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Synthesis and Characterization of 4-Phenyl Substituted 2-Aminothiazoles Coupling Intermediate for Organic Synthesis

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Abstract: A series of 4-aryl-2-aminothiazole were synthesized with an objective to develop novel and potent coupling components of synthetic origin as well as in dye synthesis. The required derivatives of 4-phenylsubstituted-2-aminothiazole were synthesized via a multicomponent condensation between thiourea, acetophenone and iodine. The intermediates were obtained using acetophenone and various substituted aldehydes to synthesize the intermediates which on cyclization with sulphur yielded the final products. Synthesized compounds were purified, characterized using UV-Visible Spectrophotometer, Infra–red, GC-MS Spectrometer and NMR. They were also evaluated for their spectra properties. All the synthesized intermediates exhibited moderate to significant properties. They were found to possess good coupling properties as well as high degree of brightness and a colour deepening effect compared to other heterocyclic couplers obtained from aniline and anthraquinone dyes.

Keywords: 4-Phenyl-2-aminothiazole; green synthesis; acetophenone; heterocyclic compounds; spectral analysis

1. Introduction

Thiazole nucleus has been established as the potential entity in the largely growing chemical world of heterocyclic compounds possessing promising characteristics.[1,2] Substituted thiazoles and their biheterocycles have received considerable attention during last two decades as they are endowed with wide range of therapeutic properties.[3] A number of thiazole derivatives have been reported to possess significant and diverse biological activities such as antimicrobial, analgesic, anti-inflammatory, antioxidant and antiallergic activities.[4-9] For instance, Isha et al.,[4] prepared various novel thiazole derivatives and used for its antimicrobial activity. Similarly, various pesticides possessing a thiazole nucleus are well known in agriculture research.[10] Large numbers of thiazole derivatives have emerged as active pharmaceutical ingredients in several drugs for their potential anti-inflammatory, anti-tumour, anti-hyperlipidemic, anti-hypertensive and several other biological properties.[11,12] Besides, thiazoles are also synthetic intermediates and common substructures in numerous biologically active compounds.[13] Thiazole derived compounds are used in various fields in cosmetic industry, production of light-emitting diodes (LEDs), light harvesting, medicinal- and agro-chemistries, catalysis, corrosion protection, photochromes and molecular switches or nonlinear optical materials. Although there is very less number of research articles on coordination chemistry of this class of polyfunctional heterocyclic ligands published, the compounds (ligands) itself played crucial role in wide range of disciplines. Thus, the thiazole nucleus has been much studied in the field of organic and medicinal chemistry.[14,15]

In continuation to these efforts and with an objective to develop potent heterocyclic intermediates of synthetic origin, it was decided to synthesize certain amino thiazole derivatives and evaluate them for their coupling potential in dye synthesis. The synthesis from acetophenone, thiourea and iodine as well as the absorption spectra of these intermediates are discussed. In addition, the compounds were confirmed by using their physical characteristics such as physical appearance and melting point. Fig. 1 shows the molecular structure of 2-amino-4-arylthiazole derivatives.

2. Experimental Section

2.1. Materials and Characterization

All the chemicals for the synthesis were purchased from different approved vendors like Aldrich, Dayang chemicals, Ievakluga, Synquest laboratory and King scientific and all chemicals were of laboratory grade.

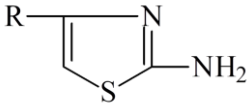


Fig. 1. Molecular structure of 2-amino-4-arylthiazole derivatives.

2.2. Synthesis of 2-amino-4-phenyl thiazole derivatives (AII)

2.2.1. Preparation of the 2-amino-4-phenylthiazole, AII_1

Resublimed iodine (7.6g, 0.03mol) was added to the slurry of the corresponding ketone (acetophenone and its para substituents - chloro, 2,4-dichloro, 3,4-dibromo, methyl, and bromo) (0.03mol) and thiourea (4.5g, 0.06mol) in toluene and the mixture was heated in an oil bath at 130°C - 140°C overnight. After cooling, the reaction mixture was diluted with distilled water (50ml) and heated to dissolve most of the solid, again cooled to ambient temperature. The aqueous solution was treated with 25% aqueous ammonium hydroxide (to pH 9-10). The precipitated thiazole was filtered, washed successively with water, collected and purified by crystallization from hot ethanol (Scheme 1).

2.2.2. Preparation of the 2- amino-4-(p-chlorophenyl) thiazole, AII_2

The procedure is the same as in 3.1 above, except that the resublime iodine and thiourea were reacted with para-chloroacetophenone in toluene. The mixture was heated in an oil bath at 130-140°C overnight. After cooling, the reaction mixture was diluted with distilled water (50ml) and heated to dissolve most of the solid, again cooled to ambient temperature. The aqueous solution containing the hydro iodide was treated with 25% aqueous ammonium hydroxide (to pH 9-10). The precipitated thiazole was filtered, washed successively with water, collected and purified by crystallization from aqueous ethanol (Kamaljit et al, 2002).

2.2.3. Preparation of the 2- amino-4-(p-aminophenyl) thiazole derivative, AII_3

The procedure is the same as in 3.1 above, except that the resublime iodine and thiourea were reacted with para-amino acetophenone in toluene and the mixture was heated in an oil bath at 130-140°C overnight.

2.2.4. Preparation of the 2- amino-4-p(2, 4-dichlorophenyl) thiazole derivative, AII_4

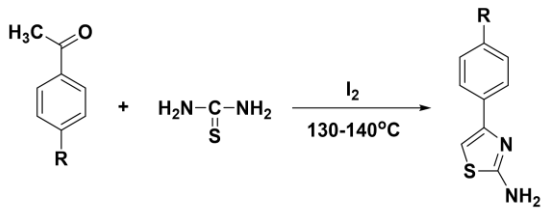
The procedure is the same as in 3.1 above, except that the resublime iodine and thiourea were reacted with 2, 4-dichloroacetophenone in toluene. The mixture was heated in an oil bath at 130-140°C overnight.

2.2.5. Preparation of the 2- amino-4-(p-bromophenyl) thiazole derivative, AII_5

The procedure is the same as in 3.1 above, except that the resublime iodine and thiourea were reacted with para-bromoacetophenone in toluene and the mixture was heated in an oil bath at 130-140°C overnight.

2.2.6. Preparation of the 2- amino-4-p(2,4-dibromophenyl) thiazole derivative, AII_6

The procedure is the same as in 3.1 above, except that the resublime iodine and thiourea were reacted with 2, 4-dibromoacetophenone in toluene. The mixture was heated in an oil bath at 130-140°C overnight.



R = $-C_6H_5$, $-C_6H_4Cl$, $-C_6H_6N$, $-C_6H_3Cl_2$, $-C_6H_4Br$, OR $-C_6H_3Br_2$

Scheme 1: Synthesis of 2-amino-4p (phenyl) thiazole derivatives (AII_1 – AII_6); R = (AII_1) - C_6H_5; (AII_2) - C_6H_4Cl; (AII_3) - C_6H_6N; (AII_4) - $C_6H_3Cl_2$; (AII_5) - C_6H_4Br; (AII_6) - $C_6H_3Br_2$.

2.3. Recrystallization and thin layer chromatograph

All aminothiazole derivatives were purified by 3- 4 recrystallizations from hot ethanol. A known weight of the intermediate was dissolved in small quantity of ethanol and heated up. It was then filtered off using a Buchner funnel with a suction pump. The crystals were collected, washed severally with water and dried. After the recrystallization, the purity of each compound was checked by spotting on a thin layer chromatography plate.

2.4. Characterization

Completion of reaction were confirmed using physical constant determination (Sharp or narrow melting ranges). Melting points were determined using melting point apparatus in open capillaries and are uncorrected. Further the compounds synthesized were proceeded for TLC wherein single spots (1st TLC run) were observed, indicating completion of reaction. After work-up was completed, (unreacted starting materials were removed by washing with ether), the products were subjected to purification by recrystallization process. Again TLC was run to find out exact Rf value. TLC plates used for final recrystallized product were pre-coated silica gel G plates. Solvent systems were developed using trial and error method by use of appropriate solvents of different polarity until the use of diethyl ether: ethanol (75%: 25%) solvent mixture was established. The Visible absorption spectra were measured using CARY 630UV-Visible spectrophotometer. Model: Agilent Technology. The infra-red spectra were carried out on FTIR Nexus 670 spectrophotometer in KBr disc (Thermo Nicolet) and absorption bands are expressed in cm-1.Mass spectra were recorded on an Agilent technologies 6460 mass spectrometer using the electron spray ionization (ESI) technique. The data shows peaks at $[M+H]^+$ and $M+Na]^+$. H^+NMR and CNMR were recorded using TMS-tetramethylsilane as internal standard and chemical shifts are given in δ (ppm), while coupling constants values were in HZ. It should however be noted that chemical shifts can be highly dependent on solvent types, concentrations and temperature.

3. Results and Discussions

3.1. Confirmation of prepared 2-amino-4-phenylthiazole derivatives

In a mixture of acetophenone and thiourea in toluene, iodine was added drop wisely with shaking. The mixture heated on an oil bath overnight at 130-140°C was afforded 2-amino-4-aryl-thiazole (1).

Table 1. Structure of the prepared 2-amino-4-phenylthiazole derivatives.

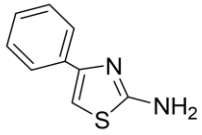
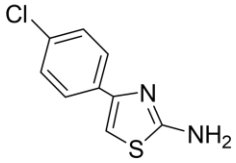
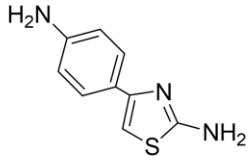
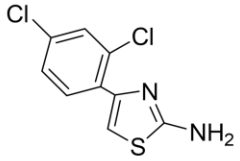
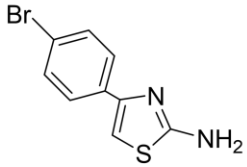
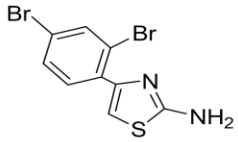
Sample code	Name and structure of the compounds
AII_1	 4-phenylthiazol-2-amine
AII_2	 4-(4-chlorophenyl)thiazol-2-amine
AII_3	 4-(4-aminophenyl)thiazol-2-amine
AII_4	 4-(2,4-dichlorophenyl)thiazol-2-amine
AII_5	 4-(4-bromophenyl)thiazol-2-amine
AII_6	 4-(2,4-dibromophenyl)thiazol-2-amine

Table 2. Physical characteristics of the prepared 2-amino-4-phenylthiazole derivatives

Compound code	Colour of crystals	Melting point (°C)	% yield
AII_1	Yellow	144-147	74
AII_2	Off white	173	85
AII_3	Yellow	187-188	52
AII_4	Brownish yellow	190-192	59
AII_5	Brownish yellow	185-187	61
AII_6	Pale yellow	199-202	67

Compound 1 on reaction with 4-chloroacetophenone give 1-{4-[(4-phenyl-1, 3-thiazol-2-yl) amino] phenyl} ethanone (2) which on further stepwise reaction with various aromatic aldehydes afforded various 2-amino-4-phenylthiazole derivatives. The primary structural difference within the series involves the nature of various substituted aldehydes. Table 1 shows the Structure of the prepared 2-amino-4-phenylthiazole derivatives (AII_1-AII_6). Table 2 shows the Physical characteristics of the prepared 2-amino-4-phenylthiazole derivatives.

Synthesized compounds were found to be crystalline in nature and easily soluble in ethyl acetate, benzene, DMSO and DMF but insoluble in hexane and toluene. With the help of analytical techniques such as melting point, UV-visible spectrophotometer and FTIR, GC- MS Spectrometer and NMR, the synthesized derivatives were characterized. These compounds showed absorption band for C-S stretching of thiazole ring between 674-745cm^{-1} and 1028- 1088 cm^{-1} for C-N. All the compounds showed absorption peaks for different kinds of functional groups at their respective regions. All of them were found to be in full consignment with assigned structures.

For Compound AII_1: Yellow crystals were obtained; it was purified by recrystallization in hot ethanol. Melting point 144-147°C, 74% yield which corresponds to literature values148°C, 80% yield (Prajapati, 2010). FTIR (KBr)/cm^{-1}):1624 (C=C), 847 (CH –Ar bend), 2283 (Ali C-H), 3435 (NH STR), 1483, 1532 (Ar C=C Str), 1340 (C-C), 1039 (C-N), 691 (C-S), 3134 (Ar-H).

For compound AII_2: Off white crystals were obtained. It was purified by recrystallization in hot ethanol. Melting point 173°C, 85% yield. FTIR (KBr)/cm^{-1}): 1621 (C=C), 827, 741 (CH –Ar bend, double), 2847 (Ali C-H), 3391 (NHSTR), 1569 (Ar C=C STR), 1088 (C-N), 1196 (C-H) 741 (C-S), 827 (C-Cl), 3119 (Ar-H).

For compound AII_3: A deep yellow crystal was obtained. It was purified by recrystallization in hot ethanol. Melting point: 187-188°C. Yield - 52%. FTIR (KBr)/cm^{-1}): 1610 (C=C), 831,738 (CH –Ar bend, double), 2776 (Ali C-H), 3410 (NH), 1490, 1427 (Ar C=C str), 1265 (C-C), 1043 (C-N), 685 (C-S), 3116 (Ar-H), 1539 (N-H Ali).

For compound AII_4: A brownish yellow crystal was obtained; it was purified by recrystallization in hot ethanol. Melting point 190-192°C, 59% yield .FTIR (KBr)/cm^{-1}):1630, 1684 (C=C Ali), 827, 764 (CH –Ar bend, double), 2922 (Ali C-H), 3309(NH), 1599 (NH band). 1520, 1580 (Ar C=C STR), 1632 (C-C), 784,723 (C-Cl), 1028 (C-N), 674 (C-S), 3116 (Ar-H), 2922 (C-H Ali), 1129, 961 (C-H).

For compound AII_5: A brownish yellow crystal was obtained. It was purified by recrystallization in hot ethanol. Melting point 185-187°C, 61% yield .FTIR (KBr)/cm^{-1}):1602 (C=C), 849, 771 (CH –Ar bend, double), 3432,(NH str), 1599 (NH band). 1159 (C=C Ar), 1028, 1326 (C-C), 1086 (C-N), 693 (C-S), 693 (C-Br), 3060 (Ar-H).

For compound AII_6: A pale yellow crystal was obtained, it was purified by recrystallization in hot ethanol. Melting point 199-202°C, 67% yield. FTIR (KBr)/cm^{-1}):1621 (C=C Ali), 823 (CH –Ar bend, double), 2847, 2948 (Ali C-H), 3384 (NHSTR), 1565 (NH band). 1490 (C=C Ar str), 1319 (C-C), 1069 (C-N), 741 (C-S), 741 (C-Br), 1189 (C-H Ar), 3116 (Ar- H).

3.2. NMR spectra of AII_{1-6} compounds

AII1 shows a singlet upfield at 2.41ppm attributed to a signal for C-H. Another singlet is seen at 4.046 ppm (slightly upfield at 4.046) attributed to N-H on the aminothiazole ring. The multiplets for the protons attached to the phenyl ring on the third position on the amino thiazole ring is seen downfield at 7.150 ppm with a coupling constant of 3.2 Hz. The NMR sepctra were recorded using $CDCl_3$ and TMS. Fig. 2 shows the ^{13}C NMR of AII1. ^{13}C NMR of AII1 showed chemical shifts values at 130, 134, 106 and 149 ppm attributed to aromatic carbon and also C=C present in thiazole ring.

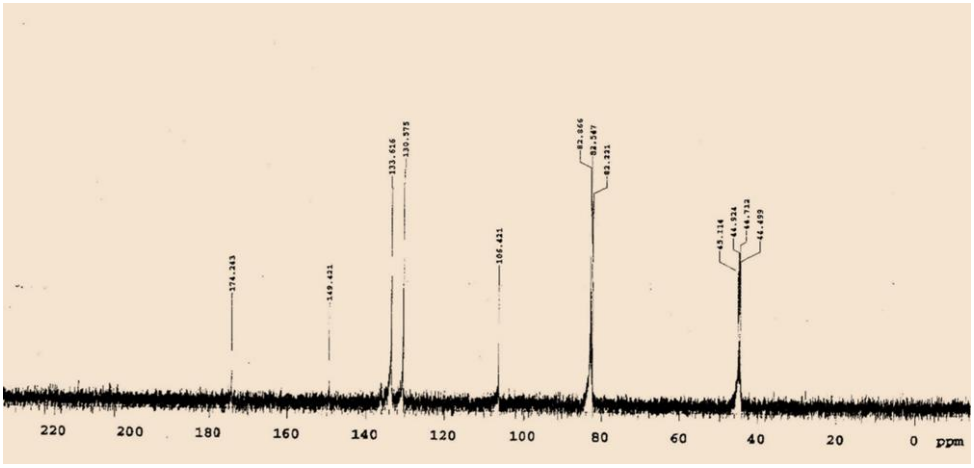


Fig. 2. ^{13}C NMR spectrum of **AII$_1$**.

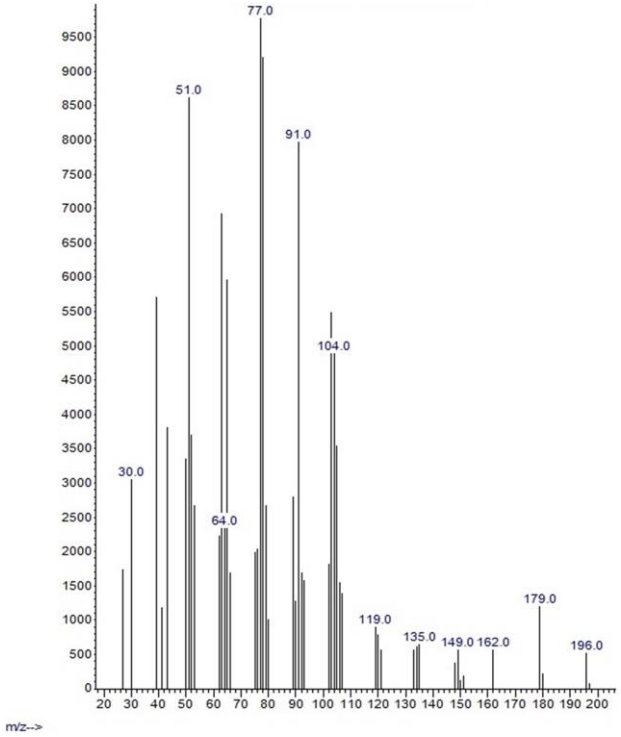


Fig. 3. Mass spectrum of **AII$_1$**.

The mass spectra data for the prepared 2-amino-4-phenylthiazole derivatives are presented in Table 3. The data showed relevant protonated peaks i.e the $(M + H)^+$ and the $(M +Na)^+$. The peaks from the intermediate AII_4 are observed at 244 and 267 m/z respective to the $(M+ H)^+$ and $(M+Na)^+$. Similarly, peaks for AII_1 and AII6, were seen at 173 and 196, 329 m/z corresponding to the $M+ H)^+$ and M+ Na)+ of AII_1 and $(M+ H)^+$ of AII_6. The mass spectra of AII_4 gave mass to charge ratio(m/z) of 55, 72, 91, 108, 135, 164, 187, 238 and parent 267 representing M and the corresponding positive fragments of….., loss of a chlorine atom (Cl_2), C_2HCl_2, $C_3H_2Cl_2$, $C_5H_5Cl_2$, M + loss of a C_6H_5, $C_8H_7N_2Cl_2S$, M+ Na)+.

The mass spectra of AII1 gave mass to charge ratio(m/z) of 30, 51, 64, 77,104, 119, 135, 149, 162, 179 and 195 representing M and the corresponding positive fragments of C-NH_2, CH_2N_2, $CSNH_2$, CH_2N_2S or tropylium C_6H_5, thiazole ring $C_3H_4N_2S$, $C_4H_4N_2S$, $C_5H_5N_2S$,$C_6H_6N_2$, $C_7H_7N_2S$, $C_8H_8N_2S$ and parent $C_9H_{10}N_2S$. The mass spectra of AII6 gave mass to charge ratio (m/z) of 61, 103, 129, 159, 199, 299, 329 and 359 representing the corresponding positive fragments of CH_2NS, $C_3H_3N_2S$ (thiazole ring), C_4H_3Br, C_6H_3Br, $C_9H_4N_2Br_2$, $C_9H_4N_2SBr_2$ and parent $(M+Na)^+$ 359.

3.3. Scope of the prepared AII$_{1-6}$ derivatives

So far, various aminothiazoles intermediates are prepared and utilized as potential candidate for various useful applications.[16-23] The aminothiazoles intermediates gave yellow colours of different shades. The dyes obtained from these intermediates gave bright colours ranging from orange, brown, majority of yellow colour.

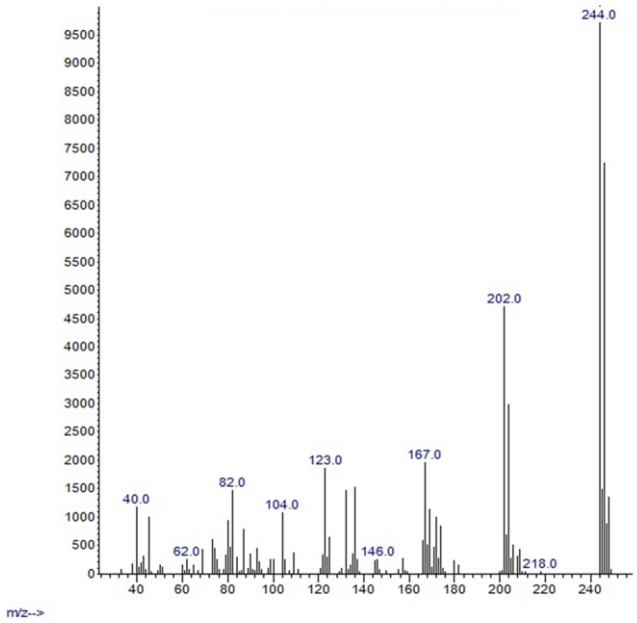


Fig. 4. Mass Spectrum for **AII$_4$**.

Table 3. Mass Spectra data of prepared 2-amino-4-phenylthiazole derivatives

Code	Molecular formula	Calculated molecular mass (m/z)	Experimental mass (m/z)
AII4	$C_9H_6Cl_2N_2S$	$(M+H)^+$ 245	244
AII1	$C_9H_8N_2S$	$(M+H)^+$ 176	173
AII6	$C_9H_6Br_2N_2S$	$(M+H)^+$ 329.8	329

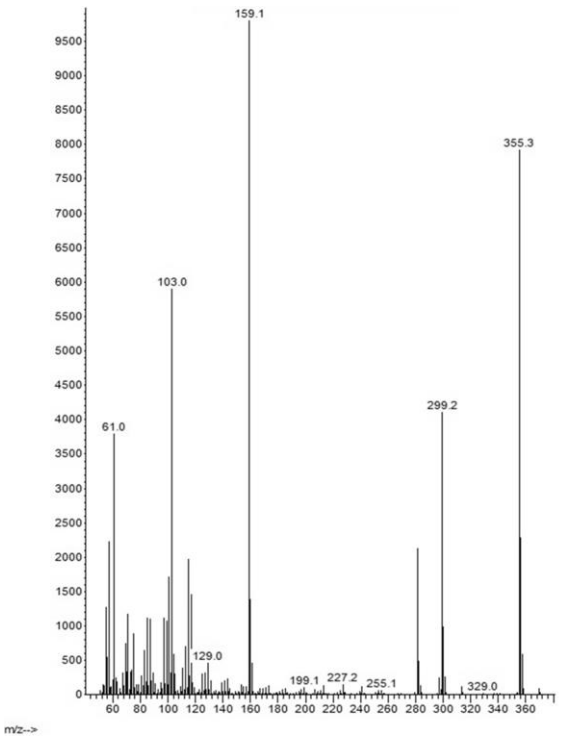


Fig. 5. Mass Spectrum for **AII$_6$**.

Derivative of 2-aminothiazole has a long history of use as heterocyclic diazo components for disperse dyes.23 Basically, thiazole nuclei have been established as potential entities in the large growing chemical world of heterocyclic compounds possessing promising coupling characteristics. In this regard, azo dyes based on heterocyclic amines have been developed, and the resultant dyes have higher tinctorial strength and give brighter dyeing than those derived from aniline-based diazo components. For instance, amino-substituted thiazole, isothiazole, thiophene, and pyrazole compounds afford very electronegative diazo components and, consequently, provide a pronounced bathochromic effect compared to the corresponding benzenoid compounds.17 In comparison with similar researchers on aminothiazole intermediates, they were also found to possess good coupling properties such as, a colour deepening effect and a high degree of brightness compared to other heterocyclic couplers. The dyes obtained from such intermediates possess high extinction coefficient and good absorption maximum values.

4. Conclusions

The analytical and other informational data, available in literature so far, have rendered thiazole significantly important class of heterocyclic compounds and their applications in ever challenging dye synthesis of various classifications. All the synthesized intermediates exhibited moderate to significant properties. They were found to possess good coupling properties as well as high degree of brightness and a colour deepening effect compared to other heterocyclic couplers obtained from aniline and anthraquinone dyes. This particular research study, in reference, would extend great deal of help to researchers in reckoning and determining the best and most productive, economical, suggestive and conclusive access various thiazoles of organic and synthetic importance superseding other coupling compounds of their class.

Conflicts of Interest

The authors declare no conflict of interest.

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