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Synthesis and Crystal Structure Analysis of 2-(Fluorobenzyl)-6-(4-Nitrophenyl) Imidazo[2,1-b][1,3,4]Thiadiazole

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Synthesis and Crystal Structure Analysis of 2-(Fluorobenzyl)-6-(4-Nitrophenyl) Imidazo[2,1-b][1,3,4]Thiadiazole

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Preparation of 2-(4-fluorobenzyl)-6-(4-nitrophenyl)imidazo[2,1-b][1,3,4]thiadiazole is described and its crystal structure is discussed. The compound crystallizes in the monoclinic space group C2/c with $a = 39.941(6)$ Å, $b = 5.698(2)$ Å, $c = 13.272(5)$ Å, $\beta = 90.880^\circ$, $V = 3020(2)$ Å³, $z = 8$. The crystal structure is stabilized by weak intermolecular C-H...N, C-H...O, C-H...S, and C-H...F interactions.

Keywords C-H...N, C-H...O, C-H...S, and C-H...F; crystal structure; imidazo[2,1-b] 1,3,4-thiadiazoles derivative; weak interactions

Introduction

1,3,4-Thiadiazoles are known for their promising biological and pharmacological activities, possibly due to the presence of a pharmacophoric isothioamide (S–C=N–) unit [1,2] in the thiadiazole nucleus. Secondly, the thiadiazole ring is bioisosteric with the thiazole moiety of the novel broad-spectrum anthelmintic tetramisole [3,4]. Some thiadiazole derivatives are reported to possess anticancer properties [5] and hence they have potential pharmaceutical prospects.

Many imidazothiadiazole derivatives have been reported to possess diverse medicinal properties such as anthelmintic [6], antimicrobial [7], and anti-inflammatory, antipyretic, analgesic [8], and many other activities of therapeutic significance [9,10]. Moreover, the presence of fluoro substituent in the molecule provides compounds with enhanced biological activity. Accumulation of fluorine [11] on carbon leads to increased oxidative and thermal stability. Further, it leads to increased lipid solubility, thereby enhancing the rate of absorption and transport of drug *in vivo*. These findings prompted us to synthesize the title compound so that it could be screened for its pharmacological activities and to carry out its crystallographic structure elucidation. The title compound will be a good intermediate to synthesize various candidate compounds for pharmacological testing.

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Synthesis and Characterization

The title compound was prepared as shown in Scheme 1. The reaction of 2-amino-(4-fluorobenzyl)-1,3,4-thiadiazole [12] **1** and nitro phenacyl bromide (**2**) in boiling ethanol afforded the 2-amino-(4-fluorobenzyl)-6-(4-nitrophenyl)-imidazo[2,1-*b*][1,3,4]thiadiazole (**3**) as hydrobromide salt, which was neutralized by sodium carbonate solution to get the free base. It is well established that this reaction proceeds via the intermediate iminothiadiazole, which under reflux temperature spontaneously undergoes dehydrocyclization to form the desired fused heterocycle [13].

Experimental

2-(4-Fluorobenzyl)-6-arylimidazo[2,1-*b*][1,3,4]thiadiazole

A mixture of 2-amino-(4-fluorobenzyl)-1,3,4-thiadiazole (**1**) (2.69, 0.013 mol) and nitro phenacyl bromide (**2**) (0.01 mol) of equimolar quantities was refluxed in dry ethanol for 18 h. The excess of solvent was distilled off and by filtration the solid hydrobromide salt that separated was collected, suspended in water, and neutralized by aqueous sodium carbonate solution to get free base (**3**). It was filtered, washed with water, dried, and recrystallized from ethanol.

Physical Measurements

Melting points were determined in open capillaries. Infrared (IR) spectra were recorded on a Nicolet Fourier transform infrared spectrophotometer (University Science Instrument Centre, Karnatak University Dharwad, Karnataka), ^1H nuclear magnetic resonance (NMR) spectra were recorded on Varian RX-300 MHz spectrometer using trimethyl siloxane (TMS) as internal standard.

The structure of imidazothiadiazole derivative **3** was established by the absence of a $\nu_{\text{N-H}}$ band in the IR spectra and the appearance of imidazole proton ($\text{C}_5\text{-H}$) around δ 8 in the ^1H NMR spectra. The ^{13}C NMR and mass spectra of this compound further confirmed the assigned structure.

2-(4-Fluorobenzyl)-6-(4-nitrophenyl)imidazo[2,1-*b*][1,3,4]thiadiazole (**3**). Yellow crystalline solid (ethanol+diagon), yield 64% m.p. 212–214°C; UV-Vis $^{-1}$ (nm): 316. IR (KBr) ν_{cm} : 3,107, 2,924, 2,854, 1,603, 1,524, 1,501; ^1H NMR (300 MHz, CDCl_3) δ : 4.31 (s, 2H, CH_2), 7.07–7.39 (m, 4H, Ar-H), 7.83 (d, $J=8.4$ Hz, 2H, Ar-H), 7.91 (d, $J=8.4$ Hz, 2H, Ar-H), 8.12 (s, 1H, C-H, imidazole). ^{13}C NMR (75 MHz, CDCl_3) δ : 37.8, 109.6, 116.3, 121.7, 127.0, 30.9, 131.0, 132.2, 145.4, 146.2, 161.1, 164.1. Anal. calcd. for $\text{C}_{17}\text{H}_{11}\text{FN}_4\text{O}_2\text{S}$: C, 57.62; H, 3.10; N, 15.81; Found: C, 57.48; H, 3.08; N, 15.78% MS (m/z): 354.

X-Ray Analysis

The X-ray diffraction data for compound **3** were collected on a Bruker Smart CCD Area Detector System (I.I.Sc, Bangalore), using $\text{MoK}\alpha$ (0.71073 Å) radiation for the crystal. Intensity data were collected up to a maximum of 28.58° in the ω - Φ scan mode. The data were reduced using SAINTPLUS [14]. The structure was solved by direct methods using SHELXS97 [15] and difference Fourier synthesis using SHELXL97 [16]. The positions and anisotropic displacement parameters of all nonhydrogen atoms were included in the full-matrix least-squares

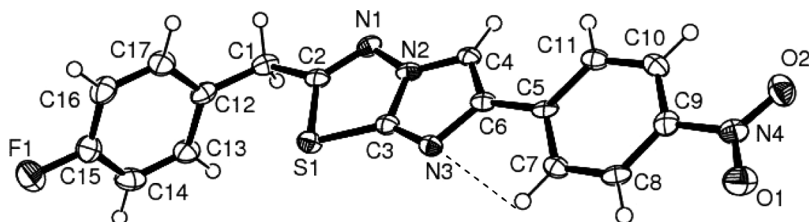


Figure 1. ORTEP diagram of compound **3** showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dotted line indicates intramolecular C7-H7...N3 interaction.

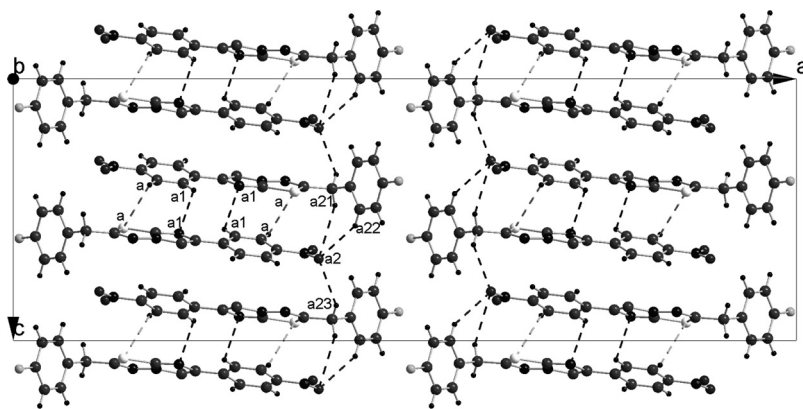


Figure 2. Packing of the molecules in crystal of **3** viewed along the *b* axis. Dotted line indicates C-H...S (*a*-*a*), and C-H...N (*a*1-*a*1), and C-H...O (*a*2-*a*21, *a*22, *a*23) intermolecular interactions.

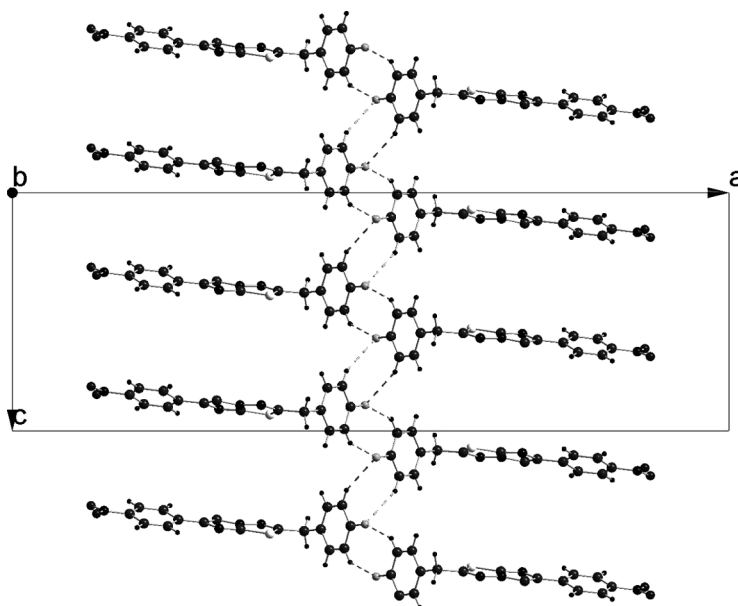


Figure 3. Packing of the molecules of **3** with dotted lines indicating C-H...F intermolecular interactions generating zig-zag tapes along the *b* axis.

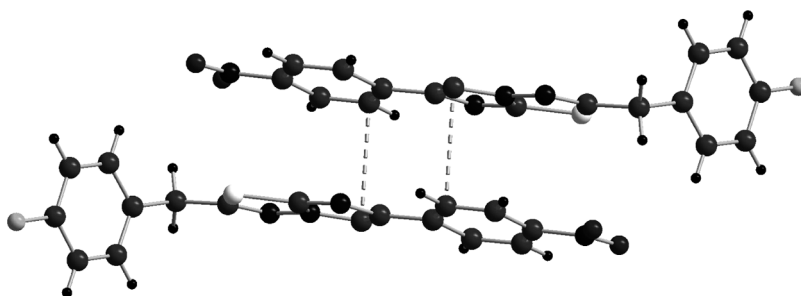


Figure 4. View of the molecular packing in **3** showing π - π stacking interactions between imidazothiadiazole and nitro benzene rings.

refinement using SHELXL97 [16] and the procedure was carried out for a few cycles until convergence was reached. A total of 9,096 reflections was collected, resulting in 3,701 [$R(\text{int})=0.0847$] independent reflections, of which the number of reflections satisfying $I > 2\sigma(I)$ criteria was 2,733.

Table 1. Crystal data and structure refinement

CCDC deposit no.	763068
Empirical formula	C17 H11 F N4 O2 S
Formula weight	354.36
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	
a (Å)	39.941(6)
b (Å)	5.698(2)
c (Å)	13.272(5)
β (°)	90.880
Volume (Å ³)	3,020(2)
Z	8
Calculated density (Mg/m ³)	1.559
Absorption coefficient (mm ⁻¹)	0.246
F(000)	1,456
Crystal size	0.4 mm × 0.35 mm × 0.3 mm
Theta range for data collection	2.04–28.58°
Limiting indices	$-52 \leq h \leq 45$, $7 \leq k \leq 6$, $17 \leq l \leq 17$
Reflection collected / unique	9,096/3,701 [$R(\text{int})=0.0847$]
Completeness of theta	28.58 95.8%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3,701/0/230
Goodness-of-fit on F^2	1.108
Final R indices [$I > 2 \sigma(I)$]	$R_1 = 0.0742$, $wR_2 = 0.1817$
R indices (all data)	$R_1 = 0.1053$, $wR_2 = 0.2283$
Largest diff. peak and hole (e Å ⁻³)	0.874 and -0.854

These were treated as observed. The H atoms were placed at calculated positions in the riding model approximation (C—H 0.93 Å); their temperature factors were set to 1.2 times those of the equivalent isotropic temperature factors of the parent atoms. All other non-H atoms were refined anisotropically. The R factor for observed data finally converged to $R = 0.0742$ with $wR_2 = 0.1990$ in the compound. The maximum and minimum values of residual electron density were 0.874 and -0.854 eÅ^{-3} . Molecular diagrams were generated using ORTEP [17]. The mean plane calculation was done using the program PARST [18].

Results and Discussion

Figure 1 shows the ORTEP diagram of the molecule with thermal ellipsoids drawn at 50% probability of the title compound. Figures 2, 3, and 4 show hydrogen-bond interactions and crystal packing of the compound. The details of crystal data and refinements are given in Table 1. Table 2 gives the list of atomic coordinates and equivalent isotropic displacement parameters of the nonhydrogen atoms. Table 3 gives the list of anisotropic displacement parameters of the nonhydrogen atoms.

Table 2. Atomic coordinate ($\times 10^4$) and equivalent isotropic displacement parameter ($\times 10^3$) nonhydrogen atoms

Atoms	x	Y	z	U(eq)
C(1)	929(1)	8,704(6)	810(2)	25(1)
C(2)	1,291(1)	7,996(6)	887(2)	21(1)
C(3)	1,818(1)	5,890(6)	882(2)	20(1)
C(4)	2,148(1)	8,836(6)	1,284(2)	19(1)
C(5)	2,685(1)	6,405(6)	1,286(2)	18(1)
C(6)	2,325(1)	6,796(6)	1,172(2)	20(1)
C(7)	2,830(1)	4,249(6)	1,013(2)	21(1)
C(8)	3,165(1)	3,849(6)	1,132(2)	22(1)
C(9)	3,363(1)	5,627(6)	1,529(2)	22(1)
C(10)	3,232(1)	7,779(6)	1,800(2)	23(1)
C(11)	2,893(1)	8,154(6)	1,685(2)	23(1)
C(12)	696(1)	6,639(6)	885(2)	26(1)
C(13)	575(1)	5,541(7)	16(3)	29(1)
C(14)	369(1)	3,612(7)	79(3)	33(1)
C(15)	282(1)	2,771(7)	1020(3)	31(1)
C(16)	399(1)	3,791(8)	1894(3)	33(1)
C(17)	604(1)	5,714(7)	1814(3)	32(1)
O(1)	3,824(1)	3,180(5)	1541(2)	35(1)
O(2)	3,901(1)	6,846(5)	1875(2)	33(1)
N(1)	1,528(1)	9,466(5)	1094(2)	23(1)
N(2)	1,825(1)	8,245(5)	1094(2)	20(1)
N(3)	2,115(1)	4,936(5)	916(2)	19(1)
N(4)	3,720(1)	5,183(5)	1662(2)	24(1)
S(1)	1,406(1)	5,057(2)	691(1)	24(1)
F(1)	77(1)	901(5)	1096(2)	43(1)

Table 3. Anisotropic displacement parameters ($\times 10^4$) of non hydrogen atoms

Atom	U11	U22	U33	U23	U13	U12
C(1)	28(2)	28(2)	20(2)	−2(1)	−1(1)	9(1)
C(2)	30(2)	22(2)	11(1)	0(1)	−1(1)	1(1)
C(3)	28(2)	20(2)	13(1)	1(1)	2(1)	−1(1)
C(4)	28(2)	19(2)	10(1)	0(1)	0(1)	−3(1)
C(5)	29(2)	21(2)	4(1)	1(1)	0(1)	2(1)
C(6)	27(2)	23(2)	9(1)	0(1)	−2(1)	1(1)
C(7)	27(2)	21(2)	16(1)	0(1)	−3(1)	−2(1)
C(8)	33(2)	22(2)	11(1)	0(1)	1(1)	5(1)
C(9)	25(2)	28(2)	14(1)	4(1)	1(1)	−1(1)
C(10)	31(2)	23(2)	14(1)	2(1)	−3(1)	−7(1)
C(11)	33(2)	20(2)	15(1)	−1(1)	0(1)	0(1)
C(12)	25(2)	33(2)	18(2)	0(1)	0(1)	7(1)
C(13)	30(2)	39(2)	18(2)	−3(1)	−4(1)	1(2)
C(14)	35(2)	38(2)	25(2)	−9(2)	−8(2)	6(2)
C(15)	22(2)	34(2)	36(2)	−2(2)	−2(1)	3(1)
C(16)	31(2)	47(2)	20(2)	4(2)	1(1)	−1(2)
C(17)	29(2)	46(2)	20(2)	−4(2)	−5(1)	−2(2)
O(1)	35(1)	36(1)	33(1)	−6(1)	−7(1)	10(1)
O(2)	28(1)	39(2)	31(1)	1(1)	−5(1)	−5(1)
N(1)	28(1)	26(2)	15(1)	1(1)	2(1)	3(1)
N(2)	29(1)	16(1)	14(1)	1(1)	0(1)	4(1)
N(3)	24(1)	18(1)	16(1)	1(1)	−1(1)	−1(1)
N(4)	31(1)	30(2)	12(1)	1(1)	−2(1)	1(1)
S(1)	25(1)	22(1)	25(1)	−3(1)	−2(1)	1(1)
F(1)	41(1)	40(2)	47(1)	−3(1)	−2(1)	−9(1)

The selected bond lengths and bond angles of all the nonhydrogen atoms are given in Tables 4 and 5. Table 6 gives selected torsion angles. Table 7 shows the respective hydrogen bond interactions for compound 3. In the molecule, the dihedral angle between fluorobenzyl and imidazothiadiazole is $89.26(5)^\circ$, which is Almost orthogonal. The nitrophenyl and imidazothiadiazole are planar with only $10.02(7)^\circ$ between them and the NO_2 group is cis to imidazothiadiazole and aryl ring. Specifically, in nitrobenzene, the nitro group is essentially coplanar with the adjacent aryl ring because the molecule is highly polar, having significant negative charge on the O atoms, with minimum energy conformation [20]. The mesomeric interaction between the nitro group and the aryl ring in nitrobenzene is rather small, so that the C-N bond is effectively a single bond with a correspondingly small barrier to rotation. Hence, the intermolecular interaction could be quite modest in solid state, if the Nitro-aryl moiety perturbs its planar conformation. The C-N bond distance is $1.461(5) \text{ \AA}$ in nitrobenzene. This value is typical of C(aryl)- NO_2 distances [19], where the mean value is 1.468 \AA . Within the nitrated aryl rings, the C-C-C angle shows significant deviations from 120° . The O-N-O angle is 123.6° , which is greater than the ideal trigonal value. This can be attributed to the substantial negative charges on the paired O atoms in this unit.

Table 4. Bond lengths (Å)

Atom	Length
C(2)-N(1)	1.289(4)
C(2)-S(1)	1.755(3)
C(3)-N(4)	1.311(4)
C(3)-N(2)	1.373(4)
C(3)-S(1)	1.720(3)
C(4)-N(2)	1.355(4)
C(4)-C(6)	1.374(4)
C(5)-C(11)	1.392(4)
C(5)-C(7)	1.409(4)
C(5)-C(6)	1.460(4)
C(6)-N(4)	1.388(4)
C(7)-C(8)	1.361(4)
C(8)-C(9)	1.378(5)
C(9)-C(10)	1.382(5)
C(9)-N(3)	1.457(4)
C(10)-C(11)	1.379(4)
C(12)-C(13)	1.387(5)
C(12)-C(17)	1.397(5)
C(13)-C(14)	1.381(5)
C(14)-C(15)	1.384(5)
C(15)-F(1)	1.352(4)
C(15)-C(16)	1.364(5)
C(16)-C(17)	1.379(5)
N(1)-N(2)	1.376(4)
N(3)-O(2)	1.220(4)
N(3)-O(1)	1.227(4)

The molecular structure is primarily stabilized by weak intramolecular C7-H7...N3 hydrogen bond (C7-H7 = 0.930(3) Å, H7...N3 = 2.557(3) Å, C7...N3 = 2.881(4) Å, and the angle C7-H7...N3 = 100.87(1)°), leading to the formation of a pseudo-five-membered hydrogen-bonded pattern with Etter's graph set analysis S(5) [21], thus locking the molecular conformation and eliminating conformational flexibility.

Further, the crystal structure is stabilized by intermolecular interactions into a three-dimensional framework structure by the combination of C-H...N, C-H...O, C-H...S, and C-H...F. In C-H...N interaction, the molecules are linked by paired C-H...N hydrogen bonds into centrosymmetric dimers corresponding to Etter's graph set notation $R_2^2(10)$ [21], the C-H...S interaction sets a bonded dimers corresponding to graph set $R_2^2(16)$ that is formed between C21 and S1 of another molecule, and the C-H...O interaction generates trifurcated bonds from three donors C1 and C17 of one molecule and O1 of another molecule to the same acceptor O1 linking the molecule into a zig-zag ribbon structure along *b* axis (Fig. 2). The C-H...F interaction generates bifurcated bonds from two donors, C14 and the C16, to the same acceptor, F1, linking the dimers so formed into zig-zag

Table 5. Bond angles(°)

Atoms	Angle
C(2)-C(1)-C(12)	112.3(3)
N(1)-C(2)-C(1)	122.6(3)
N(1)-C(2)-S(1)	117.4(2)
C(1)-C(2)-S(1)	120.1(2)
N(4)-C(3)-N(2)	112.3(3)
N(4)-C(3)-S(1)	139.0(3)
N(2)-C(3)-S(1)	108.6(2)
C(11)-C(5)-C(7)	118.3(3)
C(11)-C(5)-C(6)	120.5(3)
C(7)-C(5)-C(6)	121.1(3)
C(4)-C(6)-N(4)	111.7(3)
C(4)-C(6)-C(5)	128.6(3)
N(4)-C(6)-C(5)	119.7(3)
C(8)-C(7)-C(5)	121.5(3)
C(7)-C(8)-C(9)	118.6(3)
C(8)-C(9)-C(10)	122.0(3)
C(8)-C(9)-N(3)	118.3(3)
C(10)-C(9)-N(3)	119.7(3)
C(11)-C(10)-C(9)	119.0(3)
C(13)-C(12)-C(17)	118.1(3)
C(13)-C(12)-C(1)	120.1(3)
C(17)-C(12)-C(1)	121.8(3)
C(14)-C(13)-C(12)	120.4(3)
C(13)-C(14)-C(15)	119.3(3)
F(1)-C(15)-C(16)	117.7(3)
F(1)-C(15)-C(14)	120.0(3)
C(16)-C(15)-C(14)	122.2(3)
C(15)-C(16)-C(17)	117.7(3)
C(16)-C(17)-C(12)	122.3(3)
C(2)-N(1)-N(2)	107.5(3)
C(4)-N(2)-C(3)	107.5(3)
C(4)-N(2)-N(1)	133.8(3)
C(3)-N(2)-N(1)	118.4(3)
O(2)-N(3)-O(1)	123.6(3)
O(2)-N(3)-O(9)	117.8(3)
O(1)-N(3)-C(9)	118.5(3)
C(3)-N(4)-C(6)	103.6(3)
C(3)-S(1)-C(2)	88.11(15)

tapes leading to a two-dimensional network and resulting in centrosymmetric dimmers corresponding to graph set R_2^2 (8) along 'b' axis (Fig. 3). The molecular packing is further stabilized by π - π stacking interactions between the nitro benzene and imidazothiadiazole ring systems with C4-C7 atoms of two molecules being separated by a distance of 3.238(4) Å (Fig. 4) (symmetry code. $\frac{1}{2} -x, y, -z$).

Table 6. Selected torsion angles (°)

C(12)-C(1)-C(2)-N(1)	−162.1(3)
C(12)-C(1)-C(2)-S(1)	17.9(4)
N(2)-C(4)-C(6)-N(3)	−0.1(3)
N(2)-C(4)-C(6)-C(5)	179.9(3)
C(7)-C(8)-C(9)-N(4)	179.5(3)
N(4)-C(9)-C(10)-C(11)	178.9(3)
C(13)-C(14)-C(15)-F(1)	179.1(3)
F(1)-C(15)-C(16)-C(17)	−179.1(3)
C(1)-C(2)-N(1)-N(2)	−179.3(3)
S(1)-C(2)-N(1)-N(2)	0.7(3)
C(6)-C(4)-N(2)-C(3)	0.3(3)
C(6)-C(4)-N(2)-N(1)	179.0(3)
N(3)-C(3)-N(2)-C(4)	−0.5(3)
S(1)-C(3)-N(2)-C(4)	177.43(19)
N(3)-C(3)-N(2)-N(1)	179.4(2)
S(1)-C(3)-N(2)-C(1)	1.4(3)
C(2)-N(1)-N(2)-C(4)	178.0(3)
C(2)-N(1)-N(2)-C(3)	0.5(4)
N(2)-C(3)-N(3)-C(6)	0.4(3)
S(1)-C(3)-N(3)-C(6)	−176.6(3)
C(4)-C(6)-N(3)-C(3)	−0.2(3)
C(5)-C(6)-N(3)-C(3)	179.8(3)
C(10)-C(9)-N(4)-O(2)	−10.4(4)
C(8)-C(9)-N(4)-O(2)	169.8(3)
C(10)-C(9)-N(4)-O(1)	170.8(3)
C(8)-C(9)-N(4)-O(1)	−9.1(4)
N(3)-C(3)-S(1)-C(2)	178.5(4)
N(2)-C(3)-S(1)-C(2)	1.4(2)
N(1)-C(2)-S(1)-C(2)	−1.2(2)
C(1)-C(2)-S(1)-C(3)	−178.8(3)

Table 7. Non-bonded interaction and possible hydrogen bonds(Å, °) for compound 3

D—H...A	D—H	H...A	D...A	D—H...A
C7—H7...N3	0.930(3)	2.557(3)	2.881(4)	100
C7—H7...N3 ⁱ	0.930(3)	2.904(3)	3.509(4)	123
C8—h8...s1 ⁱ	0.930(3)	2.936(1)	3.724(3)	143(9)
C1—H1A...O2 ⁱⁱ	0.970(3)	2.955(3)	3.651(4)	129(9)
C1—H1B...O2 ⁱⁱ	0.970(3)	2.764(3)	3.613(4)	146(2)
C13—H13...O2 ⁱⁱⁱ	0.930(4)	2.776(3)	3.614(4)	150
C14—H14...F1 ^{iv}	0.930(4)	2.713(3)	3.438(5)	140(4)
C16—H16...F1 ^v	0.930(4)	2.822(3)	3.687(5)	155(3)

Symmetry code: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (ii) $-x + \frac{1}{2}, -1/2, -y + \frac{1}{2} + 1, -z$; (iii) $-x + \frac{1}{2}, +y + \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x, -y, -z$; (v) $-x, +y, -z + 1/2$.

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