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## Structure Reports

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# 1-[3-(2,4-Dichloro-5-fluorophenyl)-5-(3-methyl-2-thienyl)-4,5-dihydro-1H-pyrazol-1-yl]ethanone

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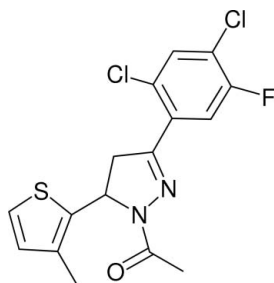
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Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å; R factor = 0.051;  $wR$  factor = 0.179; data-to-parameter ratio = 25.9.

In the title molecule,  $\text{C}_{16}\text{H}_{13}\text{Cl}_2\text{FN}_2\text{OS}$ , the dihedral angle between the thiophene and benzene rings is  $80.34$  ( $12$ )°. The pyrazoline ring is in an envelope conformation, and the plane through the four coplanar atoms makes dihedral angles of  $85.13$  ( $9$ ) and  $6.89$  ( $10$ )° with the thiophene and benzene rings, respectively. The C and O atoms of the acetyl group are nearly coplanar with the attached pyrazoline ring. In the crystal structure, inversion dimers arise from pairs of intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds. A short intermolecular  $\text{Cl}\cdots\text{S}$  contact of  $3.4250$  ( $13$ ) Å is also found.

## Related literature

For a related crystal structure, see: Thiruvalluvar *et al.* (2007).



## Experimental

### Crystal data

$\text{C}_{16}\text{H}_{13}\text{Cl}_2\text{FN}_2\text{OS}$   
 $M_r = 371.25$   
 Triclinic,  $P\bar{1}$   
 $a = 7.2240$  ( $5$ ) Å  
 $b = 8.8642$  ( $4$ ) Å  
 $c = 14.0518$  ( $9$ ) Å  
 $\alpha = 100.794$  ( $5$ )°  
 $\beta = 103.307$  ( $6$ )°

$\gamma = 101.003$  ( $5$ )°  
 $V = 833.99$  ( $10$ ) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.53$  mm<sup>-1</sup>  
 $T = 295$  ( $2$ ) K  
 $0.52 \times 0.43 \times 0.35$  mm

### Data collection

Oxford Diffraction R Gemini diffractometer  
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)

$T_{\min} = 0.786$ ,  $T_{\max} = 1.000$   
 (expected range =  $0.654$ – $0.831$ )  
 12358 measured reflections  
 5445 independent reflections  
 3028 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$   
 $wR(F^2) = 0.179$   
 $S = 1.12$   
 5445 reflections

210 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.32$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.39$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2A\cdots\text{O}1^i$	0.96	2.58	3.533 (4)	171

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; 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: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2286).

## References

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 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.  
 Thiruvalluvar, A., Subramanyam, M., Butcher, R. J. & Mahalinga, M. (2007). *Acta Cryst.* **E63**, o4770.

**supplementary materials**

*Acta Cryst.* (2008). E64, o2160 [ doi:10.1107/S1600536808033837 ]

## 1-[3-(2,4-Dichloro-5-fluorophenyl)-5-(3-methyl-2-thienyl)-4,5-dihydro-1H-pyrazol-1-yl]ethanone

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### Comment

A great deal of attention has been paid to the synthesis and structural aspects of pyrazolines, as witnessed by continued activity in this area (Thiruvalluvar *et al.*, 2007).

In the title molecule, C<sub>16</sub>H<sub>13</sub>Cl<sub>2</sub>FN<sub>2</sub>OS, Fig.1., the dihedral angle between the thiophene and benzene rings is 80.34 (12)°. The pyrazoline ring is in an envelope conformation and the plane through the four coplanar atoms makes dihedral angles of 85.13 (9)° and 6.89 (10)° with the thiophene and benzene rings, respectively. The acetyl group, except for the hydrogen atoms, is nearly coplanar with the attached pyrazoline ring. An intermolecular C2—H2A···O1(-1 - x, 1 - y, -z) hydrogen bond is found in the crystal structure (Table 1). Further, a short intermolecular Cl4···S21(1-x,1-y,1-z) contact of 3.4250 (13) Å is also found in the crystal structure.

### Experimental

A mixture of 1-(2,4-dichloro-5-fluorophenyl)-3-(3-methylthien-2-yl) prop-2-en-1-one (5 g, 0.016 mol) and a molar equivalent of hydrazine hydrate (80%) in glacial acetic acid (25 ml) was heated on a water bath at 363–365 K for 5–6 h. The reaction mass was then poured into ice-cold water. The solid obtained was filtered, washed with water, dried and crystallized from methanol to yield the title compound. Yield 5.5 g (93.5%).

### Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93, 0.96, 0.97 and 0.98 Å for Csp<sup>2</sup>, methyl, methylene and methine C, respectively;  $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$ , where  $k = 1.5$  for methyl and 1.2 for all other H atoms.

### Figures

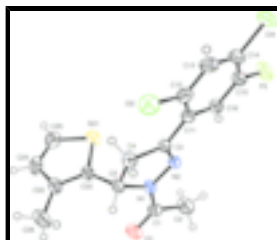


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radius.

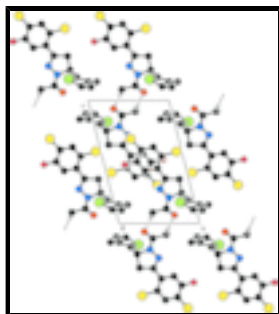


Fig. 2. The packing of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

## 1-[3-(2,4-Dichloro-5-fluorophenyl)-5-(3-methyl-2-thienyl)-4,5-dihydro-1H-pyrazol-1-yl]ethanone

### Crystal data

$C_{16}H_{13}Cl_2FN_2OS$

$M_r = 371.25$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.2240$  (5) Å

$b = 8.8642$  (4) Å

$c = 14.0518$  (9) Å

$\alpha = 100.794$  (5)°

$\beta = 103.307$  (6)°

$\gamma = 101.003$  (5)°

$V = 833.99$  (10) Å<sup>3</sup>

$Z = 2$

$F_{000} = 380$

$D_x = 1.478$  Mg m<sup>-3</sup>

Melting point: 369.5 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 4639 reflections

$\theta = 4.6$ – $32.4$ °

$\mu = 0.53$  mm<sup>-1</sup>

$T = 295$  (2) K

Chunk, pale-yellow

$0.52 \times 0.43 \times 0.35$  mm

### Data collection

Oxford Diffraction R Gemini diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 10.5081 pixels mm<sup>-1</sup>

$T = 295$ (2) K

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)

$T_{\min} = 0.786$ ,  $T_{\max} = 1.000$

12358 measured reflections

5445 independent reflections

3028 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 32.5$ °

$\theta_{\min} = 4.6$ °

$h = -10 \rightarrow 10$

$k = -13 \rightarrow 13$

$l = -21 \rightarrow 21$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.179$	$w = 1/[\sigma^2(F_o^2) + (0.083P)^2 + 0.1183P]$
$S = 1.12$	where $P = (F_o^2 + 2F_c^2)/3$
5445 reflections	$(\Delta/\sigma)_{\max} = 0.001$
210 parameters	$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

*Special details*

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C12	0.31301 (12)	0.93549 (8)	0.58594 (5)	0.0696 (3)
C14	0.45313 (12)	0.40815 (13)	0.68787 (6)	0.0875 (4)
S21	0.33962 (10)	0.82555 (8)	0.17261 (5)	0.0572 (2)
F5	0.2322 (3)	0.25555 (19)	0.47920 (15)	0.0820 (7)
O1	-0.2572 (3)	0.7121 (2)	0.06719 (14)	0.0721 (7)
N1	-0.0630 (3)	0.6973 (2)	0.21112 (13)	0.0432 (5)
N2	0.0012 (3)	0.6131 (2)	0.28019 (13)	0.0388 (5)
C1	-0.1967 (4)	0.6281 (3)	0.12037 (17)	0.0499 (7)
C2	-0.2599 (5)	0.4503 (3)	0.0905 (2)	0.0753 (10)
C3	0.1042 (3)	0.7120 (2)	0.36402 (15)	0.0352 (6)
C4	0.1162 (4)	0.8829 (3)	0.36061 (16)	0.0460 (7)
C5	0.0216 (3)	0.8696 (2)	0.24839 (16)	0.0434 (7)
C11	0.1899 (3)	0.6464 (3)	0.44812 (15)	0.0383 (6)
C12	0.2871 (3)	0.7332 (3)	0.54694 (16)	0.0464 (7)
C13	0.3677 (3)	0.6604 (4)	0.62105 (18)	0.0574 (9)
C14	0.3507 (4)	0.5009 (4)	0.5990 (2)	0.0574 (9)
C15	0.2522 (4)	0.4140 (3)	0.5020 (2)	0.0533 (8)
C16	0.1750 (3)	0.4835 (3)	0.42821 (18)	0.0457 (7)
C22	0.1646 (3)	0.9275 (3)	0.19146 (16)	0.0429 (6)
C23	0.1824 (4)	1.0568 (3)	0.15275 (18)	0.0514 (8)
C24	0.3412 (4)	1.0737 (3)	0.10905 (19)	0.0629 (9)
C25	0.4390 (4)	0.9588 (4)	0.1136 (2)	0.0634 (10)
C26	0.0455 (5)	1.1637 (3)	0.1527 (3)	0.0800 (11)
H2A	-0.39392	0.41733	0.05015	0.1130*
H2B	-0.24825	0.41020	0.14997	0.1130*
H2C	-0.17792	0.40987	0.05240	0.1130*

## supplementary materials

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H4A	0.25124	0.94454	0.38196	0.0552*
H4B	0.04343	0.93070	0.40277	0.0552*
H5	-0.08333	0.92575	0.24149	0.0520*
H13	0.43338	0.72114	0.68588	0.0688*
H16	0.11130	0.42108	0.36356	0.0549*
H24	0.37514	1.15693	0.07973	0.0755*
H25	0.54602	0.95296	0.08813	0.0761*
H26A	-0.01973	1.15300	0.20427	0.1200*
H26B	-0.05021	1.13543	0.08824	0.1200*
H26C	0.11838	1.27147	0.16557	0.1200*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C12	0.0784 (5)	0.0659 (4)	0.0470 (4)	0.0100 (3)	0.0044 (3)	-0.0045 (3)
C14	0.0679 (5)	0.1559 (8)	0.0819 (5)	0.0599 (5)	0.0340 (4)	0.0827 (5)
S21	0.0645 (4)	0.0652 (4)	0.0544 (4)	0.0279 (3)	0.0223 (3)	0.0251 (3)
F5	0.0976 (14)	0.0656 (10)	0.1059 (14)	0.0393 (10)	0.0337 (11)	0.0495 (10)
O1	0.0727 (13)	0.0737 (12)	0.0585 (11)	0.0147 (10)	-0.0122 (9)	0.0292 (10)
N1	0.0460 (10)	0.0385 (9)	0.0402 (9)	0.0047 (8)	0.0017 (8)	0.0163 (7)
N2	0.0357 (9)	0.0400 (9)	0.0378 (9)	0.0061 (7)	0.0032 (7)	0.0146 (7)
C1	0.0470 (13)	0.0536 (13)	0.0429 (12)	0.0087 (10)	0.0013 (10)	0.0142 (10)
C2	0.082 (2)	0.0553 (15)	0.0583 (16)	0.0023 (14)	-0.0197 (14)	0.0040 (13)
C3	0.0335 (10)	0.0376 (10)	0.0371 (10)	0.0093 (8)	0.0117 (8)	0.0123 (8)
C4	0.0565 (14)	0.0401 (11)	0.0394 (11)	0.0083 (10)	0.0135 (10)	0.0083 (9)
C5	0.0493 (12)	0.0368 (10)	0.0445 (12)	0.0116 (9)	0.0088 (9)	0.0150 (9)
C11	0.0345 (10)	0.0487 (11)	0.0353 (10)	0.0117 (9)	0.0116 (8)	0.0147 (9)
C12	0.0371 (11)	0.0611 (14)	0.0386 (11)	0.0083 (10)	0.0095 (9)	0.0115 (10)
C13	0.0408 (12)	0.095 (2)	0.0397 (12)	0.0185 (13)	0.0095 (10)	0.0242 (13)
C14	0.0424 (12)	0.093 (2)	0.0593 (15)	0.0309 (13)	0.0232 (11)	0.0465 (14)
C15	0.0467 (13)	0.0635 (15)	0.0669 (16)	0.0231 (11)	0.0255 (11)	0.0354 (12)
C16	0.0435 (12)	0.0524 (12)	0.0468 (12)	0.0146 (10)	0.0147 (9)	0.0195 (10)
C22	0.0489 (12)	0.0388 (10)	0.0373 (10)	0.0064 (9)	0.0049 (9)	0.0133 (8)
C23	0.0632 (15)	0.0427 (12)	0.0436 (12)	0.0093 (10)	0.0059 (11)	0.0136 (10)
C24	0.0743 (18)	0.0605 (15)	0.0468 (14)	-0.0042 (13)	0.0134 (12)	0.0209 (12)
C25	0.0626 (17)	0.0799 (19)	0.0507 (14)	0.0115 (14)	0.0215 (12)	0.0215 (13)
C26	0.100 (2)	0.0523 (15)	0.085 (2)	0.0327 (16)	0.0071 (18)	0.0184 (15)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C12—C12	1.734 (3)	C14—C15	1.382 (4)
C14—C14	1.725 (3)	C15—C16	1.367 (4)
S21—C22	1.728 (2)	C22—C23	1.355 (4)
S21—C25	1.707 (3)	C23—C24	1.417 (4)
F5—C15	1.352 (3)	C23—C26	1.495 (4)
O1—C1	1.219 (3)	C24—C25	1.349 (4)
N1—N2	1.382 (3)	C2—H2A	0.9600
N1—C1	1.360 (3)	C2—H2B	0.9600
N1—C5	1.476 (3)	C2—H2C	0.9600

N2—C3	1.293 (3)	C4—H4A	0.9700
C1—C2	1.503 (4)	C4—H4B	0.9700
C3—C4	1.511 (3)	C5—H5	0.9800
C3—C11	1.475 (3)	C13—H13	0.9300
C4—C5	1.540 (3)	C16—H16	0.9300
C5—C22	1.517 (3)	C24—H24	0.9300
C11—C12	1.399 (3)	C25—H25	0.9300
C11—C16	1.397 (4)	C26—H26A	0.9600
C12—C13	1.399 (4)	C26—H26B	0.9600
C13—C14	1.365 (5)	C26—H26C	0.9600
C12...C4	3.064 (2)	C11...C15 <sup>ii</sup>	3.391 (4)
C12...S21 <sup>i</sup>	3.6953 (10)	C14...C15 <sup>iii</sup>	3.502 (4)
C14...N1 <sup>ii</sup>	3.488 (2)	C14...C16 <sup>iii</sup>	3.514 (4)
C14...N2 <sup>ii</sup>	3.389 (2)	C15...C14 <sup>iii</sup>	3.502 (4)
C14...C11 <sup>iii</sup>	3.596 (2)	C15...C11 <sup>ii</sup>	3.391 (4)
C14...C16 <sup>iii</sup>	3.524 (3)	C16...C14 <sup>iii</sup>	3.524 (3)
C14...F5	2.917 (2)	C16...C14 <sup>iii</sup>	3.514 (4)
C14...S21 <sup>iii</sup>	3.4250 (13)	C16...C16 <sup>ii</sup>	3.600 (3)
C14...C1 <sup>ii</sup>	3.632 (3)	C22...O1	3.172 (3)
C12...H4A	2.8200	C24...O1 <sup>vi</sup>	3.408 (3)
C12...H4B	2.8400	C5...H26A	2.7500
C12...H4A <sup>i</sup>	3.0200	C24...H13 <sup>i</sup>	3.0000
C12...H4B <sup>iv</sup>	3.0600	C24...H25 <sup>x</sup>	3.0400
S21...N1	3.121 (2)	C24...H26B <sup>vi</sup>	3.0700
S21...C3	3.689 (2)	C25...H25 <sup>x</sup>	3.1000
S21...C12 <sup>i</sup>	3.6953 (10)	C26...H5	2.7600
S21...C14 <sup>iii</sup>	3.4250 (13)	H2A...O1 <sup>viii</sup>	2.5800
S21...H4A	3.1800	H2B...N2	2.4200
F5...C14	2.917 (2)	H4A...C12	2.8200
F5...C4 <sup>v</sup>	3.260 (3)	H4A...S21	3.1800
F5...H4B <sup>v</sup>	2.8200	H4A...C12 <sup>i</sup>	3.0200
O1...C22	3.172 (3)	H4B...C12	2.8400
O1...C24 <sup>vi</sup>	3.408 (3)	H4B...F5 <sup>ix</sup>	2.8200
O1...H5	2.6600	H4B...C12 <sup>iv</sup>	3.0600
O1...H25 <sup>vii</sup>	2.7900	H5...O1	2.6600
O1...H2A <sup>viii</sup>	2.5800	H5...C26	2.7600
O1...H24 <sup>vi</sup>	2.6100	H5...H26A	2.1700
N1...S21	3.121 (2)	H13...C24 <sup>i</sup>	3.0000
N1...C14 <sup>ii</sup>	3.488 (2)	H16...N2	2.4000
N2...C14 <sup>ii</sup>	3.389 (2)	H24...O1 <sup>vi</sup>	2.6100
N2...H2B	2.4200	H25...O1 <sup>xi</sup>	2.7900
N2...H16	2.4000	H25...C24 <sup>x</sup>	3.0400
C1...C14 <sup>ii</sup>	3.632 (3)	H25...C25 <sup>x</sup>	3.1000

## supplementary materials

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C3...S21	3.689 (2)	H26A...C5	2.7500
C4...C12	3.064 (2)	H26A...H5	2.1700
C4...F5 <sup>ix</sup>	3.260 (3)	H26B...C24 <sup>vi</sup>	3.0700
C11...C14 <sup>iii</sup>	3.596 (2)		
C22—S21—C25	91.98 (14)	C22—C23—C26	123.8 (3)
N2—N1—C1	123.08 (19)	C24—C23—C26	124.6 (3)
N2—N1—C5	112.89 (16)	C23—C24—C25	114.3 (3)
C1—N1—C5	123.97 (19)	S21—C25—C24	110.8 (2)
N1—N2—C3	108.80 (17)	C1—C2—H2A	109.00
O1—C1—N1	118.9 (2)	C1—C2—H2B	109.00
O1—C1—C2	123.5 (2)	C1—C2—H2C	109.00
N1—C1—C2	117.6 (2)	H2A—C2—H2B	109.00
N2—C3—C4	113.05 (18)	H2A—C2—H2C	110.00
N2—C3—C11	117.69 (18)	H2B—C2—H2C	109.00
C4—C3—C11	129.25 (19)	C3—C4—H4A	111.00
C3—C4—C5	102.60 (17)	C3—C4—H4B	111.00
N1—C5—C4	101.26 (16)	C5—C4—H4A	111.00
N1—C5—C22	110.98 (18)	C5—C4—H4B	111.00
C4—C5—C22	114.48 (19)	H4A—C4—H4B	109.00
C3—C11—C12	125.8 (2)	N1—C5—H5	110.00
C3—C11—C16	117.75 (19)	C4—C5—H5	110.00
C12—C11—C16	116.5 (2)	C22—C5—H5	110.00
C12—C12—C11	122.69 (19)	C12—C13—H13	120.00
C12—C12—C13	115.80 (19)	C14—C13—H13	120.00
C11—C12—C13	121.5 (2)	C11—C16—H16	119.00
C12—C13—C14	120.5 (2)	C15—C16—H16	119.00
C14—C14—C13	121.7 (2)	C23—C24—H24	123.00
C14—C14—C15	120.0 (3)	C25—C24—H24	123.00
C13—C14—C15	118.3 (3)	S21—C25—H25	125.00
F5—C15—C14	119.0 (3)	C24—C25—H25	125.00
F5—C15—C16	119.0 (2)	C23—C26—H26A	110.00
C14—C15—C16	122.0 (3)	C23—C26—H26B	110.00
C11—C16—C15	121.2 (2)	C23—C26—H26C	109.00
S21—C22—C5	119.58 (18)	H26A—C26—H26B	109.00
S21—C22—C23	111.36 (19)	H26A—C26—H26C	109.00
C5—C22—C23	129.0 (2)	H26B—C26—H26C	109.00
C22—C23—C24	111.6 (2)		
C25—S21—C22—C5	-177.60 (19)	C4—C5—C22—S21	66.8 (2)
C25—S21—C22—C23	0.6 (2)	C4—C5—C22—C23	-111.1 (3)
C22—S21—C25—C24	-0.2 (2)	C3—C11—C12—C12	2.4 (3)
C1—N1—N2—C3	-170.7 (2)	C3—C11—C12—C13	-178.4 (2)
C5—N1—N2—C3	6.6 (3)	C16—C11—C12—C12	-178.11 (18)
N2—N1—C1—O1	174.2 (2)	C16—C11—C12—C13	1.1 (3)
C5—N1—C1—O1	-2.8 (4)	C3—C11—C16—C15	179.5 (2)
N2—N1—C1—C2	-6.6 (4)	C12—C11—C16—C15	-0.1 (4)
C5—N1—C1—C2	176.4 (2)	C12—C12—C13—C14	178.2 (2)
N2—N1—C5—C4	-11.3 (3)	C11—C12—C13—C14	-1.0 (4)
C1—N1—C5—C4	166.0 (2)	C12—C13—C14—C14	178.5 (2)

N2—N1—C5—C22	110.7 (2)	C12—C13—C14—C15	0.0 (4)
C1—N1—C5—C22	-72.1 (3)	C14—C14—C15—F5	2.2 (4)
N1—N2—C3—C11	-179.1 (2)	C14—C14—C15—C16	-177.6 (2)
N1—N2—C3—C4	1.6 (3)	C13—C14—C15—F5	-179.3 (3)
N2—C3—C4—C5	-8.4 (3)	C13—C14—C15—C16	1.0 (4)
C4—C3—C11—C16	-173.1 (2)	F5—C15—C16—C11	179.3 (2)
N2—C3—C11—C12	-172.7 (2)	C14—C15—C16—C11	-1.0 (4)
N2—C3—C11—C16	7.8 (3)	S21—C22—C23—C24	-0.9 (3)
C11—C3—C4—C5	172.4 (2)	S21—C22—C23—C26	176.7 (2)
C4—C3—C11—C12	6.4 (4)	C5—C22—C23—C24	177.1 (2)
C3—C4—C5—N1	10.9 (2)	C5—C22—C23—C26	-5.3 (4)
C3—C4—C5—C22	-108.6 (2)	C22—C23—C24—C25	0.8 (3)
N1—C5—C22—S21	-47.0 (2)	C26—C23—C24—C25	-176.8 (3)
N1—C5—C22—C23	135.1 (2)	C23—C24—C25—S21	-0.3 (3)

Symmetry codes: (i)  $-x+1, -y+2, -z+1$ ; (ii)  $-x, -y+1, -z+1$ ; (iii)  $-x+1, -y+1, -z+1$ ; (iv)  $-x, -y+2, -z+1$ ; (v)  $x, y-1, z$ ; (vi)  $-x, -y+2, -z$ ; (vii)  $x-1, y, z$ ; (viii)  $-x-1, -y+1, -z$ ; (ix)  $x, y+1, z$ ; (x)  $-x+1, -y+2, -z$ ; (xi)  $x+1, y, z$ .

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2A $\cdots$ O1 <sup>viii</sup>	0.96	2.58	3.533 (4)	171

Symmetry codes: (viii)  $-x-1, -y+1, -z$ .

Fig. 1

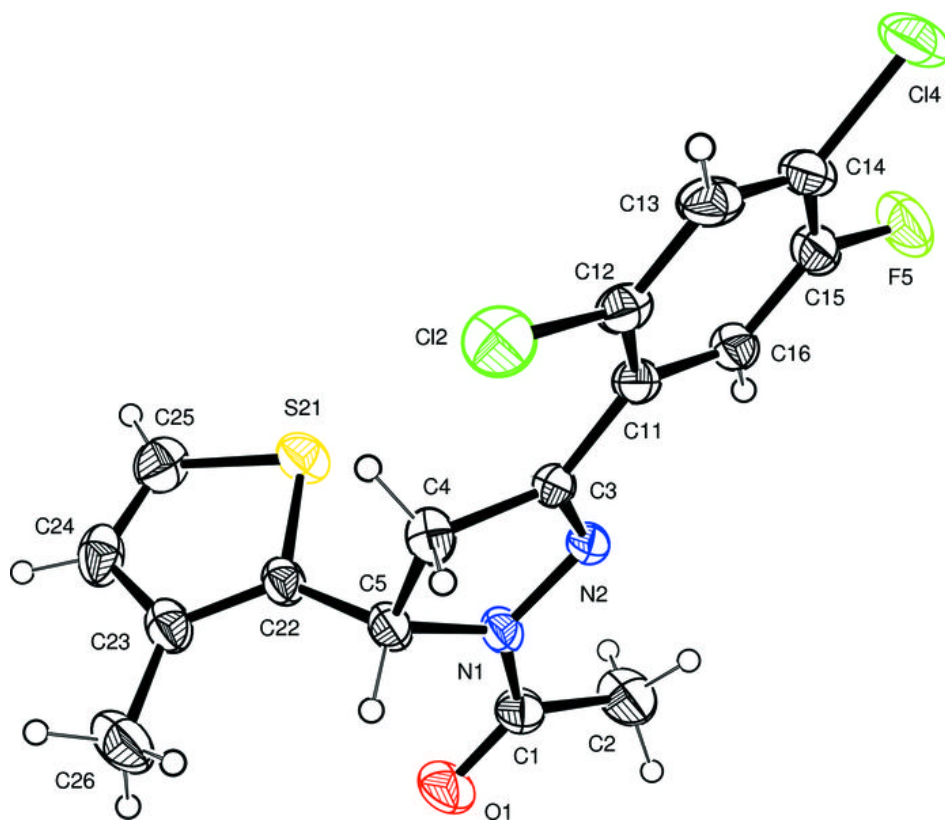


Fig. 2

