Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-4-Chloro-2-[(2,3-dimethyl-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrazol-4-yl)-iminomethyl]-6-methoxyphenyl 4-methylbenzenesulfonate acetonitrile solvate

Xiao-Li Zhen,[a] Xia Tian,[a] Zhen-Chao Li,[a] Jian-Rong Han[a]* and Shou-Xin Liu[b]‡

[a]College of Sciences, Hebei University of Science and Technology, Shijiazhuang 050018, People's Republic of China, and [b]College of Chemical and Pharmaceutical Engineering, Hebei University of Science and Technology, Shijiazhuang 050018, People's Republic of China
Correspondence e-mail: han_jianrong@163.com

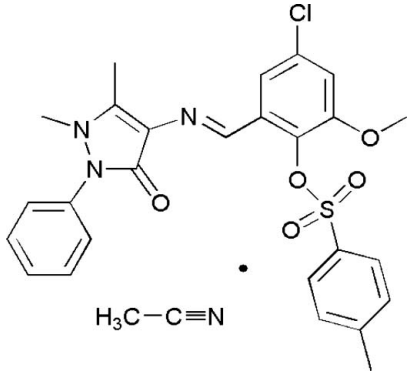
Received 16 October 2007; accepted 16 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.133; data-to-parameter ratio = 13.7.

In the title compound, $C_{26}H_{24}ClN_3O_5S\cdot C_2H_3N$, the *o*-vanillin group makes dihedral angles of 25.87 (4), 11.93 (3) and 72.05 (7)° with the pyrazolone, benzene and phenyl rings, respectively. The crystal structure is stabilized by weak intermolecular C—H···O interactions that link molecules into centrosymmetric dimers.

Related literature

For related structures, see: Han *et al.* (2007); Hu (2006). For general background, see: Kahwa *et al.* (1986); Santos *et al.* (2001). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{26}H_{24}ClN_3O_5S\cdot C_2H_3N$
M_r = 567.06
Triclinic, $P\overline{1}$
a = 7.3242 (9) Å
b = 13.2452 (17) Å
c = 15.3145 (19) Å
α = 108.731 (2)°
β = 93.303 (2)°
γ = 94.910 (2)°
V = 1396.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.955
7152 measured reflections
4893 independent reflections
4057 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.133
S = 1.02
4893 reflections
358 parameters
19 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O2^{i}	0.93	2.57	3.386 (3)	146

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

This project was supported by the Foundation of the Education Department of Hebei Province (grant No. 606022).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2589).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
Han, J.-R., Zhen, X.-L., Tian, X., Li, F. & Liu, S.-X. (2007). *Acta Cryst.* E**63**, o4035.
Hu, T.-P. (2006). *Acta Cryst.* E**62**, o2270–o2271.
Kahwa, I. A., Selbin, J., Hsieh, T. C.-Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

‡ Additional correspondence author: liu_shouxin@163.com.

 © 2007 International Union of Crystallography

supplementary materials

Acta Cryst. (2007). E**63**, o4397 [**doi:10.1107/S1600536807051021**]

(*E*)-4-Chloro-2-[(2,3-dimethyl-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrazol-4-yl)iminomethyl]-6-methoxyphenyl 4-methylbenzenesulfonate acetonitrile solvate

X.-L. Zhen, X. Tian, Z.-C. Li, J.-R. Han and S.-X. Liu

Comment

Schiff-base ligands have received a good deal of attention in biology and chemistry (Kahwa *et al.*, 1986) in areas such as protein and enzyme mimics (Santos *et al.*, 2001). Among the large number of compounds, 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them, such as (*E*)-4-[(1,5-Dimethyl-3-oxo-2-phenyl-2,3- dihydro-1*H*-pyrazol-4-ylimino)methyl]-phenyl 4-chlorobenzoate (Han *et al.*, 2007) and (*E*)-4-[4-(4-Chlorobenzyloxy)benzylideneamino]-1,5- dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Hu, 2006) have been reported. We now report the synthesis and molecular structure of the title Schiff base compound, (I), (Fig. 1)

The bond lengths and angles of (I) are within normal ranges (Allen *et al.*, 1987). The pyrazolone ring (C16—C18/N1/N2/N3/O5) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.030 Å. It makes a dihedral angle of 54.45 (9)° with the attached phenyl ring (C21—C26). The *o*-vanillin group (C8—C13/C15/O3/O4) is almost planar, with an r.m.s. deviation for fitted atoms of 0.020 Å. This group makes dihedral angles of 25.87 (4)°, 11.93 (3)° and 72.05 (7)°, with the the pyrazolone ring (C16—C18/N1/N2/N3/O5), the terminal C1—C6 benzene ring and the terminal C21—C26 phenyl ring, respectively.

The crystal packing is stabilized by weak intermolecular C10—H10···O2═S1 interaction (Table 1, Fig. 2) that form inversion dimers.

Experimental

An anhydrous ethanol solution (50 ml) of 4-chloro-2-formyl-6-methoxyphenyl 4-methylbenzenesulfonate (3.41 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture stirred at 350 K for 3 h under N_2, giving a yellow precipitate. The product was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give pure compound (I) in 85% yield. Yellow blocks of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Refinement

The H atoms were included in calculated positions (C—H = 0.93–0.96 Å) and refined as riding U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

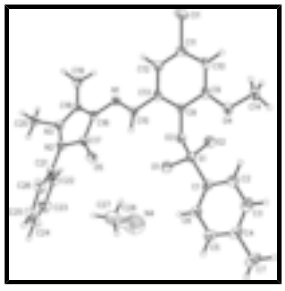


Fig. 1. The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

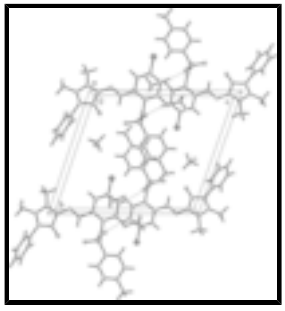


Fig. 2. A packing diagram for (I), with hydrogen bonds drawn as dashed lines.

(*E*)-4-Chloro-2-[(2,3-dimethyl-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrazol-4-\ yl)iminomethyl]-6-methoxyphenyl 4-methylbenzenesulfonate acetonitrile solvate

Crystal data

$C_{26}H_{24}ClN_3O_5S·C_2H_3N$	$Z = 2$
$M_r = 567.06$	$F_{000} = 592$
Triclinic, $P\overline{1}$	$D_x = 1.349$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.3242$ (9) Å	Cell parameters from 4430 reflections
$b = 13.2452$ (17) Å	$\theta = 2.8$–26.4º
$c = 15.3145$ (19) Å	$\mu = 0.26$ mm^{-1}
$\alpha = 108.731$ (2)º	$T = 294$ (2) K
$\beta = 93.303$ (2)º	Block, yellow
$\gamma = 94.910$ (2)º	0.24 × 0.22 × 0.18 mm
$V = 1396.1$ (3) Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	4893 independent reflections
Radiation source: fine-focus sealed tube	4057 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 294$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan	$h = -8 \rightarrow 8$

(SADABS; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.955
7152 measured reflections
$k = -15 \rightarrow 13$
$l = -18 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.133$
$S = 1.02$
4893 reflections
358 parameters
19 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
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.7683P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0160 (19)

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 $F^2 > 2\sigma(F^2)$ 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	0.62434 (8)	0.24085 (4)	0.38970 (4)	0.03804 (18)
Cl1	0.72326 (14)	−0.28513 (6)	0.33595 (5)	0.0783 (3)
O1	0.6098 (2)	0.25614 (14)	0.30172 (11)	0.0514 (4)
O2	0.4724 (2)	0.18514 (14)	0.41509 (12)	0.0509 (4)
O3	0.8015 (2)	0.17747 (11)	0.39394 (10)	0.0372 (4)
O4	0.7707 (2)	0.11000 (14)	0.53785 (10)	0.0505 (4)
O5	0.9284 (2)	0.18328 (12)	0.11286 (10)	0.0453 (4)
N1	0.7377 (2)	−0.03192 (14)	0.12557 (11)	0.0326 (4)
N2	0.8337 (2)	0.09923 (14)	−0.04314 (11)	0.0352 (4)
N3	0.7241 (2)	0.00446 (14)	−0.09482 (11)	0.0346 (4)
C1	0.6988 (3)	0.36377 (18)	0.47552 (15)	0.0405 (5)
C2	0.7231 (5)	0.3682 (2)	0.56658 (18)	0.0637 (8)
H2	0.7062	0.3062	0.5827	0.076*

C3	0.7733 (5)	0.4671 (2)	0.63347 (19)	0.0702 (8)
H3	0.7900	0.4711	0.6952	0.084*
C4	0.7994 (3)	0.5602 (2)	0.61129 (19)	0.0533 (6)
C5	0.7738 (4)	0.5527 (2)	0.52022 (19)	0.0559 (7)
H5	0.7901	0.6147	0.5042	0.067*
C6	0.7242 (4)	0.45531 (19)	0.45123 (17)	0.0494 (6)
H6	0.7082	0.4516	0.3896	0.059*
C7	0.8544 (4)	0.6671 (2)	0.6855 (2)	0.0747 (9)
H7A	0.9848	0.6758	0.7013	0.112*
H7B	0.7915	0.6699	0.7394	0.112*
H7C	0.8218	0.7236	0.6631	0.112*
C8	0.7729 (3)	0.06603 (17)	0.37770 (14)	0.0342 (5)
C9	0.7623 (3)	0.03145 (18)	0.45460 (14)	0.0386 (5)
C10	0.7456 (3)	−0.07720 (19)	0.44131 (15)	0.0438 (5)
H10	0.7382	−0.1023	0.4913	0.053*
C11	0.7400 (3)	−0.14827 (18)	0.35172 (16)	0.0445 (5)
C12	0.7497 (3)	−0.11512 (18)	0.27572 (15)	0.0392 (5)
H12	0.7457	−0.1652	0.2168	0.047*
C13	0.7657 (3)	−0.00529 (17)	0.28775 (14)	0.0338 (5)
C14	0.7599 (4)	0.0782 (2)	0.61864 (16)	0.0544 (6)
H14A	0.6451	0.0352	0.6139	0.082*
H14B	0.7678	0.1408	0.6727	0.082*
H14C	0.8597	0.0372	0.6234	0.082*
C15	0.7817 (3)	0.03317 (17)	0.20836 (13)	0.0332 (4)
H15	0.8235	0.1045	0.2179	0.040*
C16	0.7603 (3)	0.00425 (16)	0.05086 (13)	0.0306 (4)
C17	0.8511 (3)	0.10494 (17)	0.04996 (13)	0.0329 (4)
C18	0.6926 (3)	−0.05439 (16)	−0.03873 (13)	0.0318 (4)
C19	0.6029 (3)	−0.16592 (17)	−0.07583 (15)	0.0410 (5)
H19A	0.4963	−0.1690	−0.1165	0.062*
H19B	0.5665	−0.1893	−0.0256	0.062*
H19C	0.6877	−0.2119	−0.1095	0.062*
C20	0.7379 (3)	−0.0387 (2)	−0.19409 (14)	0.0458 (6)
H20A	0.8540	−0.0670	−0.2052	0.069*
H20B	0.7292	0.0173	−0.2208	0.069*
H20C	0.6398	−0.0950	−0.2217	0.069*
C21	0.8404 (3)	0.19099 (17)	−0.07269 (14)	0.0365 (5)
C22	0.6866 (3)	0.2147 (2)	−0.11566 (17)	0.0474 (6)
H22	0.5772	0.1695	−0.1279	0.057*
C23	0.6975 (4)	0.3068 (2)	−0.1402 (2)	0.0638 (8)
H23	0.5953	0.3229	−0.1701	0.077*
C24	0.8578 (5)	0.3746 (2)	−0.1208 (2)	0.0693 (8)
H24	0.8634	0.4370	−0.1365	0.083*
C25	1.0115 (4)	0.3500 (2)	−0.0779 (2)	0.0651 (8)
H25	1.1201	0.3961	−0.0648	0.078*
C26	1.0043 (3)	0.2575 (2)	−0.05454 (18)	0.0499 (6)
H26	1.1081	0.2401	−0.0270	0.060*
C27	0.7594 (6)	0.4169 (3)	0.1580 (3)	0.1074 (14)
H27A	0.8269	0.4824	0.1979	0.161*

H27B	0.8110	0.3952	0.0995	0.161*
H27C	0.7666	0.3619	0.1861	0.161*
C28	0.5735 (7)	0.4333 (3)	0.1443 (4)	0.128 (2)
N4	0.4221 (10)	0.4429 (5)	0.1437 (5)	0.186 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0442 (3)	0.0362 (3)	0.0308 (3)	−0.0002 (2)	−0.0012 (2)	0.0088 (2)
Cl1	0.1364 (8)	0.0419 (4)	0.0635 (5)	0.0086 (4)	0.0092 (4)	0.0272 (3)
O1	0.0684 (11)	0.0506 (10)	0.0334 (9)	0.0052 (8)	−0.0071 (7)	0.0135 (7)
O2	0.0449 (9)	0.0478 (10)	0.0571 (10)	−0.0033 (7)	0.0066 (8)	0.0148 (8)
O3	0.0424 (8)	0.0357 (8)	0.0318 (8)	−0.0017 (6)	0.0012 (6)	0.0105 (6)
O4	0.0740 (12)	0.0533 (10)	0.0236 (8)	0.0054 (8)	0.0044 (7)	0.0124 (7)
O5	0.0603 (10)	0.0392 (9)	0.0311 (8)	−0.0092 (7)	−0.0047 (7)	0.0095 (7)
N1	0.0352 (9)	0.0366 (9)	0.0270 (9)	0.0047 (7)	0.0025 (7)	0.0117 (7)
N2	0.0425 (10)	0.0353 (9)	0.0273 (9)	−0.0016 (7)	−0.0020 (7)	0.0120 (7)
N3	0.0436 (10)	0.0360 (9)	0.0228 (8)	0.0016 (8)	−0.0008 (7)	0.0089 (7)
C1	0.0443 (12)	0.0382 (12)	0.0340 (11)	0.0014 (9)	0.0008 (9)	0.0063 (9)
C2	0.102 (2)	0.0456 (15)	0.0395 (14)	−0.0013 (14)	−0.0023 (14)	0.0125 (12)
C3	0.103 (2)	0.0603 (18)	0.0353 (14)	0.0017 (16)	−0.0083 (14)	0.0029 (12)
C4	0.0465 (14)	0.0434 (14)	0.0578 (16)	0.0052 (11)	0.0011 (11)	0.0004 (12)
C5	0.0641 (17)	0.0380 (13)	0.0615 (17)	0.0027 (11)	0.0072 (13)	0.0111 (12)
C6	0.0612 (15)	0.0435 (13)	0.0434 (13)	0.0041 (11)	0.0049 (11)	0.0143 (11)
C7	0.074 (2)	0.0527 (17)	0.073 (2)	0.0020 (14)	−0.0036 (16)	−0.0105 (15)
C8	0.0371 (11)	0.0353 (11)	0.0302 (11)	0.0017 (9)	0.0008 (8)	0.0120 (9)
C9	0.0413 (12)	0.0490 (13)	0.0263 (11)	0.0025 (10)	0.0012 (9)	0.0143 (9)
C10	0.0525 (14)	0.0520 (14)	0.0342 (12)	0.0054 (11)	0.0042 (10)	0.0241 (10)
C11	0.0557 (14)	0.0384 (12)	0.0433 (13)	0.0037 (10)	0.0033 (10)	0.0194 (10)
C12	0.0489 (13)	0.0366 (11)	0.0314 (11)	0.0047 (9)	0.0015 (9)	0.0105 (9)
C13	0.0341 (10)	0.0404 (11)	0.0272 (10)	0.0011 (9)	0.0011 (8)	0.0125 (9)
C14	0.0636 (16)	0.0758 (18)	0.0277 (12)	0.0086 (13)	0.0048 (10)	0.0221 (12)
C15	0.0365 (11)	0.0351 (11)	0.0287 (10)	0.0033 (8)	0.0033 (8)	0.0115 (9)
C16	0.0350 (10)	0.0319 (10)	0.0252 (10)	0.0048 (8)	0.0025 (8)	0.0095 (8)
C17	0.0360 (11)	0.0367 (11)	0.0261 (10)	0.0040 (9)	0.0024 (8)	0.0105 (9)
C18	0.0333 (10)	0.0340 (11)	0.0282 (10)	0.0062 (8)	0.0041 (8)	0.0096 (8)
C19	0.0499 (13)	0.0365 (12)	0.0325 (11)	−0.0012 (10)	−0.0010 (9)	0.0079 (9)
C20	0.0576 (14)	0.0519 (14)	0.0235 (11)	−0.0006 (11)	0.0042 (9)	0.0080 (10)
C21	0.0460 (12)	0.0376 (11)	0.0284 (10)	0.0045 (9)	0.0054 (9)	0.0137 (9)
C22	0.0497 (13)	0.0488 (14)	0.0466 (14)	0.0025 (11)	−0.0028 (10)	0.0214 (11)
C23	0.0738 (19)	0.0610 (17)	0.0662 (18)	0.0118 (14)	−0.0092 (14)	0.0357 (15)
C24	0.088 (2)	0.0541 (17)	0.080 (2)	0.0037 (15)	0.0027 (17)	0.0433 (16)
C25	0.0662 (18)	0.0571 (17)	0.080 (2)	−0.0095 (14)	0.0029 (15)	0.0378 (15)
C26	0.0478 (14)	0.0514 (14)	0.0552 (15)	−0.0004 (11)	0.0029 (11)	0.0256 (12)
C27	0.116 (4)	0.075 (3)	0.134 (4)	0.009 (2)	0.029 (3)	0.036 (3)
C28	0.123 (4)	0.046 (2)	0.217 (6)	0.015 (2)	0.049 (4)	0.038 (3)
N4	0.185 (3)	0.183 (3)	0.190 (3)	0.0223 (11)	0.0167 (10)	0.0609 (12)

Geometric parameters (Å, °)

S1—O2	1.4240 (17)	C10—H10	0.9300
S1—O1	1.4247 (17)	C11—C12	1.373 (3)
S1—O3	1.6139 (16)	C12—C13	1.400 (3)
S1—C1	1.751 (2)	C12—H12	0.9300
Cl1—C11	1.743 (2)	C13—C15	1.468 (3)
O3—C8	1.411 (2)	C14—H14A	0.9600
O4—C9	1.356 (3)	C14—H14B	0.9600
O4—C14	1.434 (3)	C14—H14C	0.9600
O5—C17	1.233 (2)	C15—H15	0.9300
N1—C15	1.288 (3)	C16—C18	1.380 (3)
N1—C16	1.388 (3)	C16—C17	1.443 (3)
N2—C17	1.401 (3)	C18—C19	1.479 (3)
N2—N3	1.401 (2)	C19—H19A	0.9600
N2—C21	1.425 (3)	C19—H19B	0.9600
N3—C18	1.349 (3)	C19—H19C	0.9600
N3—C20	1.455 (3)	C20—H20A	0.9600
C1—C2	1.377 (3)	C20—H20B	0.9600
C1—C6	1.379 (3)	C20—H20C	0.9600
C2—C3	1.384 (4)	C21—C22	1.381 (3)
C2—H2	0.9300	C21—C26	1.385 (3)
C3—C4	1.381 (4)	C22—C23	1.385 (4)
C3—H3	0.9300	C22—H22	0.9300
C4—C5	1.367 (4)	C23—C24	1.372 (4)
C4—C7	1.509 (4)	C23—H23	0.9300
C5—C6	1.383 (3)	C24—C25	1.386 (4)
C5—H5	0.9300	C24—H24	0.9300
C6—H6	0.9300	C25—C26	1.380 (4)
C7—H7A	0.9600	C25—H25	0.9300
C7—H7B	0.9600	C26—H26	0.9300
C7—H7C	0.9600	C27—C28	1.411 (6)
C8—C13	1.393 (3)	C27—H27A	0.9600
C8—C9	1.398 (3)	C27—H27B	0.9600
C9—C10	1.381 (3)	C27—H27C	0.9600
C10—C11	1.388 (3)	C28—N4	1.126 (7)
O2—S1—O1	118.58 (11)	C12—C13—C15	120.92 (19)
O2—S1—O3	107.79 (9)	O4—C14—H14A	109.5
O1—S1—O3	107.52 (9)	O4—C14—H14B	109.5
O2—S1—C1	111.84 (11)	H14A—C14—H14B	109.5
O1—S1—C1	109.30 (11)	O4—C14—H14C	109.5
O3—S1—C1	100.09 (9)	H14A—C14—H14C	109.5
C8—O3—S1	118.42 (12)	H14B—C14—H14C	109.5
C9—O4—C14	117.70 (19)	N1—C15—C13	119.96 (19)
C15—N1—C16	119.45 (18)	N1—C15—H15	120.0
C17—N2—N3	109.06 (16)	C13—C15—H15	120.0
C17—N2—C21	123.68 (17)	C18—C16—N1	123.36 (18)
N3—N2—C21	120.39 (16)	C18—C16—C17	107.84 (17)

C18—N3—N2	107.95 (15)	N1—C16—C17	128.80 (17)
C18—N3—C20	125.35 (18)	O5—C17—N2	123.64 (19)
N2—N3—C20	118.97 (17)	O5—C17—C16	131.51 (19)
C2—C1—C6	121.1 (2)	N2—C17—C16	104.84 (16)
C2—C1—S1	119.46 (19)	N3—C18—C16	109.78 (18)
C6—C1—S1	119.36 (18)	N3—C18—C19	121.14 (18)
C1—C2—C3	118.3 (3)	C16—C18—C19	129.05 (19)
C1—C2—H2	120.9	C18—C19—H19A	109.5
C3—C2—H2	120.9	C18—C19—H19B	109.5
C4—C3—C2	122.0 (3)	H19A—C19—H19B	109.5
C4—C3—H3	119.0	C18—C19—H19C	109.5
C2—C3—H3	119.0	H19A—C19—H19C	109.5
C5—C4—C3	118.1 (2)	H19B—C19—H19C	109.5
C5—C4—C7	121.0 (3)	N3—C20—H20A	109.5
C3—C4—C7	120.9 (3)	N3—C20—H20B	109.5
C4—C5—C6	121.7 (3)	H20A—C20—H20B	109.5
C4—C5—H5	119.1	N3—C20—H20C	109.5
C6—C5—H5	119.1	H20A—C20—H20C	109.5
C1—C6—C5	118.8 (2)	H20B—C20—H20C	109.5
C1—C6—H6	120.6	C22—C21—C26	120.9 (2)
C5—C6—H6	120.6	C22—C21—N2	121.1 (2)
C4—C7—H7A	109.5	C26—C21—N2	117.9 (2)
C4—C7—H7B	109.5	C21—C22—C23	119.1 (2)
H7A—C7—H7B	109.5	C21—C22—H22	120.4
C4—C7—H7C	109.5	C23—C22—H22	120.4
H7A—C7—H7C	109.5	C24—C23—C22	120.5 (3)
H7B—C7—H7C	109.5	C24—C23—H23	119.7
C13—C8—C9	122.4 (2)	C22—C23—H23	119.7
C13—C8—O3	119.87 (18)	C23—C24—C25	119.9 (3)
C9—C8—O3	117.69 (18)	C23—C24—H24	120.0
O4—C9—C10	125.18 (19)	C25—C24—H24	120.0
O4—C9—C8	115.8 (2)	C26—C25—C24	120.3 (3)
C10—C9—C8	119.0 (2)	C26—C25—H25	119.8
C9—C10—C11	118.6 (2)	C24—C25—H25	119.8
C9—C10—H10	120.7	C25—C26—C21	119.1 (2)
C11—C10—H10	120.7	C25—C26—H26	120.5
C12—C11—C10	122.8 (2)	C21—C26—H26	120.5
C12—C11—Cl1	118.97 (18)	C28—C27—H27A	109.5
C10—C11—Cl1	118.23 (18)	C28—C27—H27B	109.5
C11—C12—C13	119.4 (2)	H27A—C27—H27B	109.5
C11—C12—H12	120.3	C28—C27—H27C	109.5
C13—C12—H12	120.3	H27A—C27—H27C	109.5
C8—C13—C12	117.77 (19)	H27B—C27—H27C	109.5
C8—C13—C15	121.27 (19)	N4—C28—C27	170.8 (7)
O2—S1—O3—C8	28.54 (16)	O3—C8—C13—C12	175.72 (18)
O1—S1—O3—C8	−100.38 (15)	C9—C8—C13—C15	−178.51 (19)
C1—S1—O3—C8	145.53 (15)	O3—C8—C13—C15	−1.9 (3)
C17—N2—N3—C18	−7.5 (2)	C11—C12—C13—C8	0.6 (3)
C21—N2—N3—C18	−159.44 (18)	C11—C12—C13—C15	178.3 (2)

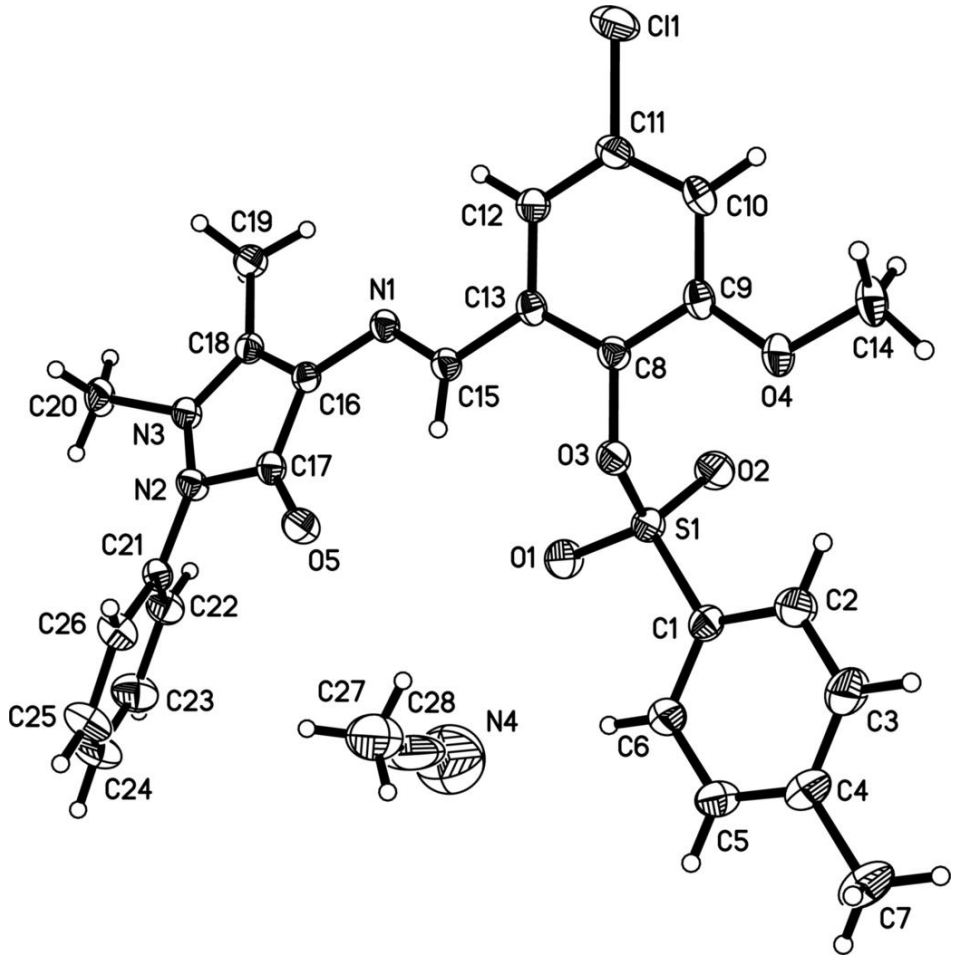
C17—N2—N3—C20	−158.46 (19)	C16—N1—C15—C13	−177.41 (17)
C21—N2—N3—C20	49.6 (3)	C8—C13—C15—N1	−165.84 (19)
O2—S1—C1—C2	46.8 (2)	C12—C13—C15—N1	16.6 (3)
O1—S1—C1—C2	−179.9 (2)	C15—N1—C16—C18	−170.18 (19)
O3—S1—C1—C2	−67.1 (2)	C15—N1—C16—C17	8.9 (3)
O2—S1—C1—C6	−130.3 (2)	N3—N2—C17—O5	−174.35 (19)
O1—S1—C1—C6	3.0 (2)	C21—N2—C17—O5	−23.5 (3)
O3—S1—C1—C6	115.8 (2)	N3—N2—C17—C16	5.0 (2)
C6—C1—C2—C3	0.2 (4)	C21—N2—C17—C16	155.87 (19)
S1—C1—C2—C3	−176.9 (2)	C18—C16—C17—O5	178.4 (2)
C1—C2—C3—C4	−0.1 (5)	N1—C16—C17—O5	−0.8 (4)
C2—C3—C4—C5	0.2 (5)	C18—C16—C17—N2	−0.9 (2)
C2—C3—C4—C7	−179.9 (3)	N1—C16—C17—N2	179.94 (19)
C3—C4—C5—C6	−0.4 (4)	N2—N3—C18—C16	6.9 (2)
C7—C4—C5—C6	179.7 (3)	C20—N3—C18—C16	155.5 (2)
C2—C1—C6—C5	−0.4 (4)	N2—N3—C18—C19	−171.31 (18)
S1—C1—C6—C5	176.7 (2)	C20—N3—C18—C19	−22.7 (3)
C4—C5—C6—C1	0.5 (4)	N1—C16—C18—N3	175.48 (18)
S1—O3—C8—C13	88.5 (2)	C17—C16—C18—N3	−3.7 (2)
S1—O3—C8—C9	−94.75 (19)	N1—C16—C18—C19	−6.5 (3)
C14—O4—C9—C10	−0.6 (3)	C17—C16—C18—C19	174.3 (2)
C14—O4—C9—C8	179.9 (2)	C17—N2—C21—C22	−112.6 (2)
C13—C8—C9—O4	−179.84 (19)	N3—N2—C21—C22	35.1 (3)
O3—C8—C9—O4	3.5 (3)	C17—N2—C21—C26	65.5 (3)
C13—C8—C9—C10	0.6 (3)	N3—N2—C21—C26	−146.8 (2)
O3—C8—C9—C10	−176.11 (19)	C26—C21—C22—C23	−0.4 (4)
O4—C9—C10—C11	−179.5 (2)	N2—C21—C22—C23	177.7 (2)
C8—C9—C10—C11	0.0 (3)	C21—C22—C23—C24	−1.1 (4)
C9—C10—C11—C12	−0.3 (4)	C22—C23—C24—C25	1.2 (5)
C9—C10—C11—Cl1	178.83 (18)	C23—C24—C25—C26	0.0 (5)
C10—C11—C12—C13	−0.1 (4)	C24—C25—C26—C21	−1.4 (5)
Cl1—C11—C12—C13	−179.15 (17)	C22—C21—C26—C25	1.6 (4)
C9—C8—C13—C12	−0.9 (3)	N2—C21—C26—C25	−176.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O2^{i}	0.93	2.57	3.386 (3)	146

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

Fig. 1


Cl1
C11
C12
C10
C19
N1
C13
C9
C18
C8
O4
C14
C20
C16
C15
N3
O3
O2
C17
N2
S1
O5
O1
C21
C22
C2
C26
C1
C27
C28
N4
C23
C3
C25
C6
C24
C4
C5
C7

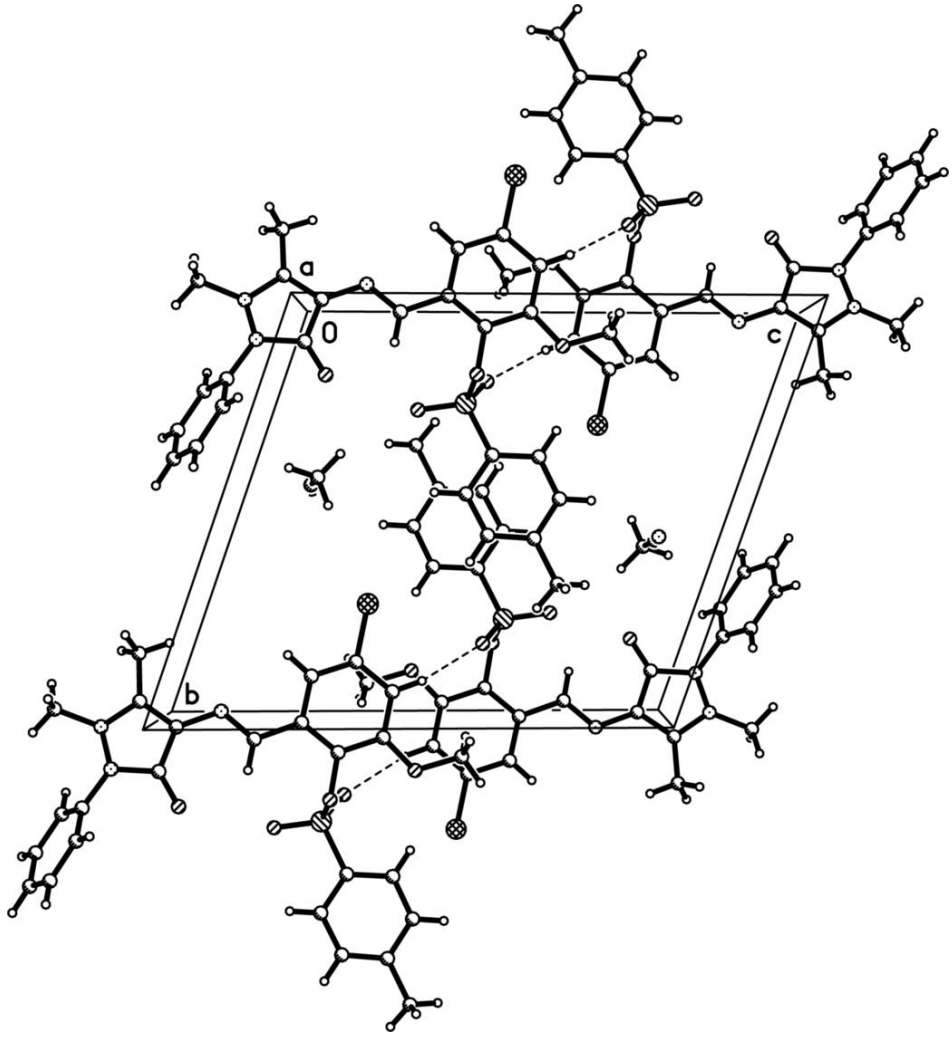



Fig. 2