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PHOTOPHYSICAL PROPERTIES OF SOME BENZOXAZOLE AND BENZOTHIAZOLE DERIVATIVES

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Abstract: The photophysical properties of the solution of benzoxazole (BNX_S) and benzothiazole (BNT_S) derivatives have been studied; the fluorescence properties have been studied in chloroform solvent, as well as, fluorescence quantum yield calculated using fluorescein as standard material. The changing of their color and UV-vis absorption spectra according to pH solution variation was studied in ethanol 95%. Laboratory prepared mixture of phosphorus acid/phosphorus pentoxide were used as catalyst.

***Keywords*:** benzoxazole, benzothiazole, 2,6-di (benzo[d]oxazol-2-yl) pyridine, 2,6-di(benzo[d]thiazol-2-yl) pyridine

Introduction

The heterocyclic compounds are compounds that possess a cyclic structure with at least two different kinds of heteroatoms in their ring.[1] Oxygen heterocyclic compounds form an important class in organic

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chemistry. Especially, those fused to benzene ring.[2] Furthermore, compounds with thiazole ring have great value in medicinal field. Besides to their responsibility for variety of biological activities.[3] Benzoxazole ring is one of the most common heterocyclic in medicinal chemistry, it has remarkable biological and photochromic activities like anti-hypertensive, antimicrobial, antiviral, antifungal, anticancer like *NSC-693638 and 3-Substituted 2-(4-aminophenyl)benzothiazoles* figure 1, antihistamine, anti-helminthic, anti-parasitic, anticoagulant, in addition to their herbicidal and insecticidal activities.[4-9]

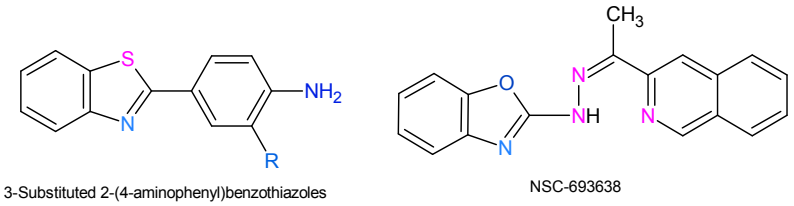


Figure 1. Chemical structure of NSC-693638 and 3-Substituted 2-(4-aminophenyl) benzothiazoles.

(BNT_S) have a vital role in the pharmaceutical industry.[10] Benzothiazole nucleus constitutes the active part of several biologically active compounds, including antifungal, antibacterial, antimicrobial, anticancer, antitumor, anti-inflammatory and anti-oxidant. In addition to their bioactivity, (BNT_S) find use in various branches of chemical research. For instance, in polymer chemistry, organic optoelectronic materials, dyes, ligands for phosphorescent complexes and pH indicators.[11-15] Industrially, (BNT_S) is used in vulcanization of industrial rubber and corrosion inhibition.[16,17] It have been noticed that (BNX_S) and (BNT_S) have combined applications like pharmaceutical uses, CO_2 storage, catalytic applications.[18]

(BNX_S) have gained special attention because of their optical applications like photoluminescents, whitening agents and laser dye. Furthermore, metal complexes of their hydroxyl-substituted derivatives is used as luminescent materials for Organic Light-Emitting Diodes ($OLED_S$), also, as sensors for the metals detection.[19-21] Excited-State Intramolecular Proton Transfer (ESIPT) is photoreaction frequently observed in organic bifunctional molecules, which contain an H-bond close enough to an acceptor group. Such as, molecules possess phenolic –OH group bonded in hydrogen nature with nearby –N= group.[22] Benzothiazole molecules show a sizable reorganization consisting of keto-enol phototautomerization occurrence, which sees the migration of proton from the hydrogen bond donor to the hydrogen bond acceptor, leading to radiative keto isomer in the excited-state.[23] Herein, we report a synthesis of six compound of (BNX_S) and (BNT_S) derivatives and studying their photophysical properties.

Results and Discussion

P_4O_{10} is a good dehydrating agent, that can be used to remove water from the phosphoric acid.[24] Here, (BNX_S) and (BNT_S) derivatives have been synthesized[25] using laboratory prepared catalyst of phosphoric acid and phosphorus pentoxide.

1. Evaluation photophysical properties

1.1. UV-vis properties

UV-vis spectra at studied pH scale (1, 4, 7, 10, 13) were recorded, and the obtained spectra were represented in figure 2.

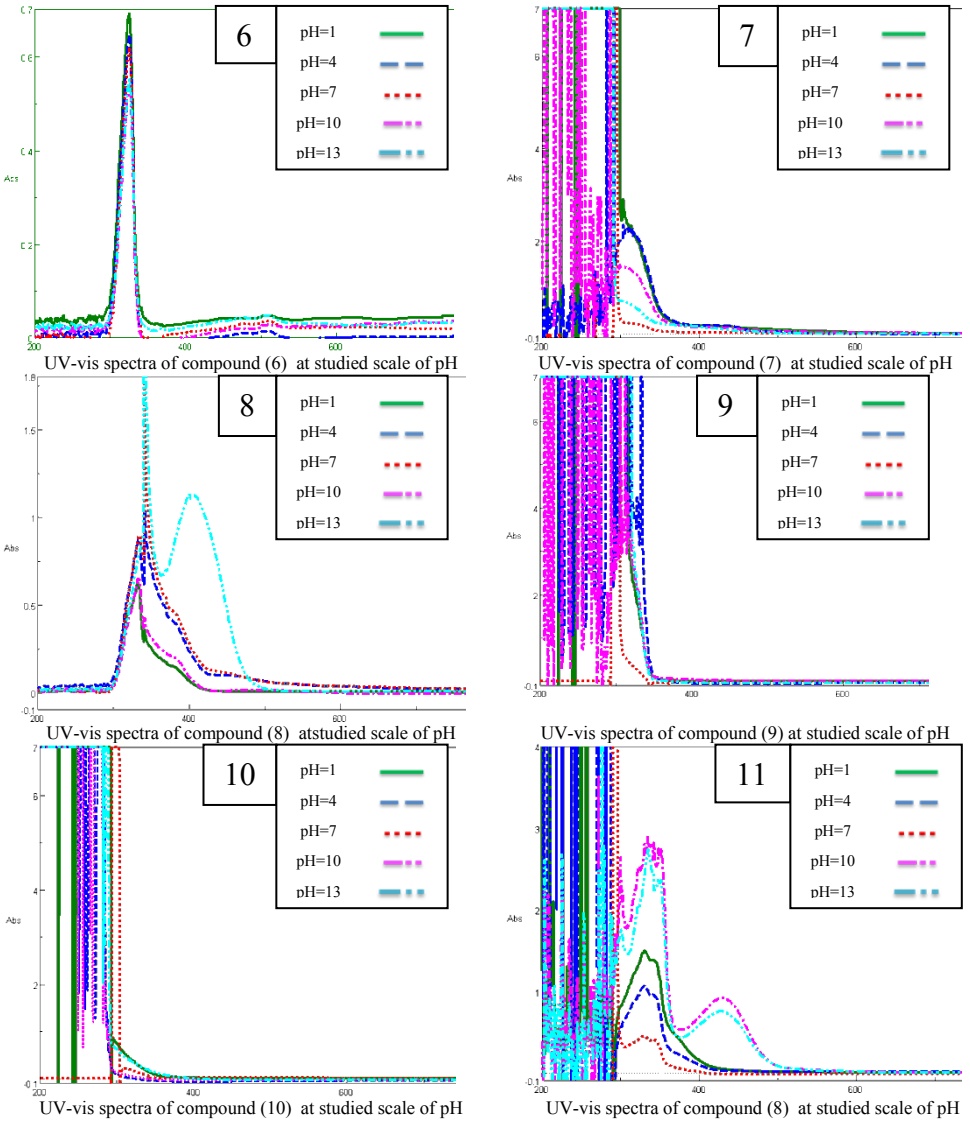
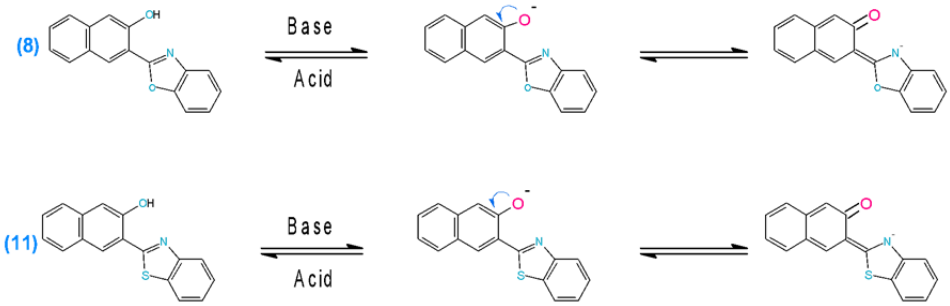


Figure 2. UV-vis spectra of compounds 6, 7, 8, 9, 10 and 11 at studied scale of pH.

Figure (2) shows that no important transition in spectra concerning compounds **6**, **7**, **9** and **10**, whereas, new absorption band appears in spectra of **8** compound at pH > 10 and so 11 at pH ≥ 10. The variance in spectrum is accompanied with changing from transparent toward yellow color relating to compounds **8** and **11**. However, this occurrence is unnoticeable at other compounds. This phenomenon can be attributed to the existence of hydroxyl

group, which stay suppressed in acidic media. In contrary, basic media excites protons' migration by the effect of light, from oxygen in naphthalene ring to nitrogen in (BNX_S) and (BNT_S) ring, changing electronic transition $\pi \rightarrow \pi^*$ toward red shift accompanied with keto-enol tautomerism as shown in scheme 1.



Scheme 1. Keto-enol tautomerism of compounds **8** and **11**

1.2. Fluorescence properties

Fluorescence spectra for every compound and standard have been recorded at their special excitation wavelength (λ_{abs}) by spectrofluorometer (Shimadzo RF-530 1PC). Quantum yield computed from the equation $\Phi = \Phi^{\circ} \frac{FA_0}{F_0 A} \frac{n^2}{n_0^2}$ where; F= fluorescence intensity, A = absorbance at excitation wavelength, n = refractive index and the subscript 0 stand for standard. [26] Parameters and result of fluorescence study were outlined in table 1.

Table 1 shows that all prepared compounds have fluorescence property except compound **9**. All compounds have good stake's shift which reaches 194 (nm) at compound **11**, whereas, quantum yield shows a little value in general, and reaches 0.39 % at the same compound.

Table 1. Fluorescence properties of prepared compounds

Compound	λ_{abs}(nm)	λ_{em}(nm)	Stake's shift (nm)	Quantum yield
6	334	468	134	0.022
7	310	367	57	0.077
8	318	389	71	0.01
9	316	---	---	---
10	329	436	107	0.019
11	331	525	194	0.39

2. Experimental Section

2.1. Catalyst preparation

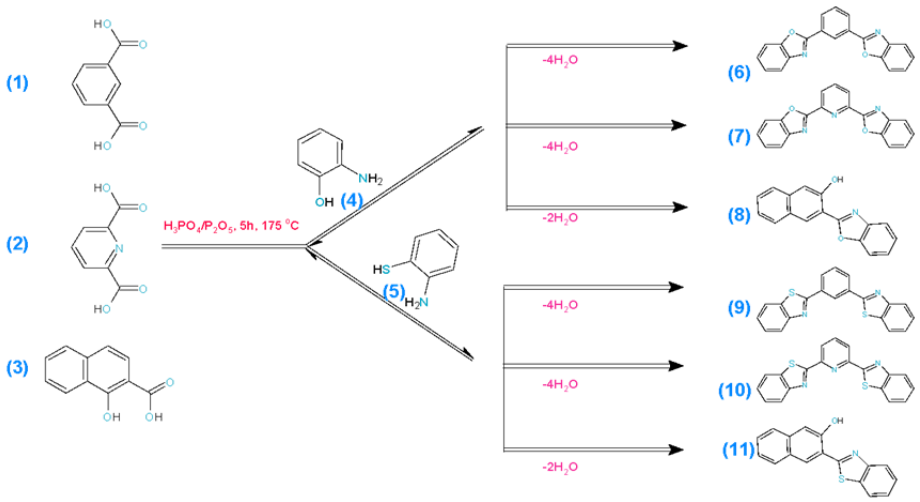
200 mL of phosphorous acid 85 % were put in 500 mL size flask and heated to 100 °C for 1 hour, then, 30 g of phosphorous pentoxide were added until the concentration of P_2O_5 reached 100% in the mixture, the temperature of mixture was risen to 300 °C for 30 min where the mixture became homogenous. The heating turned off and the mixture cooled to 100 °C until became viscous greenish liquid, preserved in closed can for subsequent reaction.

2.2. Preparation UV-vis and fluorescence solutions

A (95%) ethanolic solutions of compounds were prepared in concentration of $2 \cdot 10^{-4}$ mol/L, and UV-vis spectra at studied pH scale (1, 4, 7, 10, 13) have been recorded using spectrophotometer (Jasco V 630, Japan) between 200 and 800 nm with 2 min server scan velocity. Fluorescence solution were prepared in concentration of ($2 \cdot 10^{-4}$ mol/L) using chloroform as solvent and fluorescein as standard material, λ_{max} (λ_{abs}) of each compound was determined from UV-vis spectra.

General procedure for synthesis (BNX_S) and (BNT_S) derivatives

All synthesized products were prepared by the reaction of stoichiometric ratio between 2-aminophenol or 2-aminothiophenol and corresponding carboxylic acid to produce (BNX_S) or (BNT_S) derivatives respectively as scheme 2 shows.



Scheme 2. Preparation reactions of (BNX_S) and ($BENT_S$) derivatives

2.3. Synthesis of 1,3-di(benzo[d]oxazol-2-yl)benzene 6

0.848 g (5 mmol) of Isophthalic acid 98 % and 1.114 g (10 mmol) of 2-aminophenol 98% were taken in 50 mL flask contain 10 mL of preheated catalyst H_3PO_4/P_2O_5. Then reaction temperature was risen to 175 °C for 5 hour. Then, the reaction stopped and reaction mixture poured into iced basin until the next day where filtered, naturalized with diluted solution of sodium bicarbonate until pH=7, then washed three times with water for removing traces of catalyst and bicarbonate. Product purified by column chromatography using chloroform: ethyl acetate (90:10) as mobile phase. The obtained product has yellowish white color. **mp** (°C) 216-217, yield (%) 77; **IR** (cm^{-1}) 3056 ($\nu_{Ar-H\ str}$), 1618 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm): 9.17 (s, 1H), 8.53 (d, J=21, 2H), 8.36 (d, J=24, 2H), 7.78 (m,

4H), 7.29 (m, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 162.08, 150.92, 142.08, 130.24, 129.63, 128.18, 126.64, 125.50, 124.79, 120.26, 110.73.

2.4. Synthesis of 2,6-di(benzo[d]oxazol-2-yl) pyridine 7

0.844 g (5 mmol) of 2,6-pyridine dicarboxylic acid 99% and 1.114 g (10 mmol) of 2-aminophenol 98% were taken in (50 mL) flask contain (10 mL) of preheated catalyst (H_3PO_4/P_2O_5). After 5 hour of reaction's beginning, the reaction stopped and reaction mixture processed. The pure product was eluted by column chromatography using chloroform: ethyl acetate (90:10) as a mobile phase. The obtained product has pink color. **mp** (°C) 270-271, yield (%) 24; **IR** (cm^{-1}) 3056 ($\nu_{Ar-H\ str}$), 1614 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm): 8.47 (d, J=8 Hz, 2H), 8.13 (d, J=8 Hz, 2H), 8.00 (m, 3H), 7.53 (ddd, J_1=8 Hz, J_2=4 Hz, J_3=1.2 Hz, 2H), 7.45 (ddd, J_1=8 Hz, J_2=4 Hz, J_3=1.2 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 168.62, 154.08, 151.16, 138.30, 136.28, 126.49, 125.99, 123.71, 122.12, 122.05.

2.5. Synthesis of 3-(benzo[d]oxazol-2-yl) naphthalen-2-ol 8

1.92 g (10 mmol) of 3-(2-hydroxynaphthoic) acid 98% and 1.114 g (10 mmol) of 2-aminophenol 98% were added into (50 mL) flask contain (10 mL) of preheated catalyst (H_3PO_4/P_2O_5). The reaction stopped 5 hour after its beginning and product eluted by column chromatography using mobile phase of chloroform: ethyl acetate (90:10). The gained product has slightly yellowish color. **mp** (°C) 154-155, yield (%) 85; **IR** (cm^{-1}) 3442 ($\nu_{O-H\ str}$), 3041 ($\nu_{Ar-H\ str}$), 1644 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm); 11.27 (s, 1H), 8.60 (s, 1H), 7.86 (d, J=8 Hz, 1H), 7.73 (dd, J_1=19 Hz, J_2= 8.4 Hz, 2H), 7.64(d, J=5.2 Hz, 1H), 7.48 (t, J=8 Hz, 1H), 7.41 (m, 3H) 7.34 (t, J= 8 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 162.52, 154.27, 149.34, 140.13, 136.73, 128.70, 128.63, 128.45, 127.49, 126.52, 125.82, 125.18, 124.04, 119.57, 112.67, 111.65, 110.73.

2.6. Synthesis of 1,3-di(benzo[d]thiazol-2-yl)benzene 9

0.848 g (5 mmol) of isophathlic acid 98% and 1.28 g (10 mmol) of 2-aminothiophenol 98 % were added into (50 mL) flask contain (10 mL) of preheated catalyst (H_3PO_4/P_2O_5). The reaction stopped after 5 hours and reaction's mixture was processed and pure product eluted by column chromatography using mobile phase of (85:15) chloroform: n-hexane. The gained has slightly bluish white color. **mp** (°C) 163, yield (%) 61; **IR** (cm^{-1}) 3056 ($\nu_{Ar-H\ str}$), 1634, 1608 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm): 8.79 (t, J= 1.8 Hz, 1H), 8.21 (dd, J_1= 7.8 Hz, J_2= 1.8 Hz, 2H), 8.12 (dd, J_1= 8.1 Hz, J_2= 1.8 Hz, 2H), 7.93 (dd, J_1= 8 Hz, J_2= 1.3 Hz, 2H), 7.61 (t, J= 7.8 Hz, 1H), 7.51 (ddd, J_1= 8.3 Hz, J_2= 7.2 Hz, J_3= 1.3 Hz, 2H), 7.41 (ddd, J_1= 8.2 Hz, J_2= 7.2 Hz, J_3= 1.2 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 166.97, 153.97, 135.14, 134.5, 129.81, 129.79, 126.57, 126.55, 125.55, 123.44, 121.74.

2.7. Synthesis of 2,6-di(benzo[d]thiazol-2-yl)pyridine 10

0.844 g (5 mmol) of 2,6-pyridine dicarboxylic acid 99% and 1.28 g (10 mmol) of 2-aminothiophenol 98 % were added into (50 mL) flask contain (10 mL) of preheated catalyst (H_3PO_4/P_2O_5). After 5 hour of reaction's beginning, reaction's mixture was worked up and the pure product eluted by column chromatography using (85:15) chloroform: n-hexane as a mobile phase. The obtained product has pink color. **mp** (°C) 251-253, yield (%) 24; **IR** (cm^{-1}) 3056 ($\nu_{Ar-H\ str}$), 1614 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm): 8.55 (d, J= 7.9, 2H), 8.11 (t, J= 7.9, 1H), 7.87 (m, 2H), 7.73 (dd, J_1= 7.6 Hz, J_2= 1.8 Hz, 2H), 7.43 (m, 4H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 160.73, 151.23, 146.64, 141.50, 138.37, 126.46, 125.31, 125.18, 120.77, 111.65.

2.8. Synthesis of 3-(benzo[d]thiazol-2-yl) naphthalen-2-ol 11

1.92 g (10 mmol) of 3-(2-hydroxynaphthoic) acid 98% and 1.28 g (10 mmol) of 2-aminothiophenol 98 % were taken in (50 mL) flask contain (10 mL) of preheated catalyst (H_3PO_4/P_2O_5). The reaction lasted 5 hours, product eluted by column chromatography using mobile phase of Chloroform: n-hexane (85:15). The gained has reddish brown color. **mp** (°C) 173, yield (%) 29; **IR** (cm^{-1}) 3442 ($\nu_{O-H\ str}$), 3041 ($\nu_{Ar-H\ str}$), 1645 ($\nu_{C=N-\ str}$); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ (ppm): 8.27 (s, 1H), 8.03 (dd, J_1= 8 Hz, J_2= 1 Hz, 1H), 7.92 (dd, J_1= 8 Hz, J_2= 1 Hz, 1H), 7.81 (d, J= 8.2 Hz, 1H), 7.68 (d, J= 8.3 Hz, 1H), 7.52 (ddd, J_1= 8.3 Hz, J_2= 7.6 Hz, J_3= 1.3 Hz, 1H), 7.44 (m, 3H), 7.32 (ddd, J_1= 8.1 Hz, J_2= 6.8 Hz, J_3= 1.2 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ (ppm): 168.27, 158.04, 144.33, 136.24, 132.88, 129.36, 128.33, 128.25, 127.57, 126.89, 126.38, 126.00, 123.97, 122.50, 121.58, 119.04, 111.92.

3. Conclusions

Six derivatives of benzoxazole and benzothiazole have been synthesized using mixture of phosphoric acid /phosphorus pentoxide as catalyst. The synthesized derivatives were characterized by spectroscopic technique. The UV-vis spectra of compounds **8** and **11** show difference in their colors, which inspire us to use them as pH indicators in acid base titration within their color changing range. The fluorescence properties revealed a large Stake's shift reached at 194 nm for compound **11**, that may suggest a possible practical application especially as probe in medicinal field (if their photophysical properties in the range of blood pH are suitable for such applications). Prepared compounds have low quantum yields, the most promising one being registered by compound **11** (0.39).

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Supplementary Data

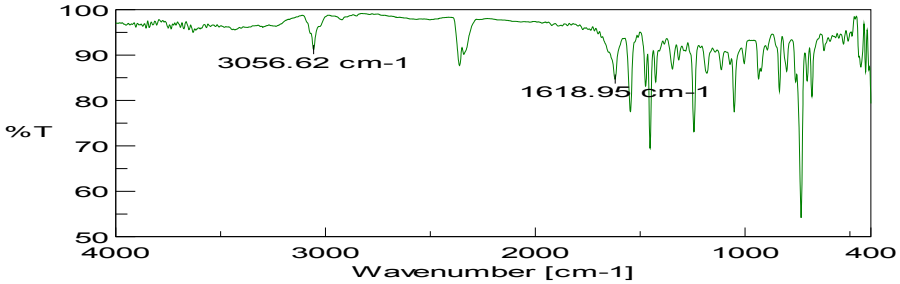

Figure 1. IR spectra 1,3-di(benzo[d]oxazol-2-yl)benzene (6).

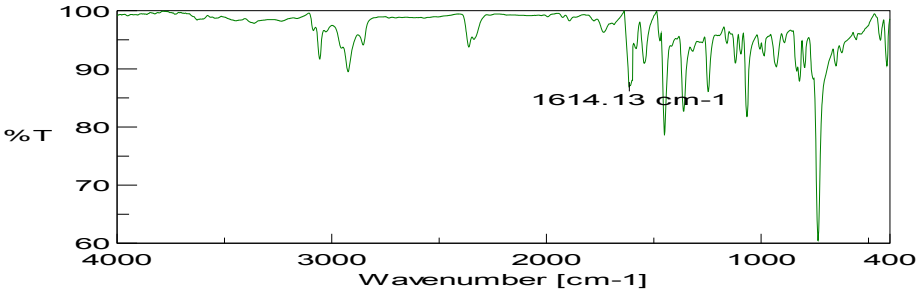

Figure 2. IR spectra of 2,6-di(benzo[d]oxazol-2-yl) pyridine (7).

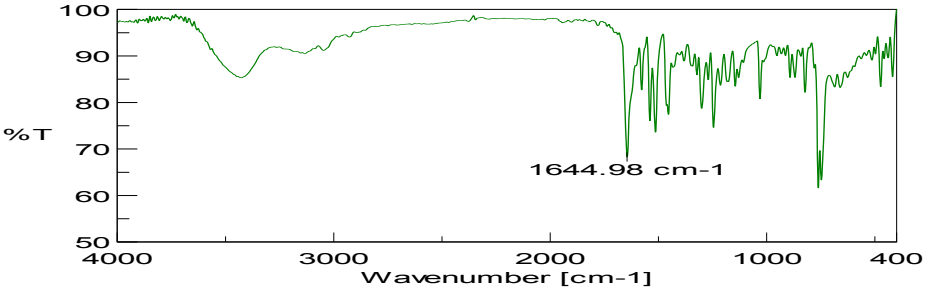

Figure 3. IR spectra of 3-(benzo[d]oxazol-2-yl) naphthalen-2-ol (8).

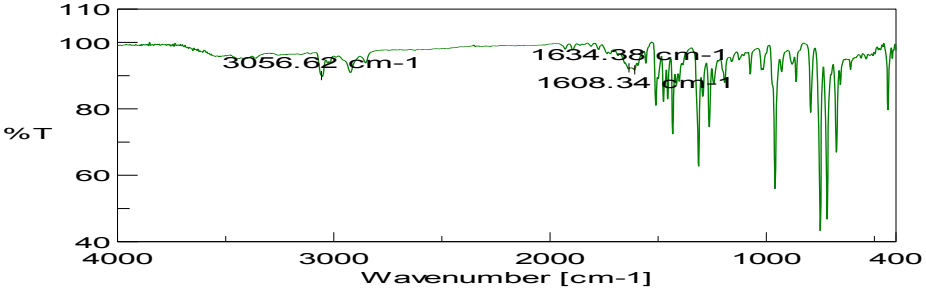

Figure 4. IR spectra 1,3-di(benzo[d]thiazol-2-yl)benzene (9).

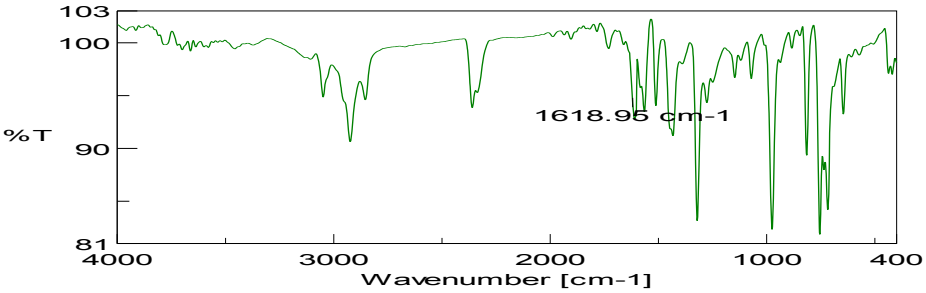

Figure 5. IR spectra of 2,6-di(benzo[d]thiazol-2-yl)pyridine (10).

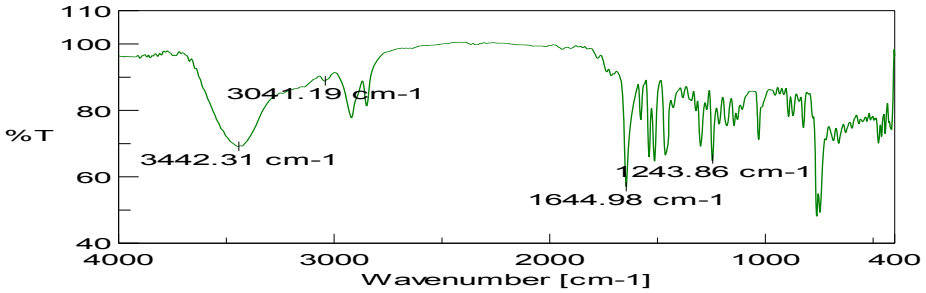

Figure 6. IR spectra of 3-(benzo[d]thiazol-2-yl) naphthalen-2-ol (11).

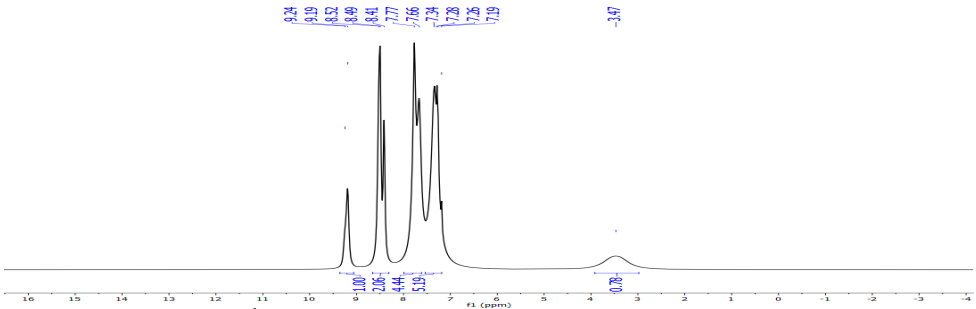

Figure 14. ^{1}H-NMR spectra of 1,3-di(benzo[d]oxazol-2-yl)benzene (6).

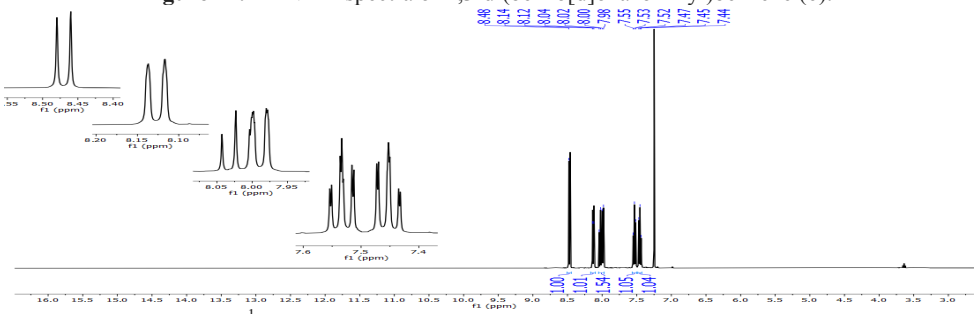

Figure 15. ^{1}H-NMR spectra of 2,6-di(benzo[d]oxazol-2-yl) pyridine (7).

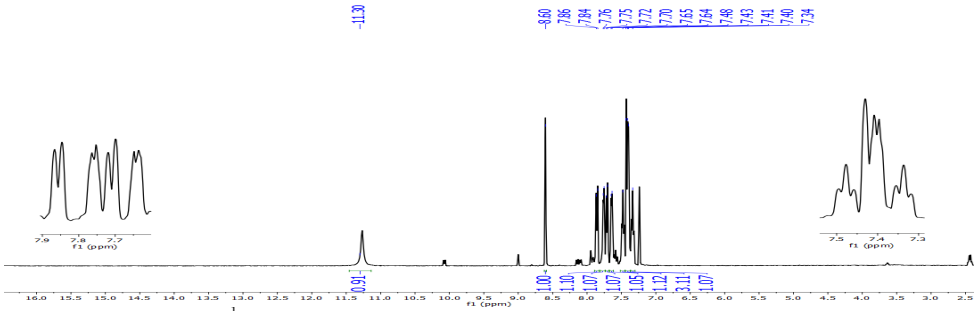

Figure 16. [1]H-NMR spectra of 3-(benzo[d]oxazol-2-yl) naphthalen-2-ol (8).

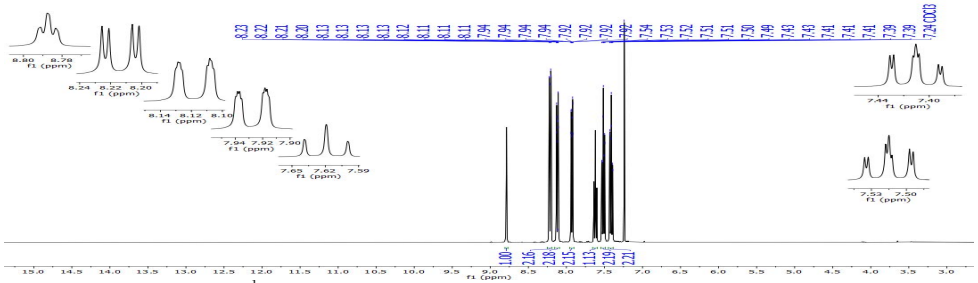

Figure 17. [1]H-NMR spectra of 1,3-di(benzo[d]thiazol-2-yl)benzene (9).

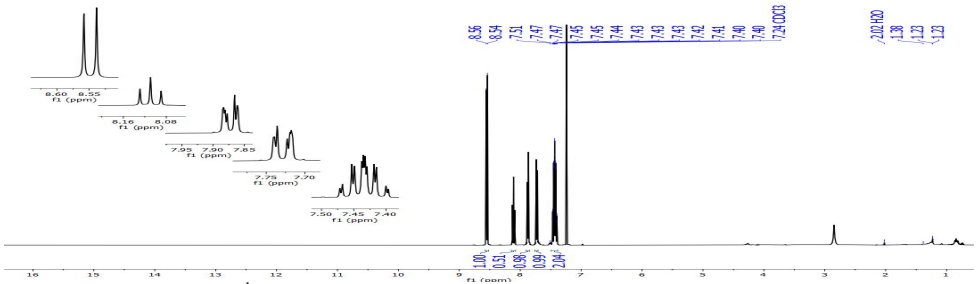

Figure 18. [1]H-NMR spectra of 2,6-di(benzo[d]thiazol-2-yl)pyridine (10).

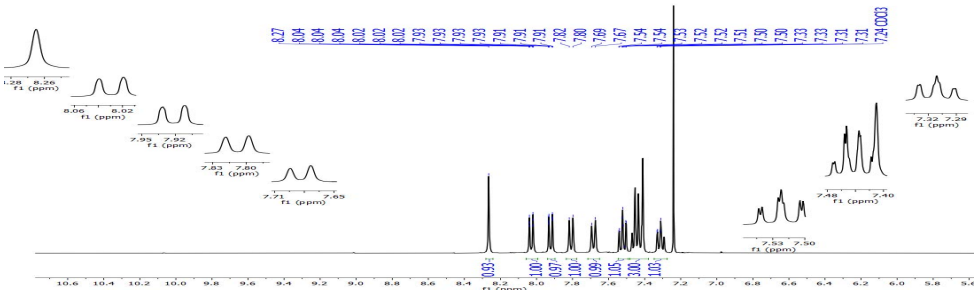

Figure 19. [1]H-NMR spectra of 3-(benzo[d]thiazol-2-yl) naphthalen-2-ol (11).

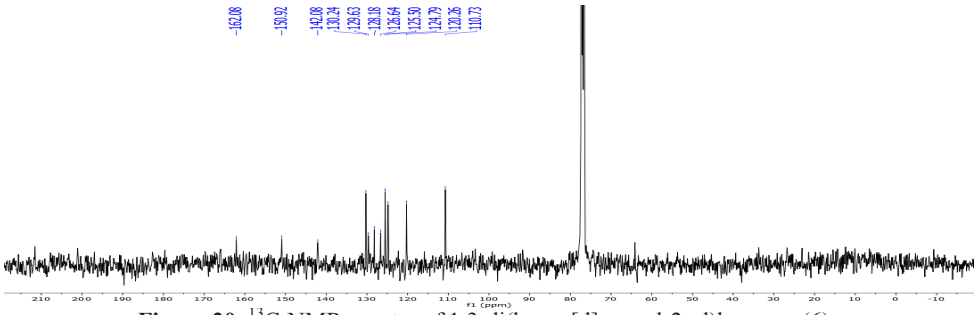

Figure 20. ^{13}C-NMR spectra of 1,3-di(benzo[d]oxazol-2-yl)benzene (6).

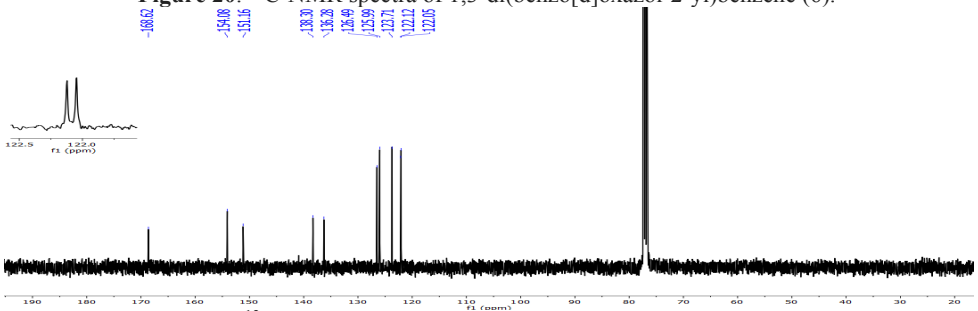

Figure 21. ^{13}C-NMR spectra of 2,6-di(benzo[d]oxazol-2-yl) pyridine (7).

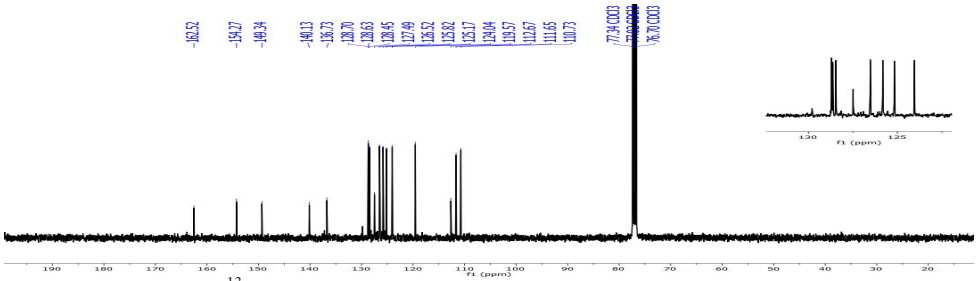

Figure 22. ^{13}C-NMR spectra of 3-(benzo[d]oxazol-2-yl) naphthalen-2-ol (8).

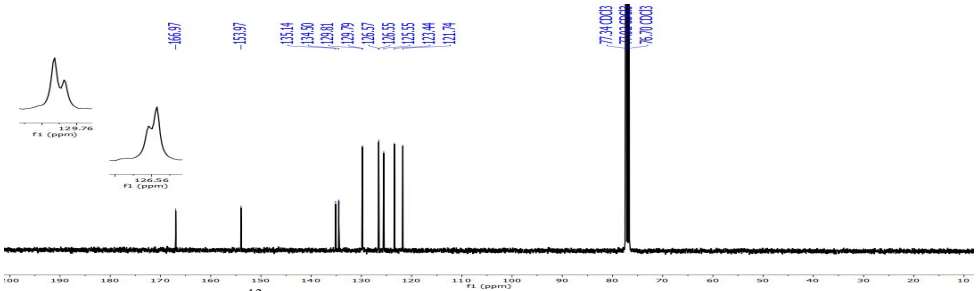

Figure 23. ^{13}C-NMR spectra of 1,3-di(benzo[d]thiazol-2-yl)benzene (9).

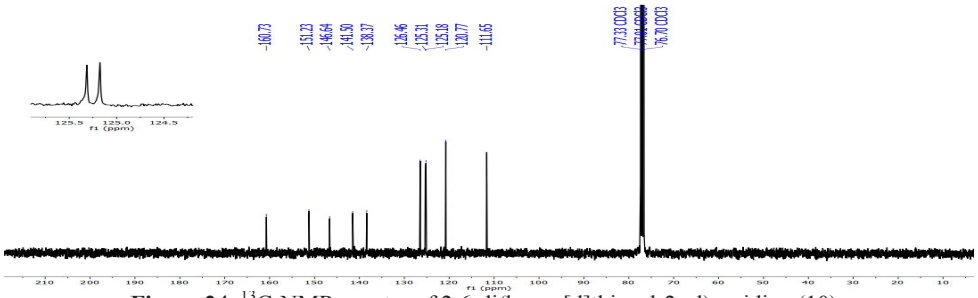

Figure 24. ^{13}C-NMR spectra of 2,6-di(benzo[d]thiazol-2-yl)pyridine (10).

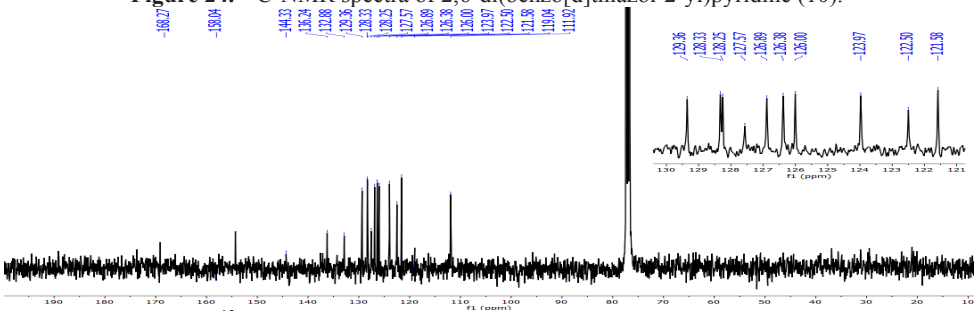

Figure 25. ^{13}C-NMR spectra of 3-(benzo[d]thiazol-2-yl) naphthalen-2-ol (11).

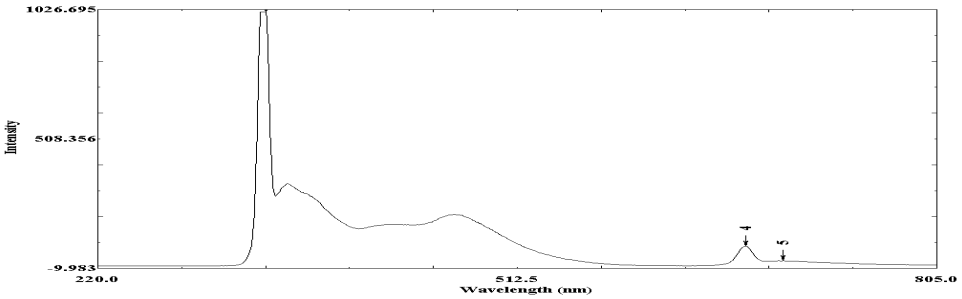

Figure 26. Fluorescence spectra of 1,3-di(benzo[d]oxazol-2-yl)benzene (6).

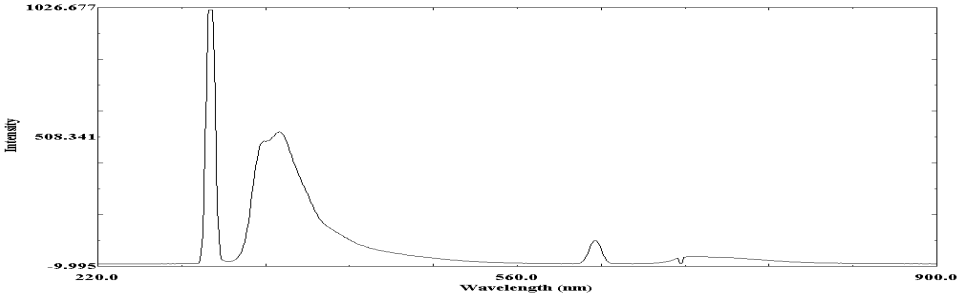

Figure 27. Fluorescence spectra of 2,6-di(benzo[d]oxazol-2-yl) pyridine (7).

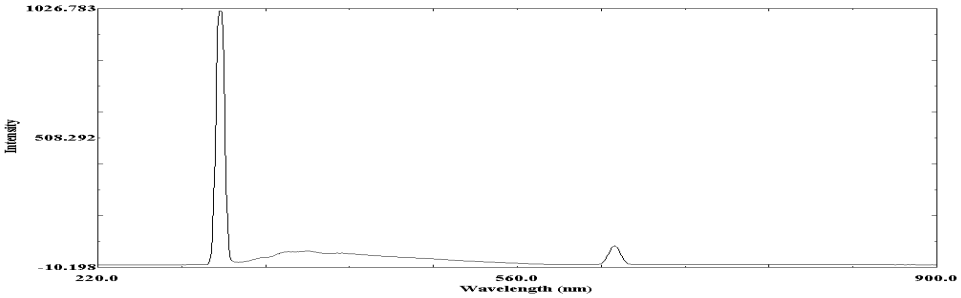

Figure 28. Fluorescence spectra of 3-(benzo[d]oxazol-2-yl) naphthalen-2-ol (8).

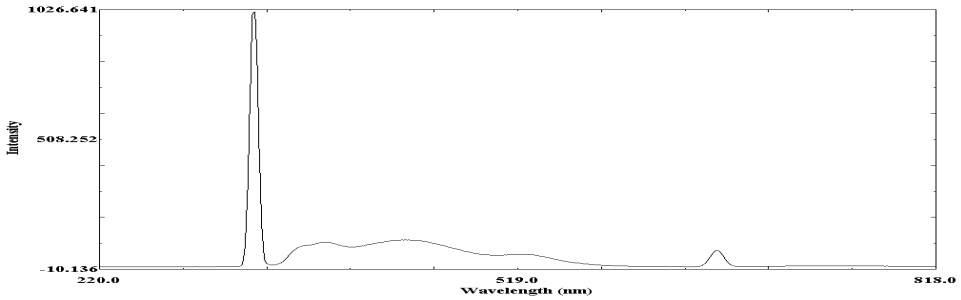

Figure 29. Fluorescence spectra of 2,6-di(benzo[d]thiazol-2-yl)pyridine (10).

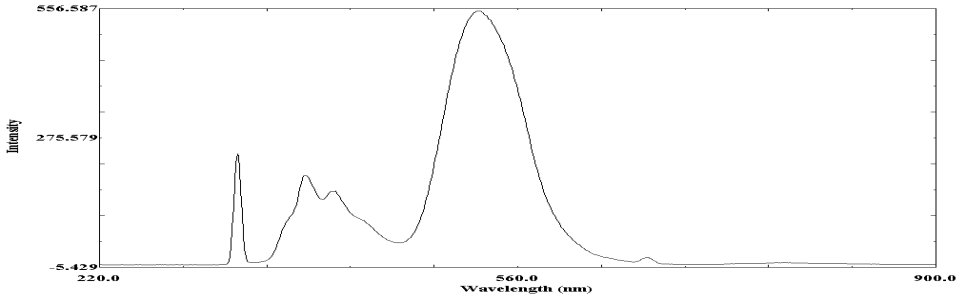

Figure 30. Fluorescence spectra of 3-(benzo[d]thiazol-2-yl) naphthalen-2-ol (11).