



Preparation and Characterization of Some Schiff Base Compounds

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Abstract

Schiff bases (imines) have been frequently used in various fields such as medicine, pharmaceutical purposes due to their various biological properties. In this study, nine new imine compounds (3a-h) were synthesized from 1-naphthyl amine with aromatic aldehydes in MeOH and their chemical structures were defined by ¹H/¹³C NMR, IR and elemental analysis studies. We observed a singlet one hydrogen of the imines (–CH=N–) at 8.56–8.86 ppm in the ¹H NMR spectra and also carbon of the Schiff bases (–CH=N–) at 158.2-163.4 ppm in the ¹³C NMR spectra. Also the IR spectra displayed the (–C=N–) characteristic absorption band at around 1600 cm⁻¹. The obtained characteristic peaks at the expected locations proved the structural accuracy of the synthesized new derivative Schiff bases.

Keywords: Schiff bases; Imines; 2-naphthylamine; Aromatic Aldehyde.

Bazı Schiff Baz Bileşiklerinin Hazırlanması ve Karakterizasyonu

Öz

Schiff bazları (iminler), çeşitli biyolojik özellikleri nedeniyle tıp, farmasötik amaçlar gibi çeşitli alanlarda sıkça kullanılmaktadır. Bu çalışmada, 1-naftil aminden MeOH içerisinde aromatik aldehitler ile dokuz yeni imin bileşiğinin (3a-h) sentezi yapılarak ve kimyasal yapıları;

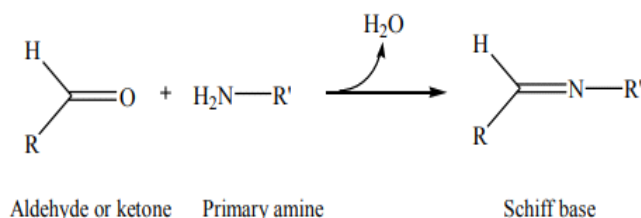


¹H/¹³C NMR, IR ve elementel analiz yöntemleri aydınlatılmıştır. ¹H/¹³C NMR analiz sonuçları incelendiğinde iminlerin (–CH=N–) protonu 8.56-8.86 ppm ve karbonunun ise 158.2-163.4 ppm de geldiği gözlemlenmiştir. Ayrıca IR spektrumundada (–C=N–) piki 1600 cm⁻¹ civarında görülmüştür. Elde edilen bu karakteristik piklerin beklenen yerlerde gelmesi sentezlenen yeni türev Schiff bazların yapısal doğruluğunu kanıtlamıştır.

Anahtar Kelimeler: Schiff bazları; İminler; 2-naftilamin; Aromatik aldehit.

1. Introduction

Generally, Schiff bases (imines) occur from primary amines and carbonyl compounds (aldehydes or ketones, in Scheme 1) and were first synthesized in 1869 by the German chemist Hugo Schiff [1, 2]. The bond formed by reaction with aldehyde is called azomethine or aldimine, while the bond formed by reaction with ketone is called imine or ketimine.



Scheme 1: General route for synthesis of a imines

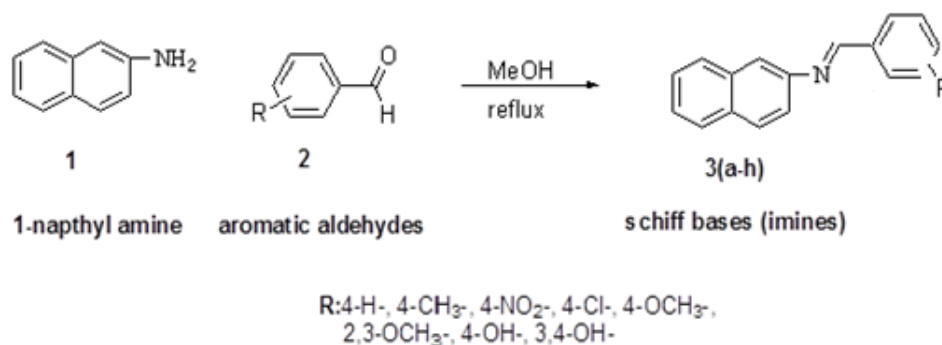
Imines can also be represented by the general formula RCH = NR', wherein R and R' are alkyl or alkyl substituents [3-5]. Many imines have been worked due to their very varying structural properties and used as chelating ligands in coordination Chemistry [6-12]. This interest in Schiff bases can be explained due to their availability in many areas, such as biological systems, medicine, and also in new technologies [9, 13-15]. In addition these, imines are important intermediates and multipurpose starting materials for the synthesis of many reaction such as Mannich bases [16, 17], indoles [18, 19], betalactam [20, 21], pyrimidine derivatives [22]. Compounds obtained from these reactions accompanied by Schiff bases have been used in the treatment of various diseases due to their biological activity [23]. Many reagents have been used for the synthesis of imines such as Lewis acids [24-27], metal complex [28, 29], metal-free conditions [30, 31], promoted by microwave irradiation [32] and ultrasound radiation [33].

In this study, nine new imine compounds (3a-h) were synthesized from 1- naphthyl amine with aromatic aldehydes in MeOH and their chemical structures were defined by ¹H/¹³C NMR, IR, and elemental analysis studies. The evaluation of the analysis results had proved the accuracy of the synthesized structures.

2. Materials and Methods

All starting materials and reagents were commercially available and used without further purification except where indicated. Analytical thin-layer chromatography (TLC) was carried out on precoated plates (silica gel 60 F254), and spots were visualised with ultraviolet (UV) light. Melting points were determined on a Yanagimoto micro-melting point apparatus and were uncorrected. IR spectra were measured on a SHIMADZU Prestige-21 (200 VCE) spectrometer. ¹H/¹³C NMR spectra were measured on spectrometer at VARIAN Infinity Plus 300 and at 75 MHz, respectively. ¹H/¹³C chemical shifts are referenced to the internal deuterated solvent. Chemical shift values (δ) are given in ppm. The elemental analysis was carried out with a Leco CHNS-932 (St. Joseph, Michigan) instrument. All chemicals were purchased from Merck (Darmstadt, Germany), Alfa Aesar (Ward Hill, MA), and Sigma-Aldrich (Taufkirchen, Germany).

2.1. General procedure for synthesis of compounds



Scheme 2: Synthesis of new schiff base compounds (3a-h)

2.1.1. General procedure for the synthesis of substituted benzylidenyl amino naphthalenes(3a-h):

Mixture of 1-naphthylamine (1) (1.0 mmol) and aromatic aldehydes derivatives (2) (1.0 mmol) were stirred and refluxed for 3 hours in MeOH (15 ml). After completion of the reaction, the mixture was left cooling to room temperature and poured on cold water (50 ml). The product (3) was filtered and dried (in Scheme 2).

N-(benzylidene)naphthyl-amine(3a); yellow solid; m.p. 121-123 °C; (91%) yield. IR (KBr, cm^{-1}): 3018 (aromatic C-H), 2926 (azomethine C-H), 1600 (Ar-CH=N-Ar), 1560 (-C=C-). ¹H NMR (300 MHz, DMSO, δ , ppm): 7.26-8.27 (12H, m, Ar-H), 8.80 (1H, s, Ar-CH=N-). ¹³C NMR (75 MHz, DMSO, δ , ppm): 158.2 (Ar-CH=N-Ar), 142.3 (-Ar-N=), 133.5 (-Ar-H), 130.1-

119.3 (other aromatic karbon). Anal. Calcd. for $C_{17}H_{13}N$ %: C 88.28, H 5.66, N 6.06; found: C 88.30, H 5.20, N 6.09.

N-(4-methylbenzylidene)naphthylamine(3b); light yellow solid; m.p. 69-71 °C; (89%) yield. IR(KBr, cm^{-1}): 3023 (aromatic C-H), 2935 (azomethine C-H), 1605 (Ar-CH=N-Ar), 1562 (-C=C-), 1354 (-CH₃). ¹H NMR (300 MHz, DMSO, δ , ppm): 2.35 (s, 3H, -CH₃), 7.24-8.20 (11H, m, Ar-H), 8.78 (1H, s, Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ , ppm): 158.2 (Ar-CH=N-Ar), 142.3 (-Ar-N=), 140.5 (-Ar-CH₃), 130.1-119.3 (other aromatic karbon). Anal. Calcd. for $C_{18}H_{15}N$ %: C 88.13, H 6.16, N 5.71; found: C 88.20, H 6.20, N 5.80.

N-(4-Nitrobenzylidene)naphthalene-1-amine (3c); dark yellow solid; m.p. 160-162 °C; (85%) yield. IR(KBr, cm^{-1}): 3018 (aromatic C-H), 2955 (azomethine C-H), 1603 (Ar-CH=N-Ar), 1568 (-C=C-), 1332 (-N=O). ¹H NMR (300 MHz, DMSO, δ , ppm): 7.36-8.57 (11H, m, Ar-H), 8.86 (1H, s, -Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ , ppm): 158.2 (-CH=N-), 142.3 (-Ar-N=), 143.5 (-Ar-NO₂), 130.1-119.3 (other aromatic karbon). Anal. Calcd. for $C_{17}H_{12}N_2O_2$ %: C 73.90, H 4.38, N 10.14, O 11.58; found: C 73.92, H 4.25, N 10.07, O 11.50.

N-(4-Chlorobenzylidene)naphthalene-1-amine (3d); yellow solid; m.p. 103-105 °C; (78%) yield. IR (KBr, cm^{-1}): 3020 (aromatic C-H), 2947 (azomethine C-H), 1620 (Ar-CH=N-Ar), 1558 (-C=C-), 1108-1035 (-C-Cl). ¹H NMR (300 MHz, DMSO, δ , ppm): 7.23-8.21 (11H, m, Ar-H), 8.64 (1H, s, Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ , ppm): 158.2 (-C=N-), 142.3 (-Ar-N=), 135.3 (-Ar-Cl), 130.1-119.3 (other aromatic karbon). Anal. Calcd. for $C_{17}H_{12}NCl$ %: C 77.84, H 4.55, Cl 13.34, N 5.30; found: C 76.99, H 4.49, Cl 13.30, N 5.26.

N-(4-methoxybenzylidene)naphthylamine(3e); yellow solid; m.p. 75-77 °C; (79%) yield. IR (KBr, cm^{-1}): 3024 (aromatic C-H), 2943 (azomethine C-H), 1654 (Ar-CH=N-Ar), 1547 (-C=C-), 1128 (CH₃-O-). ¹H NMR (300 MHz, DMSO, δ , ppm): 3.68 (s, 3H, -OCH₃), 7.12-7.90 (11H, m, Ar-H), 8.60 (1H, s, Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ , ppm): 162.4 (4-OMe-Ar-), 160.0 (-CH=N-), 142.3 (-Ar-N=), 130.1-119.3 (other aromatic karbon), 56.7 (-O-CH₃). Anal. Calcd. for $C_{18}H_{15}NO$ %: C 82.73, H 5.50, N 5.36, O 6.12; found: C 82.80, H 5.49, N 5.20, O 6.09.

N-(2,3-dimethoxybenzylidene)naphthylamine(3f); yellow solid; m.p. 85-87 °C; (74%) yield. IR (KBr, cm^{-1}): 3018 (aromatic C-H), 2945 (azomethine C-H), 1660 (Ar-CH=N-Ar), 1547 (-C=C-), 1137 (CH₃-O-). ¹H NMR (300 MHz, DMSO, δ , ppm): 3.63 (s, 6H, -OCH₃), 7.12-7.90 (10H, m, Ar-H), 8.58 (1H, s, Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ , ppm): 160.2 (-CH=N-), 142.3 (-Ar-N=), 141.3 (2-CH₃O-Ar-), 139.3 (3-CH₃O-Ar-), 130.1-119.3 (other aromatic karbon).

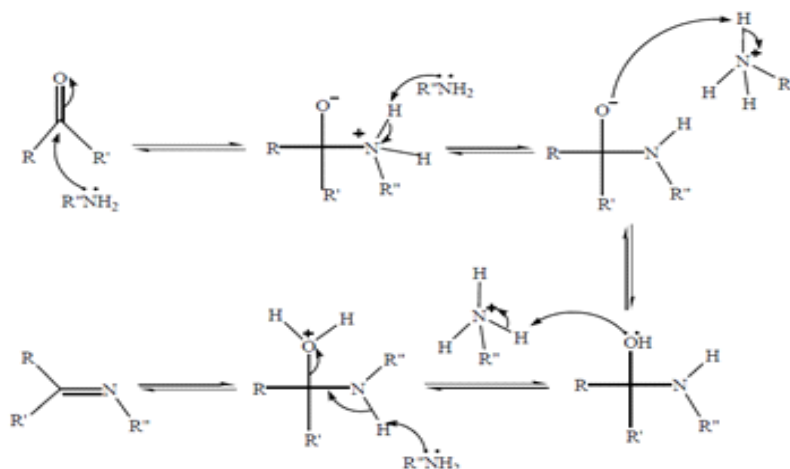
karbon), 58.2 (-O-CH₃). Anal. Calcd. for C₁₉H₁₇NO₂ %: C 78.73, H 5.80, N 4.81, O 10.98; found: C 78.80, H 5.79, N 4.80, O 10.95.

N-(4-hydroxybenzylidene)naphy-amine(3g); yellow solid; m.p. 91-93 °C; (85%) yield. IR (KBr, cm⁻¹): 3320 (O-H), 3024 (aromatic C-H), 2958 (azomethine C-H), 1615 (Ar-CH=N-Ar), 1558 (-C=C-). ¹H NMR (300 MHz, DMSO, δ, ppm): 4.68 (s,1H,-OH), 6.90-7.80 (11H,m,Ar-H), 8.56 (1H,s,Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ, ppm): 161.3 (4-OH-Ar), 160.2 (-CH=N-), 142.3 (-Ar-N=), 130.1-119.3 (other aromatic karbon), 58.2 (-O-CH₃). Anal. Calcd. for C₁₇H₁₃NO %: C 82.73, H 5.50, N 5.36, O 6.12; found: C 82.80, H 5.49, N 5.20, O 6.09.

N-(3,4-dihydroxybenzylidene)naphy-amine(3h); yellow solid; m.p. 98-100 °C; (78%) yield. IR (KBr, cm⁻¹): 3400-3320 (-O-H), 3028 (aromatic C-H), 2960 (azomethine C-H), 1610 (Ar-CH=N-Ar), 1560 (-C=C-). ¹H NMR(300 MHz, DMSO, δ, ppm): 4.73 (s,2H,-OH), 6.90-7.80 (10H,m,Ar-H), 8.56 (1H,s,Ar-CH=N-Ar). ¹³C NMR (75 MHz, DMSO, δ, ppm): 160.2 (Ar-CH=N-Ar), 142.3 (-Ar-N=), 139.3 (4-OH-Ar-), 136.9 (3-OH-Ar-), 130.1-119.3 (other aromatic karbon). Anal. Calcd. for C₁₇H₁₃NO₂ %: C 77.55, H 4.98, N 5.32, O 12.15; found: C 77.63, H 5.00, N 5.20, O 12.09.

3. Results

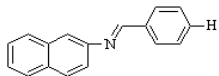
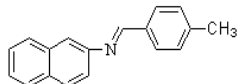
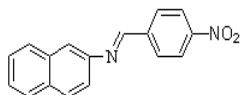
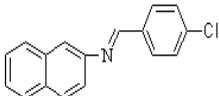
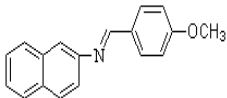
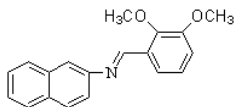
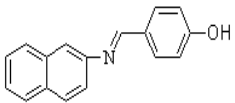
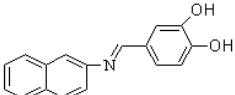
In our research,the Schiff base benzylidene aminonaphthalenes (3a-h) were prepared by refluxing an appropriate amount of 1-naphthylamine with the different aromatic aldehydes in methanol under mild experimental conditions. [34]. For purification, the products obtained were poured on cold water and filtered. The efficiency of the products obtained was between 74-91% conversion. In addition, the reaction mechanism of the Schiff bases obtained was considered to be as outlined in Scheme 3.



Scheme 3: Reaction mechanism of schiff base synthesis

The structures of the synthesized all compounds were characterized with the help of their $^1\text{H}/^{13}\text{C}$ NMR, IR and Mass spectroscopic studies. The spectral data results showed that all the compounds (3a-h) were successfully synthesized. Some physical properties and analytical data of the imines compounds were summarized in Table 1.

Table 1: New Schiff base compounds (3a-h)

Compound	Product	Melting Point (°C)	Yield (%)
3a		121-123	91
3b		69-71	89
3c		160-162	85
3d		103-105	78
3e		75-77	79
3f		85-87	74
3g		91-93	85
3h		98-100	78

4. Discussion

According to the literature research, all spectral data of the synthesized Schiff Bases (3a-h) were found to be consistent with the expected results. In the IR spectra displayed characteristic absorption bands at around 3438, 3020, 2960, 1620, 1480, 1330 cm^{-1} regions, confirming the presence of (-O-H), aromatic (C-H), azomethine (C-H), (C=N), (C=C) respectively [35, 18]. A strong absorption band at 1558-1660 cm^{-1} was due to (C=N) vibration [36]. The absorption bands at 3012- 3139 cm^{-1} and 1568-1547 cm^{-1} belonged to the stretching frequency of the aromatic ring. The strong absorption band in the region 1137-1128 cm^{-1} arose from vibrations of the (CH_3O -)

group present in 3e, 3f. Compounds 3g and 3h gave rise to a strong band at 3400-3320 cm^{-1} due to stretching vibrations of the (-O-H) bond [31]. Also, compound 3d had strong (-C-Cl) peak nearly at 850 cm^{-1} . Compound 3c has (-NO₂) group. This stretching was observed at 1548 cm^{-1} as very strong peak. The infrared spectral data of the Schiff base matched the expected range and total IR results were given in Table 2.

Table 2: IR spectral data (cm^{-1})

Compound	Aromatic C-H	Azomethine C-H	C=N	Aromatic C=C
3a	3018	2926	1600	1560
3b	3023	2935	1605	1562
3c	3018	2955	1603	1568
3d	3020	2947	1620	1558
3e	3024	2943	1654	1547
3f	3018	2945	1660	1547
3g	3024	2958	1615	1558
3h	3028	2960	1610	1560

Further, we observed a singlet of integration intensity equivalent to one hydrogen at 8.56–8.86 ppm in the ¹H NMR spectra of the Schiff bases, indicating the presence of the azomethine proton (-CH=N-) [37, 38]. The peaks of naphthalene aromatic and phenyl group protons appeared as multiple signal at 7.26-8.57 ppm. Also compounds 3e and 3f (-O-CH₃) protons were found 3.63 ppm as singlet signal. The peak at 2.35 ppm was due to three methyl protons (s,-CH₃) in 3b. Finally, signal of (-OH) groups protons in compounds 3g and 3h were observed nearly at 4.00 ppm. The ¹H NMR spectroscopy provided an additional support for the formation of Schiff base derivatives and total ¹H NMR results were given in Table 3.

Table 3: ¹H NMR chemical shifts (ppm)

Compound	Aromatic C-H	Azomethine C-H
3a	8.80	7.26-8.27
3b	8.78	7.24-8.20
3c	8.86	7.36-8.57
3d	8.64	7.23-8.21
3e	8.60	7.12-7.90
3f	8.58	7.12-7.90
3g	8.56	6.90-7.80
3h	8.56	6.90-7.80

For compound 3(a-h), characteristic ^{13}C -NMR signal of the azomethine group ($-\text{C}=\text{NH}$) was observed at 158.2-163.4 ppm. This characteristic peak has been found for the others at 162.4(4-OMe-Ar-), 143.5(-Ar-NO₂), 142.3(-Ar-N=), 135.3(-Ar-Cl), 139.3(4-OH-Ar), 136.9(3-OH-Ar), and 58.2(-O-CH₃), respectively. Furthermore, signal of aromatic carbons (compound 3) were observed at 130.1-119.3 ppm. The purity of all imine compounds was confirmed by mass analysis.

5. Conclusions

The structure of the newly synthesized compounds was elucidated on the basis of elemental analysis and spectral data. When all analysis results of the synthesized compounds were examined, the presence of characteristic peaks proving the formation of imine was observed and the synthesis of the compounds was successful. Also, this synthesis has quite environmentally friendly synthesis method because methanol was used as solvent and no need to any catalysts.

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