Synthesis and Characterization of Some New 2-Aminobenzothiazole Derivatives

Souad J. Lafta and Suzanne J. Abass
Department of chemistry, college of Science, Mustansiriyha University

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INTRODUCTION

2- substituted benzothiazole has immerged in its usage as a core structure in the diverse therapeutic applications. The studies of structure – activity relationship interestingly reveal that change of the structure of substituent group at C-2 position commonly results the change of its bioactivity[1]. Since most of the benzothiazole derivatives were reported for their diversified activity such as antitumor, antitubercular, antimalarial, anticonvulsant, anthelmintic, analgesic, anti-

ABSTRACT

Compound S$_1$ (2-aminobenzothiazole) was prepared through the condensation of aniline with ammoniumthiocyanate in presence of bromine as a catalyst[3], its derivatives were synthesized through four different lines as follows.

1: 2-aminobenzothiazole was treated with $m$-nitro banzaldehyde in absolute ethanol to obtain shiff's base S$_2$. Shiff's base was treated with succinic anhydride in dry benzene to obtain a compound with seven membered heterocyclic ring S$_3$.

2: 2-aminobenzothiazole was treated with $p$-chlorophenylisothiocyanate to obtain thiourea derivative compound S$_5$ and the later was treated with chloroacetic acid to obtain a compound with two thiazole rings compound S$_6$.

3: 2-aminobenzothiazole was treated with two different anhydrides (succinic anhydride & phthalic anhydride) to obtain 2-aminobenzothiazole derivatives with carboxylic acid moiety S$_7a$-$b$ and these two compounds were treated with o-phenylenediamine to obtain heterocyclic compounds with imidazole ring S$_8a$-$b$.

4: 2-aminobenzothiazole was treated with chloroacetyl chloride to obtain compound S$_9$ which was treated with different primary and secondary aromatic amines to obtain different amide derivatives S$_{10a}$-$e$.
inflammatory and antifungal [2]. Recently, several new methods have been reported, some of the most common methods for synthesis of 2-aminobenzothiazole and its derivatives are as follows: 1) Hofmann method 2) Jacobson cyclization 3) Using Bromine as catalyst 4) Using Sulfuric acid as a catalyst 5) Using Benzen as a catalyst 6) Copper and palladium catalyzed cyclization and many other methods.

**MATERIALS AND METHODS**

**Materials:**
All materials were from BDH, FLUKA and REDLE–DE HAEN. All other solvents were analar grade.

**Instruments:**
Melting points were measured on a Gallan Kamp MFB-600 Melting point apparatus and were uncorrected.
FTIR spectra were recorded as potassium bromide (KBr) disk on FTIR-8400S Fourier Transform Infrared Spectrophotometer "SHIMADZU".
UV-Visible spectra were recorded on CARY 100 Conc UV-Visible Spectrophotometer "VARIAN".
H1 NMR spectra were recorded on Burker DMX-500 NMR (300-600 MHz) Spectrophotometer with using DMSO as a solvent in Jordan University.

**Preparation of compound (S1) [3]**
(2-aminobenzothiazole)
Aniline (4.6g, 0.05mol) and ammonium thiocyanat (3.8g, 0.05 mol) were dissolved in absolute ethanol containing 4 ml of con. HCl. To this mixture bromine in glacial acetic acid (6.75ml, 0.125 mol) was added and the reaction mixture was refluxed for 1 hr. Then it was cooled in ice bath. The precipitate obtained was filtered, washed with cold water and dried. The crude product was recrystallized from ethanol. The physical properties are listed in table 2.

**Preparation of compound (S2) [4]**
(N-(3-nitrobenzylidene)-1,3-benzothiazol-2-amine)
To a mixture of compound (S1) (1.50g, 0.01mol) and m-nitro benzaldehyde (1.51g, 0.01mol) in (30ml) ethanol, 3 drops of glacial acetic acid were added. The mixture was refluxed for 7 hrs. The solvent was evaporated under reduced pressure and the precipitated solid was washed with petroleum ether (range of B.P =60-80°C) and recrystallized from ethanol. The physical properties are listed in table 2.

**Preparation of compound (S3) [5]**
(3-(1,3-benzothiazol-2-yl)-2-(3-nitrophenyl)-1,3-oxazepane-4,7-dione)
Mixture of compound (S2) (0.28g, 0.001mol) with succinic anhydride (0.1g, 0.001mol) in (10 ml) of dry benzene was refluxed on
water bath for 1hr. The solvent was evaporated and the precipitated solid was recrystallized from tetrahydrofuran (THF). The physical properties are listed in table 2.

**Preparation of compounds (S₄)** [6]
(3-(1,3-benzothiazol-2-yl)-2-(3-nitrophenyl)-1,3-thiazolidin-4-one)

(\(N\)-(1,3-benzothiazol-2-yl)-2-\{(2-(3-nitrophenyl)-4-oxo-1,3-thiazolidin-3-yl)amin\}acetamide)

Compound S₂(0.15g ,0.001mol)and mercaptoacetic acid (0.001mol) was heated under reflux for 8hrs . The reaction mixture was neutralized with 10% sodium carbonate solution .The solid was filtered off, washed with water, dried and recrystallized from toluene The physical properties are listed in table 2.

**Preparation of compound( S₅)** [7]
(1-(1,3-benzothiazol-2-yl)-3-(4-chlorophenyl)thiourea)

A mixture of compound S₁ (1.5g , 0.01mol) and p-chloro phenyl isothiocyanate (1.7g ,0.01mol) in (50ml) dry dioxan was refluxed for 7hrs. The reaction mixture was concentrated and the obtained solid was filtered off, dried and recrystallized from ethanol-dioxan mixture . The physical properties are listed in table 2.

**Preparation of compound (S₆)** [7]
((2E)-3-(1,3-benzothiazol-2-yl)-2-\{(4-chlorophenyl)imino\}-1,3-thiazolidin-4-one)

A mixture of compound (S₅) (0.32g , 0.001mol) and chloro acetic acid (0.094g, 0.001mol) in glacial acetic acid was refluxed for 3hrs and then cooled . The formed solid filtered off, dried and recrystallized from dioxan . The physical properties are listed in table 2.

**2.2.7 Preparation of compounds ( S₇a-b)** [8]
(2-(1,3-benzothiazol-2-ylcarbamoyl)benzoic acid) \&( 4-(1,3-benzothiazol-2-ylamino)-4-oxobutanoic acid)

A mixture of compound (S₁) (0.76g , 0.005mol) and anhydride (0.005mol) in glacial acetic acid was refluxed for 3hrs and then cooled . The formed solid filtered off, dried and recrystallized from dioxan . The physical properties are listed in table 2.

**2.2.8 Preparation of compounds (S₈a-b)** [9]
(\(N\)-(1,3-benzothiazol-2-yl)-2-(2,7a-dihydro-1H-benzimidazol-2-yl)benzamide) \& \((N\)-(1,3-benzothiazol-2-yl)-3-(2,7a-dihydro-1H-benzimidazol-2-yl)propanamide)

Compounds (S₇a-b) (0.005mol) and o-phenylenediamine (0.005mol) were dissolved in absolute ethanol , the reaction mixture was refluxed for 12hrs. It was cooled and poured onto (30ml) ice cold water containing (1ml) of conc. HCl .The precipitate which was allowed to settle down for 1hr at room temperature was filtered off , dried and recrystallized from ethanol . The physical properties are listed in table 2.
2.2.9 Preparation of compound (S₉) [10]
(N-(1,3-benzothiazol-2-yl)-2-chloroacetamide)
To a solution of compound (S₉) (2.5 g, 0.016mol) in (30ml) glacial acetic acid, chloroacetyl chloride (3.7g, 0.032mol) was added dropwise with constant stirring. The reaction mixture was refluxed for 5 hrs then it was powered onto crushed ice. The precipitated solid that obtained was filtered off, washed with cold water, dried and recrystallized from aqueous ethanol. The physical properties are listed in table 2.

2.2.10 Preparation of compounds (S₁₀a-e) [11]
A mixture of compound (S₉) (0.001mol) and amines (0.001mol) in DMF in presence of (0.2g) sodium carbonate was refluxed for 10hrs, then it was poured onto ice-cold water and stirred for 30 minutes. The reaction mixture was filtered and the precipitated solid was dried and recrystallized from ethanol. The physical properties are listed in table 2.
Table 1: the structure & the scientific name for compounds S<sub>10a-e</sub>

<table>
<thead>
<tr>
<th>Comp. No.</th>
<th>Comp. Structure</th>
<th>Comp. Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>S&lt;sub&gt;10a&lt;/sub&gt;</td>
<td><img src="image" alt="Structure S&lt;sub&gt;10a&lt;/sub&gt;" /></td>
<td>N-(benzo[d]thiazol-2-yl)-2-((3-(5-(4-hydroxyphenyl)-1-phenyl-4,5-dihydro-1H-pyrazol-3-yl)phenyl)(amino) acetamide</td>
</tr>
<tr>
<td>S&lt;sub&gt;10b&lt;/sub&gt;</td>
<td><img src="image" alt="Structure S&lt;sub&gt;10b&lt;/sub&gt;" /></td>
<td>N-(1,3-benzothiazol-2-yl)-2-[(4-nitrophenyl)(phenyl)(amino)acetamide</td>
</tr>
<tr>
<td>S&lt;sub&gt;10c&lt;/sub&gt;</td>
<td><img src="image" alt="Structure S&lt;sub&gt;10c&lt;/sub&gt;" /></td>
<td>N-(1,3-benzothiazol-2-yl)-2-[[4-methylphenyl)sulfonyl]amino}acetamide</td>
</tr>
<tr>
<td>S&lt;sub&gt;10d&lt;/sub&gt;</td>
<td><img src="image" alt="Structure S&lt;sub&gt;10d&lt;/sub&gt;" /></td>
<td>N-(1,3-benzothiazol-2-yl)-2-(2-phenylhydrazinyl)acetamide</td>
</tr>
<tr>
<td>S&lt;sub&gt;10e&lt;/sub&gt;</td>
<td><img src="image" alt="Structure S&lt;sub&gt;10e&lt;/sub&gt;" /></td>
<td>N-(1,3-benzothiazol-2-yl)-2-hydrazinylacetamide</td>
</tr>
</tbody>
</table>

RESULTS AND DISCUTION

2-aminobenzothiazole was prepared by condensation reaction between equimolar quantities of aniline and ammonium thiocyanate in presence of bromine as a catalyst [3]. The structure of this compound is confirmed by its spectral data. FTIR spectrum (Fig. No. 1) shows appearance of doublet band of (NH<sub>2</sub>) stretching frequency at 3394-3271 cm<sup>-1</sup> and (C= N) stretching frequency at 1641 cm<sup>-1</sup>. UV spectrum (Fig. No. 2) shows appearance of three absorption peaks, at 262 nm for (n-
π*) transitions, at 222 nm for (π - π*) of aromatic benzene ring and 205 nm for other (π - π*) transitions.

Schiff's base (S2) was prepared by condensation reaction between m-nitro benzaldehyde and 2-aminobenzothiazole. The structure of this compound is confirmed by its spectral data. FTIR spectrum (Fig. No. 3) shows appearance of (C=N) stretching frequency at about 1604 cm⁻¹, beside that it shows disappearance of doublet band of (NH₂) group of 2-aminobenzothiazole and disappearance of the stretching frequency that belongs to (C=O) group of m-nitro benzaldehyde. H¹NMR spectrum shows peaks at : 7.2-8.5 ppm (8H, Ar-H), 8.6 ppm (1H, CH=N). UV spectrum shows appearance of three absorption peaks, at 345 nm for (n - π*) transitions, at 260 nm for (π - π*) of aromatic benzene ring and at 217 nm for other (π - π*).

Reaction of Schiff's base with succinic anhydride leads to prepare compound S3. The structure of this compound is confirmed by its spectral data. FTIR spectrum shows appearance of (C=O) stretching frequency at 1693 cm⁻¹ and disappearance of (C=N) stretching frequency that belongs to Schiff's base. UV spectrum shows appearance of three absorption peaks, at 346 nm for (n - π*) transitions, at 263 nm for (π - π*) transitions of aromatic benzene ring and at 214 nm for other (π - π*).

Compound S₄ was prepared through the condensation between 2-aminobenzothiazole and mercaptoacetic acid. The structure of this compound is confirmed by its spectral data. FTIR spectrum shows appearance of (C=O) stretching frequency at 1705 cm⁻¹. UV spectrum shows appearance of two absorption peaks, at 259 nm for (n - π*) transitions, at 210 nm for (π - π*) transitions of aromatic benzene ring.

2-aminobenzothiazole reacts with 4-chloro phenyl isothiocyanate in dry dioxan to produce thiourea derivative (compound S₅). The structure of this compound is confirmed by spectral data. FTIR spectrum (Fig. No. 4) shows appearance of singlet band of (N=H) stretching frequency at 3171 cm⁻¹ instead of the doublet band of (NH₂) group, it also shows appearance of (C=S) stretching frequency at 1188 cm⁻¹. UV spectrum shows appearance of three absorption peaks, at 342 nm for (n - π*) transitions, at 315 nm for (π - π*) transitions of aromatic benzene ring and at 216 nm for other (π - π*) transitions.

Condensation reaction between compound S₅ and chloroacetic acid produces compound S₆ through ring closure reaction. The structure of this compound is confirmed by its spectral data. FTIR spectrum shows appearance of (C=O) stretching frequency at 1728 cm⁻¹ and (C-S) stretching frequency at 1141 cm⁻¹ and disappearance the stretching frequencies for (N-H) group and (C=S) group. H¹NMR spectrum (Fig. No. 6) shows : 4.2 ppm (2H, CH₂), 7.3-7.9 ppm (8H, Ar-H). UV spectrum
shows appearance of three absorption peaks, at 320 nm for \((n - \pi^*)\) transitions, at 261 nm for \((\pi - \pi^*)\) transitions of aromatic benzene ring and at 208 nm for other \((\pi - \pi^*)\) transitions.

2-aminobenzothiazole was acylated by succinic and phthalic anhydride in glacial acetic acid to form compounds \(S_{7a-b}\). The structures of these compounds are confirmed by their spectral data. FTIR spectrum shows appearance of \((O-H)\) stretching frequency at 3254 cm\(^{-1}\) for compound \(S_{7a}\) and at 3259 cm\(^{-1}\) for compound \(S_{7a}\). It also shows appearance of the stretching frequencies of \((C=O)\) of carboxylic group at 1693 cm\(^{-1}\) for compound \(S_{7a}\) and at 1734 cm\(^{-1}\) for compound \(S_{7b}\) while \((C=O)\) stretching frequency of amide group appears at 1630 cm\(^{-1}\) for compound \(S_{7a}\) and at 1695 cm\(^{-1}\) for compound \(S_{7b}\). UV spectrum shows appearance of two absorption peaks for each compound, the first at 273 nm of \((n - \pi^*)\) transitions for compound \(S_{7a}\) and at 274 nm for compound \(S_{7b}\). The second at 244 nm of \((\pi - \pi^*)\) transitions for compound \(S_{7a}\) and at 220 nm for compound \(S_{7b}\).

The condensation reaction between compounds \((S_{7a-b})\) and \(o\)-phenylene diamine produces benzimidazole derivatives (compounds \(S_{8a-b}\)). The structures of these compounds are confirmed by their spectral data. FTIR spectrum shows disappearance of stretching frequencies of each \((O-H)\) group and \((C=O)\) group that belong to carboxylic groups in the precursor compounds, while \((C=O)\) stretching frequencies that belong to amide groups are apparent at 1656 cm\(^{-1}\) for compound \(S_{8a}\) and at 1648 cm\(^{-1}\) for compound \(S_{8b}\). UV spectrum shows appearance of three absorption peaks for each compound, at 297 nm of \((n - \pi^*)\) transitions for compound \(S_{8a}\) and at 287 nm for compound \(S_{8b}\), at 274 nm for \((\pi - \pi^*)\) transitions of aromatic benzene ring for compound \(S_{7a}\) and at 220 nm for the same moiety for compound \(S_{8b}\) and at 206 nm of other \((\pi - \pi^*)\) transitions for compound \(S_{8a}\) and at 205 nm for compound \(S_{8b}\).

2-aminobenzothiazole on reaction with chloro acetylchloride gives amide derivative (compound \(S_9\)) through \(S_{N2}\) mechanism. The structure of this compound is confirmed by its spectral data. FTIR spectrum shows appearance of singlet band of \((N-H)\) stretching frequency at 3178 cm\(^{-1}\) instead of the doublet band of \((NH_2)\) group which belongs to (2-aminobenzothiazole), it also shows appearance of \((C=O)\) stretching frequency at 1701 cm\(^{-1}\). UV spectrum shows appearance of three absorption peaks, at 297 nm for \((n - \pi^*)\) transitions, at 274 nm for \((\pi - \pi^*)\) transitions of aromatic benzene ring and at 208 nm for other \((\pi - \pi^*)\) transitions.

The reaction of primary and secondary amines with chloroacetylated derivative of (2-aminobenzothiazole) proceeds through \(S_{N2}\) mechanism and produces different amide derivatives (\(S_{10a-c}\)). The structures of these compounds are confirmed by their spectral data. FTIR spectrum for
compound $S_{10e}$ (Fig. No. 5) shows appearance of doublet band for $(\text{NH}_2)$ stretching frequency at 3201 (asymmetric), 3128 cm$^{-1}$ (asymmetric) and a singlet band for (NH) stretching frequency at 3317 cm$^{-1}$, it also shows appearance of (C=O) stretching frequency at 1649 cm$^{-1}$. UV spectrum for compound $S_{10e}$ shows appearance of two absorption peaks, at 340 nm for $(n-\pi^*)$ transitions and at 221 nm for $(\pi-\pi^*)$ transitions.

Table-2 The physical properties for the synthesized compounds

<table>
<thead>
<tr>
<th>Comp. No.</th>
<th>Color</th>
<th>m.p°C</th>
<th>Yield%</th>
<th>Mol. Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_1$</td>
<td>pale yellow</td>
<td>117-120</td>
<td>55</td>
<td>$C_7H_6N_2S$</td>
</tr>
<tr>
<td>$S_2$</td>
<td>Yellowish green</td>
<td>179-182</td>
<td>77</td>
<td>$C_{14}H_9N_3SO_2$</td>
</tr>
<tr>
<td>$S_3$</td>
<td>yellow</td>
<td>158-161</td>
<td>56</td>
<td>$C_{18}H_{13}N_3SO_3$</td>
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<tr>
<td>$S_4$</td>
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<td>150-153</td>
<td>60</td>
<td>$C_{16}H_{10}N_3S_2O_3$</td>
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<tr>
<td>$S_5$</td>
<td>pale white</td>
<td>207-210</td>
<td>53</td>
<td>$C_{14}H_{10}N_3Cl$</td>
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<tr>
<td>$S_6$</td>
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<td>240-243</td>
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<td>$C_{16}H_{12}N_3S_2OCl$</td>
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<tr>
<td>$S_{7a}$</td>
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<td>$C_{11}H_{10}N_2SO_3$</td>
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<td>$S_{7b}$</td>
<td>White</td>
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<td>$C_{17}H_{10}N_2SO_3$</td>
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<td>$S_{8a}$</td>
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<td>$C_{17}H_{14}N_4SO$</td>
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<td>$S_{8b}$</td>
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<td>$S_{10a}$</td>
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<td>$C_9H_{10}N_4SO$</td>
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Table 3 The spectral data for the synthesized compounds

<table>
<thead>
<tr>
<th>Comp No.</th>
<th>UV (EtOH)</th>
<th>Characteristic bands of FT-IR ( cm⁻¹, KBr disk )</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>λ max</td>
<td>v (N-H) cm⁻¹</td>
</tr>
<tr>
<td>S₁</td>
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<td>(NH₃) =3394-3271</td>
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Synthesis and Characterization of Some New 2-Aminobenzothiazole Derivatives

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Figure-1: FTIR Spectrum for compound $S_1$

Figure-2: UV Spectrum for compound $S_1$
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Figure-3: FTIR Spectrum for compound S_2

Figure-4: FTIR Spectrum for compound S_5
Figure-5: FTIR Spectrum for compound S_{10e}

Figure-6: H$^1$NMR Spectrum for compound S_{66}
REFRENCES