Synthesis of *N*-(5-aryl-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3*H*)-yl)acetamide derivatives promoted by carbodiimide condensation

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Novel *N*-(5-aryl-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3*H*)-yl)acetamide derivatives were prepared by 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride and *N*-hydroxybenzotrizole condensation catalysis in a convenient and fast method. These compounds were identified by IR, ^{1}H NMR and elemental analyses and the intermediate compound 5-(2-chlorophenyl)-1,3,4-thiadiazol-2-amine was confirmed by single-crystal X-ray diffraction.

Keywords: 1,3,4-thiadiazole, 1,2-benzisothiazolin-3-one, acetamide synthesis

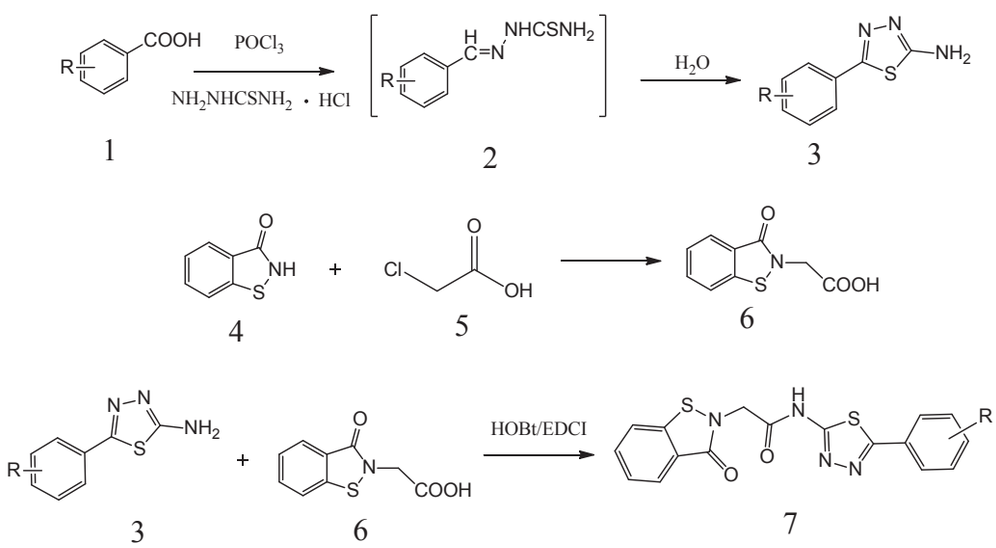
Among various biologically heterocyclic scaffolds, 1,3,4-thiadiazole derivatives have been intensely investigated during recent years. Many possess interesting biological properties such as antimicrobial, anti-inflammatory, analgesic, antituberculosis, anti-depressant, and anticonvulsant activities.[1–6] In addition, the corrosion inhibition activities were found to be associated with 1,3,4-thiadiazole and their derivatives.[7,8]

Isothiazolinone derivatives have attracted considerable attention in chemical and medicinal research because of their diverse biological activities. For example, some benzisothiazolinone derivatives possess antibacterial, anti-corrosion activities and anti-fungal activities.[9–11] Organic compounds containing acetamide are of increasing interest because of their diverse biological properties such as anti-angiogenic, anti-inflammatory, anti-HIV and antitumoural activities.[12–15]

In connection with our research interest directed toward the synthesis of novel *N*-(5-aryl-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(*3H*)-yl)acetamide derivatives, we now report the synthesis of a series of new compounds containing 1,3,4-thiadiazole and benzisothiazolinone (Scheme 1).

Results and discussion

N-(5-Aryl-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(*3H*)-yl) (**7a**–**l**) was prepared by the reaction of 2-amino-5-substitute phenyl-1,3,4-thiadiazole and 2-(3-oxo-1,2-benzothiazol-2(*3H*)-yl) acetic acid (Table 1).



7a R=H 7b R=3-methyl 7c R=4-methoxy 7d R=3,4,5-trimethoxy
7e R=2-bromine 7f R=4-chloro 7g R=2-bromine-4-nitro 7h R= 3,4-difloro
7i R=3,5-difloro 7j R=2-chloro 7k R=4-n-pentyl 7l R= 3,5-dinitro-4-methyl

Scheme 1 The synthetic route of compounds **7a**–**l.**

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Table 1 Synthesis of compounds **7a–l**

Entry	R	Time/h	Yield/%
7a	H	16	76
7b	3-Methyl	20	82
7c	4-Methoxy	18	71
7d	3,4,5-Trimethoxy	18	80
7e	2-Bromine	16	85
7f	4-Chloro	24	77
7g	2-Bromine-4-nitro	20	80
7h	3,4-Difluoro	18	85
7i	3,5-Difluoro	20	80
7j	2-Chloro	18	73
7k	4-n-Pentyl	18	70
7l	3,5-Nitro-4-methyl	24	68

Experimental

Melting points were recorded on an X-4 binocular microscope melting point apparatus. ^{1}H NMR spectra were recorded on an Avance Bruker-500 instrument or Avance Bruker-300 instrument and chemical shifts in ppm are reported with TMS as the internal standard. IR spectra in KBr were recorded by a PerkinElmer PE-683 IR spectrometer. Elemental analyses were performed on an Elementer Vario EL III elementary analysis instrument.

Synthesis of **3;** *general procedure*[16,17]

A mixture of substituted benzoic acid **1** (0.1 mol) and thiosemicarbazide (0.1 mol) was treated with $POCl_3$ (0.3 mol) dropwise at 0–5 °C and maintained for 30 minutes. The reaction mixture was allowed to raise temperature to 70 °C and stirred for 4 h. After cooling, water (50 mL) was added dropwise into the reaction mixture. The pH of the reaction solution was adjusted to the range of 8–9 with 50% NaOH solution. The crude product precipitated, filtered, washed with water, dried and recrystallised from ethanol to afford compound **3** as shown in Table 2.

Table 2 Physical constants of products **3a–l**

Compd	R	Yield/%	M.p. /°C	M.p. (lit.)/°C
3a	H	80	225–227	225–226[18]
3b	3-Methyl	85	238–240	240–241[19]
3c	4-Methoxy	70	209–211	210–212[18]
3d	3,4,5-Trimethoxy	75	221–223	220[20]
3e	2-Bromine	79	191–192	192–194[21]
3f	4-Chloro	74	213–215	214–216[22]
3g	2-Bromine-4-nitro	68	218–220	/
3h	3,4-Difluoro	82	202–204	/
3i	3,5-Difluoro	77	227–228	/
3j	2-Chloro	82	220–222	220–221[18]
3k	4-n-Pentyl	78	186–189	/
3l	3,5-Dinitro-4-methyl	72	218–222	/

5-(2-Bromo-4-nitrophenyl)-1,3,4-thiadiazol-2-amine (**3g**): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 7.67 (s, 2H, NH_2), 8.21–8.54 (m, 3H, ArH); IR(KBr)υ: 3261 (N–H), 3095 (C–H), 1625 (C=N), 1496 (C=C), 1300 (C–N), 1147 (C–C), 514 (C–Br) cm^{-1}. Anal calcd for $C_8H_5BrN_4O_2S$: C, 31.91; H, 1.67; N, 18.61; found: C, 31.88; H, 1.61; N, 18.58%.

5-(3,4-Difluorophenyl)-1,3,4-thiadiazol-2-amine (**3h**): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 7.48 (s, 2H, NH_2), 7.51–7.87 (m, 3H, ArH); IR(KBr)υ: 3271 (N–H), 3088 (C–H), 1633 (C=N), 1512 (C=C), 1307 (C–N), 1178 (C–F) cm^{-1}. Anal calcd for $C_8H_5F_2N_3S$: C, 45.07; H, 2.36; N, 19.71; found: C, 45.13; H, 2.31; N, 19.64%.

5-(3,5-Difluorophenyl)-1,3,4-thiadiazol-2-amine (**3i**): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 7.48 (s, 2H, NH_2), 7.28–7.58 (m, 3H, ArH); IR(KBr)υ: 3278 (N–H), 3091 (C–H), 1625 (C=N), 1502 (C=C), 1330 (C–N), 1193 (C–F) cm^{-1}. Anal calcd for $C_8H_5F_2N_3S$: C, 45.07; H, 2.36; N, 19.71; found: C, 45.01; H, 2.39; N, 19.68%.

5-(4-Pentylphenyl)-1,3,4-thiadiazol-2-amine (**3k**): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 0.86 (t, 3H, CH_3), 1.27 (m, 4H, –CH_2CH_2–), 1.58 (m, 2H, –CH_2–), 2.60 (t, 2H, Ar–CH_2), 7.26 (s, 2H, NH_2), 7.28–7.66 (m, 4H, ArH), 7.64; IR(KBr)υ: 3253 (N–H), 3095 (C–H), 1616 (C=N), 1508 (C=C), 1328 (C–N) cm^{-1}. Anal calcd for $C_{13}H_{17}N_3S$: C, 63.12; H, 6.93; N, 16.99; found: C, 63.17; H, 6.90; N, 16.91%.

5-(3,5-Dinitro-4-methylphenyl)-1,3,4-thiadiazol-2-amine (**3l**): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 2.48 (s, 3H, CH_3), 7.72 (s, 2H, NH_2), 8.52 (s, 2H, ArH); IR(KBr)υ: 3267 (N–H), 3086 (C–H), 1624 (C=N), 1504 (C=C), 1348 (C–N) cm^{-1}. Anal calcd for $C_9H_7N_5O_4S$: C, 38.43; H, 2.51; N, 24.90; found: C, 38.38; H, 2.56; N, 24.82%.

Synthesis of **6***; general procedure*

The solution of KOH (0.012 mol) with 5 mL H_2O was added dropwise to the mixture of benzisothiazolinone **4** (0.01 mol) and 10 mL H_2O. The mixture was stirred for 1 h at the temperature of 60 °C. Then, the solution of chloroacetic acid 5 (0.01 mol) with 10 mL H_2O was added into the mixture dropwise. The mixture was heated and stirred for another 5 h at 90 °C. The pH was adjusted by 50% KOH solution to 9–10. After completion of the reaction, crude compound **6** was obtained by cooling and filtering. The pure compound was obtained by recrystallisation from ethanol with a yield of 79% as a white solid, m.p. 232–233 °C (lit.[23] 229–230 °C).

Synthesis of **7a–l***; general procedure*

A mixture of compounds **3a–l** (0.01 mol) and **6** (0.0105 mol) in DMF (30 mL) was added to EDCI (0.012 mol) and HOBT (0.012 mol). The mixture was stirred for 24 h at room temperature. After completion of the reaction, the mixture was poured into water. The crude precipitated was filtered and washed with water to obtain the crude product. Using column chromatography (ethyl acetate: petroleum ether = 1 : 1) the crude products were identified as compounds **7a–l**.

2-(3-Oxo-1,2-benzothiazol-2(3H)-yl)-N-(5-phenyl-1,3,4-thiadiazole-2-yl)acetamide (**7a**): Yield 76%; m.p. 238–239 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.37 (s, 2H, N–CH_2), 7.52–8.11 (m, 9H, ArH), 13.07 (s, 1H, N–H); IR(KBr)υ: 3177 (N–H), 2919 (C–H), 1722, 1640 (C=O), 1568 (C=C), 1310 (C–N) cm^{-1}. Anal calcd for $C_{17}H_{12}N_4O_2S_2$: C, 55.42; H, 3.28; N, 15.21; S, 17.41; found: C, 55.26; H, 3.11; N, 15.50; S, 17.11%.

2-(3-Oxo-1,2-benzothiazol-2(3H)-yl)-N-(5-(m-tolyl)-1,3,4-thiadiazole-2-yl)acetamide (**7b**): Yield 82%; m.p. 217–218 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 2.38 (s, 3H, CH_3), 5.36 (s, 2H, N–CH_2), 7.33–8.11 (m, 8H, ArH), 13.05 (s, 1H, N–H); IR(KBr)υ: 3177 (N–H), 2918 (C–H), 1709, 1672 (C=O), 1568 (C=C), 1307 (C–N) cm^{-1}. Anal. calcd for $C_{18}H_{14}N_4O_2S_2$: C, 56.53; H, 3.69; N, 14.65; S, 16.77; found: C, 56.58; H, 3.55; N, 14.61; S, 16.43%.

N-(5-(4-Methoxyphenyl)-1,3,4-thiadiazole-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7c**): Yield 71%; m.p. 239–241 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 3.82 (s, 3H, OCH_3), 5.35 (s, 2H, N–CH_2), 7.06–8.11 (m, 8H, ArH), 12.98 (s, 1H, N–H); IR(KBr)υ: 3178 (N–H), 2917 (C–H), 1718, 1691 (C=O), 1570 (C=C), 1318 (C–N) cm^{-1}. Anal. calcd for $C_{18}H_{14}N_4O_3S_2$: C, 54.26; H, 3.54; N, 14.06; S, 16.09; found: C, 54.30; H, 3.72; N, 14.82; S, 16.01%.

2-(3-Oxo-1,2-benzothiazol-2(3H)-yl)-N-(5-(3,4,5-trimethoxyphenyl)-1,3,4-thiadiazole-2-yl)acetamide (**7d**): Yield 80%; m.p. 232–234 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 3.79 (s, 9H, OCH_3), 5.36 (s, 2H, N–CH_2), 7.19–8.11 (m, 6H, ArH), 13.05 (s, 1H, N–H); IR(KBr)υ: 3172 (N–H), 2918 (C–H), 1704, 1676 (C=O), 1586 (C=C), 1310 (C–N) cm^{-1}. Anal. calcd for $C_{20}H_{18}N_4O_5S_2$: C, 52.39; H, 3.96; N, 12.22; S, 13.99; found: C, 52.17; H, 3.52; N, 12.29; S, 13.51%.

N-(5-(2-Bromophenyl)-1,3,4-thiadiazole-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7e**): Yield 85%; m.p. 248–49 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.38 (s, 2H, N–CH_2), 7.46–8.11 (m, 8H, ArH), 13.13 (s, 1H, N–H); IR(KBr)υ: 3160 (N–H), 2918 (C–H), 1711, 1668 (C=O), 1568 (C=C), 1307 (C–N), 1152 (C–Br) cm^{-1}. Anal. calcd for $C_{17}H_{11}BrN_4O_2S_2$: C, 45.64; H 2.48; N 12.52, S, 14.34; found: C, 45.55; H, 2.28; N, 12.72, S, 14.14%.

N-(5-(4-Chlorophenyl)-1,3,4-thiadiazole-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7f**): Yield 77%; m.p. 248–250 °C;

^{1}H NMR(DMSO-d_6, 300 MHz) δ 4.82 (s, 2H, N–CH_2), 7.11–8.11 (m, 8H, ArH), 13.12 (s, 1H, N–H); IR(KBr)υ: 3159 (N–H), 2921 (C–H), 1729, 1704 (C=O), 1563 (C=C), 1300 (C–N), 1191 (C–Cl) cm^{-1}. Anal. calcd for $C_{17}H_{11}ClN_4O_2S_2$: C, 50.68; H 2.75; N 13.91; S, 15.92 found: C, 50.32; H, 2.88; N, 14.01; S, 15.80%.

N-(5-(2-Bromo-4-nitrophenyl)-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7g**): Yield 80%; m.p. 204–206 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.40 (s, 2H, N–CH_2), 7.52–8.61 (m, 7H, ArH), 13.29 (s, 1H, N–H); IR(KBr)υ: 3165 (N–H), 2918 (C–H), 1720, 1657 (C=O), 1570 (C=C), 1309 (C–N) cm^{-1}. Anal. calcd for $C_{17}H_{10}BrN_5O_4S_2$: C, 41.47; H, 2.05; N, 14.23; S, 13.03; found: C, 41.32; H, 2.38; N, 14.51; S, 13.11%.

N-(5-(3,4-Difluorophenyl)-1,3,4-thiadiazole-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7h**): Yield 85%; m.p. 262–264 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.37 (s, 2H, N–CH_2), 7.52–8.11 (m, 7H, ArH), 13.14 (s, 1H, N–H); IR(KBr)υ: 3156 (N–H), 2920 (C–H), 1721, 1664 (C=O), 1586 (C=C), 1312 (C–N), 1170 (C–F) cm^{-1}. Anal. calcd for $C_{17}H_{10}F_2N_4O_2S_2$: C, 50.49; H 2.49; N 13.85, S, 15.86; found: C, 50.62; H, 2.38; N, 13.61; S, 15.52%.

N-(5-(3,5-Difluorophenyl)-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7i**): Yield 80%; m.p. 220–221 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.37 (s, 2H, N–CH_2), 7.42–8.11 (m, 7H, ArH), 13.20 (s, 1H, N–H); IR(KBr)υ: 3159 (N–H), 2920 (C–H), 1722, 1661 (C=O), 1598 (C=C), 1311 (C–N), 1170 (C–F) cm^{-1}. Anal. calcd for $C_{17}H_{10}F_2N_4O_2S_2$: C, 50.49; H 2.49; N 13.85, S, 15.86; found: C, 50.82; H, 2.45; N, 13.71; S, 15.45%.

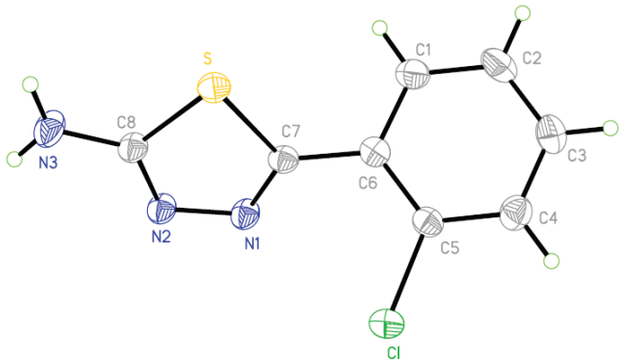

Fig. 1 ORTEP diagram of **3j** thermal ellipsoids is shown at the 30% probability level.

N-(5-(2-Chlorophenyl)-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7j**): Yield 73%; m.p. 236–238 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 5.38 (s, 2H, N–CH_2), 7.11–8.15 (m, 8H, ArH), 13.18 (s, 1H, N–H); IR(KBr)υ: 3168 (N–H), 2851 (C–H), 1703, 1681 (C=O), 1568 (C=C), 1312 (C–N), 1206 (C–Cl) cm^{-1}. Anal. calcd for $C_{17}H_{11}ClN_4O_2S_2$: C, 50.68; H 2.75; N 13.91; S, 15.92; found: C50.62; H, 2.38; N, 13.81; S, 15.52%.

2-(3-Oxo-1,2-benzothiazol-2(3H)-yl)-N-(5-(4-pentylphenyl)-1,3,4-thiadiazol-2-yl)acetamide (**7k**): Yield 70%; m.p. 202–204 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 0.85 (t, 3H, CH_3), 1.28 (m, 4H, –CH_2CH_2–), 1.59 (m, 2H, –CH_2–), 2.62 (t, 2H, Ar–CH_2), 5.36 (s, 2H, N–CH_2), 7.33–8.11 (m, 8H, ArH), 13.03 (s, 1H, N–H); IR(KBr)υ: 3168 (N–H), 2921 (C–H), 1710, 1668 (C=O), 1564 (C=C), 1297 (C–N)cm^{-1}. Anal. calcd for $C_{22}H_{22}N_4O_2S_2$: C, 60.25; H 5.06; N 12.78, S, 14.62; found: C, 60.62; H, 5.38; N, 12.41; S, 14.50%.

N-(5-(4-Methyl-3,5-dinitrophenyl)-1,3,4-thiadiazol-2-yl)-2-(3-oxo-1,2-benzothiazol-2(3H)-yl)acetamide (**7l**): Yield 68%; m.p. 216–217 °C; ^{1}H NMR(DMSO-d_6, 500 MHz) δ 2.45 (s, 3H, CH_3), 5.39 (s, 2H, N–CH_2), 7.53–8.74 (m, 6H, ArH), 13.31 (s, 1H, N–H); IR(KBr)υ: 3152 (N–H), 2868 (C–H), 1721, 1616 (C=O), 1597 (C=C), 1303 (C–N) cm^{-1}. Anal. calcd for $C_{18}H_{12}N_6O_6S_2$: C, 45.76; H 2.56; N 17.79, S, 13.57; found: C, 45.62; H, 2.48; N, 17.81; S, 13.47%.

The intermediate compound **3j** was subjected to single crystal X-ray crystallography and intensity data were collected 298(2) K on the Enraf-Nonius CAD-4 diffractometer, using graphite Monochromatic MoKα adiation (λ=0.71073Å). The structure was solved directly using the SHELXL-97 program[24] and refined with the SHELXL-97 program.[25] All H atoms bonded to the C atoms were placed geometrically and constrained to ride on their parent atoms. The thermal ellipsoids were plotted with the SHELXL-97 program at 30% probability. The molecular structure is shown in Fig. 1. The molecular packing is shown in Fig. 2. Selected crystal data and structure refinement details are presented in Table 3. Hydrogen bonds are shown in Table 4.

In the crystal of compound **3j**, the benzene ring was, of course, planar, with an root mean square (RMS) deviation of 0.0013(3) and the thiadiazole ring was a planar five-membered ring with an RMS deviation of 0.0018(2). The dihedral angle between them was 44.8º. Two intermolecular N–H…N hydrogen bonds linked the molecules to form a three-dimensional network, in which they may be effective in the stabilisation of the structure.

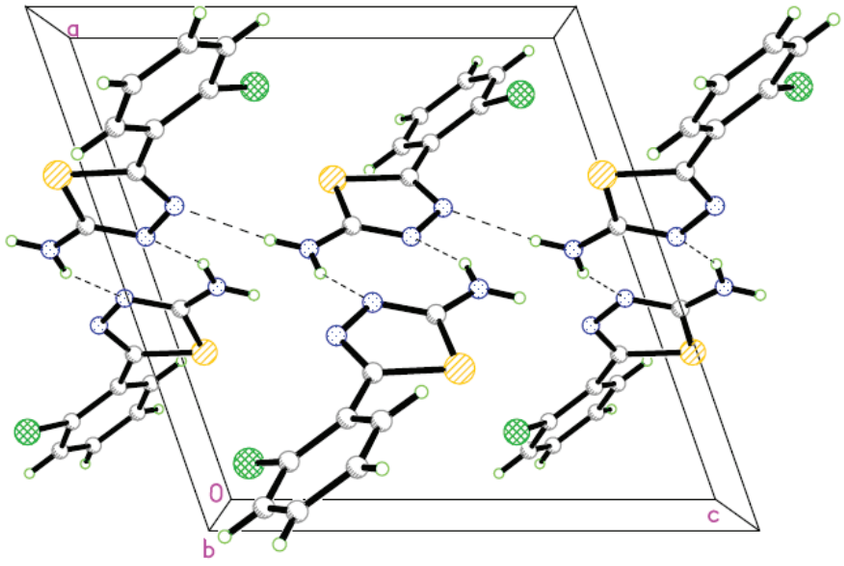

Fig. 2 A view of the molecular packing of **3j**. Hydrogen bonds are shown as dashed lines.

Table 3 Crystal data and structure refinement for $C_8H_6ClN_3S$

Empirical formula	$C_8H_6ClN_3S$
Formula weight	211.67/g mol^{-1}
Temperature	293(2)/K
Wavelength	0.71073/Å
Crystal system	Monoclinic
Space group	$P2_1/C$
Unit cell dimensions	a=11.619(2)/Å b=7.1500(14)/Å c=11.519(2)/Å β=109.31(3)/°
Volume	903.1(3)/Å^{-3}
Z	4
Absorption correction	Psi-scan
F(000)	432
Absorption coefficient	0.60 mm^{-1}
θ range for entire data collection	1.9° to 25.4°
Reflections collected	1734
Independent reflections	1650 (R_{int}=0.0334)
Data/restraints/parameters	1650/0/119
Final R indices [$I>2\sigma>(I)$]	R^1=0.0413, wR^2=0.1391
Goodness-of-fit on F^2	1.009
Final residual electron density	0.404 and −0.343/e Å^{-3}

Table 4 Selected bond distances (Å) and angles (°) for compound (**3j**)

D–H···*A*	*D*–H	H···*A*	*D*···*A*	*D*–H···*A*
N3–H3A···N2^{i}	0.86	2.25	3.057 (4)	157
N3–H3B···N1^{ii}	0.86	2.16	3.016 (3)	171

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) *x*, −*y*+3/2, *z*−1/2.

Crystallographic data for the structure reported in this paper has been deposited with the Cambridge Data Centre. The deposition number is CCDC 980924 (compound **3j**).

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