) | −0.0033 (9) | 0.0002 (9) |
| C5 | 0.0284 (10) | 0.0275 (10) | 0.0246 (10) | −0.0028 (8) | 0.0002 (8) | −0.0012 (8) |
| C6 | 0.0272 (9) | 0.0230 (9) | 0.0184 (9) | −0.0020 (8) | −0.0031 (7) | 0.0003 (7) |
| C7 | 0.0237 (9) | 0.0258 (10) | 0.0236 (9) | −0.0026 (8) | −0.0019 (7) | −0.0011 (8) |
| C8 | 0.0195 (8) | 0.0245 (9) | 0.0224 (9) | −0.0010 (7) | −0.0012 (7) | 0.0006 (7) |
| C9 | 0.0227 (9) | 0.0267 (10) | 0.0295 (10) | 0.0019 (8) | 0.0008 (8) | 0.0025 (8) |
| C10 | 0.0232 (9) | 0.0243 (10) | 0.0202 (9) | 0.0030 (7) | −0.0015 (7) | 0.0023 (7) |
| C11 | 0.0268 (9) | 0.0233 (10) | 0.0214 (9) | 0.0014 (8) | −0.0041 (7) | 0.0015 (7) |
| C12 | 0.0293 (10) | 0.0300 (11) | 0.0292 (10) | 0.0032 (9) | −0.0006 (8) | −0.0032 (9) |
| C13 | 0.0391 (12) | 0.0302 (11) | 0.0325 (12) | 0.0065 (9) | 0.0008 (9) | −0.0042 (9) |
| C14 | 0.0454 (13) | 0.0241 (10) | 0.0313 (11) | −0.0014 (9) | −0.0036 (10) | −0.0025 (9) |
| C15 | 0.0342 (11) | 0.0287 (11) | 0.0334 (11) | −0.0042 (9) | −0.0025 (9) | 0.0030 (9) |
| C16 | 0.0300 (10) | 0.0249 (10) | 0.0278 (10) | 0.0002 (8) | −0.0001 (8) | −0.0002 (8) |
| Cl2 | 0.0278 (3) | 0.0471 (3) | 0.0356 (3) | 0.0053 (2) | −0.0013 (2) | −0.0076 (2) |
| S2 | 0.0236 (2) | 0.0383 (3) | 0.0298 (3) | 0.0043 (2) | −0.00573 (19) | −0.0034 (2) |
| N4 | 0.0273 (8) | 0.0264 (9) | 0.0226 (8) | −0.0035 (7) | −0.0023 (6) | −0.0020 (7) |
| N5 | 0.0219 (8) | 0.0343 (10) | 0.0274 (9) | 0.0002 (7) | −0.0024 (7) | −0.0091 (7) |
| N6 | 0.0212 (8) | 0.0261 (9) | 0.0254 (8) | −0.0010 (7) | 0.0002 (6) | −0.0030 (7) |
| C17 | 0.0278 (10) | 0.0262 (10) | 0.0227 (9) | 0.0019 (8) | −0.0022 (7) | 0.0030 (8) |
| C18 | 0.0400 (12) | 0.0246 (10) | 0.0257 (10) | 0.0031 (9) | −0.0001 (9) | −0.0003 (8) |
| C19 | 0.0420 (12) | 0.0261 (10) | 0.0270 (11) | −0.0054 (9) | −0.0074 (9) | −0.0020 (9) |
| C20 | 0.0304 (10) | 0.0339 (11) | 0.0337 (11) | −0.0046 (9) | −0.0068 (9) | 0.0008 (9) |
| C21 | 0.0297 (10) | 0.0290 (11) | 0.0275 (11) | 0.0003 (9) | −0.0007 (8) | 0.0000 (9) |
| C22 | 0.0295 (10) | 0.0228 (10) | 0.0211 (9) | −0.0003 (8) | −0.0027 (8) | 0.0024 (8) |
| C23 | 0.0253 (9) | 0.0281 (10) | 0.0238 (10) | −0.0016 (8) | −0.0012 (7) | 0.0007 (8) |
| C24 | 0.0204 (9) | 0.0232 (9) | 0.0259 (9) | −0.0020 (7) | −0.0010 (7) | −0.0002 (8) |
| C25 | 0.0241 (10) | 0.0354 (12) | 0.0302 (11) | 0.0075 (9) | −0.0012 (8) | −0.0008 (9) |
| C26 | 0.0233 (9) | 0.0203 (9) | 0.0272 (10) | 0.0006 (7) | 0.0012 (7) | 0.0013 (8) |
| C27 | 0.0241 (9) | 0.0224 (9) | 0.0234 (9) | −0.0015 (8) | 0.0024 (7) | 0.0014 (8) |
| C28 | 0.0284 (10) | 0.0263 (10) | 0.0296 (11) | 0.0010 (8) | 0.0011 (8) | 0.0013 (9) |
| C29 | 0.0386 (12) | 0.0287 (11) | 0.0304 (11) | 0.0028 (9) | 0.0056 (9) | −0.0021 (9) |
| C30 | 0.0463 (13) | 0.0361 (12) | 0.0235 (10) | −0.0012 (10) | 0.0006 (9) | −0.0017 (9) |
| C31 | 0.0348 (11) | 0.0369 (12) | 0.0287 (11) | 0.0022 (9) | −0.0025 (9) | 0.0020 (9) |
| C32 | 0.0256 (9) | 0.0283 (10) | 0.0269 (10) | 0.0029 (8) | 0.0014 (8) | −0.0002 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C1 | 1.737 (2) | Cl2—C17 | 1.735 (2) |
| S1—C9 | 1.722 (2) | S2—C25 | 1.722 (2) |
| S1—C8 | 1.7301 (19) | S2—C24 | 1.723 (2) |
| N1—C7 | 1.282 (3) | N4—C23 | 1.278 (3) |
| N1—N2 | 1.363 (2) | N4—N5 | 1.370 (2) |
| N2—C8 | 1.344 (2) | N5—C24 | 1.363 (2) |
| N2—H2A | 0.9098 | N5—H5A | 0.9100 |
| N3—C8 | 1.305 (2) | N6—C24 | 1.306 (2) |
| N3—C10 | 1.389 (2) | N6—C26 | 1.387 (2) |
| C1—C2 | 1.385 (3) | C17—C18 | 1.384 (3) |
| C1—C6 | 1.394 (3) | C17—C22 | 1.395 (3) |
| C2—C3 | 1.378 (3) | C18—C19 | 1.374 (3) |
| C2—H2 | 0.9500 | C18—H18 | 0.9500 |
| C3—C4 | 1.380 (3) | C19—C20 | 1.384 (3) |
| C3—H3 | 0.9500 | C19—H19 | 0.9500 |
| C4—C5 | 1.371 (3) | C20—C21 | 1.382 (3) |
| C4—H4 | 0.9500 | C20—H20 | 0.9500 |
| C5—C6 | 1.402 (3) | C21—C22 | 1.401 (3) |
| C5—H5 | 0.9500 | C21—H21 | 0.9500 |
| C6—C7 | 1.458 (3) | C22—C23 | 1.459 (3) |
| C7—H7 | 0.9500 | C23—H23 | 0.9500 |
| C9—C10 | 1.356 (3) | C25—C26 | 1.354 (3) |
| C9—H9 | 0.9500 | C25—H25 | 0.9500 |
| C10—C11 | 1.468 (3) | C26—C27 | 1.472 (3) |
| C11—C16 | 1.391 (3) | C27—C28 | 1.394 (3) |
| C11—C12 | 1.393 (3) | C27—C32 | 1.398 (3) |
| C12—C13 | 1.376 (3) | C28—C29 | 1.375 (3) |
| C12—H12 | 0.9500 | C28—H28 | 0.9500 |
| C13—C14 | 1.384 (3) | C29—C30 | 1.390 (3) |
| C13—H13 | 0.9500 | C29—H29 | 0.9500 |
| C14—C15 | 1.379 (3) | C30—C31 | 1.380 (3) |
| C14—H14 | 0.9500 | C30—H30 | 0.9500 |
| C15—C16 | 1.391 (3) | C31—C32 | 1.381 (3) |
| C15—H15 | 0.9500 | C31—H31 | 0.9500 |
| C16—H16 | 0.9500 | C32—H32 | 0.9500 |
| | | | |
| C9—S1—C8 | 88.38 (9) | C25—S2—C24 | 88.24 (9) |
| C7—N1—N2 | 116.74 (16) | C23—N4—N5 | 116.15 (17) |
| C8—N2—N1 | 118.06 (16) | C24—N5—N4 | 116.89 (16) |
| C8—N2—H2A | 122.8 | C24—N5—H5A | 119.7 |
| N1—N2—H2A | 118.7 | N4—N5—H5A | 118.9 |
| C8—N3—C10 | 110.30 (16) | C24—N6—C26 | 110.19 (16) |
| C2—C1—C6 | 121.89 (19) | C18—C17—C22 | 121.71 (19) |
| C2—C1—Cl1 | 117.90 (16) | C18—C17—Cl2 | 117.79 (16) |
| C6—C1—Cl1 | 120.21 (16) | C22—C17—Cl2 | 120.50 (15) |
| C3—C2—C1 | 119.3 (2) | C19—C18—C17 | 119.6 (2) |

| | | | |
|---|---|---|---|
| C3—C2—H2 | 120.4 | C19—C18—H18 | 120.2 |
| C1—C2—H2 | 120.4 | C17—C18—H18 | 120.2 |
| C2—C3—C4 | 120.3 (2) | C18—C19—C20 | 120.2 (2) |
| C2—C3—H3 | 119.9 | C18—C19—H19 | 119.9 |
| C4—C3—H3 | 119.9 | C20—C19—H19 | 119.9 |
| C5—C4—C3 | 120.1 (2) | C21—C20—C19 | 119.9 (2) |
| C5—C4—H4 | 119.9 | C21—C20—H20 | 120.0 |
| C3—C4—H4 | 119.9 | C19—C20—H20 | 120.0 |
| C4—C5—C6 | 121.5 (2) | C20—C21—C22 | 121.2 (2) |
| C4—C5—H5 | 119.3 | C20—C21—H21 | 119.4 |
| C6—C5—H5 | 119.3 | C22—C21—H21 | 119.4 |
| C1—C6—C5 | 116.98 (18) | C17—C22—C21 | 117.30 (18) |
| C1—C6—C7 | 122.36 (18) | C17—C22—C23 | 122.05 (18) |
| C5—C6—C7 | 120.66 (18) | C21—C22—C23 | 120.59 (18) |
| N1—C7—C6 | 119.91 (18) | N4—C23—C22 | 120.50 (18) |
| N1—C7—H7 | 120.0 | N4—C23—H23 | 119.8 |
| C6—C7—H7 | 120.0 | C22—C23—H23 | 119.8 |
| N3—C8—N2 | 123.26 (17) | N6—C24—N5 | 122.14 (17) |
| N3—C8—S1 | 115.56 (15) | N6—C24—S2 | 115.85 (15) |
| N2—C8—S1 | 121.18 (14) | N5—C24—S2 | 122.01 (14) |
| C10—C9—S1 | 111.14 (15) | C26—C25—S2 | 111.26 (15) |
| C10—C9—H9 | 124.4 | C26—C25—H25 | 124.4 |
| S1—C9—H9 | 124.4 | S2—C25—H25 | 124.4 |
| C9—C10—N3 | 114.57 (18) | C25—C26—N6 | 114.46 (18) |
| C9—C10—C11 | 126.78 (18) | C25—C26—C27 | 126.99 (18) |
| N3—C10—C11 | 118.58 (17) | N6—C26—C27 | 118.41 (17) |
| C16—C11—C12 | 118.49 (19) | C28—C27—C32 | 118.67 (19) |
| C16—C11—C10 | 120.61 (18) | C28—C27—C26 | 121.19 (18) |
| C12—C11—C10 | 120.88 (18) | C32—C27—C26 | 120.11 (18) |
| C13—C12—C11 | 120.9 (2) | C29—C28—C27 | 120.7 (2) |
| C13—C12—H12 | 119.6 | C29—C28—H28 | 119.6 |
| C11—C12—H12 | 119.6 | C27—C28—H28 | 119.6 |
| C12—C13—C14 | 120.3 (2) | C28—C29—C30 | 120.3 (2) |
| C12—C13—H13 | 119.9 | C28—C29—H29 | 119.9 |
| C14—C13—H13 | 119.9 | C30—C29—H29 | 119.9 |
| C15—C14—C13 | 119.7 (2) | C31—C30—C29 | 119.4 (2) |
| C15—C14—H14 | 120.1 | C31—C30—H30 | 120.3 |
| C13—C14—H14 | 120.1 | C29—C30—H30 | 120.3 |
| C14—C15—C16 | 120.1 (2) | C30—C31—C32 | 120.7 (2) |
| C14—C15—H15 | 119.9 | C30—C31—H31 | 119.7 |
| C16—C15—H15 | 119.9 | C32—C31—H31 | 119.7 |
| C11—C16—C15 | 120.5 (2) | C31—C32—C27 | 120.23 (19) |
| C11—C16—H16 | 119.8 | C31—C32—H32 | 119.9 |
| C15—C16—H16 | 119.8 | C27—C32—H32 | 119.9 |
| | | | |
| C7—N1—N2—C8 | 173.77 (18) | C23—N4—N5—C24 | −170.95 (18) |
| C6—C1—C2—C3 | 0.2 (3) | C22—C17—C18—C19 | −0.1 (3) |
| Cl1—C1—C2—C3 | 179.50 (17) | Cl2—C17—C18—C19 | −179.82 (17) |

| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | 0.6 (3) | C17—C18—C19—C20 | −0.1 (3) |
| C2—C3—C4—C5 | −0.8 (3) | C18—C19—C20—C21 | −0.2 (3) |
| C3—C4—C5—C6 | 0.2 (3) | C19—C20—C21—C22 | 0.6 (3) |
| C2—C1—C6—C5 | −0.8 (3) | C18—C17—C22—C21 | 0.5 (3) |
| Cl1—C1—C6—C5 | 179.94 (15) | Cl2—C17—C22—C21 | −179.80 (15) |
| C2—C1—C6—C7 | 179.12 (19) | C18—C17—C22—C23 | 177.84 (19) |
| Cl1—C1—C6—C7 | −0.1 (3) | Cl2—C17—C22—C23 | −2.4 (3) |
| C4—C5—C6—C1 | 0.6 (3) | C20—C21—C22—C17 | −0.7 (3) |
| C4—C5—C6—C7 | −179.34 (19) | C20—C21—C22—C23 | −178.12 (19) |
| N2—N1—C7—C6 | −179.33 (17) | N5—N4—C23—C22 | 175.08 (17) |
| C1—C6—C7—N1 | −172.59 (19) | C17—C22—C23—N4 | 177.70 (19) |
| C5—C6—C7—N1 | 7.3 (3) | C21—C22—C23—N4 | −5.0 (3) |
| C10—N3—C8—N2 | −177.70 (18) | C26—N6—C24—N5 | 179.78 (18) |
| C10—N3—C8—S1 | 2.3 (2) | C26—N6—C24—S2 | 0.6 (2) |
| N1—N2—C8—N3 | −179.50 (18) | N4—N5—C24—N6 | 167.05 (18) |
| N1—N2—C8—S1 | 0.5 (3) | N4—N5—C24—S2 | −13.8 (3) |
| C9—S1—C8—N3 | −1.55 (16) | C25—S2—C24—N6 | −0.41 (17) |
| C9—S1—C8—N2 | 178.42 (17) | C25—S2—C24—N5 | −179.58 (18) |
| C8—S1—C9—C10 | 0.33 (16) | C24—S2—C25—C26 | 0.07 (17) |
| S1—C9—C10—N3 | 0.9 (2) | S2—C25—C26—N6 | 0.3 (2) |
| S1—C9—C10—C11 | −175.89 (16) | S2—C25—C26—C27 | −175.32 (16) |
| C8—N3—C10—C9 | −2.0 (2) | C24—N6—C26—C25 | −0.6 (3) |
| C8—N3—C10—C11 | 175.04 (17) | C24—N6—C26—C27 | 175.43 (17) |
| C9—C10—C11—C16 | 168.6 (2) | C25—C26—C27—C28 | −24.5 (3) |
| N3—C10—C11—C16 | −8.0 (3) | N6—C26—C27—C28 | 160.09 (18) |
| C9—C10—C11—C12 | −10.1 (3) | C25—C26—C27—C32 | 153.4 (2) |
| N3—C10—C11—C12 | 173.30 (18) | N6—C26—C27—C32 | −22.0 (3) |
| C16—C11—C12—C13 | 0.2 (3) | C32—C27—C28—C29 | 0.6 (3) |
| C10—C11—C12—C13 | 178.94 (19) | C26—C27—C28—C29 | 178.54 (19) |
| C11—C12—C13—C14 | 0.6 (3) | C27—C28—C29—C30 | −0.5 (3) |
| C12—C13—C14—C15 | −0.6 (3) | C28—C29—C30—C31 | 0.2 (3) |
| C13—C14—C15—C16 | −0.3 (3) | C29—C30—C31—C32 | 0.0 (3) |
| C12—C11—C16—C15 | −1.2 (3) | C30—C31—C32—C27 | 0.1 (3) |
| C10—C11—C16—C15 | −179.87 (18) | C28—C27—C32—C31 | −0.4 (3) |
| C14—C15—C16—C11 | 1.2 (3) | C26—C27—C32—C31 | −178.36 (19) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···N6 | 0.91 | 2.02 | 2.901 (2) | 163 |
| N5—H5*A*···N3 | 0.91 | 2.05 | 2.946 (2) | 166 |
| C9—H9···S1$^{i}$ | 0.95 | 2.97 | 3.696 (2) | 134 |
| C25—H25···Cl1$^{ii}$ | 0.95 | 2.92 | 3.720 (2) | 143 |

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