Syntheses and X-ray structure of *N*-(Benzothiazol-2-yl)-3-chlorobenzamide.

ABSTRACT
The reaction of 3-chloro-benzoyl chloride and 2-aminobenzothiazole in the presence of potassium thiocyanate yielded a yellowish solid $C_{15}H_{10}ClN_3OS_2$ (**1**). Reaction of **1** with cobalt chloride yield a yellowish solid $C_{14}H_{11}ClN_2O_2S$ (**2**) which gives crystal suitable for X-ray analyses. The compounds were characterized by elemental analyses FTIR spectroscopy and melting point measurement. Compound **2** crystallizes in the triclinic space group $P\bar{1}$ with the following parameters: a = 7.0299(2) Å b = 7.0466(2) Å c = 14.5490(2) Å α = 103.844(3)° β = 92.929(2)° γ = 107.291(3) V = 662.33(4) $Å^3$ Z= 2 R_1 = 0.030 wR_2 = 0.034. The asymmetric unit of (**2**) consists of one molecule in which the 3-chloro phenyl moiety and the benzothiazole-2-yl moiety form a dihedral angle of 6.417(4)°. An intramolecular O—H···O hydrogen bond occurs. In the crystal O—H···N and N—H···O hydrogen bonds link the molecules forming layers parallel to the *ac* plane.

Key words: Cobalt(II)3-chloro-benzoyl chloride2-aminobenzothiazoledethiocyanationFTIR, X-ray diffraction.

1. INTRODUCTION

Thiourea derivatives can be synthesized with different methods [1 2]and uses in different applications such as the extraction of certain transition metals in acidic medium [3–5]. Multiple biological activities such as antibacterial [56]antifungal [6]antituberculosis[7]anticancer[8] antiviral [9] anticonvulsant[10] antioxidant [11] insecticidal [12]and herbicidal [13 14].Benzoylthiourea derivatives are widely and successfully used in coordination chemistry. Indeed these compounds have C=O and C=S functions which can serve as donors in the chelation of metal ions [15–18].Thiourea derivatives often have versatile behavior and are good intermediates in organic synthesis. In the presence of metal ions formation of heterocycles including the carbon atom of the C=S group or the oxygen atom of the C=O group can occur. In our recent work we reported triazolic and oxadiazolic compounds from 1-isonicotinoyl-4-phenylthiosemicarbazide in the presence of Fe^{2+} or Mn^{2+} ions respectively[19]. A heterocyclic compound in which the sulfur atom of the C=S group is included in the ring is reported in the literature [20].On continuing our work in the synthesis coordination complexes from thiourea derivative the ligand (13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea was reacted with cobalt chloride (II). This reaction led to the dethiocyanation of the organic molecule to give *N*-(Benzothiazol-2-yl)-3-chlorobenzamide. In this article we report the synthesis and structural characterization by a spectroscopic study and by x-ray diffraction of the compounds.

2. METHODOLOGY

2.1 Starting materials and Instrumentation

Potassium thiocyanate3-chloro-benzoyl chlorideand 2-aminobenzothiazole were purchased from Sigma-Aldrich and used as received without further purification. All solvents used were of reagent grade. Elemental analyses of C H N Sand Cl were recorded on a VxRio EL Instrument. Infrared spectra were obtained on a FTIR Spectrum Two of Perkin Elmer spectrometer in the 4000-400 cm^{-1} region.The compounds were synthetized following the procedure reported by Odame et al. [21 22] with slight modification.

2.2 Synthesis of3-(13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea

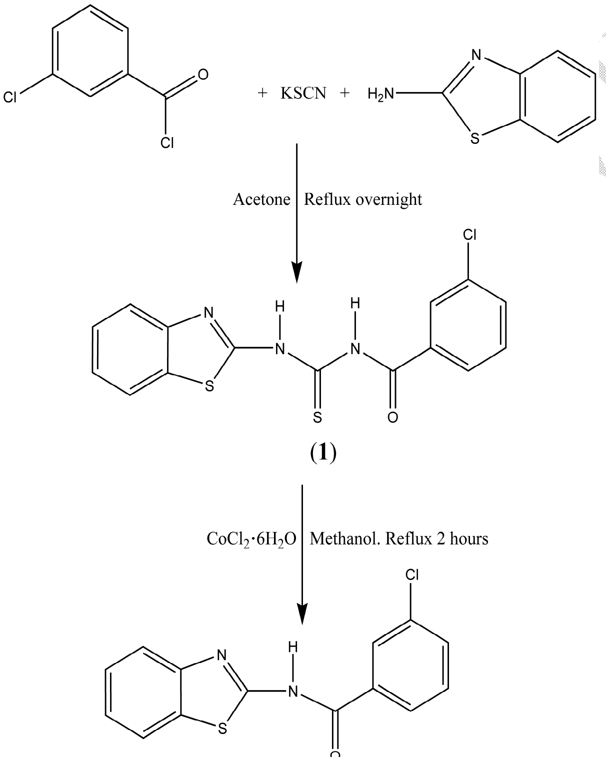
The thiourea derivative was obtained by the reaction of potassium thiocyanate (1.9388 g 20 mmol) with 3-chloro-benzoyl chloride (3.3492 g 20 mmol)in 25 mL of acetone and heating under reflux for 2 h to yield the 3-chlorobenzoyl isothiocyanate. To the above solution was added a solution of2-aminobenzothiazole (3 g 20 mmol) in 25 mL of acetone. The resulting mixture was heated overnight. The solvent was removed by evaporation and the crude product was recrystallized in methanol. Yield : 75.7 % . MP : 141-142 °C. Anal. calcd. for $C_{15}H_{10}ClN_3OS_2$: Calculated % C 51.79; % H 2.90; % N 12.08; % S 18.44; % Cl 10.19. Found C 51.81; % H 2.92; % N 12.03; % S 18.41; % Cl 10.16. FTIR: (ν cm^{-1}) : 3290 (N−H) 3055 (N−H) 1675 (C=O) 1592 (C=C) 1546 (C=C) 1451(C−N) 1439 (C−N).

2.3 Synthesis of*N*-(Benzothiazol-2-yl)-3-chlorobenzamide

3-(13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea(0.3068 g1 mmol) was dissolved in 20 mL of methanol and a solution of $CoCl_2{\cdot}6H_2O$ (0.2369g1 mmol) in 5 mL of methanol was added. The mixture was refluxed for 2 hours and filtered while hot. Slow evaporation of the resulting solution yield crystal suitable for X-ray analyses after a week.Yield : 80.3 % . MP : 155 °C. Anal. calcd. for $C_{14}H_{11}ClN_2O_2S$: . Calculated % C 54.81; % H 3.61; % N 9.13; % S 10.45; % Cl 11.56. Found % C 54.79; % H 3.58; % N 9.10; % S 10.39; % Cl 11.53. FTIR:(ν cm^{-1}) : 3280 (N–H amide) 3039 (C–H aromatic) 1702 (C=O) 1590 (C=C aromatic)1450 (C–N amide) 1441 (C–N thiazole) 725 (C–Cl).

2.4 X-ray data collection structure determination and refinement

Crystals suitable for X-diffraction of the reported compound were grown by slow evaporation of MeOH solution of the complex. Details of the X-rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using a SuperNova Rigaku OxfordDiffraction diffractometer single diffractometer with graphite monochromatized Mo*Kα* radiation (λ = 0.71073 Å). All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package *SHELXTL* [23]. The structures were solved by direct methods which revealed the position of all non-hydrogen atoms. All the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms [24]. The hydrogen atoms were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using *ORTEP-3* [25].



Scheme 1. General scheme for the synthesis of the compounds

Table 1. Crystal data and structure refinement details for (**2**).

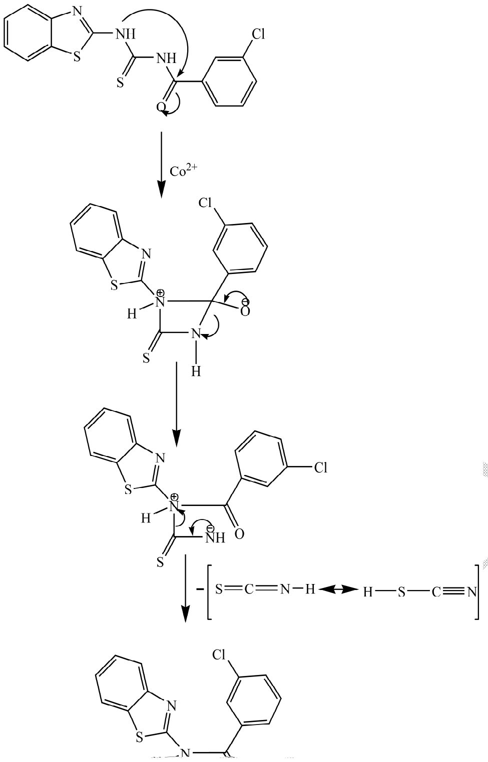
Chemical formula	$C_{14}H_9ClN_2OS \cdot H_2O$
M_r	306.76
Crystal system space group	Triclinic$P\bar{1}$
Temperature (K)	100
a (Å)	7.0299 (2)
b (Å)	7.0466 (2)

c (Å)	14.5490 (4)
α	103.843 (2)
β	92.929 (2)
γ (°)	107.291 (2)
V (Å^3)	662.32 (3)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.45
Crystal size (mm)	0.22 × 0.11 × 0.07
$T_{\mathrm{min}}T_{\mathrm{max}}$	0.4991.000
Measured reflections	12431
Independent reflections	3437
Reflections [I > 2σ(I)]	2964
R_{int}	0.030
R [I > 2σ(I)]	0.034
wR_2	0.087
Goodness–of–Fit	1.06
Data/parameters/restraint	3437/184/0
$\Delta\rho_{\mathrm{max}}$ $\Delta\rho_{\mathrm{min}}$ (e Å^{-3})	0.38−0.33

3. Results and discussion

In the present study the compound 3-(13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea(**1**)was synthesized by the reaction of 3-chlorobenzoyl isothiocyanategenerated in situ and 2-aminobenzothiazole(Scheme 1). *N*-3-(13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea (**1**)was used to synthetize coordination complex owing to the presence of multiple donor sites such as O N and S atoms.(Benzothiazol-2-yl)-3-chlorobenzamide (**2**)was obtained during our attempt to synthesize complex using Co(II) salt. It seems that the metal cation act as catalyst for the dethiocyanation reaction. This phenomena was observed inanalogous reactions. In fact inour previous work in attempt to synthetize Mn(II) and Fe(II) complexes using 1-isonicotinoyl-4-phenylthiosemicarbazidewe have isolated two news organic ligands 4-phenyl-3-(pyridin-4-yl)-1*H*-124-triazole-5(4*H*)-thione and *N*-phenyl-5-(pyridin-4-yl)-134-oxadiazol-2-amine hydrochloride respectively [19].The reaction sequences depicted in the

Scheme **1**were followed to obtain (**1**)and (**2**) respectively. A mechanism for the dethiocyanation and the formation of (**2**) is proposed in Scheme **2**.



Scheme 2. Proposed mecanism for the formation of (2)

3.1 General study

The infrared spectrum of compound (**1**) exhibited characteristic bands. The bands of medium intensities appearing at 3290 cm^{-1} and 3055cm^{-1} are attributed to N–H stretching vibrations of the amide and the thioamide groups[26]. The bands pointed at 1675 cm^{-1} is attributed to the C=O linked to the phenyl ring. Bands due to the aromatic units are pointed in the range 1592-1546 cm^{-1}[27]. The bands pointed at 1451 cm^{-1} and 1439cm^{-1}are attributed to the C–N stretching vibrations[28]. Upon reaction of (**1**) and Co(**II**) salt compound (**2**) was recovered.On the infrared spectrum of compound (**2**) the band pointed at 3280 cm^{-1}is indicative of the presence of N–H of the amide group. The band due to the C=O moiety is shifted to high frequencies and is pointed at 1702 cm^{-1}. The presence of aromatic rings is confirmed by the bands which appear in the range 1590-1540 cm^{-1}. Bands at 1450 cm^{-1} and 1441 cm^{-1} are attributed to the C–N stretching vibrations. Band due to C–Cl is pointed at 730 cm^{-1}[29].

3.2 Description of the structure of (2)

Compound (**2**)was obtained by mixing methanolic solution of Cobalt(**II**) chloride with methanol solution of 3-(13-Benzothiazol-2-yl)-1-(3-chlorobenzoyl)thiourea. After the reaction gradual evaporation of the filtered solution yields crystals of (**2**) suitable for X-ray diffraction. The title compound $C_{14}H_{11}ClN_2O_2S$(**2**) crystallizes in the triclinic space group $P\bar{1}$. The crystallographic data selected bond lengths and bond angles for the crystal structure are provided in Table 2. The ORTEP diagram for compounds if presented in Fig. 1. The molecule consists of a 3-chlorophenyl moiety linked through a carbonyl unit to a 2-aminobenzothiazole moiety yielding themonohydrated*N*-(Benzothiazol-2-yl)-3-chlorobenzamide derivative. The benzothiazole-2-yl moiety is quite planar (rms deviations of fitted atoms of 0.0099 (3) Å) and form a dihedral angle of 6.417 (4)° with the 3-chlorophenyl moiety. The bond distance of O1–C8 is 1.2305 (17) Å which is consistent with a carbonyl whilst the S1–C1 bond distance value of 1.7504 (14) Å is consistent with double bond character. These value are comparable to those found for *N*-(benzothiazol-2-yl)benzamide [1.750 (1) Å for S–C and 1.222 (2) Å for O1–C2] [22]. The C7—N2 and C8—N2 bond lengths are 1.3867 (18) and 1.3657 (18) Å] are shorter than the normal single C—N bond length indicating the sp^2 hybridization of the nitrogen atom [22 30]. The bond angle value of 122.67(12)° for C8-N2-C7 confirms the sp^2 hybridization of N2. The bond angle of C1–S1–C7 is 88.22 (7)° whilst the bond angle of S1–C7–N3 in is 117.13 (11)°. The benzothiazolyl moiety is *cis* with respect to the carbonyl oxygen atom across the C8–N2 bond. The independent water molecule donates one intramolecular hydrogen bond to the carbonyl atom of the ligand [O2—H2B⋯O1] (Fig. 2, Table 3). Molecular layers running almost parallel to the *ac* plane are formed by intermolecular hydrogen bonds involving the second H atom of the independent water molecule group and the N atom of the benzothiazolyl moiety as acceptor [O2—H2C⋯N3^{i} : i = $-x+1\ -y+1\ -z+1$] and NH groups and the oxygen atom of the water molecule as acceptor [N2—H2⋯O2^{ii} : ii =$xy+1\ z$] (Fig. 3, Table 3).

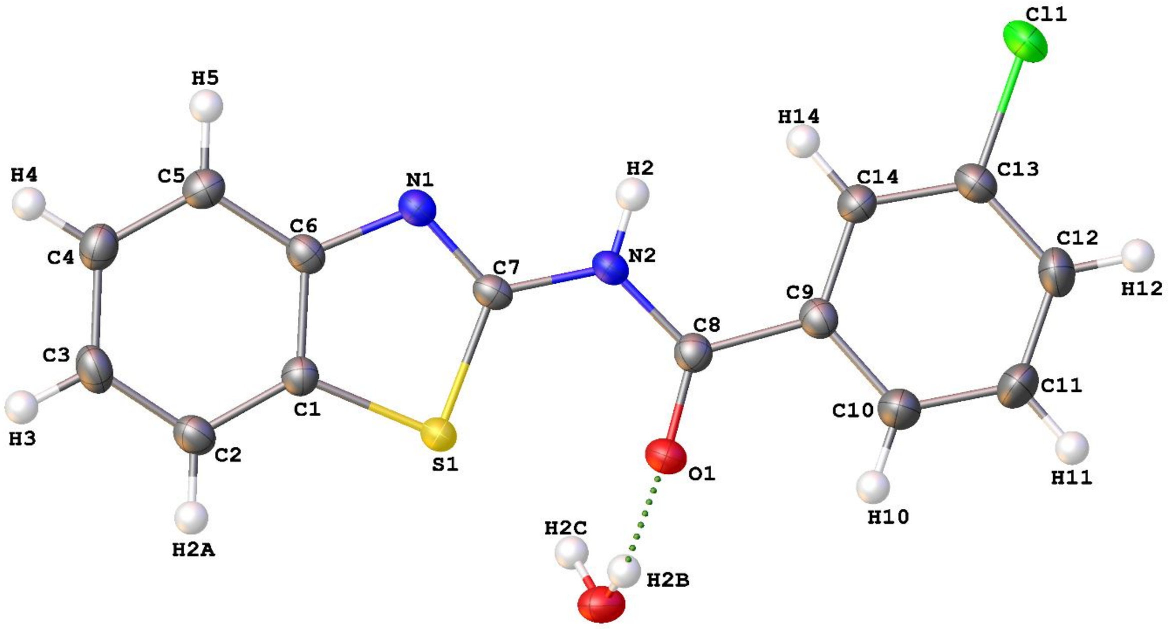


Figure 1. ORTEP plot (30% probability ellipsoids) showing the structure of (2).

Table 2. Selected geometric parameters (Å °)for (2).

S1—C1	1.7359 (14)	N3—C7	1.3018 (18)
S1—C7	1.7504 (14)	N2—C7	1.3867 (18)
O1—C8	1.2305 (17)	N2—C8	1.3657 (18)
N3—C6	1.3940 (18)	C13—C12	1.382 (2)
C1—S1—C7	88.22 (7)	N3—C7—N2	120.34 (12)
C7—N3—C6	109.67 (12)	O1—C8—N2	120.84 (13)
C8—N2—C7	122.67 (12)	O1—C8—C9	120.11 (12)
N3—C7—S1	117.13 (11)	N2—C8—C9	119.05 (12)

Table 3. Hydrogen-bonding geometry (Å °) of compound (2).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2B···O1	0.87	2.00	2.8542(15)	165.0
O2—H2C···N3$^{\text{i}}$	0.87	2.19	2.9472(16)	145.1
N2—H2···O2$^{\text{ii}}$	0.88	1.99	2.8541(16)	167.1

Symmetry codes: (i) −*x*+1 −*y*+1 −*z*+1; (ii) *xy*+1 *z*.

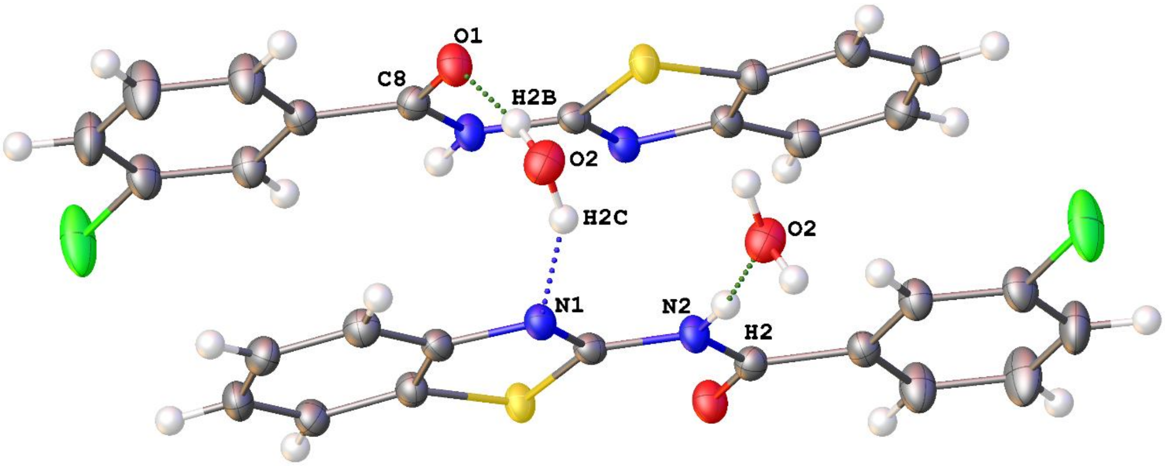


Figure 2.Intra and intermolecular hydrogen contacts showing the dimer in the crystal.

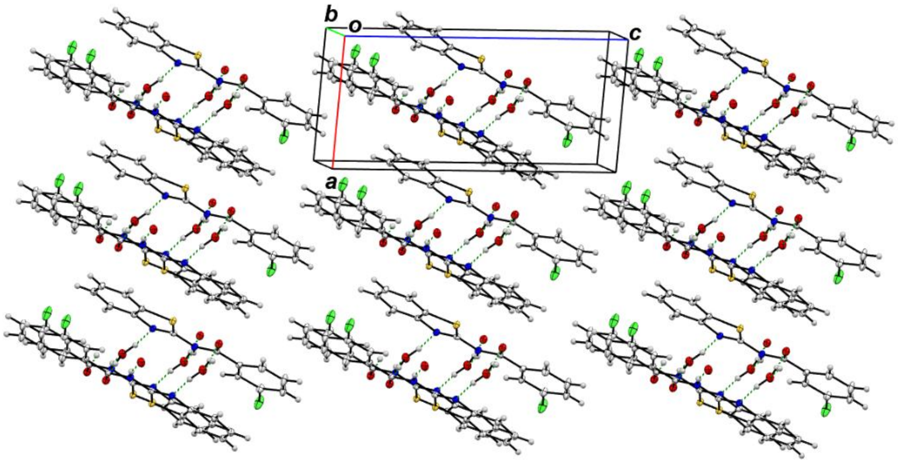


Figure 3. View along the *b*axis of the crystal packing of compound (**2**)

4. Conclusion

Compound (**1**) synthesized from the reaction between 2-aminothiazole and 3-chlorobenzoyl isothiocyanate yields compound (**2**) when it reacts with Co(II). It appears that the action of the metal is determinate in the dethiocyanation reaction. The structures of compounds (**1**) and (**2**) were confirmed by elemental analysis and spectroscopic techniques. The molecular structure of compound (**2**) is determined by X-ray diffraction.

SUPPLEMENTARY MATERALS

CCDC-2340894contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/ structures/ or by contacting The Cambridge Crystallographic Data Centre 12 Union Road Cambridge CB2 1EZ UK.

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