Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{4-[(5-methylthien-2-yl)methyleneamino]phenyl} sulfide

Yun-Fa Zheng,* Liang-Gui Wang and Chun-Niu Zhang

Department of Chemistry, Lishui University, Lishui 323000, People's Republic of China
Correspondence e-mail: xraylab@hotmail.com

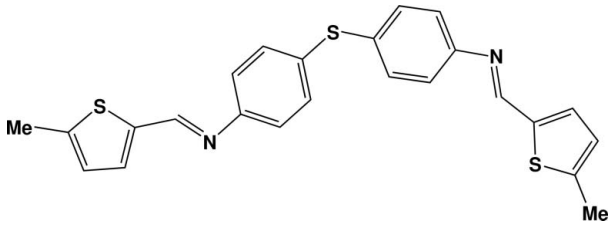
Received 19 June 2007; accepted 19 June 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.065; wR factor = 0.134; data-to-parameter ratio = 16.2.

In the title compound, $C_{24}H_{20}N_2S_3$, all geometric parameters are in the usual ranges. The two benzene rings are almost perpendicular [85.80 (11)°]. The dihedral angle between the benzene and thiophene rings are 45.16 (15) and 37.43 (14)° in the two halves. The C=N double bonds are coplanar with the thiophene rings.

Related literature

For related literature, see: Corey *et al.* (1980); Luly *et al.* (1987); Vicini *et al.* (2003).



Experimental

Crystal data

$C_{24}H_{20}N_2S_3$
M_r = 432.60
Triclinic, $P\overline{1}$
a = 5.315 (3) Å
b = 14.275 (4) Å
c = 15.106 (3) Å
α = 72.828 (3)°
β = 88.344 (4)°
γ = 83.314 (4)°
V = 1087.6 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 291 (2) K
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.901, T_{max} = 0.926
11209 measured reflections
4283 independent reflections
3075 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.134
S = 1.06
4283 reflections
264 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Natural Science Foundation of Zhejiang Province (No. M203052) and the Research Foundation of Lishui University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2400).

References

Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.22), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Corey, E. J., Clark, D. A., Goto, G., Marfat, A., Mioskowski, C., Samuelsson, B. & Hammarström, S. (1980). *J. Am. Chem. Soc.* **102**, 1436–1439.
Luly, J. R., Yi, N., Soderquist, J., Stein, H., Cohen, J., Perun, T. J. & Plattner, J. J. (1987). *J. Med. Chem.* **30**, 1609–1616.
Vicini, P., Geronikaki, A., Incerti, M., Busonera, B., Poni, G., Cabras, C. A. & Colla, P. L. (2003). *Bioorg. Med. Chem.* **11**, 4785–4789.

 doi:10.1107/S1600536807029984

supplementary materials

Acta Cryst. (2007). E**63**, o3307 [**doi:10.1107/S1600536807029984**]

Bis{4-[(5-methylthien-2-yl)methyleneamino]phenyl} sulfide

Y.-F. Zheng, L.-G. Wang and C.-N. Zhang

Comment

Organosulfides are important intermediates in the field of medicinal chemistry and organic synthesis (Luly *et al.*, 1987; Corey *et al.*, 1980). Thiazole Schiff bases are also important lead compounds active against emergent and re-emergent human and cattle infectious diseases, such as AIDS, hepatitis B, hepatitis C, tuberculosis and bovine viral diarrhea, (Vicini *et al.*, 2003). Recently, we have synthesized the title ligand, a new schiff base containing thiophen component.

All bond lengths and angles have normal values (Fig. 1), the bond length of C–N are 1.267 (4)Å and 1.271 (4) Å, respectively, blong to typical double bonds. The dihedral angle between the two phenyl rings is 85.80 (11)°.

Experimental

Under nitrogen, a mixture of 4,4'-thiodianiline (1.08 g, 5 mmol) and 5-methylthiophene-2-carbaldehyde (1.26 g, 10 mmol) in anhydrous ethanol (20 ml) was refluxed for 6 h, yielding a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. After being dried a yellow solid was obtained in yield 85% (1.84 g). Yellow single crystals suitable for X-ray analysis were grown by slow evaporation of anhydrous ethanol at room temperature.

Refinement

H atoms bonded to C atoms were positioned geometrically and refined using a riding model (including free rotation about the methyl C—C bond), with C—H = 0.93–0.96 Å and with U_{iso}(H) = 1.2(1.5 for methyl groups) times U_{eq}(C).

Figures

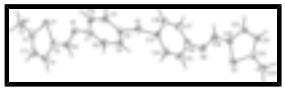


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

Bis{4-[(5-methylthien-2-yl)methyleneamino]phenyl} sulfide

Crystal data

$C_{24}H_{20}N_2S_3$	$Z = 2$
$M_r = 432.60$	$F_{000} = 452$
Triclinic, $P\overline{1}$	$D_x = 1.321$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.315$ (3) Å	Cell parameters from 2294 reflections
$b = 14.275$ (4) Å	$\theta = 2.3$–25.5º

c = 15.106 (3) Å	μ = 0.35 mm^{-1}
α = 72.828 (3)º	T = 291 (2) K
β = 88.344 (4)º	Block, yellow
γ = 83.314 (4)º	0.30 × 0.24 × 0.22 mm
V = 1087.6 (11) Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	4283 independent reflections
Radiation source: sealed tube	3075 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.053
T = 291(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −6→6
T_{min} = 0.901, T_{max} = 0.926	k = −17→17
11209 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.065	H-atom parameters constrained
$wR(F^2)$ = 0.134	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.55P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
4283 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
264 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)

3.9093 (0.0067) x − 7.3689 (0.0222) y + 2.0066 (0.0224) z = 2.2004 (0.0243)

* 0.0017 (0.0017) S1 * −0.0037 (0.0022) C2 * 0.0043 (0.0027) C3 * −0.0027 (0.0026) C4 * 0.0003 (0.0022) C5

Rms deviation of fitted atoms = 0.0029

3.1052 (0.0064) x + 0.2728 (0.0209) y + 11.7286 (0.0163) z = 12.8848 (0.0204)

Angle to previous plane (with approximate e.s.d.) = 45.16 (0.15)

* 0.0011 (0.0024) C7 * 0.0019 (0.0025) C8 * −0.0005 (0.0025) C9 * −0.0039 (0.0024) C10 * 0.0070 (0.0026) C11 * −0.0056 (0.0027) C12

Rms deviation of fitted atoms = 0.0041

3.5500 (0.0058) *x* + 8.3600 (0.0174) *y* − 5.2434 (0.0231) *z* = 7.6913 (0.0305)

Angle to previous plane (with approximate e.s.d.) = 85.80 (0.11)

* −0.0008 (0.0023) C13 * 0.0034 (0.0025) C14 * −0.0053 (0.0026) C15 * 0.0046 (0.0025) C16 * −0.0021 (0.0026) C17 * 0.0001 (0.0026) C18

Rms deviation of fitted atoms = 0.0033

3.7905 (0.0067) *x* + 11.0723 (0.0165) *y* + 4.4432 (0.0214) *z* = 17.2182 (0.0267)

Angle to previous plane (with approximate e.s.d.) = 37.43 (0.14)

* 0.0009 (0.0016) S3 * −0.0045 (0.0021) C20 * 0.0067 (0.0025) C21 * −0.0059 (0.0025) C22 * 0.0027 (0.0021) C23

Rms deviation of fitted atoms = 0.0046

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.2614 (9)	0.0886 (3)	0.9139 (3)	0.0857 (13)
H1A	0.2240	0.0362	0.9674	0.129*
H1B	0.3734	0.0622	0.8739	0.129*
H1C	0.1071	0.1195	0.8811	0.129*
C2	0.3849 (7)	0.1630 (2)	0.9435 (3)	0.0637 (10)
C3	0.3779 (8)	0.1806 (3)	1.0259 (3)	0.0747 (11)
H3	0.2889	0.1460	1.0767	0.090*
C4	0.5197 (8)	0.2571 (3)	1.0272 (3)	0.0675 (10)
H4	0.5318	0.2786	1.0792	0.081*
C5	0.6357 (7)	0.2966 (2)	0.9476 (2)	0.0550 (8)
C6	0.7838 (7)	0.3786 (2)	0.9234 (2)	0.0578 (8)
H6	0.8086	0.4097	0.9681	0.069*
C7	1.0027 (6)	0.4972 (2)	0.8216 (2)	0.0483 (7)
C8	0.9070 (6)	0.5813 (2)	0.8451 (2)	0.0558 (8)
H8	0.7653	0.5797	0.8828	0.067*
C9	1.0204 (6)	0.6678 (2)	0.8129 (2)	0.0564 (8)
H9	0.9541	0.7236	0.8292	0.068*
C10	1.2311 (6)	0.6715 (2)	0.7567 (2)	0.0431 (7)
C11	1.3271 (7)	0.5885 (2)	0.7341 (2)	0.0562 (8)

H11	1.4709	0.5898	0.6974	0.067*
C12	1.2126 (7)	0.5029 (2)	0.7654 (3)	0.0630 (9)
H12	1.2786	0.4477	0.7480	0.076*
C13	1.1345 (6)	0.8760 (2)	0.6981 (2)	0.0467 (7)
C14	1.0991 (7)	0.9302 (2)	0.7597 (3)	0.0594 (9)
H14	1.2080	0.9166	0.8101	0.071*
C15	0.8991 (7)	1.0059 (2)	0.7467 (3)	0.0602 (9)
H15	0.8740	1.0418	0.7893	0.072*
C16	0.7382 (6)	1.0280 (2)	0.6711 (2)	0.0486 (7)
C17	0.7769 (7)	0.9728 (3)	0.6105 (3)	0.0618 (9)
H17	0.6691	0.9860	0.5598	0.074*
C18	0.9732 (7)	0.8979 (2)	0.6236 (2)	0.0610 (9)
H18	0.9969	0.8616	0.5814	0.073*
C19	0.4707 (7)	1.1612 (3)	0.5835 (3)	0.0599 (9)
H19	0.5624	1.1525	0.5326	0.072*
C20	0.2608 (6)	1.2376 (2)	0.5676 (2)	0.0513 (8)
C21	0.1764 (8)	1.3004 (3)	0.4856 (2)	0.0646 (10)
H21	0.2484	1.2998	0.4289	0.077*
C22	−0.0331 (8)	1.3671 (2)	0.4953 (2)	0.0645 (10)
H22	−0.1150	1.4141	0.4452	0.077*
C23	−0.1032 (6)	1.3566 (2)	0.5832 (2)	0.0492 (7)
C24	−0.3108 (7)	1.4138 (3)	0.6194 (2)	0.0575 (8)
H24A	−0.3612	1.4749	0.5728	0.086*
H24B	−0.2528	1.4272	0.6735	0.086*
H24C	−0.4527	1.3762	0.6352	0.086*
N1	0.8823 (6)	0.41080 (19)	0.84441 (19)	0.0553 (7)
N2	0.5373 (5)	1.10504 (19)	0.6635 (2)	0.0564 (7)
S1	0.5706 (2)	0.23984 (7)	0.86657 (6)	0.0630 (3)
S2	1.38975 (16)	0.77884 (6)	0.71406 (6)	0.0547 (2)
S3	0.08630 (18)	1.26159 (7)	0.65792 (6)	0.0591 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.095 (3)	0.063 (2)	0.098 (3)	−0.026 (2)	−0.024 (3)	−0.013 (2)
C2	0.065 (2)	0.0417 (17)	0.075 (3)	−0.0114 (16)	−0.0046 (18)	−0.0008 (16)
C3	0.083 (3)	0.069 (3)	0.068 (3)	−0.017 (2)	0.020 (2)	−0.013 (2)
C4	0.090 (3)	0.061 (2)	0.053 (2)	−0.017 (2)	0.0128 (19)	−0.0194 (17)
C5	0.066 (2)	0.0445 (17)	0.0536 (19)	−0.0078 (15)	0.0000 (16)	−0.0127 (14)
C6	0.063 (2)	0.0511 (19)	0.061 (2)	−0.0100 (16)	0.0017 (17)	−0.0184 (16)
C7	0.0500 (18)	0.0465 (17)	0.0467 (17)	−0.0004 (14)	−0.0016 (14)	−0.0130 (13)
C8	0.0474 (19)	0.0500 (18)	0.072 (2)	−0.0072 (15)	0.0172 (16)	−0.0221 (16)
C9	0.053 (2)	0.0464 (17)	0.075 (2)	−0.0053 (15)	0.0135 (17)	−0.0284 (16)
C10	0.0426 (16)	0.0391 (15)	0.0461 (16)	0.0022 (12)	0.0002 (12)	−0.0128 (12)
C11	0.052 (2)	0.0504 (18)	0.067 (2)	−0.0021 (15)	0.0178 (16)	−0.0223 (16)
C12	0.079 (3)	0.0405 (17)	0.071 (2)	0.0017 (16)	0.0194 (19)	−0.0241 (16)
C13	0.0441 (17)	0.0373 (15)	0.0579 (19)	−0.0071 (13)	0.0091 (14)	−0.0127 (13)
C14	0.062 (2)	0.0539 (19)	0.065 (2)	−0.0011 (16)	−0.0156 (17)	−0.0224 (16)

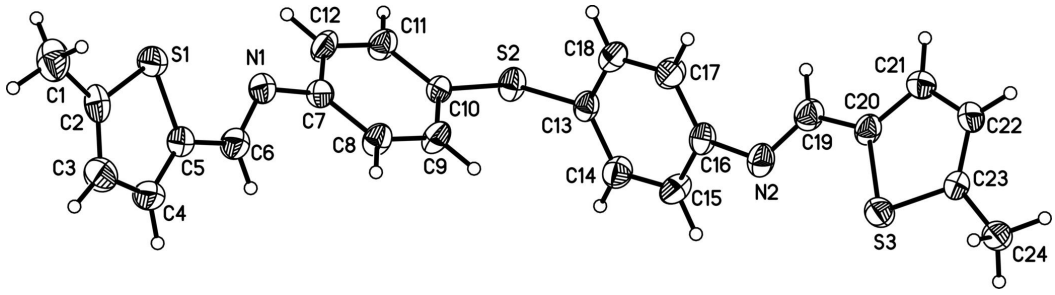
C15	0.064 (2)	0.057 (2)	0.069 (2)	−0.0027 (17)	−0.0020 (18)	−0.0341 (18)
C16	0.0406 (17)	0.0417 (16)	0.062 (2)	−0.0036 (13)	−0.0010 (14)	−0.0136 (14)
C17	0.067 (2)	0.057 (2)	0.064 (2)	0.0052 (17)	−0.0145 (17)	−0.0250 (17)
C18	0.075 (2)	0.0487 (18)	0.064 (2)	0.0009 (17)	−0.0018 (18)	−0.0263 (16)
C19	0.059 (2)	0.0530 (19)	0.067 (2)	−0.0016 (16)	0.0128 (17)	−0.0187 (17)
C20	0.0473 (18)	0.0432 (16)	0.062 (2)	0.0014 (14)	0.0112 (15)	−0.0161 (15)
C21	0.085 (3)	0.056 (2)	0.0471 (19)	0.0053 (18)	0.0109 (18)	−0.0122 (16)
C22	0.085 (3)	0.0481 (19)	0.0498 (19)	0.0138 (18)	0.0003 (18)	−0.0065 (15)
C23	0.0559 (19)	0.0396 (15)	0.0518 (18)	−0.0013 (14)	0.0014 (14)	−0.0147 (13)
C24	0.056 (2)	0.059 (2)	0.0521 (19)	0.0071 (16)	0.0073 (15)	−0.0140 (15)
N1	0.0662 (18)	0.0438 (14)	0.0542 (16)	−0.0097 (13)	0.0059 (13)	−0.0111 (12)
N2	0.0501 (16)	0.0455 (15)	0.0703 (19)	0.0012 (12)	0.0054 (14)	−0.0150 (14)
S1	0.0819 (7)	0.0545 (5)	0.0542 (5)	−0.0172 (4)	0.0015 (4)	−0.0150 (4)
S2	0.0445 (5)	0.0455 (4)	0.0723 (6)	−0.0040 (3)	0.0100 (4)	−0.0156 (4)
S3	0.0609 (5)	0.0588 (5)	0.0509 (5)	0.0044 (4)	0.0039 (4)	−0.0106 (4)

Geometric parameters (Å, °)

C1—C2	1.489 (5)	C13—C14	1.371 (4)
C1—H1A	0.9600	C13—C18	1.374 (5)
C1—H1B	0.9600	C13—S2	1.791 (3)
C1—H1C	0.9600	C14—C15	1.399 (5)
C2—C3	1.341 (5)	C14—H14	0.9300
C2—S1	1.721 (4)	C15—C16	1.385 (5)
C3—C4	1.402 (5)	C15—H15	0.9300
C3—H3	0.9300	C16—C17	1.370 (5)
C4—C5	1.334 (5)	C16—N2	1.422 (4)
C4—H4	0.9300	C17—C18	1.378 (5)
C5—C6	1.438 (5)	C17—H17	0.9300
C5—S1	1.718 (3)	C18—H18	0.9300
C6—N1	1.267 (4)	C19—N2	1.271 (4)
C6—H6	0.9300	C19—C20	1.439 (4)
C7—C12	1.378 (5)	C19—H19	0.9300
C7—C8	1.386 (4)	C20—C21	1.350 (5)
C7—N1	1.404 (4)	C20—S3	1.722 (3)
C8—C9	1.387 (5)	C21—C22	1.412 (5)
C8—H8	0.9300	C21—H21	0.9300
C9—C10	1.382 (4)	C22—C23	1.337 (5)
C9—H9	0.9300	C22—H22	0.9300
C10—C11	1.367 (4)	C23—C24	1.484 (4)
C10—S2	1.777 (3)	C23—S3	1.725 (3)
C11—C12	1.379 (5)	C24—H24A	0.9600
C11—H11	0.9300	C24—H24B	0.9600
C12—H12	0.9300	C24—H24C	0.9600
C2—C1—H1A	109.5	C13—C14—C15	120.0 (3)
C2—C1—H1B	109.5	C13—C14—H14	120.0
H1A—C1—H1B	109.5	C15—C14—H14	120.0
C2—C1—H1C	109.5	C16—C15—C14	120.5 (3)
H1A—C1—H1C	109.5	C16—C15—H15	119.7

H1B—C1—H1C	109.5	C14—C15—H15	119.7
C3—C2—C1	129.2 (4)	C17—C16—C15	118.5 (3)
C3—C2—S1	111.1 (3)	C17—C16—N2	124.4 (3)
C1—C2—S1	119.7 (3)	C15—C16—N2	117.1 (3)
C2—C3—C4	112.4 (3)	C16—C17—C18	121.0 (3)
C2—C3—H3	123.8	C16—C17—H17	119.5
C4—C3—H3	123.8	C18—C17—H17	119.5
C5—C4—C3	114.5 (4)	C13—C18—C17	120.8 (3)
C5—C4—H4	122.7	C13—C18—H18	119.6
C3—C4—H4	122.7	C17—C18—H18	119.6
C4—C5—C6	129.1 (3)	N2—C19—C20	123.4 (3)
C4—C5—S1	110.3 (3)	N2—C19—H19	118.3
C6—C5—S1	120.5 (3)	C20—C19—H19	118.3
N1—C6—C5	123.5 (3)	C21—C20—C19	127.5 (3)
N1—C6—H6	118.3	C21—C20—S3	110.9 (2)
C5—C6—H6	118.3	C19—C20—S3	121.5 (3)
C12—C7—C8	117.7 (3)	C20—C21—C22	112.6 (3)
C12—C7—N1	118.7 (3)	C20—C21—H21	123.7
C8—C7—N1	123.3 (3)	C22—C21—H21	123.7
C7—C8—C9	120.7 (3)	C23—C22—C21	114.0 (3)
C7—C8—H8	119.6	C23—C22—H22	123.0
C9—C8—H8	119.6	C21—C22—H22	123.0
C10—C9—C8	120.4 (3)	C22—C23—C24	128.9 (3)
C10—C9—H9	119.8	C22—C23—S3	110.6 (2)
C8—C9—H9	119.8	C24—C23—S3	120.5 (2)
C11—C10—C9	118.9 (3)	C23—C24—H24A	109.5
C11—C10—S2	117.9 (2)	C23—C24—H24B	109.5
C9—C10—S2	123.1 (2)	H24A—C24—H24B	109.5
C10—C11—C12	120.6 (3)	C23—C24—H24C	109.5
C10—C11—H11	119.7	H24A—C24—H24C	109.5
C12—C11—H11	119.7	H24B—C24—H24C	109.5
C7—C12—C11	121.6 (3)	C6—N1—C7	120.9 (3)
C7—C12—H12	119.2	C19—N2—C16	118.8 (3)
C11—C12—H12	119.2	C5—S1—C2	91.71 (19)
C14—C13—C18	119.2 (3)	C10—S2—C13	102.28 (15)
C14—C13—S2	120.4 (3)	C20—S3—C23	91.82 (16)
C18—C13—S2	120.4 (2)		

Fig. 1


C12
C11
S1
N1
S2
C18
C17
C21
C1
C2
C7
C10
C13
C20
C22
C3
C5
C6
C8
C9
C14
C16
C19
C23
C4
C15
N2
S3
C24