Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-(Cyclohexylsulfanyl)-1-[(*E*)-2-(cyclohexylsulfanyl)-1-phenylethenyl]-3-phenyl-1*H*-pyrazole

**P. Ramesh,[a] A. Subbiahpandi,[a] Ramaiyan Manikannan,[b] S. Muthusubramanian[b] and M. N. Ponnuswamy[c]***

[a]Department of Physics, Presidency College (Autonomous), Chennai 600 005, India, [b]Department of Organic Chemistry, School of Chemistry, Madurai Kamaraj University, Madurai 625 021, India, and [c]Centre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India
Correspondence e-mail: mnpsy2004@yahoo.com

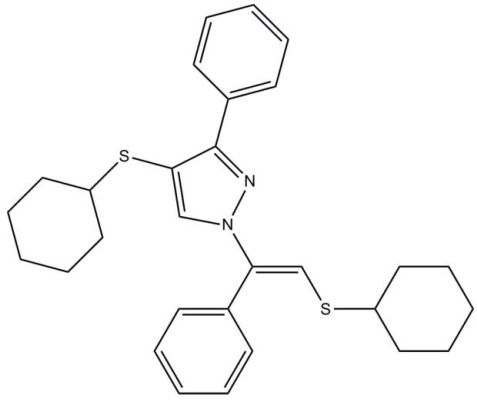
Received 4 September 2008; accepted 13 October 2008

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.051; $wR$ factor = 0.149; data-to-parameter ratio = 26.1.

In the title compound, $C_{29}H_{34}N_2S_2$, the pyrazole ring is planar and both cyclohexane rings adopt chair conformations. The dihedral angles between the pyrazole ring and the two benzene rings are 59.9 (2) and 19.8 (2)°. The conformation and packing of the molecules in the unit cell are stabilized by a weak intramolecular C—H···S and C—H···N interactions, in addition to van der Waals forces.

## Related literature

For pharmacological and medicinal properties of pyrazole derivatives, see: Baraldi *et al.* (1998); Bruno *et al.* (1990); Cottineau *et al.* (2002); Londershausen (1996); Chen & Li (1998); Mishra *et al.* (1998); Smith *et al.* (2001). For hybridization, see: Beddoes *et al.* (1986). For puckering and asymmetry analysis, see: Cremer & Pople (1975); Nardelli (1983). Manikannan (2008) describes other compounds formed along with the title compound in its synthesis.




## Experimental

### Crystal data

$C_{29}H_{34}N_2S_2$
$M_r$ = 474.70
Orthorhombic, $P2_12_12_1$
$a$ = 6.3859 (5) Å
$b$ = 19.1596 (17) Å
$c$ = 21.337 (2) Å
$V$ = 2610.7 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 293 (2) K
0.25 × 0.21 × 0.19 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min}$ = 0.936, $T_{max}$ = 0.965
19948 measured reflections
7787 independent reflections
4945 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.149
$S$ = 1.02
7787 reflections
298 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$
Absolute structure: Flack (1983), 3381 Friedel pairs
Flack parameter: −0.01 (8)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···N1 | 0.93 | 2.40 | 2.760 (4) | 103 |
| C27—H27···S2 | 0.93 | 2.80 | 3.450 (4) | 128 |
| C31—H31···N1 | 0.93 | 2.46 | 2.786 (4) | 101 |

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

PR thanks Dr Babu Varghese, SAIF, IIT Madras, Chennai, India, for his help in data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2138).

## References

Baraldi, P. G., Manfredini, S., Romagnoli, R., Stevanato, L., Zaid, A. N. & Manservigi, R. (1998). *Nucleosides Nucleotides*, **17**, 2165–2171.
Beddoes, R. L., Dalton, L., Joule, T. A., Mills, O. S., Street, J. D. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 787–797.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, O., Bondavalli, F., Ranise, A., Schenone, P., Losasso, C., Cilenti, L., Matera, C. & Marmo, E. (1990). *Farmaco*, **45**, 147–166.
Chen, H. S. & Li, Z. M. (1998). *Chem. J. Chin. Univ.* **19**, 572–576.
Cottineau, B., Toto, P., Marot, C., Pipaud, A. & Chenault, J. (2002). *Bioorg. Med. Chem.* **12**, 2105–2108.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Londershausen, M. (1996). *Pestic. Sci.* **48**, 269–274.
Manikannan, R. (2008). PhD thesis, Madurai Kamaraj University, Madurai, India.

Mishra, P. D., Wahidullah, S. & Kamat, S. Y. (1998). *Indian J. Chem. Sect. B*, **37**, 199.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Sheldrick, G. M. (2001). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, S. R., Denhardt, G. & Terminelli, C. (2001). *Eur. J. Pharmacol.* **432**, 107–119.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

# supplementary materials

*Acta Cryst.* (2008). E**64**, o2132-o2133 [ **doi:10.1107/S1600536808033126** ]

# 4-(Cyclohexylsulfanyl)-1-[(*E*)-2-(cyclohexylsulfanyl)-1-phenylethenyl]-3-phenyl-1*H*-pyrazole

**P. Ramesh, A. Subbiahpandi, R. Manikannan, S. Muthusubramanian and M. N. Ponnuswamy**

## Comment

Pyrazole derivatives possess significant antiarrhythmic and sedative (Bruno *et al.*, 1990), hypoglycemic (Cottineau *et al.*, 2002), antiviral (Baraldi *et al.*, 1998), and pesticidal (Londershausen, 1996) properties. Some pyrazole derivatives were successfully tested for their antifungal (Chen & Li, 1998), antihistaminic (Mishra *et al.*, 1998) and anti-inflammatory (Smith *et al.*, 2001) properties.

An *ORTEP* plot of the molecule is shown in Fig. 1 and a packing plot in Fig. 2. The pyrazole ring adopts a planar conformation. The sum of the angles at N1 of the pyrazole ring (359.95°) is in accordance with $sp^2$ hybridization (Beddoes *et al.*, 1986). Both the cyclohexane rings in the molecule adopt chair conformations which can be seen from the puckering (Cremer & Pople, 1975) and the asymmetry parameters (Nardelli, 1983). The values for the ring C8-C13 are: $q_2$ = 0.010 (4) Å, $q_3$ = - 0.562 (4) Å, $\pi$ = 186 (22)°, $\Delta$s(C9) and $\Delta$s(C12) =0.5 (4)° and for ring C20-C25 are: $q_2$ = 0.014 (4) Å, $q_3$ = - 0.572 (4) Å, $\pi$ = 132 (14)°, $\Delta$s(C22) and $\Delta$s(C25) = 0.3 (3)°.

The best least-squares planes calculated for the two cyclohexane rings (atoms C8, C9, C11 & C12 lie in the plane and C10 & C13 deviate for one of the rings; atoms C21, C22, C24 & C25 lie in the plane and C20 & C23 deviate for the other ring ) are twisted from the pyrazole ring by 50.06 (17)° and 69.71 (15), respectively. The crystal packing is augmented by weak intramolecular C—H···N and C—H···S interactions in addition to van der Waals forces.

## Experimental

A mixture of 2-(cyclohexylsulfanyl)-1-phenyl-1-ethanone N-[(E)-2- (cyclohexylsulfanyl)-1-phenylethylidene]hydrazone (0.003 mole) and 3 ml of dimethyl formamide were kept in an ice bath at 273 K and phosphorus oxychloride (0.024 mole) was added dropwise for 5 to 10 minutes. The reaction mixture was then kept in a microwave oven at 600 W for 30–60 sec. The process of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was poured into crushed ice and extracted with dichloromethane. The organic layer was dried with anhydrous sodium sulfate. The different compounds present in the mixture were separated by column chromatography using petroleum ether and ethyl acetate as the eluent (99:1 v/v, $R_f$ index of the title compound: 0.8336). The isolated title compound was recrystallized from dichloromethane to obtain 4-(cyclohexylsulfanyl) -1-[(*E*)-2-(cyclohexylsulfanyl)-1-phenylethenyl]-3 -phenyl-1*H* -pyrazole (title compound) and 4-(cyclohexyl sulfanyl)-1-[*Z*-2 -(cyclohexylsulfanyl)-1-phenyl-1-ethenyl]-3-phenyl-1*H*-pyrazole in 38% and 60% yield. The compounds identified through column are characterized by NMR studies (Manikannan, 2008).

## Refinement

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C) for all H atoms.

**Figures**

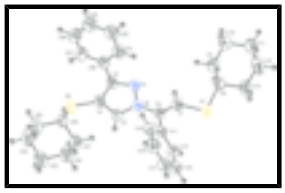


Fig. 1. Perspective view of the molecule with thermal ellipsoids drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radius.

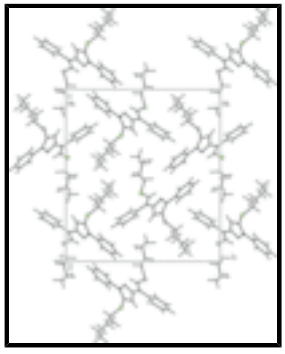


Fig. 2. The packing of the molecules in the unit cell viewed down the *a* axis.

**4-(Cyclohexylsulfanyl)-1-[(*E*)-2-(cyclohexylsulfanyl)-1-phenylethenyl]-\ 3-phenyl-1*H*-pyrazole**

*Crystal data*

| | |
|---|---|
| $C_{29}H_{34}N_2S_2$ | $F_{000}$ = 1016 |
| $M_r$ = 474.70 | $D_x$ = 1.208 Mg m$^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2ac 2ab | Cell parameters from 2320 reflections |
| $a$ = 6.3859 (5) Å | θ = 1.4–30.3º |
| $b$ = 19.1596 (17) Å | μ = 0.22 mm$^{-1}$ |
| $c$ = 21.337 (2) Å | $T$ = 293 (2) K |
| $V$ = 2610.7 (4) Å$^3$ | Block, colorless |
| $Z$ = 4 | 0.25 × 0.21 × 0.19 mm |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 7787 independent reflections |
| Radiation source: fine-focus sealed tube | 4945 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.031 |
| $T$ = 293(2) K | $\theta_{max}$ = 30.3º |
| ω and φ scans | $\theta_{min}$ = 1.4º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2001) | $h$ = −9→8 |
| $T_{min}$ = 0.936, $T_{max}$ = 0.965 | $k$ = −27→27 |
| 19948 measured reflections | $l$ = −30→21 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.051$ | $w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.1893P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.149$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.02$ | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 7787 reflections | $\Delta\rho_{min} = -0.17$ e Å$^{-3}$ |
| 298 parameters | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 3381 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack parameter: −0.01 (8) |

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 1.40978 (12) | 0.48628 (4) | 0.39564 (3) | 0.0608 (2) |
| S2 | 0.40247 (11) | 0.64092 (3) | 0.23133 (3) | 0.05671 (18) |
| N1 | 0.8399 (3) | 0.61798 (10) | 0.36277 (10) | 0.0481 (5) |
| N2 | 0.8853 (3) | 0.56795 (10) | 0.32015 (9) | 0.0467 (4) |
| C3 | 0.7544 (4) | 0.57046 (13) | 0.27104 (12) | 0.0490 (5) |
| H3 | 0.7592 | 0.5416 | 0.2360 | 0.059* |
| C4 | 0.6130 (4) | 0.62265 (12) | 0.28137 (11) | 0.0480 (5) |
| C5 | 0.6735 (4) | 0.65155 (12) | 0.33978 (11) | 0.0466 (5) |
| C6 | 1.0582 (4) | 0.52201 (12) | 0.33064 (11) | 0.0450 (5) |
| C7 | 1.1971 (4) | 0.53826 (14) | 0.37473 (12) | 0.0532 (6) |
| H7 | 1.1795 | 0.5803 | 0.3959 | 0.064* |
| C8 | 1.4367 (4) | 0.51105 (14) | 0.47716 (13) | 0.0548 (6) |
| H8 | 1.4470 | 0.5620 | 0.4798 | 0.066* |
| C9 | 1.2542 (5) | 0.4872 (2) | 0.51640 (15) | 0.0790 (10) |
| H9A | 1.2377 | 0.4372 | 0.5117 | 0.095* |
| H9B | 1.1275 | 0.5093 | 0.5011 | 0.095* |
| C10 | 1.2808 (6) | 0.5042 (3) | 0.58490 (18) | 0.1023 (14) |

| | | | | |
|---|---|---|---|---|
| H10A | 1.1638 | 0.4854 | 0.6085 | 0.123* |
| H10B | 1.2823 | 0.5544 | 0.5906 | 0.123* |
| C11 | 1.4836 (5) | 0.4733 (2) | 0.60921 (17) | 0.0874 (10) |
| H11A | 1.5042 | 0.4877 | 0.6524 | 0.105* |
| H11B | 1.4741 | 0.4228 | 0.6084 | 0.105* |
| C12 | 1.6674 (5) | 0.49605 (19) | 0.57090 (18) | 0.0821 (10) |
| H12A | 1.7925 | 0.4729 | 0.5862 | 0.098* |
| H12B | 1.6870 | 0.5460 | 0.5758 | 0.098* |
| C13 | 1.6387 (4) | 0.47926 (18) | 0.50208 (15) | 0.0691 (8) |
| H13A | 1.6350 | 0.4290 | 0.4965 | 0.083* |
| H13B | 1.7566 | 0.4974 | 0.4785 | 0.083* |
| C14 | 1.0638 (4) | 0.45828 (13) | 0.29141 (11) | 0.0477 (5) |
| C15 | 1.2456 (4) | 0.44122 (15) | 0.25948 (13) | 0.0584 (7) |
| H15 | 1.3617 | 0.4705 | 0.2614 | 0.070* |
| C16 | 1.2534 (5) | 0.38010 (18) | 0.22451 (14) | 0.0707 (8) |
| H16 | 1.3757 | 0.3683 | 0.2033 | 0.085* |
| C17 | 1.0840 (6) | 0.33739 (16) | 0.22097 (15) | 0.0765 (9) |
| H17 | 1.0904 | 0.2965 | 0.1975 | 0.092* |
| C18 | 0.9053 (6) | 0.35457 (15) | 0.25173 (15) | 0.0728 (8) |
| H18 | 0.7890 | 0.3255 | 0.2488 | 0.087* |
| C19 | 0.8942 (5) | 0.41443 (14) | 0.28721 (13) | 0.0589 (6) |
| H19 | 0.7713 | 0.4252 | 0.3085 | 0.071* |
| C20 | 0.5010 (4) | 0.71168 (13) | 0.18284 (13) | 0.0502 (6) |
| H20 | 0.5267 | 0.7524 | 0.2096 | 0.060* |
| C21 | 0.6984 (5) | 0.69469 (18) | 0.14829 (16) | 0.0715 (9) |
| H21A | 0.6779 | 0.6526 | 0.1238 | 0.086* |
| H21B | 0.8100 | 0.6860 | 0.1781 | 0.086* |
| C22 | 0.7614 (5) | 0.7546 (2) | 0.10494 (18) | 0.0823 (10) |
| H22A | 0.7965 | 0.7953 | 0.1299 | 0.099* |
| H22B | 0.8850 | 0.7413 | 0.0814 | 0.099* |
| C23 | 0.5874 (6) | 0.7732 (2) | 0.05995 (16) | 0.0823 (9) |
| H23A | 0.6285 | 0.8133 | 0.0351 | 0.099* |
| H23B | 0.5630 | 0.7344 | 0.0316 | 0.099* |
| C24 | 0.3890 (5) | 0.78953 (16) | 0.09496 (17) | 0.0755 (9) |
| H24A | 0.2776 | 0.7990 | 0.0653 | 0.091* |
| H24B | 0.4097 | 0.8310 | 0.1202 | 0.091* |
| C25 | 0.3260 (4) | 0.72955 (15) | 0.13670 (15) | 0.0613 (7) |
| H25A | 0.2947 | 0.6890 | 0.1112 | 0.074* |
| H25B | 0.2004 | 0.7419 | 0.1597 | 0.074* |
| C26 | 0.5914 (4) | 0.71108 (11) | 0.37568 (11) | 0.0478 (5) |
| C27 | 0.3886 (5) | 0.73724 (14) | 0.36781 (15) | 0.0626 (7) |
| H27 | 0.2979 | 0.7165 | 0.3392 | 0.075* |
| C28 | 0.3239 (5) | 0.79383 (15) | 0.40263 (17) | 0.0736 (9) |
| H28 | 0.1905 | 0.8119 | 0.3961 | 0.088* |
| C29 | 0.4492 (6) | 0.82374 (16) | 0.44595 (16) | 0.0739 (9) |
| H29 | 0.4020 | 0.8617 | 0.4692 | 0.089* |
| C30 | 0.6466 (6) | 0.79767 (16) | 0.45541 (16) | 0.0755 (9) |
| H30 | 0.7335 | 0.8175 | 0.4855 | 0.091* |
| C31 | 0.7160 (5) | 0.74201 (14) | 0.42013 (14) | 0.0625 (7) |

| | | | | |
|---|---|---|---|---|
| H31 | 0.8506 | 0.7250 | 0.4266 | 0.075* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0537 (3) | 0.0746 (4) | 0.0542 (4) | 0.0223 (3) | −0.0076 (3) | −0.0031 (3) |
| S2 | 0.0476 (3) | 0.0660 (4) | 0.0565 (4) | −0.0031 (3) | −0.0134 (3) | 0.0156 (3) |
| N1 | 0.0514 (11) | 0.0490 (10) | 0.0439 (11) | 0.0077 (8) | −0.0057 (9) | 0.0028 (9) |
| N2 | 0.0481 (11) | 0.0523 (10) | 0.0396 (10) | 0.0112 (9) | −0.0039 (9) | 0.0015 (8) |
| C3 | 0.0512 (13) | 0.0583 (13) | 0.0375 (12) | 0.0060 (11) | −0.0024 (11) | 0.0043 (11) |
| C4 | 0.0468 (12) | 0.0525 (12) | 0.0446 (13) | 0.0036 (10) | −0.0073 (11) | 0.0103 (10) |
| C5 | 0.0483 (12) | 0.0474 (12) | 0.0440 (13) | 0.0046 (10) | −0.0028 (10) | 0.0095 (10) |
| C6 | 0.0436 (12) | 0.0500 (12) | 0.0413 (12) | 0.0083 (10) | 0.0026 (10) | 0.0077 (10) |
| C7 | 0.0522 (14) | 0.0550 (13) | 0.0524 (15) | 0.0113 (11) | −0.0070 (12) | 0.0019 (12) |
| C8 | 0.0525 (14) | 0.0580 (13) | 0.0539 (14) | 0.0098 (11) | −0.0140 (12) | −0.0015 (12) |
| C9 | 0.0445 (14) | 0.131 (3) | 0.0620 (19) | 0.0160 (17) | −0.0067 (13) | −0.0031 (19) |
| C10 | 0.076 (2) | 0.172 (4) | 0.059 (2) | 0.041 (3) | −0.0054 (18) | 0.001 (2) |
| C11 | 0.073 (2) | 0.124 (3) | 0.065 (2) | 0.014 (2) | −0.0145 (18) | 0.017 (2) |
| C12 | 0.0695 (19) | 0.091 (2) | 0.085 (2) | −0.0139 (17) | −0.0374 (18) | 0.0172 (19) |
| C13 | 0.0418 (15) | 0.091 (2) | 0.075 (2) | −0.0011 (13) | −0.0132 (13) | 0.0102 (17) |
| C14 | 0.0514 (13) | 0.0534 (12) | 0.0383 (12) | 0.0087 (11) | 0.0007 (10) | 0.0052 (10) |
| C15 | 0.0529 (14) | 0.0738 (17) | 0.0485 (15) | 0.0122 (12) | 0.0076 (12) | 0.0020 (13) |
| C16 | 0.0710 (18) | 0.089 (2) | 0.0520 (17) | 0.0305 (17) | 0.0058 (15) | −0.0081 (15) |
| C17 | 0.095 (2) | 0.0715 (18) | 0.0629 (19) | 0.0181 (19) | −0.008 (2) | −0.0199 (15) |
| C18 | 0.078 (2) | 0.0649 (17) | 0.076 (2) | −0.0043 (16) | −0.0062 (19) | −0.0089 (15) |
| C19 | 0.0571 (14) | 0.0597 (14) | 0.0598 (16) | 0.0037 (13) | 0.0053 (14) | −0.0028 (12) |
| C20 | 0.0465 (12) | 0.0551 (13) | 0.0489 (14) | 0.0034 (10) | −0.0100 (11) | 0.0050 (11) |
| C21 | 0.0566 (16) | 0.094 (2) | 0.0641 (19) | 0.0183 (15) | 0.0000 (15) | 0.0214 (17) |
| C22 | 0.0532 (16) | 0.115 (3) | 0.079 (2) | 0.0054 (17) | 0.0047 (16) | 0.034 (2) |
| C23 | 0.082 (2) | 0.101 (2) | 0.0641 (19) | 0.003 (2) | −0.0034 (19) | 0.0330 (17) |
| C24 | 0.0654 (18) | 0.0767 (19) | 0.084 (2) | 0.0102 (16) | −0.0185 (18) | 0.0206 (16) |
| C25 | 0.0490 (14) | 0.0675 (16) | 0.0673 (18) | 0.0021 (12) | −0.0159 (13) | 0.0154 (14) |
| C26 | 0.0527 (13) | 0.0439 (11) | 0.0469 (13) | 0.0055 (11) | 0.0008 (11) | 0.0104 (10) |
| C27 | 0.0570 (15) | 0.0603 (15) | 0.0705 (18) | 0.0097 (13) | −0.0064 (15) | 0.0051 (13) |
| C28 | 0.075 (2) | 0.0659 (17) | 0.080 (2) | 0.0235 (15) | 0.0031 (18) | 0.0023 (16) |
| C29 | 0.095 (2) | 0.0579 (16) | 0.069 (2) | 0.0210 (16) | 0.0033 (19) | −0.0031 (15) |
| C30 | 0.094 (2) | 0.0654 (18) | 0.067 (2) | 0.0087 (16) | −0.0096 (17) | −0.0061 (15) |
| C31 | 0.0674 (18) | 0.0601 (16) | 0.0600 (17) | 0.0098 (14) | −0.0075 (14) | −0.0014 (13) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C7 | 1.742 (2) | C16—C17 | 1.359 (5) |
| S1—C8 | 1.811 (3) | C16—H16 | 0.9300 |
| S2—C4 | 1.752 (2) | C17—C18 | 1.357 (5) |
| S2—C20 | 1.818 (3) | C17—H17 | 0.9300 |
| N1—C5 | 1.335 (3) | C18—C19 | 1.376 (4) |
| N1—N2 | 1.353 (3) | C18—H18 | 0.9300 |
| N2—C3 | 1.341 (3) | C19—H19 | 0.9300 |
| N2—C6 | 1.429 (3) | C20—C21 | 1.496 (4) |

| | | | |
|---|---|---|---|
| C3—C4 | 1.365 (3) | C20—C25 | 1.528 (3) |
| C3—H3 | 0.9300 | C20—H20 | 0.9800 |
| C4—C5 | 1.417 (3) | C21—C22 | 1.529 (4) |
| C5—C26 | 1.471 (3) | C21—H21A | 0.9700 |
| C6—C7 | 1.330 (3) | C21—H21B | 0.9700 |
| C6—C14 | 1.481 (3) | C22—C23 | 1.511 (4) |
| C7—H7 | 0.9300 | C22—H22A | 0.9700 |
| C8—C9 | 1.506 (4) | C22—H22B | 0.9700 |
| C8—C13 | 1.522 (4) | C23—C24 | 1.504 (5) |
| C8—H8 | 0.9800 | C23—H23A | 0.9700 |
| C9—C10 | 1.507 (5) | C23—H23B | 0.9700 |
| C9—H9A | 0.9700 | C24—C25 | 1.508 (4) |
| C9—H9B | 0.9700 | C24—H24A | 0.9700 |
| C10—C11 | 1.515 (5) | C24—H24B | 0.9700 |
| C10—H10A | 0.9700 | C25—H25A | 0.9700 |
| C10—H10B | 0.9700 | C25—H25B | 0.9700 |
| C11—C12 | 1.495 (5) | C26—C31 | 1.373 (4) |
| C11—H11A | 0.9700 | C26—C27 | 1.399 (4) |
| C11—H11B | 0.9700 | C27—C28 | 1.378 (4) |
| C12—C13 | 1.514 (5) | C27—H27 | 0.9300 |
| C12—H12A | 0.9700 | C28—C29 | 1.350 (5) |
| C12—H12B | 0.9700 | C28—H28 | 0.9300 |
| C13—H13A | 0.9700 | C29—C30 | 1.371 (5) |
| C13—H13B | 0.9700 | C29—H29 | 0.9300 |
| C14—C19 | 1.374 (4) | C30—C31 | 1.379 (4) |
| C14—C15 | 1.385 (3) | C30—H30 | 0.9300 |
| C15—C16 | 1.390 (4) | C31—H31 | 0.9300 |
| C15—H15 | 0.9300 | | |
| C7—S1—C8 | 99.80 (12) | C15—C16—H16 | 119.7 |
| C4—S2—C20 | 103.31 (11) | C18—C17—C16 | 119.8 (3) |
| C5—N1—N2 | 105.4 (2) | C18—C17—H17 | 120.1 |
| C3—N2—N1 | 111.50 (19) | C16—C17—H17 | 120.1 |
| C3—N2—C6 | 128.7 (2) | C17—C18—C19 | 120.8 (3) |
| N1—N2—C6 | 119.76 (19) | C17—C18—H18 | 119.6 |
| N2—C3—C4 | 108.2 (2) | C19—C18—H18 | 119.6 |
| N2—C3—H3 | 125.9 | C14—C19—C18 | 120.3 (3) |
| C4—C3—H3 | 125.9 | C14—C19—H19 | 119.8 |
| C3—C4—C5 | 104.3 (2) | C18—C19—H19 | 119.8 |
| C3—C4—S2 | 123.7 (2) | C21—C20—C25 | 110.3 (2) |
| C5—C4—S2 | 131.83 (19) | C21—C20—S2 | 114.20 (19) |
| N1—C5—C4 | 110.6 (2) | C25—C20—S2 | 106.29 (18) |
| N1—C5—C26 | 117.8 (2) | C21—C20—H20 | 108.6 |
| C4—C5—C26 | 131.6 (2) | C25—C20—H20 | 108.6 |
| C7—C6—N2 | 118.8 (2) | S2—C20—H20 | 108.6 |
| C7—C6—C14 | 125.2 (2) | C20—C21—C22 | 110.9 (3) |
| N2—C6—C14 | 116.0 (2) | C20—C21—H21A | 109.5 |
| C6—C7—S1 | 124.6 (2) | C22—C21—H21A | 109.5 |
| C6—C7—H7 | 117.7 | C20—C21—H21B | 109.5 |
| S1—C7—H7 | 117.7 | C22—C21—H21B | 109.5 |

| | | | |
|---|---|---|---|
| C9—C8—C13 | 109.9 (2) | H21A—C21—H21B | 108.0 |
| C9—C8—S1 | 112.4 (2) | C23—C22—C21 | 111.6 (3) |
| C13—C8—S1 | 108.1 (2) | C23—C22—H22A | 109.3 |
| C9—C8—H8 | 108.8 | C21—C22—H22A | 109.3 |
| C13—C8—H8 | 108.8 | C23—C22—H22B | 109.3 |
| S1—C8—H8 | 108.8 | C21—C22—H22B | 109.3 |
| C8—C9—C10 | 112.8 (3) | H22A—C22—H22B | 108.0 |
| C8—C9—H9A | 109.0 | C24—C23—C22 | 110.7 (3) |
| C10—C9—H9A | 109.0 | C24—C23—H23A | 109.5 |
| C8—C9—H9B | 109.0 | C22—C23—H23A | 109.5 |
| C10—C9—H9B | 109.0 | C24—C23—H23B | 109.5 |
| H9A—C9—H9B | 107.8 | C22—C23—H23B | 109.5 |
| C9—C10—C11 | 110.1 (3) | H23A—C23—H23B | 108.1 |
| C9—C10—H10A | 109.6 | C23—C24—C25 | 111.0 (3) |
| C11—C10—H10A | 109.6 | C23—C24—H24A | 109.4 |
| C9—C10—H10B | 109.6 | C25—C24—H24A | 109.4 |
| C11—C10—H10B | 109.6 | C23—C24—H24B | 109.4 |
| H10A—C10—H10B | 108.1 | C25—C24—H24B | 109.4 |
| C12—C11—C10 | 111.7 (3) | H24A—C24—H24B | 108.0 |
| C12—C11—H11A | 109.3 | C24—C25—C20 | 110.9 (2) |
| C10—C11—H11A | 109.3 | C24—C25—H25A | 109.5 |
| C12—C11—H11B | 109.3 | C20—C25—H25A | 109.5 |
| C10—C11—H11B | 109.3 | C24—C25—H25B | 109.5 |
| H11A—C11—H11B | 107.9 | C20—C25—H25B | 109.5 |
| C11—C12—C13 | 111.9 (3) | H25A—C25—H25B | 108.1 |
| C11—C12—H12A | 109.2 | C31—C26—C27 | 117.7 (2) |
| C13—C12—H12A | 109.2 | C31—C26—C5 | 119.2 (2) |
| C11—C12—H12B | 109.2 | C27—C26—C5 | 123.1 (2) |
| C13—C12—H12B | 109.2 | C28—C27—C26 | 119.7 (3) |
| H12A—C12—H12B | 107.9 | C28—C27—H27 | 120.2 |
| C12—C13—C8 | 110.9 (3) | C26—C27—H27 | 120.2 |
| C12—C13—H13A | 109.5 | C29—C28—C27 | 121.7 (3) |
| C8—C13—H13A | 109.5 | C29—C28—H28 | 119.1 |
| C12—C13—H13B | 109.5 | C27—C28—H28 | 119.1 |
| C8—C13—H13B | 109.5 | C28—C29—C30 | 119.4 (3) |
| H13A—C13—H13B | 108.1 | C28—C29—H29 | 120.3 |
| C19—C14—C15 | 119.0 (2) | C30—C29—H29 | 120.3 |
| C19—C14—C6 | 121.5 (2) | C29—C30—C31 | 119.8 (3) |
| C15—C14—C6 | 119.5 (2) | C29—C30—H30 | 120.1 |
| C14—C15—C16 | 119.5 (3) | C31—C30—H30 | 120.1 |
| C14—C15—H15 | 120.2 | C26—C31—C30 | 121.7 (3) |
| C16—C15—H15 | 120.2 | C26—C31—H31 | 119.2 |
| C17—C16—C15 | 120.6 (3) | C30—C31—H31 | 119.2 |
| C17—C16—H16 | 119.7 | | |
| C5—N1—N2—C3 | 1.2 (3) | C7—C6—C14—C15 | −51.7 (4) |
| C5—N1—N2—C6 | 179.9 (2) | N2—C6—C14—C15 | 129.7 (2) |
| N1—N2—C3—C4 | −1.5 (3) | C19—C14—C15—C16 | −0.6 (4) |
| C6—N2—C3—C4 | 179.9 (2) | C6—C14—C15—C16 | 177.7 (2) |
| N2—C3—C4—C5 | 1.1 (3) | C14—C15—C16—C17 | 0.6 (4) |

| | | | |
|---|---|---|---|
| N2—C3—C4—S2 | −175.90 (18) | C15—C16—C17—C18 | 0.1 (5) |
| C20—S2—C4—C3 | −96.9 (2) | C16—C17—C18—C19 | −0.8 (5) |
| C20—S2—C4—C5 | 86.9 (3) | C15—C14—C19—C18 | −0.1 (4) |
| N2—N1—C5—C4 | −0.4 (3) | C6—C14—C19—C18 | −178.3 (3) |
| N2—N1—C5—C26 | −178.1 (2) | C17—C18—C19—C14 | 0.8 (5) |
| C3—C4—C5—N1 | −0.5 (3) | C4—S2—C20—C21 | 57.0 (2) |
| S2—C4—C5—N1 | 176.23 (19) | C4—S2—C20—C25 | 178.85 (19) |
| C3—C4—C5—C26 | 176.9 (3) | C25—C20—C21—C22 | 56.0 (4) |
| S2—C4—C5—C26 | −6.4 (4) | S2—C20—C21—C22 | 175.7 (2) |
| C3—N2—C6—C7 | 164.4 (2) | C20—C21—C22—C23 | −55.8 (4) |
| N1—N2—C6—C7 | −14.1 (3) | C21—C22—C23—C24 | 55.3 (4) |
| C3—N2—C6—C14 | −16.9 (3) | C22—C23—C24—C25 | −56.3 (4) |
| N1—N2—C6—C14 | 164.5 (2) | C23—C24—C25—C20 | 57.3 (4) |
| N2—C6—C7—S1 | 177.14 (18) | C21—C20—C25—C24 | −57.3 (3) |
| C14—C6—C7—S1 | −1.4 (4) | S2—C20—C25—C24 | 178.4 (2) |
| C8—S1—C7—C6 | −150.7 (2) | N1—C5—C26—C31 | 18.0 (3) |
| C7—S1—C8—C9 | 67.3 (2) | C4—C5—C26—C31 | −159.2 (3) |
| C7—S1—C8—C13 | −171.3 (2) | N1—C5—C26—C27 | −160.5 (2) |
| C13—C8—C9—C10 | 56.4 (4) | C4—C5—C26—C27 | 22.3 (4) |
| S1—C8—C9—C10 | 176.9 (3) | C31—C26—C27—C28 | 2.3 (4) |
| C8—C9—C10—C11 | −55.9 (5) | C5—C26—C27—C28 | −179.1 (3) |
| C9—C10—C11—C12 | 54.5 (5) | C26—C27—C28—C29 | −2.1 (5) |
| C10—C11—C12—C13 | −55.3 (4) | C27—C28—C29—C30 | 0.5 (5) |
| C11—C12—C13—C8 | 55.5 (4) | C28—C29—C30—C31 | 0.9 (5) |
| C9—C8—C13—C12 | −55.1 (3) | C27—C26—C31—C30 | −1.0 (4) |
| S1—C8—C13—C12 | −178.1 (2) | C5—C26—C31—C30 | −179.6 (3) |
| C7—C6—C14—C19 | 126.5 (3) | C29—C30—C31—C26 | −0.6 (5) |
| N2—C6—C14—C19 | −52.1 (3) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···N1 | 0.93 | 2.40 | 2.760 (4) | 103 |
| C27—H27···S2 | 0.93 | 2.80 | 3.450 (4) | 128 |
| C31—H31···N1 | 0.93 | 2.46 | 2.786 (4) | 101 |

**Fig. 1**

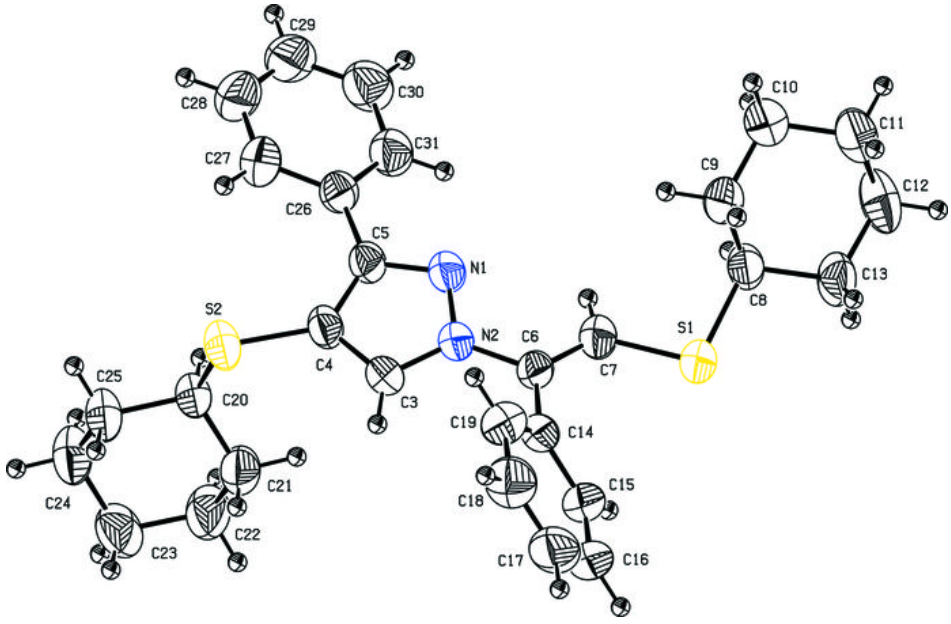

C29
C28
C30
C27
C31
C26
C5
N1
S2
C4
N2
C6
C7
S1
C10
C11
C9
C12
C8
C13
C25
C20
C3
C19
C14
C24
C21
C18
C15
C22
C23
C17
C16

**Fig. 2**