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# 8b,8c-Diphenyl-2,6-bis(4-pyridylmethyl)-perhydro-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]fluorene-4,8-dithione chloroform solvate

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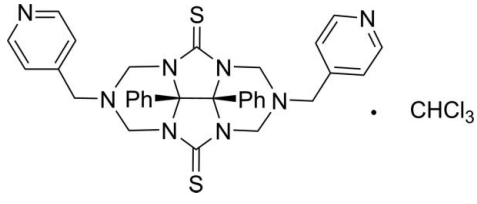
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.062; $wR$ factor = 0.210; data-to-parameter ratio = 13.7.

In the thioglycoluril system of the title compound, $C_{32}H_{30}N_8S_2\cdot CHCl_3$, the two pyridine rings are roughly parallel, forming a dihedral angle of 7.2 (1)°, and the distance between the centroids of the two phenyl rings is 3.951 (5) Å. The chloroform solvent molecule is linked to the main molecule *via* a weak C—H···N hydrogen bond.

## Related literature

For applications of glycoluril derivatives, see: Rowan *et al.* (1999). For the preparation of the title compound, see: Broan *et al.* (1989); Li *et al.* (2008).




## Experimental

### Crystal data

$C_{32}H_{30}N_8S_2\cdot CHCl_3$ $M_r$ = 710.13
Triclinic, $P\overline{1}$
$a$ = 9.5381 (6) Å
$b$ = 12.1712 (8) Å
$c$ = 14.8765 (9) Å
$\alpha$ = 100.978 (1)°
$\beta$ = 91.699 (1)°
$\gamma$ = 98.500 (1)°
$V$ = 1673.81 (18) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.44 mm$^{-1}$
$T$ = 294 K
0.20 × 0.10 × 0.10 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer
11061 measured reflections
5690 independent reflections
2136 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.109

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.210
$S$ = 0.85
5690 reflections
415 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C33—H33···N8$^{i}$ | 0.98 | 2.33 | 3.168 (9) | 142 (8) |

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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*.

The authors are grateful to the Central China Normal University for financial support and to Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2714).

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# supplementary materials

*Acta Cryst.* (2010). E**66**, o1524 [ **doi:10.1107/S1600536810020040** ]

# 8b,8c-Diphenyl-2,6-bis(4-pyridylmethyl)perhydro-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]fluorene-4,8-dithione chloroform solvate

**C. Deng, W. Shu and D. Zhang**

## Comment

Recently, molecular clips based on concave glycoluril unit have been widely investigated in supramolecular chemistry (Rowan *et al.*, 1999). We report here the structure of the title compound (Fig. 1), which is a derivative of thioglycoluril with two pyridine units. We believe the title compound would offer the possibility in construction of coordination framework with novel patterns (Li *et al.*, 2008). The crystal packing exhibits weak intermolecular C—H···N hydrogen bond (Table 1) between the chloroform solvent molecule and the main molecule.

## Experimental

The title compound was synthesized according to the literature (Broan *et al.*, 1989; Li *et al.*, 2008). Crystals of (I) suitable for X-ray diffraction were grown by slow evaporation of a chloroform-methanol (1:2) solution of the title compound under 293 K.

## Refinement

All H atoms were positioned in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93-0.98 Å and $U_{iso}(H) = 1.2\text{-}1.5U_{eq}(C)$. The low ratio observed/unique reflections (0.38) was mainly caused by poor quality of the crystal selected for measurements.

## Figures

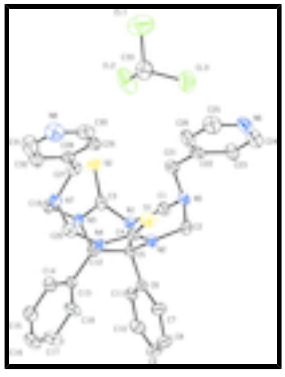


Fig. 1. A view of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 30% probability level.

## 8b,8c-Diphenyl-2,6-bis(4-pyridylmethyl)perhydro-2,3a,4a,6,7a,8a- hexaazacyclopenta[*def*]fluorene-4,8-dithione chloroform solvate

### *Crystal data*

| | |
|---|---|
| $C_{32}H_{30}N_8S_2{\cdot}CHCl_3$ | $V$ = 1673.81 (18) Å$^3$ |
| $M_r$ = 710.13 | $Z$ = 2 |
| Triclinic, $P\overline{1}$ | $F(000)$ = 736 |
| Hall symbol: -P 1 | $D_x$ = 1.409 Mg m$^{-3}$ |
| $a$ = 9.5381 (6) Å | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| $b$ = 12.1712 (8) Å | μ = 0.44 mm$^{-1}$ |
| $c$ = 14.8765 (9) Å | $T$ = 294 K |
| α = 100.978 (1)° | Block, colourless |
| β = 91.699 (1)° | 0.20 × 0.10 × 0.10 mm |
| γ = 98.500 (1)° | |

### *Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 2136 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.109 |
| graphite | $\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.7° |
| phi and ω scans | $h$ = −11→11 |
| 11061 measured reflections | $k$ = −14→13 |
| 5690 independent reflections | $l$ = −17→17 |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)]$ = 0.062 | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2)$ = 0.210 | H-atom parameters constrained |
| $S$ = 0.85 | $w = 1/[\sigma^2(F_o^2) + (0.1041P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 5690 reflections | $(\Delta/\sigma)_{max}$ = 0.001 |
| 415 parameters | $\Delta\rho_{max}$ = 0.47 e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min}$ = −0.39 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 $F^2 > \sigma(F^2)$ 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. Because of the poor quality of crystal, the ratio of Observed/Unique Reflections is 38%.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.4638 (5) | 0.4696 (4) | 0.1361 (4) | 0.0426 (14) |
| H1A | 0.4056 | 0.4515 | 0.0790 | 0.051* |
| H1B | 0.4486 | 0.5435 | 0.1685 | 0.051* |
| C2 | 0.6423 (6) | 0.3623 (4) | 0.0735 (4) | 0.0461 (15) |
| H2A | 0.7434 | 0.3659 | 0.0658 | 0.055* |
| H2B | 0.5934 | 0.3385 | 0.0133 | 0.055* |
| C3 | 0.4314 (5) | 0.4077 (4) | 0.2860 (4) | 0.0395 (14) |
| C4 | 0.6795 (5) | 0.2382 (4) | 0.1877 (4) | 0.0391 (14) |
| C5 | 0.4489 (5) | 0.2700 (4) | 0.1553 (3) | 0.0295 (12) |
| C6 | 0.3463 (5) | 0.2069 (4) | 0.0750 (3) | 0.0344 (13) |
| C7 | 0.3908 (6) | 0.1281 (5) | 0.0063 (4) | 0.0518 (16) |
| H7 | 0.4868 | 0.1222 | 0.0040 | 0.062* |
| C8 | 0.2952 (6) | 0.0585 (5) | −0.0584 (4) | 0.0565 (17) |
| H8 | 0.3262 | 0.0045 | −0.1031 | 0.068* |
| C9 | 0.1541 (7) | 0.0688 (5) | −0.0572 (4) | 0.0590 (17) |
| H9 | 0.0895 | 0.0223 | −0.1014 | 0.071* |
| C10 | 0.1082 (6) | 0.1478 (5) | 0.0096 (4) | 0.0546 (16) |
| H10 | 0.0125 | 0.1551 | 0.0104 | 0.065* |
| C11 | 0.2042 (6) | 0.2160 (4) | 0.0753 (4) | 0.0436 (15) |
| H11 | 0.1725 | 0.2690 | 0.1205 | 0.052* |
| C12 | 0.4498 (5) | 0.2132 (4) | 0.2406 (3) | 0.0322 (13) |
| C13 | 0.3367 (5) | 0.1100 (4) | 0.2397 (3) | 0.0351 (13) |
| C14 | 0.2062 (6) | 0.1236 (5) | 0.2759 (4) | 0.0567 (17) |
| H14 | 0.1877 | 0.1954 | 0.3020 | 0.068* |
| C15 | 0.1037 (7) | 0.0290 (6) | 0.2726 (4) | 0.0675 (19) |
| H15 | 0.0167 | 0.0376 | 0.2975 | 0.081* |
| C16 | 0.1289 (7) | −0.0762 (6) | 0.2335 (5) | 0.077 (2) |
| H16 | 0.0588 | −0.1389 | 0.2304 | 0.093* |
| C17 | 0.2577 (7) | −0.0896 (5) | 0.1986 (5) | 0.072 (2) |
| H17 | 0.2765 | −0.1614 | 0.1730 | 0.086* |
| C18 | 0.3601 (6) | 0.0048 (5) | 0.2017 (4) | 0.0570 (17) |
| H18 | 0.4470 | −0.0045 | 0.1770 | 0.068* |
| C19 | 0.4813 (6) | 0.2929 (4) | 0.4068 (3) | 0.0455 (15) |
| H19A | 0.4783 | 0.3621 | 0.4511 | 0.055* |
| H19B | 0.4162 | 0.2327 | 0.4245 | 0.055* |
| C20 | 0.6388 (6) | 0.1716 (4) | 0.3368 (4) | 0.0469 (15) |
| H20A | 0.5837 | 0.1033 | 0.3497 | 0.056* |
| H20B | 0.7376 | 0.1611 | 0.3364 | 0.056* |
| C21 | 0.7120 (6) | 0.5325 (5) | 0.1933 (4) | 0.0530 (16) |

| | | | | |
|---|---|---|---|---|
| H21A | 0.7130 | 0.4839 | 0.2379 | 0.064* |
| H21B | 0.6786 | 0.6012 | 0.2227 | 0.064* |
| C22 | 0.8592 (6) | 0.5619 (4) | 0.1653 (4) | 0.0482 (16) |
| C23 | 0.8900 (6) | 0.6050 (5) | 0.0882 (4) | 0.0577 (17) |
| H23 | 0.8157 | 0.6157 | 0.0505 | 0.069* |
| C24 | 1.0254 (7) | 0.6325 (5) | 0.0652 (5) | 0.068 (2) |
| H24 | 1.0400 | 0.6619 | 0.0124 | 0.081* |
| C25 | 1.1118 (7) | 0.5806 (6) | 0.1887 (5) | 0.073 (2) |
| H25 | 1.1882 | 0.5730 | 0.2261 | 0.088* |
| C26 | 0.9754 (8) | 0.5499 (6) | 0.2152 (5) | 0.075 (2) |
| H26 | 0.9635 | 0.5204 | 0.2682 | 0.090* |
| C27 | 0.7389 (6) | 0.3621 (5) | 0.4180 (4) | 0.0481 (15) |
| H27A | 0.7105 | 0.4275 | 0.4570 | 0.058* |
| H27B | 0.7554 | 0.3804 | 0.3582 | 0.058* |
| C28 | 0.8721 (5) | 0.3375 (4) | 0.4579 (4) | 0.0384 (14) |
| C29 | 1.0046 (6) | 0.3720 (4) | 0.4278 (4) | 0.0505 (16) |
| H29 | 1.0126 | 0.4087 | 0.3784 | 0.061* |
| C30 | 1.1231 (6) | 0.3516 (5) | 0.4715 (5) | 0.0576 (18) |
| H30 | 1.2098 | 0.3761 | 0.4491 | 0.069* |
| C31 | 0.9976 (8) | 0.2679 (6) | 0.5694 (5) | 0.084 (2) |
| H31 | 0.9927 | 0.2311 | 0.6189 | 0.101* |
| C32 | 0.8720 (7) | 0.2838 (6) | 0.5309 (5) | 0.0676 (19) |
| H32 | 0.7865 | 0.2582 | 0.5542 | 0.081* |
| C33 | 0.6754 (7) | 0.8554 (6) | 0.3853 (5) | 0.076 (2) |
| H33 | 0.6949 | 0.7839 | 0.4001 | 0.091* |
| Cl1 | 0.7802 (3) | 0.9693 (2) | 0.4618 (2) | 0.1451 (11) |
| Cl2 | 0.4992 (2) | 0.86364 (19) | 0.39764 (19) | 0.1222 (10) |
| Cl3 | 0.7234 (2) | 0.86119 (18) | 0.27531 (15) | 0.1061 (8) |
| N1 | 0.4182 (4) | 0.3843 (3) | 0.1924 (3) | 0.0370 (11) |
| N2 | 0.5961 (4) | 0.2785 (3) | 0.1304 (3) | 0.0380 (12) |
| N3 | 0.4351 (4) | 0.3080 (3) | 0.3151 (3) | 0.0349 (11) |
| N4 | 0.5925 (4) | 0.1866 (3) | 0.2456 (3) | 0.0342 (11) |
| N5 | 0.6126 (5) | 0.4746 (4) | 0.1154 (3) | 0.0446 (12) |
| N6 | 1.1383 (6) | 0.6195 (4) | 0.1144 (5) | 0.0697 (17) |
| N7 | 0.6238 (4) | 0.2652 (3) | 0.4087 (3) | 0.0388 (11) |
| N8 | 1.1278 (6) | 0.3010 (5) | 0.5420 (5) | 0.0756 (17) |
| S1 | 0.85378 (14) | 0.24229 (12) | 0.18583 (10) | 0.0545 (5) |
| S2 | 0.43168 (16) | 0.53272 (12) | 0.35358 (10) | 0.0527 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.042 (3) | 0.042 (3) | 0.044 (3) | 0.002 (3) | 0.006 (3) | 0.013 (3) |
| C2 | 0.042 (3) | 0.056 (4) | 0.040 (3) | 0.002 (3) | 0.015 (3) | 0.009 (3) |
| C3 | 0.026 (3) | 0.050 (4) | 0.046 (4) | 0.009 (3) | 0.014 (3) | 0.012 (3) |
| C4 | 0.038 (3) | 0.028 (3) | 0.046 (3) | 0.004 (3) | 0.014 (3) | −0.006 (2) |
| C5 | 0.025 (3) | 0.032 (3) | 0.031 (3) | 0.006 (2) | 0.004 (2) | 0.003 (2) |
| C6 | 0.039 (3) | 0.032 (3) | 0.032 (3) | 0.001 (2) | 0.010 (3) | 0.006 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.039 (3) | 0.055 (4) | 0.052 (4) | −0.002 (3) | 0.013 (3) | −0.009 (3) |
| C8 | 0.047 (4) | 0.063 (4) | 0.047 (4) | 0.000 (3) | 0.006 (3) | −0.012 (3) |
| C9 | 0.061 (4) | 0.058 (4) | 0.046 (4) | −0.009 (3) | −0.003 (3) | −0.005 (3) |
| C10 | 0.034 (3) | 0.055 (4) | 0.072 (5) | −0.003 (3) | −0.002 (3) | 0.014 (3) |
| C11 | 0.042 (3) | 0.040 (3) | 0.046 (4) | 0.002 (3) | 0.014 (3) | 0.005 (3) |
| C12 | 0.026 (3) | 0.032 (3) | 0.037 (3) | 0.001 (2) | 0.007 (2) | 0.005 (2) |
| C13 | 0.035 (3) | 0.038 (3) | 0.034 (3) | −0.002 (3) | 0.000 (3) | 0.015 (2) |
| C14 | 0.050 (4) | 0.060 (4) | 0.054 (4) | −0.006 (3) | 0.018 (3) | 0.007 (3) |
| C15 | 0.046 (4) | 0.090 (5) | 0.058 (4) | −0.019 (4) | 0.009 (3) | 0.013 (4) |
| C16 | 0.066 (5) | 0.066 (5) | 0.091 (5) | −0.026 (4) | 0.008 (4) | 0.020 (4) |
| C17 | 0.066 (5) | 0.038 (4) | 0.104 (6) | −0.006 (3) | −0.002 (4) | 0.007 (4) |
| C18 | 0.044 (4) | 0.039 (4) | 0.083 (5) | −0.007 (3) | 0.009 (3) | 0.009 (3) |
| C19 | 0.053 (4) | 0.047 (3) | 0.036 (3) | 0.006 (3) | 0.014 (3) | 0.008 (3) |
| C20 | 0.038 (3) | 0.039 (3) | 0.066 (4) | 0.008 (3) | 0.008 (3) | 0.013 (3) |
| C21 | 0.050 (4) | 0.047 (4) | 0.053 (4) | −0.010 (3) | 0.012 (3) | 0.002 (3) |
| C22 | 0.039 (4) | 0.040 (3) | 0.060 (4) | −0.006 (3) | 0.006 (3) | 0.004 (3) |
| C23 | 0.041 (4) | 0.067 (4) | 0.066 (4) | 0.001 (3) | 0.012 (3) | 0.022 (3) |
| C24 | 0.055 (4) | 0.070 (5) | 0.086 (5) | 0.008 (4) | 0.010 (4) | 0.034 (4) |
| C25 | 0.034 (4) | 0.101 (6) | 0.083 (5) | 0.000 (4) | −0.019 (4) | 0.026 (5) |
| C26 | 0.065 (5) | 0.082 (5) | 0.077 (5) | −0.017 (4) | 0.012 (4) | 0.032 (4) |
| C27 | 0.051 (4) | 0.048 (4) | 0.044 (4) | 0.008 (3) | 0.002 (3) | 0.005 (3) |
| C28 | 0.030 (3) | 0.040 (3) | 0.040 (3) | 0.003 (3) | −0.006 (3) | 0.000 (3) |
| C29 | 0.040 (4) | 0.047 (4) | 0.057 (4) | −0.007 (3) | 0.009 (3) | 0.002 (3) |
| C30 | 0.025 (3) | 0.064 (4) | 0.075 (5) | 0.006 (3) | −0.008 (3) | −0.006 (4) |
| C31 | 0.069 (5) | 0.110 (6) | 0.084 (6) | 0.004 (5) | −0.016 (5) | 0.056 (5) |
| C32 | 0.042 (4) | 0.085 (5) | 0.078 (5) | −0.004 (4) | 0.007 (4) | 0.031 (4) |
| C33 | 0.083 (5) | 0.081 (5) | 0.075 (5) | 0.032 (4) | 0.004 (4) | 0.029 (4) |
| Cl1 | 0.178 (3) | 0.1084 (19) | 0.139 (2) | 0.0305 (19) | −0.036 (2) | 0.0012 (17) |
| Cl2 | 0.0895 (16) | 0.1205 (19) | 0.178 (2) | 0.0245 (14) | 0.0594 (16) | 0.0679 (17) |
| Cl3 | 0.1197 (18) | 0.1078 (16) | 0.0969 (16) | 0.0181 (14) | 0.0459 (14) | 0.0292 (12) |
| N1 | 0.041 (3) | 0.034 (2) | 0.034 (3) | 0.003 (2) | 0.012 (2) | 0.0037 (19) |
| N2 | 0.029 (3) | 0.039 (3) | 0.044 (3) | −0.001 (2) | 0.013 (2) | 0.005 (2) |
| N3 | 0.033 (2) | 0.038 (3) | 0.032 (3) | 0.003 (2) | 0.011 (2) | 0.0018 (19) |
| N4 | 0.028 (2) | 0.032 (2) | 0.040 (3) | 0.0028 (19) | 0.003 (2) | 0.0026 (19) |
| N5 | 0.041 (3) | 0.044 (3) | 0.046 (3) | −0.004 (2) | 0.011 (2) | 0.010 (2) |
| N6 | 0.062 (4) | 0.056 (3) | 0.090 (5) | −0.003 (3) | 0.027 (4) | 0.017 (3) |
| N7 | 0.030 (2) | 0.043 (3) | 0.042 (3) | 0.003 (2) | 0.002 (2) | 0.008 (2) |
| N8 | 0.057 (4) | 0.079 (4) | 0.095 (5) | 0.019 (3) | −0.005 (4) | 0.021 (4) |
| S1 | 0.0312 (8) | 0.0584 (10) | 0.0680 (11) | 0.0046 (7) | 0.0157 (8) | −0.0022 (8) |
| S2 | 0.0588 (10) | 0.0434 (9) | 0.0501 (9) | 0.0087 (8) | 0.0122 (8) | −0.0072 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N5 | 1.456 (6) | C18—H18 | 0.9300 |
| C1—N1 | 1.479 (6) | C19—N7 | 1.449 (6) |
| C1—H1A | 0.9700 | C19—N3 | 1.474 (6) |
| C1—H1B | 0.9700 | C19—H19A | 0.9700 |
| C2—N5 | 1.462 (6) | C19—H19B | 0.9700 |
| C2—N2 | 1.472 (6) | C20—N7 | 1.434 (6) |

| | | | |
|---|---|---|---|
| C2—H2A | 0.9700 | C20—N4 | 1.466 (7) |
| C2—H2B | 0.9700 | C20—H20A | 0.9700 |
| C3—N1 | 1.366 (6) | C20—H20B | 0.9700 |
| C3—N3 | 1.368 (6) | C21—N5 | 1.471 (7) |
| C3—S2 | 1.656 (5) | C21—C22 | 1.488 (7) |
| C4—N2 | 1.355 (6) | C21—H21A | 0.9700 |
| C4—N4 | 1.386 (6) | C21—H21B | 0.9700 |
| C4—S1 | 1.657 (5) | C22—C26 | 1.357 (8) |
| C5—N2 | 1.457 (5) | C22—C23 | 1.372 (8) |
| C5—N1 | 1.470 (5) | C23—C24 | 1.356 (8) |
| C5—C6 | 1.527 (7) | C23—H23 | 0.9300 |
| C5—C12 | 1.557 (7) | C24—N6 | 1.329 (8) |
| C6—C11 | 1.376 (6) | C24—H24 | 0.9300 |
| C6—C7 | 1.385 (6) | C25—N6 | 1.300 (8) |
| C7—C8 | 1.373 (7) | C25—C26 | 1.390 (8) |
| C7—H7 | 0.9300 | C25—H25 | 0.9300 |
| C8—C9 | 1.370 (7) | C26—H26 | 0.9300 |
| C8—H8 | 0.9300 | C27—N7 | 1.469 (6) |
| C9—C10 | 1.374 (7) | C27—C28 | 1.481 (7) |
| C9—H9 | 0.9300 | C27—H27A | 0.9700 |
| C10—C11 | 1.377 (7) | C27—H27B | 0.9700 |
| C10—H10 | 0.9300 | C28—C32 | 1.369 (8) |
| C11—H11 | 0.9300 | C28—C29 | 1.386 (7) |
| C12—N4 | 1.448 (6) | C29—C30 | 1.365 (8) |
| C12—N3 | 1.467 (5) | C29—H29 | 0.9300 |
| C12—C13 | 1.528 (6) | C30—N8 | 1.316 (8) |
| C13—C18 | 1.350 (7) | C30—H30 | 0.9300 |
| C13—C14 | 1.391 (6) | C31—N8 | 1.348 (8) |
| C14—C15 | 1.389 (8) | C31—C32 | 1.368 (9) |
| C14—H14 | 0.9300 | C31—H31 | 0.9300 |
| C15—C16 | 1.361 (8) | C32—H32 | 0.9300 |
| C15—H15 | 0.9300 | C33—Cl2 | 1.711 (6) |
| C16—C17 | 1.367 (8) | C33—Cl3 | 1.724 (7) |
| C16—H16 | 0.9300 | C33—Cl1 | 1.772 (8) |
| C17—C18 | 1.386 (7) | C33—H33 | 0.9800 |
| C17—H17 | 0.9300 | | |
| N5—C1—N1 | 112.5 (4) | N7—C20—H20A | 108.9 |
| N5—C1—H1A | 109.1 | N4—C20—H20A | 108.9 |
| N1—C1—H1A | 109.1 | N7—C20—H20B | 108.9 |
| N5—C1—H1B | 109.1 | N4—C20—H20B | 108.9 |
| N1—C1—H1B | 109.1 | H20A—C20—H20B | 107.7 |
| H1A—C1—H1B | 107.8 | N5—C21—C22 | 112.7 (4) |
| N5—C2—N2 | 111.2 (4) | N5—C21—H21A | 109.0 |
| N5—C2—H2A | 109.4 | C22—C21—H21A | 109.0 |
| N2—C2—H2A | 109.4 | N5—C21—H21B | 109.0 |
| N5—C2—H2B | 109.4 | C22—C21—H21B | 109.0 |
| N2—C2—H2B | 109.4 | H21A—C21—H21B | 107.8 |
| H2A—C2—H2B | 108.0 | C26—C22—C23 | 114.0 (6) |
| N1—C3—N3 | 108.0 (4) | C26—C22—C21 | 122.6 (6) |

| | | | |
|---|---|---|---|
| N1—C3—S2 | 126.4 (4) | C23—C22—C21 | 123.4 (6) |
| N3—C3—S2 | 125.4 (4) | C24—C23—C22 | 122.0 (6) |
| N2—C4—N4 | 108.0 (4) | C24—C23—H23 | 119.0 |
| N2—C4—S1 | 126.5 (4) | C22—C23—H23 | 119.0 |
| N4—C4—S1 | 125.4 (5) | N6—C24—C23 | 123.4 (6) |
| N2—C5—N1 | 108.9 (4) | N6—C24—H24 | 118.3 |
| N2—C5—C6 | 111.8 (3) | C23—C24—H24 | 118.3 |
| N1—C5—C6 | 112.8 (4) | N6—C25—C26 | 123.5 (7) |
| N2—C5—C12 | 103.1 (4) | N6—C25—H25 | 118.2 |
| N1—C5—C12 | 103.9 (3) | C26—C25—H25 | 118.2 |
| C6—C5—C12 | 115.6 (4) | C22—C26—C25 | 121.4 (6) |
| C11—C6—C7 | 118.3 (5) | C22—C26—H26 | 119.3 |
| C11—C6—C5 | 120.8 (4) | C25—C26—H26 | 119.3 |
| C7—C6—C5 | 120.4 (4) | N7—C27—C28 | 111.6 (4) |
| C8—C7—C6 | 120.9 (5) | N7—C27—H27A | 109.3 |
| C8—C7—H7 | 119.5 | C28—C27—H27A | 109.3 |
| C6—C7—H7 | 119.5 | N7—C27—H27B | 109.3 |
| C9—C8—C7 | 120.0 (5) | C28—C27—H27B | 109.3 |
| C9—C8—H8 | 120.0 | H27A—C27—H27B | 108.0 |
| C7—C8—H8 | 120.0 | C32—C28—C29 | 115.7 (6) |
| C8—C9—C10 | 119.9 (6) | C32—C28—C27 | 121.4 (5) |
| C8—C9—H9 | 120.0 | C29—C28—C27 | 122.8 (5) |
| C10—C9—H9 | 120.0 | C30—C29—C28 | 119.3 (6) |
| C9—C10—C11 | 119.8 (5) | C30—C29—H29 | 120.3 |
| C9—C10—H10 | 120.1 | C28—C29—H29 | 120.3 |
| C11—C10—H10 | 120.1 | N8—C30—C29 | 126.9 (6) |
| C6—C11—C10 | 121.0 (5) | N8—C30—H30 | 116.5 |
| C6—C11—H11 | 119.5 | C29—C30—H30 | 116.5 |
| C10—C11—H11 | 119.5 | N8—C31—C32 | 125.7 (7) |
| N4—C12—N3 | 109.6 (4) | N8—C31—H31 | 117.2 |
| N4—C12—C13 | 112.3 (4) | C32—C31—H31 | 117.2 |
| N3—C12—C13 | 112.3 (3) | C31—C32—C28 | 120.0 (6) |
| N4—C12—C5 | 103.0 (3) | C31—C32—H32 | 120.0 |
| N3—C12—C5 | 101.7 (4) | C28—C32—H32 | 120.0 |
| C13—C12—C5 | 116.9 (4) | Cl2—C33—Cl3 | 112.1 (4) |
| C18—C13—C14 | 119.0 (5) | Cl2—C33—Cl1 | 109.7 (4) |
| C18—C13—C12 | 120.8 (4) | Cl3—C33—Cl1 | 108.3 (4) |
| C14—C13—C12 | 120.2 (4) | Cl2—C33—H33 | 108.9 |
| C15—C14—C13 | 119.3 (5) | Cl3—C33—H33 | 108.9 |
| C15—C14—H14 | 120.4 | Cl1—C33—H33 | 108.9 |
| C13—C14—H14 | 120.4 | C3—N1—C5 | 111.1 (4) |
| C16—C15—C14 | 120.8 (6) | C3—N1—C1 | 123.0 (4) |
| C16—C15—H15 | 119.6 | C5—N1—C1 | 114.7 (3) |
| C14—C15—H15 | 119.6 | C4—N2—C5 | 112.7 (4) |
| C15—C16—C17 | 119.8 (6) | C4—N2—C2 | 126.8 (4) |
| C15—C16—H16 | 120.1 | C5—N2—C2 | 116.3 (4) |
| C17—C16—H16 | 120.1 | C3—N3—C12 | 113.4 (4) |
| C16—C17—C18 | 119.5 (6) | C3—N3—C19 | 127.7 (4) |
| C16—C17—H17 | 120.3 | C12—N3—C19 | 114.3 (4) |

| | | | |
|---|---|---|---|
| C18—C17—H17 | 120.3 | C4—N4—C12 | 111.8 (4) |
| C13—C18—C17 | 121.6 (5) | C4—N4—C20 | 124.5 (4) |
| C13—C18—H18 | 119.2 | C12—N4—C20 | 113.6 (4) |
| C17—C18—H18 | 119.2 | C1—N5—C2 | 110.7 (4) |
| N7—C19—N3 | 111.8 (4) | C1—N5—C21 | 113.8 (4) |
| N7—C19—H19A | 109.3 | C2—N5—C21 | 113.6 (4) |
| N3—C19—H19A | 109.3 | C25—N6—C24 | 115.7 (6) |
| N7—C19—H19B | 109.3 | C20—N7—C19 | 111.5 (4) |
| N3—C19—H19B | 109.3 | C20—N7—C27 | 114.4 (4) |
| H19A—C19—H19B | 107.9 | C19—N7—C27 | 115.4 (4) |
| N7—C20—N4 | 113.5 (4) | C30—N8—C31 | 112.4 (6) |
| N2—C5—C6—C11 | −162.1 (4) | C12—C5—N1—C3 | 12.7 (5) |
| N1—C5—C6—C11 | −38.9 (6) | N2—C5—N1—C1 | 48.5 (6) |
| C12—C5—C6—C11 | 80.4 (6) | C6—C5—N1—C1 | −76.3 (5) |
| N2—C5—C6—C7 | 26.4 (7) | C12—C5—N1—C1 | 157.8 (4) |
| N1—C5—C6—C7 | 149.6 (4) | N5—C1—N1—C3 | 87.6 (5) |
| C12—C5—C6—C7 | −91.1 (5) | N5—C1—N1—C5 | −52.9 (6) |
| C11—C6—C7—C8 | −1.8 (8) | N4—C4—N2—C5 | 8.2 (5) |
| C5—C6—C7—C8 | 170.0 (5) | S1—C4—N2—C5 | −175.2 (3) |
| C6—C7—C8—C9 | 1.8 (9) | N4—C4—N2—C2 | 163.9 (4) |
| C7—C8—C9—C10 | −0.8 (10) | S1—C4—N2—C2 | −19.5 (7) |
| C8—C9—C10—C11 | −0.3 (9) | N1—C5—N2—C4 | 108.8 (4) |
| C7—C6—C11—C10 | 0.7 (8) | C6—C5—N2—C4 | −125.8 (4) |
| C5—C6—C11—C10 | −171.0 (5) | C12—C5—N2—C4 | −1.1 (5) |
| C9—C10—C11—C6 | 0.4 (9) | N1—C5—N2—C2 | −49.6 (5) |
| N2—C5—C12—N4 | −5.9 (4) | C6—C5—N2—C2 | 75.7 (5) |
| N1—C5—C12—N4 | −119.5 (4) | C12—C5—N2—C2 | −159.5 (4) |
| C6—C5—C12—N4 | 116.3 (4) | N5—C2—N2—C4 | −101.2 (5) |
| N2—C5—C12—N3 | 107.6 (4) | N5—C2—N2—C5 | 53.7 (6) |
| N1—C5—C12—N3 | −6.0 (4) | N1—C3—N3—C12 | 10.3 (5) |
| C6—C5—C12—N3 | −130.1 (4) | S2—C3—N3—C12 | −173.6 (3) |
| N2—C5—C12—C13 | −129.7 (4) | N1—C3—N3—C19 | 164.3 (4) |
| N1—C5—C12—C13 | 116.7 (4) | S2—C3—N3—C19 | −19.6 (7) |
| C6—C5—C12—C13 | −7.4 (5) | N4—C12—N3—C3 | 106.3 (5) |
| N4—C12—C13—C18 | −31.2 (7) | C13—C12—N3—C3 | −128.0 (4) |
| N3—C12—C13—C18 | −155.3 (5) | C5—C12—N3—C3 | −2.2 (5) |
| C5—C12—C13—C18 | 87.6 (6) | N4—C12—N3—C19 | −51.3 (5) |
| N4—C12—C13—C14 | 150.7 (5) | C13—C12—N3—C19 | 74.4 (5) |
| N3—C12—C13—C14 | 26.6 (7) | C5—C12—N3—C19 | −159.8 (4) |
| C5—C12—C13—C14 | −90.4 (5) | N7—C19—N3—C3 | −101.1 (5) |
| C18—C13—C14—C15 | 0.4 (9) | N7—C19—N3—C12 | 52.7 (5) |
| C12—C13—C14—C15 | 178.6 (5) | N2—C4—N4—C12 | −12.5 (5) |
| C13—C14—C15—C16 | −0.9 (10) | S1—C4—N4—C12 | 170.8 (3) |
| C14—C15—C16—C17 | 1.4 (11) | N2—C4—N4—C20 | −155.4 (4) |
| C15—C16—C17—C18 | −1.5 (11) | S1—C4—N4—C20 | 27.9 (6) |
| C14—C13—C18—C17 | −0.5 (9) | N3—C12—N4—C4 | −96.3 (4) |
| C12—C13—C18—C17 | −178.6 (5) | C13—C12—N4—C4 | 138.1 (4) |
| C16—C17—C18—C13 | 1.1 (10) | C5—C12—N4—C4 | 11.3 (5) |
| N5—C21—C22—C26 | −139.0 (6) | N3—C12—N4—C20 | 50.8 (5) |

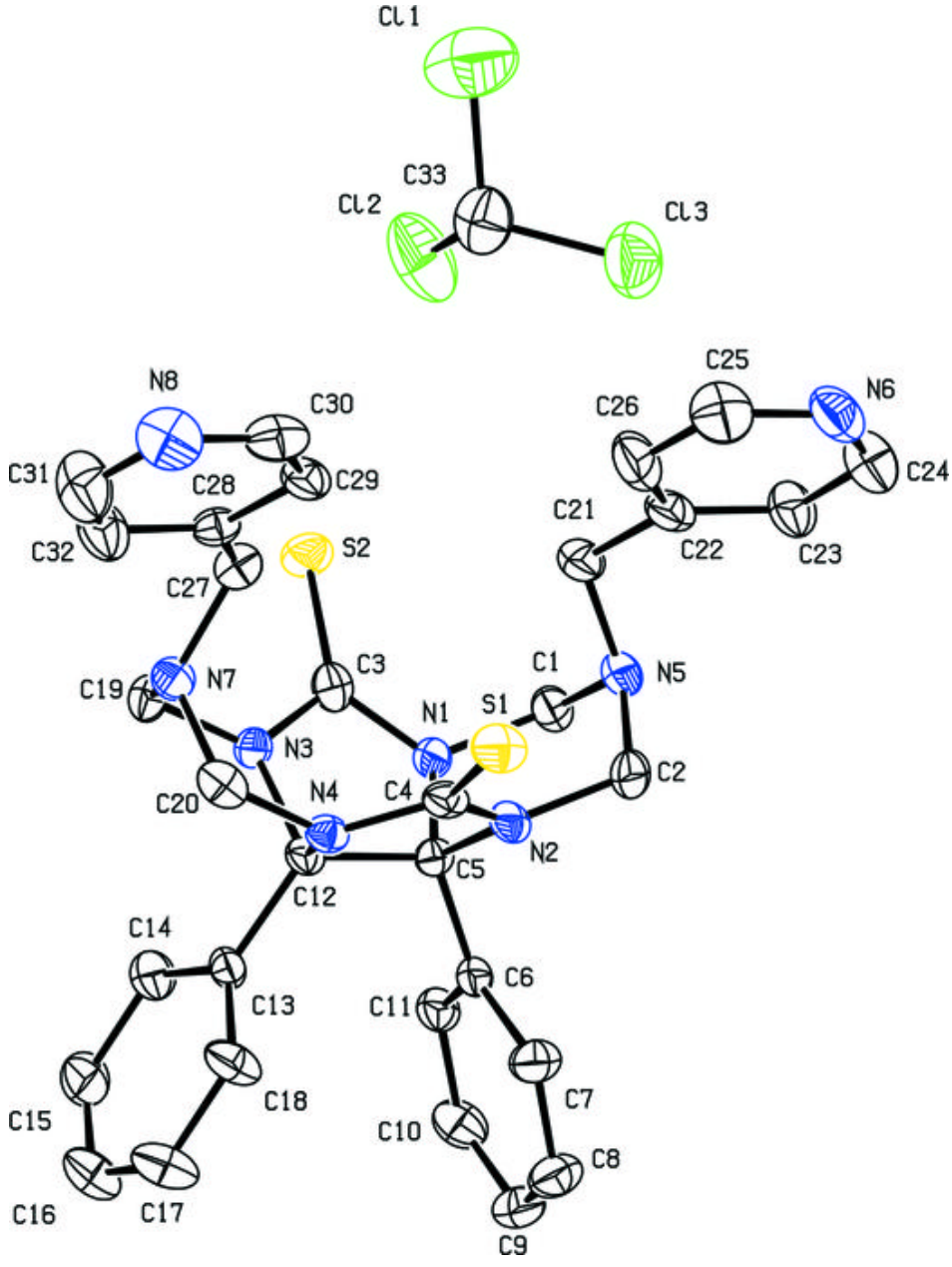
| | | | |
|---|---|---|---|
| N5—C21—C22—C23 | 42.0 (7) | C13—C12—N4—C20 | −74.8 (5) |
| C26—C22—C23—C24 | 0.0 (9) | C5—C12—N4—C20 | 158.5 (4) |
| C21—C22—C23—C24 | 179.1 (5) | N7—C20—N4—C4 | 89.0 (5) |
| C22—C23—C24—N6 | 0.5 (10) | N7—C20—N4—C12 | −53.3 (5) |
| C23—C22—C26—C25 | 0.7 (9) | N1—C1—N5—C2 | 53.9 (6) |
| C21—C22—C26—C25 | −178.4 (5) | N1—C1—N5—C21 | −75.5 (6) |
| N6—C25—C26—C22 | −2.0 (11) | N2—C2—N5—C1 | −53.5 (6) |
| N7—C27—C28—C32 | −43.3 (7) | N2—C2—N5—C21 | 76.0 (5) |
| N7—C27—C28—C29 | 140.3 (5) | C22—C21—N5—C1 | −165.7 (4) |
| C32—C28—C29—C30 | −0.2 (8) | C22—C21—N5—C2 | 66.4 (6) |
| C27—C28—C29—C30 | 176.3 (5) | C26—C25—N6—C24 | 2.3 (10) |
| C28—C29—C30—N8 | −0.1 (9) | C23—C24—N6—C25 | −1.6 (10) |
| N8—C31—C32—C28 | 0.0 (12) | N4—C20—N7—C19 | 52.5 (6) |
| C29—C28—C32—C31 | 0.3 (9) | N4—C20—N7—C27 | −80.7 (5) |
| C27—C28—C32—C31 | −176.3 (6) | N3—C19—N7—C20 | −51.7 (5) |
| N3—C3—N1—C5 | −14.4 (5) | N3—C19—N7—C27 | 81.0 (5) |
| S2—C3—N1—C5 | 169.5 (3) | C28—C27—N7—C20 | −72.7 (5) |
| N3—C3—N1—C1 | −156.2 (4) | C28—C27—N7—C19 | 155.9 (4) |
| S2—C3—N1—C1 | 27.7 (6) | C29—C30—N8—C31 | 0.4 (9) |
| N2—C5—N1—C3 | −96.7 (5) | C32—C31—N8—C30 | −0.3 (11) |
| C6—C5—N1—C3 | 138.6 (4) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C33—H33···N8$^{i}$ | 0.98 | 2.33 | 3.168 (9) | 142 (8) |

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

**Fig. 1**


Cl1
C33
Cl2
Cl3
N8
C30
C31
C28
C29
C32
S2
C27
C25
N6
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C24
C21
C22
C23
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