



ARAŞTIRMA MAKALESİ/RESEARCH ARTICLE

THE SYNTHESIS OF 4,5-DIHYDRO-4-(para-METHYLPHENYL)-1,4-NAPHTHO[6,7-b]- OXAZEPINE-3(2H)-ONES Sevim Bilgiç¹, Murat Gündüz^{1,2}

ABSTRACT

In this work the Schiff base (1) which was prepared from toluene, was reacted with NaBH₄ to synthesise the reduced amine (2). The reaction was continued till the yellow Schiff base became a colorless clear solution at room temperature. To the isolated 1-N-(para-methylphenylaminomethyl)-2-naphthol (2) which was stirred vigorously in dry benzene, chloroacetyl chloride was added slowly and refluxed for 2 hours. To the product from this reaction (3) NaOH solution was added and stirred at room temperature. Resulted solid (4) was washed with water and crystallized from ethanol to give a colorless solid. The structures of the products (2, 3, 4) were determined with IR, UV, ¹³C-NMR (DEPT), ¹H-NMR spectra.

Key words : Schiff bases, Chloroacetyl chloride, Naphthoxazepinone, Reduction

4,5-DİHİDRO-4-(para-METİLFENİL)-1,4-NAFTO[6,7-b]OKSAZEPİN-3(2H)-ON'UN SENTEZİ

ÖZ

Bu çalışmada p-CH₃ anilinden hazırlanan Schiff bazı (1), indirgenmiş amin bileşiğini (2) sentezlemek amacıyla NaBH₄ ile reaksiyona girdirilmiştir. Reaksiyon oda sıcaklığında, sarı renkli Schiff bazı berraklaşp gaz çıkışı sona erene kadar sürdürülmüştür. Elde edilen 1-N-(para-metilfenilaminometil)-2-naftol (2) susuz benzen içinde şiddetli olarak karıştırılmış ve kloroasetil klorür yavaşça eklenerek 2 saat geri soğutucu altında ısıtılmıştır. Bu reaksiyondan elde edilen ürün (3) oda sıcaklığında NaOH içine alınarak su banyosunda ılıtılmıştır. Elde edilen katı (4) su ile yıkanmış ve alkolden kristallendirilerek renksiz kristaller elde edilmiştir. Ürünlerin (2,3,4) yapıları IR, UV, ¹³C-NMR (DEPT), ¹H-NMR ile tayin edilmiştir.

Anahtar Kelimeler : Schiff bazı, Kloroasetil klorür, Naftoksazepinon, İndirgeme

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1. INTRODUCTION

The saturated benzoxazepines are known to have effects on hipnotic, muscle relaxation (Hirohashi et al., 1970), antiemetic, antagonistic effects (Okada et al., 1994), against inflammation (Mueller, 1977) and are used as anxiolytics agent (Effland et al., 1982), antiallergic and antihistaminic (Cale et al., 1982). The benzoxazepines containing carbonyl group in 3, 5 positions were also notified to have anxiolytics (Tatsuoka et al., 1990), antiemetic and antagonistic effects (Okada, et al., 1994). The benzoxazepines containing carbonyl group in 5 positions were also declared to have central depressant (Himizu et al., 1972), analgesic and antiphlogistic (Schenker, 1971), local anesthetic (Nichimizu et al., 1975), antiinflammatory (Schridhar et al., 1979), antihistaminic activities (Cale, 1987). In addition, it was also notified that the stabilizing property of benzoxazepines in photography was determined (Kaneko, 1988). There are several different methods to obtain benzoxazepines in literature (Mueller, 1977; Effland, et al., 1982; Bernard, 1957; Griengl et al., 1979).

One of the synthetic methods is to ring closure of the product obtained from the reaction of α -anilino-cresols obtained from the reduction of Schiff bases with BrCH_2COBr (Derieg and Sternbach, 1966) or ClCH_2COCl (Mohan et al., 1984) in basic medium.

2. EXPERIMENTAL

After 1-(N-para-methylphenylimino-methyl)-2-naphthol (**1**) was dissolved in 1:1 ratio methanol and dioxan, NaBH_4 was added. Yellow colored Schiff base was stirred until gas evolution was stopped (1 hour) at room temperature. When the reaction had been completed, solution was diluted with ice-water to precipitate the product and the product was crystallized from alcohol. 1-N-(para-methylphenyl-aminomethyl)-2-naphthol (**2**) was strongly stirred in dry benzene. It was refluxed with chloroacetyl chloride for two hours. Solvent was gained back by distillation, and white colored crystals were obtained.

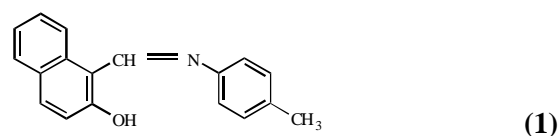
1-N-Chloroacetyl-N-(para-methyl-phenylaminomethyl)-2-naphthol (**3**) was put into 5% NaOH solution at room temperature, and was warmed in water bath for 30 minutes. Then, the obtained solid was washed with water and crystallized from alcohol.

Structures of the isolated products (**2**, **3**, **4**) were determined by IR, UV, ^{13}C -NMR (DEPT), ^1H -NMR.

3. RESULT and DISCUSSION

3.1. Synthesis of Naphtolic Schiff Bases

Naphtolic Schiff base (**1**) was firstly synthesized according to Sawich et al. (Sawich et al., 1956).



The spectroscopic data of the Schiff base was in accordance with the literature. A band for the azomethine group was observed in IR spectrum approximately in 1625 cm^{-1} .

3.2. Reduction of Naphtolic Schiff Base with NaBH_4

After 1-(N-para-methylphenylimino-methyl)-2-naphthol (**1**) was dissolved in 1:1 ratio in methanol and dioxan, it was mixed until the yellow colored Schiff base had disappeared by adding NaBH_4 and then the compound was precipitated with ice-water. (Mohan et al., 1984). Melting point of the product obtained with 78% yield, was determined as 105°C .

Absorption bands for OH group at 3464 cm^{-1} and NH group at 3280 cm^{-1} , were observed in IR spectrum of the compound. Absorption bands at $1600\text{-}1523\text{ cm}^{-1}$ was supported aromatic structure. Bands at 834 cm^{-1} were assigned to para-disubstitued phenyl ring (Figure 1).

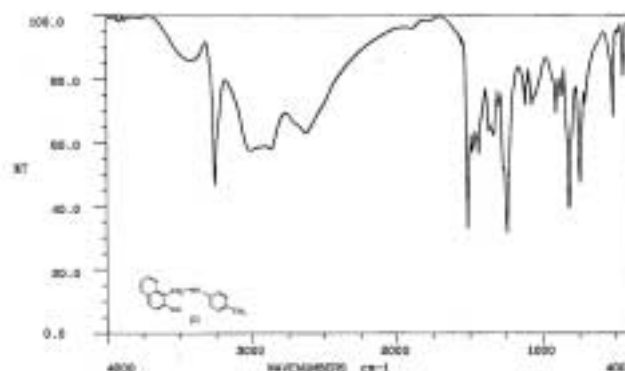


Figure 1. IR (KBr) Spectrum of 1-(N-para-methylphenyl-aminomethyl)-2-Naphtol (**2**)

Absorption peaks at 334.8, 322.4 and 289.2 nm was observed in UV spectrum of the compound (Figure 2). Absorption maximums at 356.0, 289.0 and 269.0 nm were observed, when UV spectrum of the compound had been taken in acidic medium. The spectrum taken in basic medium was the same as neutral UV spectrum.

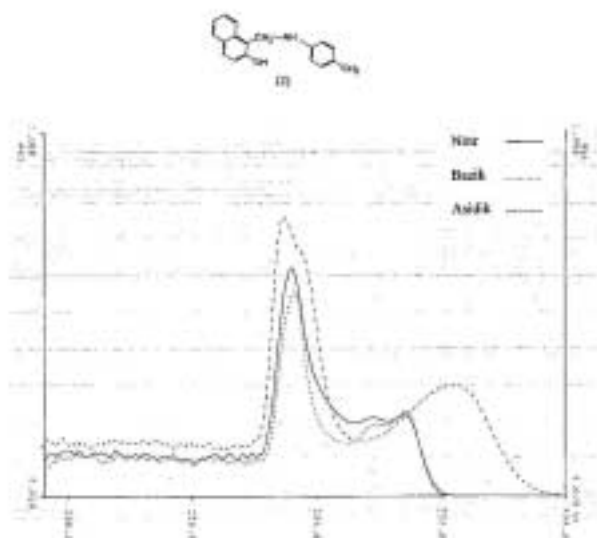


Figure 2. UV Spectrum (EtOH) of 1-(N-para-methylphenylaminomethyl)-2-Naphthol (2)

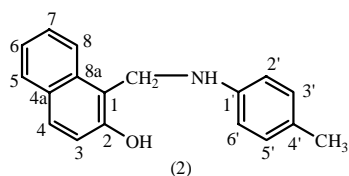


Table 1. λ_{\max} (nm) values of 1-(N-para-methylphenylaminomethyl)-2-naphthol (2) in neutral, basic and acidic medium

UV (Ethanol), λ_{\max} (abs)	UV (OH ⁻), λ_{\max} (abs)	UV (H ⁺), λ_{\max} (abs)
334.8 (0.221)	334.8 (0.213)	356.0 (0.300)
322.4 (0.214)	322.4 (0.200)	296.0 (0.660)
289.2 (0.622)	289.2 (0.550)	289.0 (0.750)

In ¹H-NMR spectrum of the compound, 3-proton singlet at 2.23 ppm, 2-proton singlet at 4.77 ppm and 8-proton multiplet signals in aromatic region were observed (Figure 3). It was thought that, signals at 2.23 and 4.77 ppm were belonging to methyl and -CH₂-NH-methylene protons.

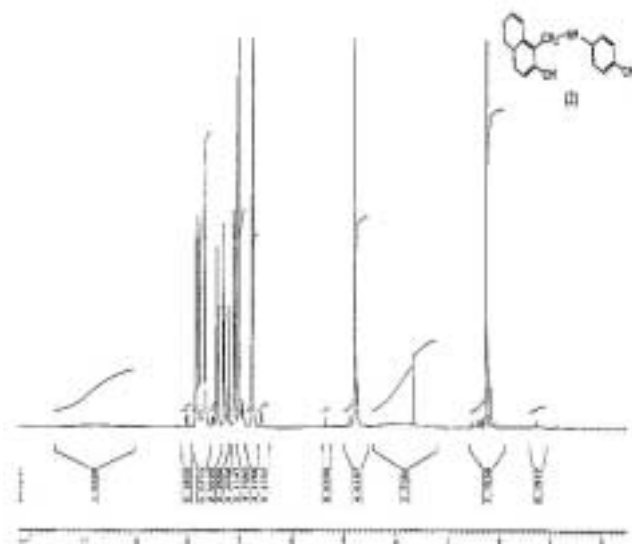


Figure 3. ¹H-NMR Spectrum of 1-(N-para-methylphenylaminomethyl)-2-naphthol (2).

Aromatic protons were marked from enlarged spectrum of aromatic region (Figure 4).

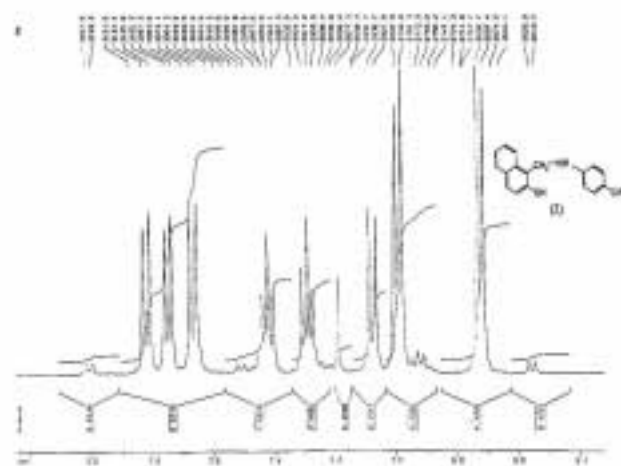


Figure 4. ¹H-NMR Spectrum of 1-(N-para-methylphenylaminomethyl)-2-naphthol (2) (In the extended NMR spectrum of this area).

In the enlarged ¹H-NMR spectrum, 1-proton doublet at 7.83 ppm ($J_{7,8}=8.6$ Hz), 7.75 ppm ($J_{3,4}=8.0$ Hz) and 7.67 ppm ($J_{5,6}=8.8$ Hz) were marked as to be belonging to H-8, H-4 and H-5 protons respectively. It was thought that, triplet of the 1-proton doublet at 7.43 ppm ($J_{6,7}=7.2$ Hz, $J_{7,8}=7.4$ Hz, $J_{5,7}=0.8$ Hz) and 1-proton triplet at 7.29 ppm ($J_{5,6}=J_{6,7}=7.4$ Hz) were belonging to H-7 and H-6 protons, and 1-proton doublet at 7.08 ppm ($J_{3,4}=8.8$ Hz) was belonging to H-3 proton. It was clear that, 2-proton doublet at 6.99 and 6.73 ppm ($J_{2,3}=8.3$ Hz ; $J_{2,3}=8.1$ Hz) were belonging to 3'-H and 2'-H protons

¹³C-NMR spectrum of the compound was supported the structure (Figure 5). 13 signals in aromatic region, 1 methylene and 1 methyl signals were observed in ¹³C-NMR spectrum. The signal at 45.220 ppm was thought to be arisen from -CH₂-NH- methyl-

ene carbon in DEPT spectrum of the compound (Figure 6). Signal at 20.948 ppm was attributed as to the methyl carbon.

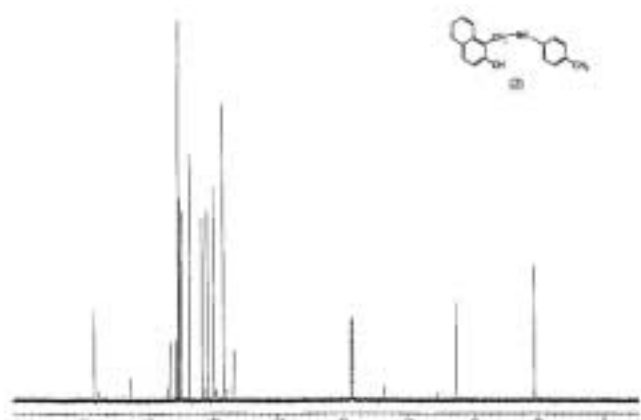


Figure 5. ^{13}C -NMR Spectrum of 1-(N-para-methylphenylaminomethyl)-2-naphthol (**2**).

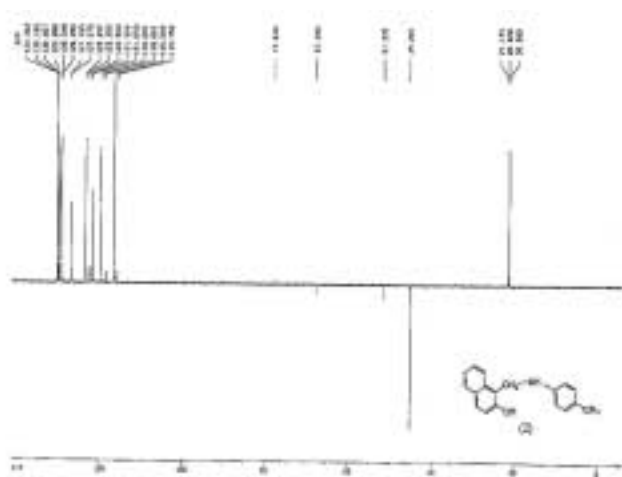
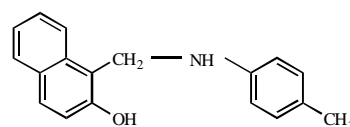


Figure 6. ^{13}C -NMR (DEPT) Spectrum of 1-(N-para-methylphenylaminomethyl)-2-Naphthol (**2**).

8 -CH- signal in DEPT spectrum were marked as below. Signals at 121.653, 129.898, 129.348, 123.297, 127.075 and 119.681 ppm were assigned as to be belonging to C-3, C-4, C-5, C-6, C-7 and C-8 carbons respectively. It was understood that, signals at 130.342 and 116.602 ppm were belonging to C-3 and C-2 carbons in phenyl ring. Remaining 6 signals were assigned by the aid using from ^{13}C -NMR and DEPT spectrums. It was clear that, signals at 155.986, 145.258, 132.056, 131.055, 129.264 and 113.242 ppm were belonging to C-2, C-1, C-4a, C-8a, C-4, and C-1 carbons respectively.

It was understood from the above described spectroscopic data that, the structure was 1-(N-para-methylphenyl- aminomethyl)-2-naphthol (**2**).



(2)

3.3. Reaction of Reduced Schiff Base with Chloroacetyl Chloride

In this part we aimed to synthesize N-acyl derivatives (**3**) by reacting amines obtained from reduction a Schiff base with NaBH_4 (**2**), with chloroacetyl chloride.

1-N-(para-methylphenylamino-methyl)-2-naphthol (2 mmol) (**2**) was refluxed in dry benzene with chloroacetyl chloride (4.6 mmol) for two hours. Solvent and excess chloroacetyl chloride were removed by distillation and the crude product was crystallized from ethanol with 54% efficiency (mp: 98 °C) (Mohan et al.,1984).

In the IR spectrum of the product signals at 1804 and 1651 cm^{-1} were due to the ester and amides carbonyls. Absorption bands belonging to aromatic structure at 1523-1472 cm^{-1} were observed. Absorption band at 834 cm^{-1} was assigned to para-disubstitution (Figure 7).

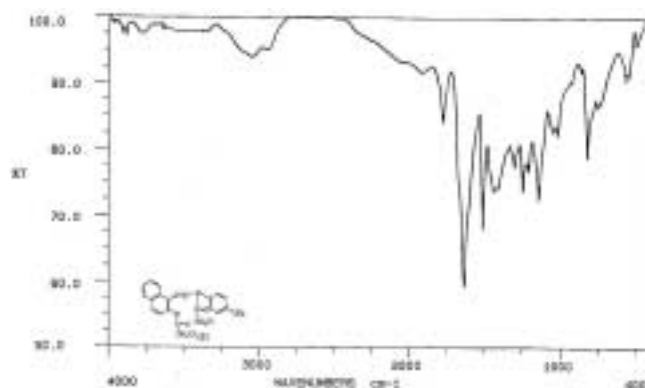


Figure 7. IR (KBr) Spectrum of (1N,2-O-dichloroacetyl)-N-(para-methylphenylaminomethyl)-2-Naphtol (**3**)

Absorption maximums were observed at 335.2, 289.6 and 273.2 nm in UV spectrum of the product.

Absorption maximums were observed at 361.2, 321.1 and 286.0 nm in acidic medium, and at 332.0, 318.0 and 286.0 nm in basic medium (Figure 8).

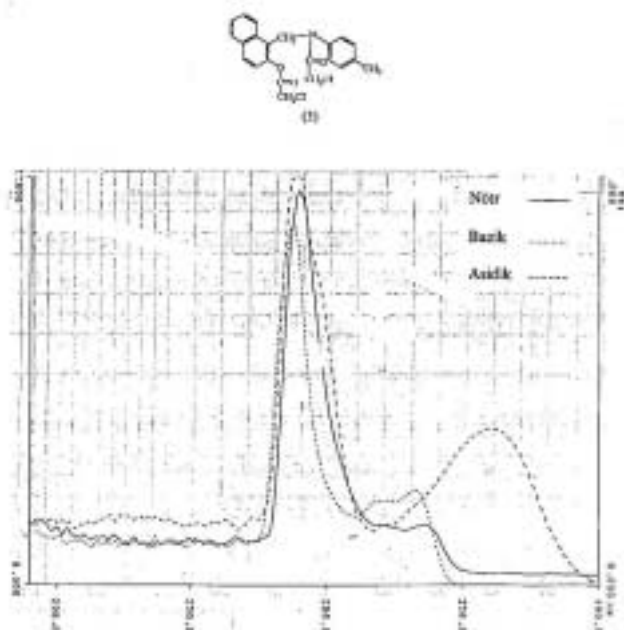


Figure 8. UV Spectrum (EtOH) of (1-N, 2-O-dichloroacetyl)-N-(para-methylphenylaminomethyl)-2-Naphtol (3)

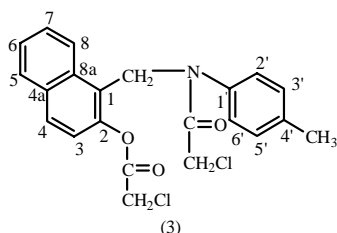


Table 2. λ_{\max} (nm) values of 1-(N- Chloro acetylchloride)-N-(para-methylphenylaminomethyl)-2-naphtol (3) in neutral, basic and acidic medium.

UV (Ethanol), λ_{\max} (abs)	UV (OH ⁻), λ_{\max} (abs)	UV (H ⁺), λ_{\max} (abs)
335.2 (0.148)	332.0 (0.233)	361.2 (0.386)
289.6 (0.966)	318.0 (0.200)	321.1 (0.133)
273.2 (0.105)	286.0 (0.880)	290.3 (0.980)
		273.0 (0.166)

In the ¹H-NMR spectrum of the compound 3-protons singlet at 2.180 ppm and 3 two proton singlets at 3.710, 4.250 and 5.360 ppm were observed. It was understood that, 3-proton signal at 2.180 ppm were attributed to the methyl protons, and three 1-proton singlets at 3.710, 4.250 and 5.360 ppm were belonging to -N-(CO)-CH₂-Cl, -O-(CO)-CH₂-Cl ve Ar-CH₂-N-Ar methylene protons (Figure 9). Aromatic protons from enlarged spectrum of aromatic region were tried to be assigned (Figure 10). Two 2-proton doublets at 6.87 ve 6.62 ppm ($J_{7,8} \approx 8.0$ Hz) were assigned to H-2, H-6 ve H-3, H-5 protons. So, markings at phenyl ring were completed. Remaining signals were -CH- 's in naphthyl ring. Three corrupted 1-proton doublets at 7.82, 7.72 and 7.71 ppm ($J_{7,8} = 5.8$ Hz; $J_{3,4} = 6.6$ Hz; $J_{5,6} = 5.2$ Hz) were marked to be belonging to H-8, H-4 and H-5 protons. H-7 and H-6 protons at 7.36 ppm

($J_{7,8} = 5.8$ Hz) were observed as 2-proton corrupted doublets 1-proton doublet at 7.06 ppm ($J_{3,4} = 8.9$ Hz) was marked to be belonging to H-3 proton.

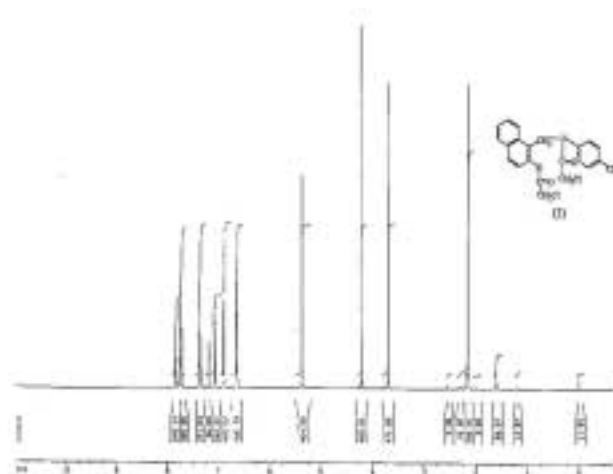


Figure 9 ¹H-NMR Spectrum of (1-N,2-O-dichloroacetyl)-N-(para-methylphenylaminomethyl)-2-Naphtol (3)

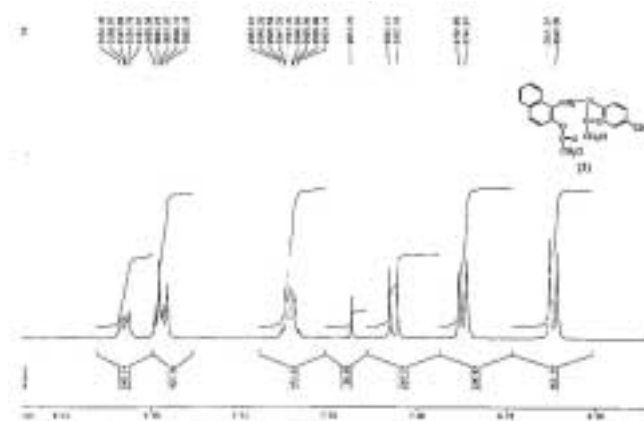


Figure 10 ¹H-HMR Spectrum of (1-N,2-O-dichloroacetyl)-N-(para-methylphenylaminomethyl)-2-Naphtol (3) (In the extended NMR spectrum of this area).

20 signals were observed in ¹³C-NMR spectrum of the compound (Figure 11). Three of these were arisen from saturated groups. Signals at 41.267, 42.325 and 42.593 ppm were thought to be belonging to -CH₂-N-, -N-(CO)-CH₂-Cl and -O-(CO)-CH₂-Cl methylene carbons respectively. Furthermore, peak belonging to CH₃- carbon was observed at 21.358 ppm. Signals at 166.346 and 166.078 ppm were marked to be belonging to 2 different peak -O-(CO)- and -N-(CO)- carbonyl carbons. For marking aromatic CH- and -C- carbons, ¹³C-DEPT spectrum was aided (Figure 12). Signals at 126.150, 130.441, 128.856, 124.382, 127.602 and 121.007 ppm were marked to be belonging to C-3, C-4, C-5, C-6, C-7 ve C-8 aromatic carbons in naphthyl ring. 2 different -CH- were observed in phenyl ring. It was thought that, signal at 128.635 and 129.014 ppm were belonging to C-2 and C-3 carbons respectively.

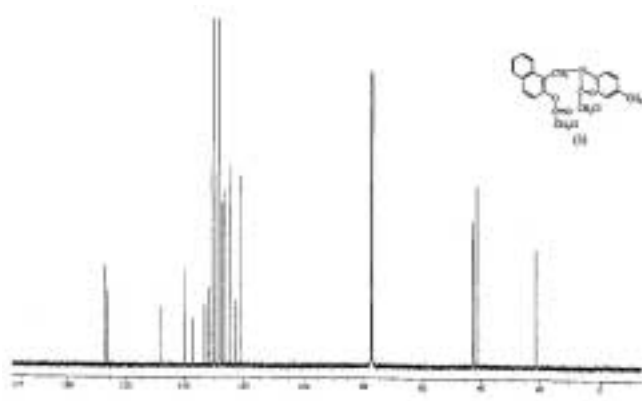


Figure 11 ^{13}C -NMR Spectrum of (1-N,2-O-dichloroacetyl)-N-(para-methylphenylamino)-2-Naphtol (**3**)

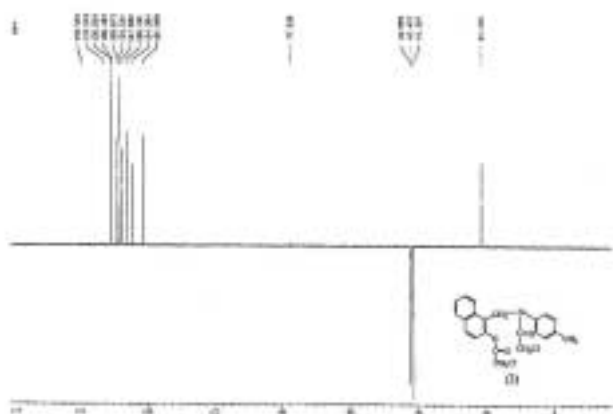
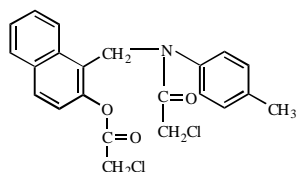


Figure 12 ^{13}C -NMR (DEPT) Spectrum of (1-N,2-O-dichloroacetyl)-N-(para-methylphenylamino)-2-Naphtol (**3**)

^{13}C -NMR spectrum, it was understood that, remaining signals at 122.830, 148.063, 132.146 and 133.323 ppm were belonging to C-1, C-2, C-4a ve C-8a aromatic carbons in naphthyl the ring, and signals at 139.537 and 130.591 ppm to aromatic carbons in the phenyl ring.

It was understood from the above described spectroscopic data that, the compound was (1-N,2-O-dichloroacetyl)-N-(para-methylphenylamino)-2-naphtol (**3**).



(**3**)

3.4. Ring Closure in Basic Medium

(1-N,2-O-dichloroacetylchloride)-N-(para-methylphenylamino)-2-naphtol (0,5 mmol) (**3**)

had been warmed in water bath by putting into 5% 0.93 ml NaOH for 30 minutes at room temperature. Then, the obtained solid was washed by water, and crystallized from alcohol. (Mohan et al., 1984) (mp: 132°C).

Absorption bands belonging to amides carbonyl was observed at 1676 cm^{-1} in IR spectrum of the compound. Absorption bands at 1549-1447 cm^{-1} had supported the aromatic structure. C-O band at 1268 cm^{-1} was observed. The band at 830 cm^{-1} was marked to be belonging to disubstituted phenyl ring (Figure 13).

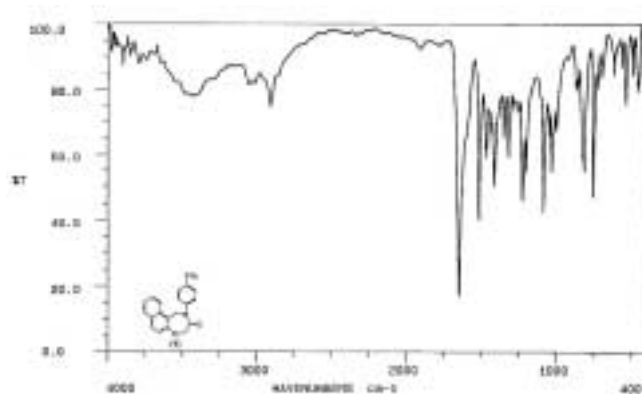


Figure 13 IR(KBr) Spectrum of 4,5-dihydro-4-(para-methylphenyl)-1,4-naphtol[6,7-b]-oxazepine-3(2H)-ones (**4**)

Absorption peaks at 319.2, 311.6 and 293.2 nm was observed in UV spectrum of the compound. Absorption peaks at 362.0, 300.8 and 276.8 nm was observed in basic medium. There was not so much difference in UV spectrum in acidic medium (Figure 14).

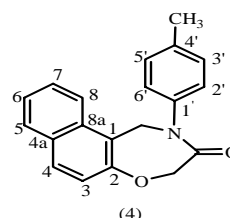


Table 3. λ_{max} (nm) values of 4,5-dihydro-4-(para-methylphenyl)-1,4-naphtol[6,7-b]oxazepine-3(2H)-ones (**4**) in neutral, basic and acidic medium.

UV (Ethanol), λ_{max} (abs)	UV (OH ⁻), λ_{max} (abs)	UV (H ⁺), λ_{max} (abs)
319.2 (0.172)	362.0 (0.629)	335.2 (0.105)
311.6 (0.127)	300.8 (0.280)	319.2 (0.255)
293.2 (0.998)	276.8 (0.140)	311.2 (0.202)
277.6 (0.054)	272.0 (0.138)	294.4 (0.998)
	268.8 (0.141)	

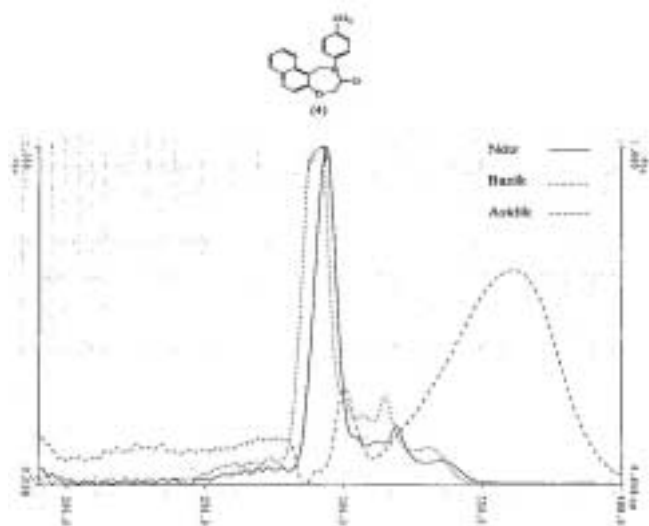


Figure 14. UV Spectrum (EtOH) of 4,5-dihydro-4-(p-methylphenyl)-1,4-naphthol [6,7-b]-oxazepine-3(2H)-ones(4).

In $^1\text{H-NMR}$ spectrum of the compound, 3-proton singlet at 2.338 ppm and two 2-protons singlets at 4.813 and 5.167 ppm, and 10-proton signals in aromatic region were observed. It was understood that, 3-proton signal at 2.388 ppm were belonging to methyl protons, and two 2-proton singlets at 4.813 and 5.167 ppm were belonging to $-\text{N-CH}_2-$ and $-\text{O-CH}_2-$ methylene protons (Figure 15). Aromatic protons were tried to be marked from enlarged spectrum of the aromatic region (Figure 16). 2-proton two doublets at 7.05 and 7.14 ppm ($J=8.0$ Hz) were marked to be belonging to H-2, H-6 ve H-3, H-5 protons. So, markings at phenyl ring were completed. It was clear that, the remaining signals were $-\text{CH=}$'s at naphthyl ring. 1-protons three doublets at 7.82, 7.80 and 7.69 ppm ($J_{7,8}=8.6$ Hz ; $J_{3,4}=8.0$ Hz ; $J_{5,6}=8.5$ Hz) were assigned to H-8, H-4 and H-5 protons. H-7 and H-6 protons were observed as 1-proton two doublets at 7.43 and 7.37 ppm ($J\cong 7.0$ Hz). 1-proton doublet at 7.26 ppm ($J_{3,4}=8.8$ Hz) was marked to be belonging to H-3 proton.

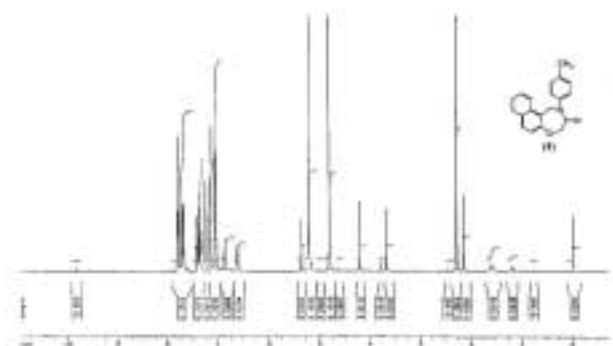


Figure 15. $^1\text{H-NMR}$ Spectrum of 4,5-dihydro-4-(p-methylphenyl)-1,4-naphthol [6,7-b]-oxazepine-3(2H)-ones(4).

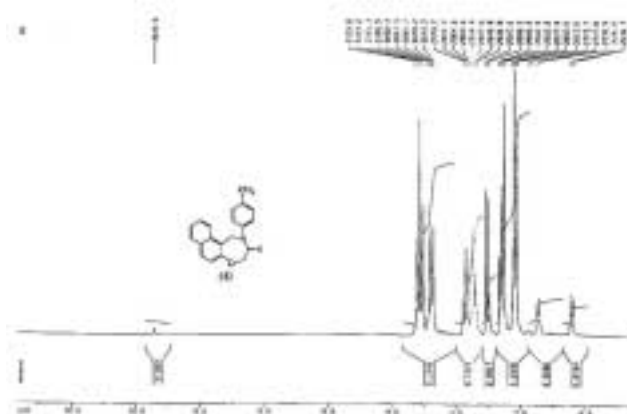


Figure 16. $^1\text{H-NMR}$ Spectrum of 4,5-dihydro-4-(p-methylphenyl)-1,4-naphthol [6,7-b]-oxazepine-3(2H)-ones(4) (In the extended NMR spectrum of this area).

Totally 19 signals were observed in $^{13}\text{C-NMR}$ spectrum of the compound (Figure 17). CH_3- , $-\text{CH}_2-$ and $-\text{CH-}$ carbons had been marked in DEPT spectrum (Figure 18). It was thought that, signal at 20.405 ppm was belonging to methyl carbon, and signals at 44.774 and 71.980 ppm were belonging to methylene carbons. 8 $-\text{CH-}$ signals were observed in DEPT spectrum. Two high amplitude signals of these at 129.181 and 125.240 ppm were marked to be belonging to C-3 and C-2 carbons. It was thought that, signals at 121.326, 126.217, 129.571, 123.841, 127.979 and 119.329 ppm were belonging to C-3, C-4, C-5, C-6, C-7 and C-8 carbons respectively. Signal at 168.037 ppm was marked to be belonging to carbonyl carbon. Signals belonging to C-2 and C-1 carbons were observed at 155.062 and 140.541 ppm. It was thought that, signals at 123.092, 136.262 and 129.947 ppm were belonging to C-1, C-4a and C-8a carbons respectively.

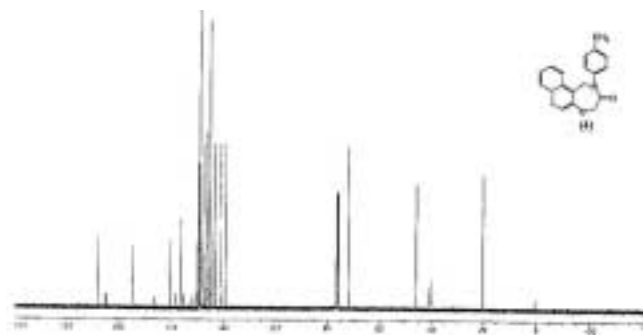


Figure 17. $^{13}\text{C-NMR}$ Spectrum of 4,5-dihydro-4-(p-methylphenyl)-1,4-naphthol [6,7-b]-oxazepine-3(2H)-ones(4).

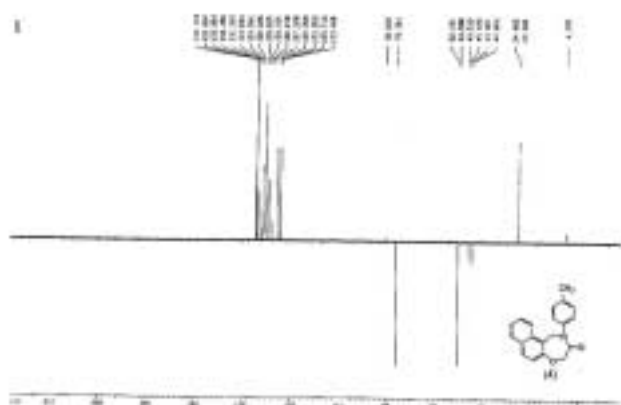
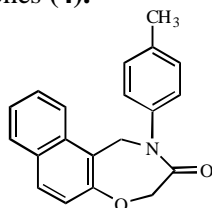


Figure 18. ^{13}C -NMR Spectrum of 4,5-dihydro-4-(para-methylphenyl)-1,4-naphtho[6,7-b]-oxazepine-3(2H)-ones (4).

It was understood from the above described spectroscopic data that, the compound was 4,5-dihydro-4-(para-methylphenyl)-1,4-naphtho[6,7-b]-oxazepine-3(2H)-ones (4).



(4)

Since naphthoxazepinones have so many application areas especially on medicine, increasing the number of the naphthoxazepinones and research of their activities will be our following aims.

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