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1-(4-Chloro-3-fluorophenyl)-2-[(3-phenylisoquinolin-1-yl)sulfanyl]ethanone

P. Manivel,^a Venkatesha R. Hathwar,^b T. Maiyalagan,^a
N. Burcu Arslan^c and F. Nawaz Khan^{a*}

^aChemistry Division, School of Science and Humanities, VIT University, Vellore 632 014, Tamil Nadu, India, ^bSolid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and ^cOndokuz Mayıs University, Arts and Sciences Faculty, Department of Physics, 55139-Samsun, Turkey

Correspondence e-mail: nawaz_f@yahoo.co.in

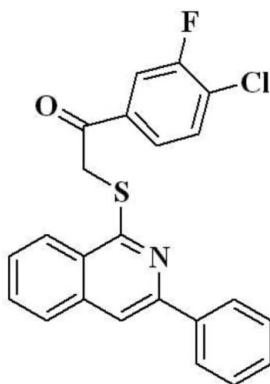
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Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.056; wR factor = 0.128; data-to-parameter ratio = 14.2.

In the title compound, $\text{C}_{23}\text{H}_{15}\text{ClFNO}$, the isoquinoline system and the 4-chloro-3-fluorophenyl ring are aligned at $80.4(1)^\circ$. The dihedral angle between the isoquinoline system and the pendant (unsubstituted) phenyl ring is $19.91(1)^\circ$.

Related literature

For related structures, see: Hathwar *et al.* (2008); Manivel *et al.* (2009a,b).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{15}\text{ClFNO}$
 $M_r = 407.87$
Orthorhombic, $Pbca$
 $a = 16.9008(11)$ Å
 $b = 9.8036(7)$ Å
 $c = 23.3226(16)$ Å
 $V = 3864.3(5)$ Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.33$ mm⁻¹
 $T = 290(2)$ K
 $0.24 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.925$, $T_{\max} = 0.965$
27428 measured reflections
3595 independent reflections
2424 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.128$
 $S = 1.04$
3595 reflections
253 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.32$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); 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: NG2534).

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supplementary materials

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Comment

In compound (I), the S atom also located in the plane. The F atom deviates by 0.014 Å from mean plane of phenyl ring containing F and Cl atoms. In this ring F—C and Cl—C bond distances are 1.348 (4) Å, 1.727 (3) Å, respectively. The orientation of isoquinoline ring system with respect to the another phenyl ring is given by the torsion angles for N1—C2—C10—C15 and C3—C2—C10—C11 are respectively -160.1 (2)°, -163.1 (3)° similarly for C16—S1—C1—N1 and C16—S1—C1—C8 are respectively -0.8 (2)° and 179.56 (19)° (Table 1).

Experimental

3-Phenylisoquinoline-1-thiol and 2-bromo-1-(3-fluoro-4-chlorophenyl)ethanone were mixed in the ratio 1:1.05 equivalents with ethanol in a round bottom flask. Then it was heated under nitrogen atmosphere on an oil bath at 323 K. After 2 h, the products were filtered and dissolved in chloroform. Further, it was washed with water, dried and concentrated. The single-crystal for X-ray structure analysis was obtained from ether solution by slow evaporation.

Refinement

All the H atoms in (I) were positioned geometrically and refined using a riding model with C—H bond lengths of 0.93 Å and 0.97 Å for aromatic and for methylene H atoms respectively and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for all carbon bound H atoms.

Figures

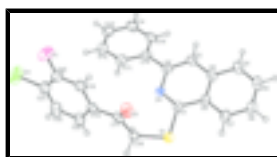


Fig. 1. ORTEP diagram of the asymmetric unit of (I) with 50% probability displacement ellipsoids.

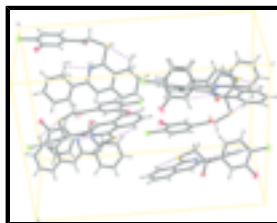


Fig. 2. The crystal packing diagram of (I). The dotted lines indicate intermolecular C—H...O hydrogen bonds. All H atoms have been omitted for clarity.

1-(4-Chloro-3-fluorophenyl)-2-[(3-phenylisoquinolin-1-yl)sulfanyl]ethanone

Crystal data

C₂₃H₁₅ClFNO_S

$F_{000} = 1680$

supplementary materials

$M_r = 407.87$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 16.9008$ (11) Å

$b = 9.8036$ (7) Å

$c = 23.3226$ (16) Å

$V = 3864.3$ (5) Å³

$Z = 8$

$D_x = 1.402$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3595 reflections

$\theta = 1.8$ – 25.5°

$\mu = 0.33$ mm⁻¹

$T = 290$ (2) K

Block, colourless

$0.24 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.925$, $T_{\max} = 0.965$

27428 measured reflections

3595 independent reflections

2424 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

$\theta_{\max} = 25.5^\circ$

$\theta_{\min} = 1.8^\circ$

$h = -18 \rightarrow 20$

$k = -11 \rightarrow 11$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.128$

$S = 1.04$

3595 reflections

253 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 1.1665P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.32$ e Å⁻³

$\Delta\rho_{\min} = -0.19$ e Å⁻³

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

x

y

z

$U_{\text{iso}}^*/U_{\text{eq}}$

O1	0.07142 (12)	0.8183 (2)	0.28005 (9)	0.0633 (6)
F1	0.09785 (13)	0.8393 (2)	0.06905 (9)	0.1057 (7)
S1	0.03138 (4)	0.61721 (7)	0.37120 (3)	0.0495 (2)
Cl1	-0.02952 (8)	0.70329 (14)	0.00961 (4)	0.1221 (5)
N1	0.12270 (12)	0.4853 (2)	0.29715 (9)	0.0399 (5)
C1	0.11497 (15)	0.5201 (2)	0.35092 (11)	0.0393 (6)
C2	0.18572 (14)	0.4054 (2)	0.28104 (11)	0.0405 (6)
C3	0.24276 (16)	0.3684 (3)	0.31906 (11)	0.0474 (7)
H3	0.2861	0.3180	0.3066	0.057*
C4	0.29398 (18)	0.3701 (3)	0.41884 (13)	0.0593 (8)
H4	0.3384	0.3205	0.4079	0.071*
C5	0.2845 (2)	0.4076 (3)	0.47468 (14)	0.0688 (9)
H5	0.3225	0.3827	0.5016	0.083*
C6	0.2188 (2)	0.4828 (3)	0.49197 (13)	0.0643 (9)
H6	0.2135	0.5085	0.5302	0.077*
C7	0.16226 (18)	0.5189 (3)	0.45299 (12)	0.0538 (7)
H7	0.1180	0.5676	0.4650	0.065*
C8	0.17027 (16)	0.4832 (2)	0.39492 (11)	0.0422 (6)
C9	0.23657 (16)	0.4063 (3)	0.37753 (11)	0.0451 (6)
C10	0.18550 (14)	0.3638 (2)	0.21970 (11)	0.0419 (6)
C11	0.13897 (17)	0.4309 (3)	0.18011 (12)	0.0515 (7)
H11	0.1085	0.5046	0.1921	0.062*
C12	0.13641 (19)	0.3920 (3)	0.12340 (12)	0.0597 (8)
H12	0.1046	0.4392	0.0976	0.072*
C13	0.18110 (19)	0.2828 (3)	0.10499 (13)	0.0641 (9)
H13	0.1805	0.2569	0.0666	0.077*
C14	0.22641 (19)	0.2130 (4)	0.14375 (14)	0.0715 (10)
H14	0.2556	0.1379	0.1317	0.086*
C15	0.22938 (17)	0.2524 (3)	0.20044 (13)	0.0598 (8)
H15	0.2609	0.2042	0.2261	0.072*
C16	-0.01524 (15)	0.6351 (3)	0.30300 (11)	0.0439 (6)
H16A	-0.0177	0.5461	0.2849	0.053*
H16B	-0.0692	0.6659	0.3089	0.053*
C17	0.02538 (15)	0.7327 (2)	0.26264 (12)	0.0426 (6)
C18	0.00791 (15)	0.7226 (2)	0.20013 (12)	0.0422 (6)
C19	0.05916 (17)	0.7874 (3)	0.16253 (13)	0.0520 (7)
H19	0.1020	0.8366	0.1766	0.062*
C20	0.0465 (2)	0.7788 (3)	0.10523 (15)	0.0647 (9)
C21	-0.0164 (2)	0.7098 (4)	0.08298 (14)	0.0679 (9)
C22	-0.0680 (2)	0.6464 (3)	0.11953 (15)	0.0714 (9)
H22	-0.1116	0.6000	0.1049	0.086*
C23	-0.05585 (18)	0.6510 (3)	0.17826 (13)	0.0572 (8)
H23	-0.0904	0.6062	0.2029	0.069*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0674 (14)	0.0532 (12)	0.0694 (14)	-0.0142 (11)	-0.0162 (11)	-0.0004 (10)

supplementary materials

F1	0.1066 (17)	0.1363 (19)	0.0742 (14)	-0.0033 (15)	0.0267 (12)	0.0272 (13)
S1	0.0554 (5)	0.0517 (4)	0.0412 (4)	0.0142 (3)	0.0010 (3)	-0.0073 (3)
C11	0.1660 (12)	0.1504 (11)	0.0500 (6)	0.0166 (9)	-0.0195 (6)	0.0006 (6)
N1	0.0408 (13)	0.0394 (12)	0.0396 (12)	0.0018 (10)	0.0016 (10)	-0.0030 (9)
C1	0.0454 (16)	0.0322 (13)	0.0404 (15)	-0.0012 (11)	0.0021 (12)	-0.0003 (11)
C2	0.0372 (15)	0.0380 (14)	0.0462 (15)	-0.0010 (11)	0.0047 (12)	-0.0020 (12)
C3	0.0381 (15)	0.0473 (15)	0.0570 (17)	0.0062 (12)	0.0002 (14)	-0.0042 (14)
C4	0.0579 (19)	0.0559 (18)	0.064 (2)	0.0096 (15)	-0.0138 (16)	0.0034 (16)
C5	0.081 (2)	0.068 (2)	0.058 (2)	0.0080 (19)	-0.0261 (18)	0.0111 (17)
C6	0.090 (2)	0.0590 (19)	0.0443 (17)	0.0110 (18)	-0.0115 (17)	0.0026 (14)
C7	0.070 (2)	0.0462 (16)	0.0448 (17)	0.0078 (15)	-0.0052 (14)	0.0015 (13)
C8	0.0501 (16)	0.0346 (13)	0.0419 (15)	-0.0011 (12)	-0.0037 (12)	0.0025 (11)
C9	0.0481 (16)	0.0368 (14)	0.0505 (16)	-0.0013 (12)	-0.0068 (13)	0.0021 (12)
C10	0.0368 (15)	0.0425 (14)	0.0464 (16)	-0.0034 (12)	0.0067 (12)	-0.0055 (12)
C11	0.0665 (19)	0.0400 (15)	0.0479 (17)	0.0057 (14)	0.0020 (15)	-0.0024 (13)
C12	0.077 (2)	0.0564 (17)	0.0457 (17)	0.0053 (16)	-0.0019 (15)	0.0004 (14)
C13	0.065 (2)	0.081 (2)	0.0468 (18)	0.0032 (18)	0.0075 (16)	-0.0157 (16)
C14	0.058 (2)	0.091 (3)	0.065 (2)	0.0256 (19)	0.0001 (17)	-0.0299 (19)
C15	0.0466 (18)	0.074 (2)	0.0585 (19)	0.0217 (16)	-0.0017 (14)	-0.0164 (16)
C16	0.0429 (16)	0.0415 (15)	0.0472 (16)	0.0076 (12)	0.0010 (12)	-0.0031 (12)
C17	0.0383 (15)	0.0353 (14)	0.0543 (17)	0.0055 (12)	-0.0039 (13)	-0.0023 (12)
C18	0.0410 (15)	0.0341 (13)	0.0514 (17)	0.0041 (12)	-0.0020 (13)	0.0016 (12)
C19	0.0485 (17)	0.0474 (17)	0.060 (2)	0.0057 (13)	0.0024 (14)	0.0034 (14)
C20	0.069 (2)	0.069 (2)	0.056 (2)	0.0130 (18)	0.0129 (18)	0.0134 (16)
C21	0.087 (3)	0.072 (2)	0.0453 (18)	0.018 (2)	-0.0032 (18)	0.0037 (16)
C22	0.081 (2)	0.070 (2)	0.064 (2)	-0.0032 (18)	-0.0258 (19)	-0.0049 (17)
C23	0.0612 (19)	0.0512 (17)	0.059 (2)	-0.0033 (14)	-0.0071 (16)	0.0041 (14)

Geometric parameters (Å, °)

O1—C17	1.215 (3)	C10—C15	1.395 (3)
F1—C20	1.348 (4)	C11—C12	1.377 (4)
S1—C1	1.768 (3)	C11—H11	0.9300
S1—C16	1.784 (3)	C12—C13	1.378 (4)
C11—C21	1.727 (3)	C12—H12	0.9300
N1—C1	1.306 (3)	C13—C14	1.368 (4)
N1—C2	1.375 (3)	C13—H13	0.9300
C1—C8	1.434 (3)	C14—C15	1.378 (4)
C2—C3	1.359 (3)	C14—H14	0.9300
C2—C10	1.487 (3)	C15—H15	0.9300
C3—C9	1.417 (3)	C16—C17	1.508 (4)
C3—H3	0.9300	C16—H16A	0.9700
C4—C5	1.363 (4)	C16—H16B	0.9700
C4—C9	1.413 (4)	C17—C18	1.491 (4)
C4—H4	0.9300	C18—C23	1.383 (4)
C5—C6	1.392 (4)	C18—C19	1.387 (4)
C5—H5	0.9300	C19—C20	1.356 (4)
C6—C7	1.366 (4)	C19—H19	0.9300
C6—H6	0.9300	C20—C21	1.363 (5)

C7—C8	1.405 (4)	C21—C22	1.369 (5)
C7—H7	0.9300	C22—C23	1.386 (4)
C8—C9	1.410 (3)	C22—H22	0.9300
C10—C11	1.380 (4)	C23—H23	0.9300
C1—S1—C16	99.63 (12)	C13—C12—H12	120.1
C1—N1—C2	119.3 (2)	C14—C13—C12	119.3 (3)
N1—C1—C8	123.8 (2)	C14—C13—H13	120.3
N1—C1—S1	118.51 (19)	C12—C13—H13	120.3
C8—C1—S1	117.71 (19)	C13—C14—C15	120.9 (3)
C3—C2—N1	121.5 (2)	C13—C14—H14	119.5
C3—C2—C10	123.8 (2)	C15—C14—H14	119.5
N1—C2—C10	114.7 (2)	C14—C15—C10	120.6 (3)
C2—C3—C9	120.4 (2)	C14—C15—H15	119.7
C2—C3—H3	119.8	C10—C15—H15	119.7
C9—C3—H3	119.8	C17—C16—S1	114.70 (19)
C5—C4—C9	120.2 (3)	C17—C16—H16A	108.6
C5—C4—H4	119.9	S1—C16—H16A	108.6
C9—C4—H4	119.9	C17—C16—H16B	108.6
C4—C5—C6	120.9 (3)	S1—C16—H16B	108.6
C4—C5—H5	119.6	H16A—C16—H16B	107.6
C6—C5—H5	119.6	O1—C17—C18	120.0 (2)
C7—C6—C5	120.2 (3)	O1—C17—C16	121.5 (3)
C7—C6—H6	119.9	C18—C17—C16	118.6 (2)
C5—C6—H6	119.9	C23—C18—C19	119.1 (3)
C6—C7—C8	120.6 (3)	C23—C18—C17	123.3 (3)
C6—C7—H7	119.7	C19—C18—C17	117.7 (2)
C8—C7—H7	119.7	C20—C19—C18	119.7 (3)
C7—C8—C9	119.2 (2)	C20—C19—H19	120.1
C7—C8—C1	124.3 (2)	C18—C19—H19	120.1
C9—C8—C1	116.5 (2)	F1—C20—C19	119.2 (3)
C8—C9—C4	118.9 (3)	F1—C20—C21	118.8 (3)
C8—C9—C3	118.4 (2)	C19—C20—C21	122.0 (3)
C4—C9—C3	122.7 (3)	C20—C21—C22	119.0 (3)
C11—C10—C15	117.4 (2)	C20—C21—C11	119.7 (3)
C11—C10—C2	120.9 (2)	C22—C21—C11	121.2 (3)
C15—C10—C2	121.6 (2)	C21—C22—C23	120.4 (3)
C12—C11—C10	121.9 (3)	C21—C22—H22	119.8
C12—C11—H11	119.0	C23—C22—H22	119.8
C10—C11—H11	119.0	C18—C23—C22	119.8 (3)
C11—C12—C13	119.8 (3)	C18—C23—H23	120.1
C11—C12—H12	120.1	C22—C23—H23	120.1
C2—N1—C1—C8	2.0 (4)	C15—C10—C11—C12	-1.1 (4)
C2—N1—C1—S1	-177.53 (17)	C2—C10—C11—C12	-178.4 (3)
C16—S1—C1—N1	-0.8 (2)	C10—C11—C12—C13	0.2 (4)
C16—S1—C1—C8	179.56 (19)	C11—C12—C13—C14	1.2 (5)
C1—N1—C2—C3	-4.0 (4)	C12—C13—C14—C15	-1.6 (5)
C1—N1—C2—C10	175.8 (2)	C13—C14—C15—C10	0.6 (5)
N1—C2—C3—C9	3.1 (4)	C11—C10—C15—C14	0.7 (4)

supplementary materials

C10—C2—C3—C9	-176.7 (2)	C2—C10—C15—C14	178.0 (3)
C9—C4—C5—C6	-0.5 (5)	C1—S1—C16—C17	-73.20 (19)
C4—C5—C6—C7	0.7 (5)	S1—C16—C17—O1	-19.3 (3)
C5—C6—C7—C8	-1.2 (4)	S1—C16—C17—C18	160.73 (18)
C6—C7—C8—C9	1.6 (4)	O1—C17—C18—C23	-164.7 (3)
C6—C7—C8—C1	-178.2 (3)	C16—C17—C18—C23	15.2 (4)
N1—C1—C8—C7	-179.4 (2)	O1—C17—C18—C19	16.0 (4)
S1—C1—C8—C7	0.1 (3)	C16—C17—C18—C19	-164.1 (2)
N1—C1—C8—C9	0.7 (4)	C23—C18—C19—C20	-0.6 (4)
S1—C1—C8—C9	-179.71 (18)	C17—C18—C19—C20	178.7 (2)
C7—C8—C9—C4	-1.5 (4)	C18—C19—C20—F1	-178.3 (2)
C1—C8—C9—C4	178.4 (2)	C18—C19—C20—C21	1.2 (5)
C7—C8—C9—C3	178.5 (2)	F1—C20—C21—C22	179.0 (3)
C1—C8—C9—C3	-1.6 (3)	C19—C20—C21—C22	-0.5 (5)
C5—C4—C9—C8	0.9 (4)	F1—C20—C21—C11	-0.8 (4)
C5—C4—C9—C3	-179.1 (3)	C19—C20—C21—C11	179.7 (2)
C2—C3—C9—C8	-0.2 (4)	C20—C21—C22—C23	-0.8 (5)
C2—C3—C9—C4	179.8 (3)	C11—C21—C22—C23	179.0 (2)
C3—C2—C10—C11	-163.1 (3)	C19—C18—C23—C22	-0.7 (4)
N1—C2—C10—C11	17.1 (3)	C17—C18—C23—C22	-180.0 (3)
C3—C2—C10—C15	19.7 (4)	C21—C22—C23—C18	1.4 (5)
N1—C2—C10—C15	-160.1 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C7—H7 \cdots S1	0.93	2.68	3.076 (3)	107
C11—H11 \cdots N1	0.93	2.47	2.795 (4)	101

Fig. 1

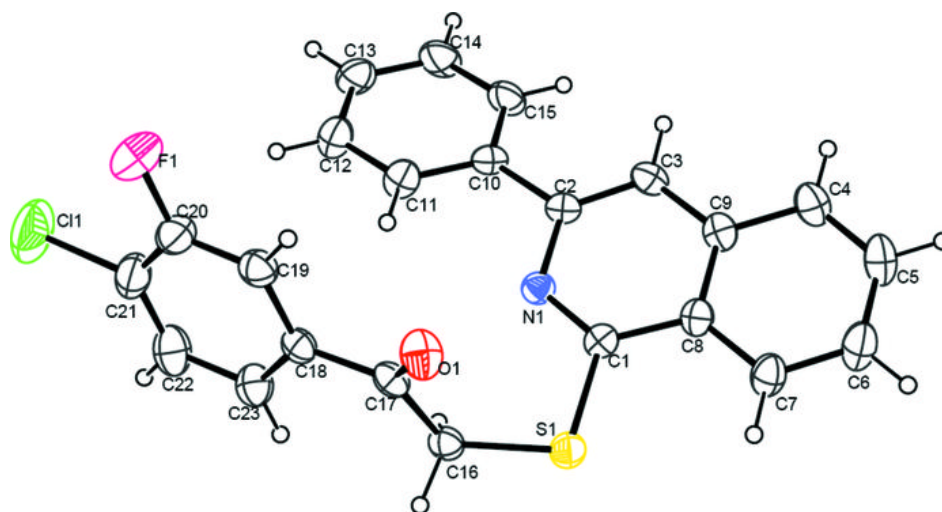


Fig. 2

