Synthesis of Some New 4-[2-(2-methylbenzoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one Derivatives with Their Antioxidant Properties

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ABSTRACT

Aims: To synthesize heterocyclic Schiff bases and evaluate their antioxidant activity.
Study Design: Synthesizing some new 4,5-dihydro-1H-1,2,4-triazol-5-one derivatives and determining their chemical structure via IR and NMR spectroscopy. The synthesized new compounds were analyzed for their in vitro potential antioxidant activities.
Place and Duration of Study: Department of Chemistry, Kafkas University, Kars, Turkey. Between February 2014 to February 2015.
Methodology: Nine new 3-alkyl(aryl)-4-[2-(2-methylbenzoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-ones (3) were synthesized by the reactions of 3-alkyl(aryl)-4-amino-4,5-dihydro-1H-1,2,4-triazol-5-ones (1) with 2-(2-methylbenzoyl)benzaldehyde (2) which had also been synthesized by the reactions of 2-hydroxybenzaldehyde with 2-methylbenzoyl chloride by using triethylamine.

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Results: Schiff bases were synthesized and their structures were determined with spectral methods. Antioxidant evaluation was carried out and discussed. Conclusion: Synthesis and structural determination of the new 3-alkyl(aryl)-4-[2-(2-methylbenzoxoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-ones (3) was successfully done. The antioxidant evaluation was determined.

Keywords: 1,2,4-Triazol-5-one; synthesis; Schiff base; antioxidant activity.

1. INTRODUCTION

In the last two decades there has been a growing attention in the role of reactive oxygen species (ROS) and nitrogen species (RNS) in food, drugs, and even living system. Therefore, scientists in diverse disciplines have become more curious about naturally-occurring antioxidant as well as in related synthetic derivatives that could supply active components which prohibit or decrease the effect of oxidative stress [1].

External chemicals and internal metabolic processes in the human body or in the food system may generate highly reactive free radicals. At high concentrations, they could be important mediators of damage among cell structures, including lipids and membranes, proteins, and nucleic acids [2]. In this regard, it is important to search for and synthesize new classes of compounds that have antioxidant properties.

1,2,4-Triazole derivatives are recorded to own a wide variety of pharmacological activities like antibacterial, antioxidant, anti-inflammatory, antiparasitic, analgesic, antiviral, antitumor, anti-HIV, antihypertensive and diuretic properties [3-11]. Besides, a few articles declaring the synthesis of several N-arylidenediamino-4,5-dihydro-1H-1,2,4-triazol-5-one derivatives have been reported so far [3,4].

In this study, nine new 3-alkyl(aryl)-4-[2-(2-methylbenzoxoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-ones (3a-i) were synthesized from the reactions of 3-alkyl(aryl)-4-amino-4,5-dihydro-1H-1,2,4-triazol-5-ones (1a-i) with 2-(2-methylbenzoxoxy)-benzaldehyde (2). In addition, due to a wide range of applications to find their possible antioxidant activity, the newly synthesized 3 type compounds were investigated by using different antioxidant assayss like; reducing power, 1,1-diphenyl-2-picryl-hydrazyl (DPPH) free radical scavenging activity and iron binding effect.

2. METHODOLOGY

2.1 Chemical Reagents and Apparatus

Chemical reagents used in this study were purchased from Merck AG, Aldrich and Fluka. Melting points were determined in open glass capillaries using a Stuart SMP30 melting point apparatus and are uncorrected. The IR spectra were recorded on an Alpha-P Bruker FT-IR spectrometer. 1H and 13C NMR spectra were recorded in deuterated dimethyl sulfoxide with TMS as internal standard using a Bruker Ultrashield Plus Biospin spectrometer. Extinction coefficients (ε) are expressed in L·mol⁻¹·cm⁻¹.

2.2 Synthesis

2.2.1 Procedure for the synthesis of 2-benzyloxy-3-ethoxy-benzoaldehyde (2)

2-hydroxybenzaldehyde (0.01 mol) dissolved in ethyl acetate (100 mL) was treated with 2-methylbenzolyl chloride (0.01 mol), and to this solution was slowly added triethylamine (0.02 mol) with stirring at 0-5°C. Stirring was continued for 2 h, and then the mixture was refluxed for 3 h and filtered. The filtrate was evaporated in vacuo, and the crude product was washed with water and recrystallized from ethanol to afford compound 2, yield 94.76%. Mp: 64°C. IR (cm⁻¹) 2863 and 2763 (C=O), 1737, 1690 (C=O), 1237 (N=O), 1H NMR (400MHz, DMSO-d₆) δ 2.62 (s, 3H, PhCHO), 7.42-7.50 (m, 3H, ArH), 7.55-7.63 (m, 2H, ArH), 7.80-7.84 (m, 1H, ArH), 8.01 (d, 1H, ArH; J=7.60 Hz), 8.21 (d, 1H, ArH; J=8.00 Hz), 10.13 (s, 1H, CHO), 13C NMR (100MHz, DMSO-d₆) δ 21.15 (PhCHO), 123.99, 126.17, 126.72, 127.87, 128.18, 131.06, 131.76, 131.83, 133.08, 135.65, 140.46, 150.79 (Ar-C), 165.06 (C=O); UV (C₂H₅OH) λ max (log ε) 286 (6597), 242 (15905) nm.
2.2.2 General procedure for the synthesis of compounds 3

The corresponding compound 1 (0.01 mol) was dissolved in acetic acid (15 mL) and treated with 2-(2-methylbenzoxy) benzaldehyde 2 (0.01 mol). The mixture was refluxed for 1.5 h and then evaporated at 50-55°C in vacuo. Several recrystallizations of the residue from appropriate solvent gave pure compounds 3a-i as colorless crystals.

2.2.2.1 3-Methyl-4-[2-(2-methylbenzoxo)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3a)

Yield 98.21%. Mp: 230°C. IR (cm⁻¹) 3169 (O=H), 1737, 1706 (C=O), 1601, 1578 (C=O), 1231 (O=C=O); ¹H NMR (400MHz, DMSO-d₆) δ 7.50 (m, 4H, ArH), 7.58-7.64 (m, 2H, ArH), 8.06 (d, 1H, ArH; J=0.60 Hz), 8.16-8.18 (m, 1H, ArH), 9.91 (CH=O); ¹³C NMR (100MHz, DMSO-d₆) δ 123.14 (CH₃), 129.27, 129.10, 130.35, 130.88, 131.28, 133.36, 140.09, 149.89 (Ar-C), 149.26, 148.72 (N=CH), 152.09 (triazole C₆), 152.24 (triazole C₅), 215.10 (C=O); UV (C₆H₅OH) λ_max (log ν) 290 (14011), 243 (17862), 224 (16019) nm.

2.2.2.2 3-Ethyl-4-[2-(2-methylbenzoxo)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3b)

Yield 99.41%. Mp: 202°C. IR (cm⁻¹) 3159 (O=H), 1727, 1702 (C=O), 1595, 1574 (C=O), 1232 (O=C=O); ¹H NMR (400MHz, DMSO-d₆) δ 7.57-7.69 (m, 2H, ArH), 8.05 (d, 1H, ArH; J=7.50 Hz), 8.16-8.18 (m, 1H, ArH), 8.12 (s, 1H, N=CH), 11.28 (s, 1H, NH); ¹³C NMR (100MHz, DMSO-d₆) δ 112.59 (CH₃), 18.27 (CH₃), 122.74, 126.91, 126.35, 126.88, 127.01, 127.77, 130.49, 131.87, 132.77, 133.18, 140.42, 149.87 (Ar-C), 147.94 (triazole C₆), 148.38 (N=CH), 151.37 (triazole C₅), 164.09 (C=O); UV (C₆H₅OH) λ_max (log ν) 290 (13412), 240 (22315) nm.

2.2.2.3 3-Propyl-4-[2-(2-methylbenzoxo)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3c)

Yield 96.88%. Mp: 174°C. IR (cm⁻¹) 3163 (O=H), 1739, 1709 (C=O), 1595 (C=O), 1249 (C=O); ¹H NMR (400MHz, DMSO-d₆) δ 0.86 (t, 3H, CH₃CH₂CH₃), J=7.20 Hz), 1.60 (sext, 2H, CH₂CH₂CH₃), J=7.20 Hz), 2.50 (t, 2H, CH₂CH₂CH₃), J=7.20 Hz), 2.61 (s, 3H, PhCH₃), 7.42-7.51 (m, 4H, ArH), 7.59-7.67 (m, 2H, ArH), 8.04 (d, 1H, ArH; J=8.00 Hz), 8.17-8.19 (m, 1H, ArH), 9.96 (s, 1H, N=CH), 11.83 (s, 1H, NH); ¹³C NMR (100MHz, DMSO-d₆) δ 13.26 (CH₃CH₂CH₃), 18.90 (CH₃CH₂CH₃), 21.13 (PhCH₃), 26.48 (CH₃CH₂CH₃), 123.76, 126.23, 126.35, 126.71, 127.10, 127.79, 130.91, 131.88, 132.49, 133.18, 140.40, 149.85 (Ar-C), 146.80 (triazole C₆), 148.58 (N=CH), 151.05 (triazole C₅), 165.08 (C=O); UV (C₆H₅OH) λ_max (log ν) 290 (11491), 242 (14578), 224 (16205) nm.

2.2.2.4 3-Benzyl-4-[2-(2-methylbenzoxo)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3d)

Yield 96.68%. Mp: 227°C. IR (cm⁻¹) 3161 (O=H), 1727, 1706 (C=O), 1590 (C=O), 1231 (O=C=O); ¹H NMR (400MHz, DMSO-d₆) δ 2.60 (s, 3H, PhCH₃), 3.99 (s, 2H, CH₂Ph), 7.23-7.35 (m, 5H, ArH), 7.41-7.50 (m, 4H, ArH), 7.58-7.67 (m, 2H, Ar-H), 8.01-8.03 (m, 1H, Ar-H), 8.11-8.18 (m, 1H, Ar-H), 9.08 (s, 1H, N=CH), 10.95 (s, 1H, NH); ¹³C NMR (100MHz, DMSO-d₆) δ 21.12 (PhCH₃), 30.88 (CH₂Ph), 123.67, 126.16, 126.24, 126.47, 126.71, 127.76, 128.43 (2C), 128.76, 129.81 (2C), 130.86, 131.88, 132.58, 133.20, 135.66, 140.37, 150.05 (Ar-C), 146.16 (triazole C₆), 147.87 (N=CH), 151.23 (triazole C₅), 165.14 (C=O); UV (C₆H₅OH) λ_max (log ν) 292 (7905), 238 (14198) nm.

2.2.2.5 3-p-Methylbenzyl-4-[2-(2- methybenzoxo)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3e)

Yield 98.73%. Mp: 226°C. IR (cm⁻¹) 3166 (O=H), 1732, 1701 (C=O), 1594 (C=O), 1231 (O=C=O); ¹H NMR (400MHz, DMSO-d₆) δ 2.25 (s, 3H, p-PhCH₃), 2.60 (s, 3H, o-PhCH₃), 3.93 (s, 2H, CH₂Ph), 7.12 (d, 2H, J=8.00 Hz), 7.19 (d, 2H, Ar-H; J=8.00 Hz), 7.40-7.50 (m, 4H, ArH), 7.58-7.66 (m, 2H, ArH), 8.03-8.05 (m, 1H, Ar-H), 8.16-8.18 (m, 1H, Ar-H), 9.67 (s, 1H, N=CH); ¹³C NMR (100MHz, DMSO-d₆) δ 20.58 (p-PhCH₃), 21.11 (o-PhCH₃), 30.49 (CH₂Ph), 123.67, 126.18, 126.22, 126.46, 126.68, 127.76, 128.63 (2C), 128.98 (2C), 130.86, 131.87, 132.48, 132.54, 133.18, 135.77, 140.37, 150.05 (Ar-C), 146.30 (triazole C₆), 147.81 (N=CH), 151.24 (triazole C₅), 165.15 (C=O); UV (C₆H₅OH) λ_max (log ν) 290 (7834), 238 (14242), 222 (14844) nm.
2.2.2.6 3-p-Methoxybenzyl-4-[2-(2-methylbenzoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3f)

Yield 98.33%. Mp: 206°C. IR (cm⁻¹) 3163 (νNH), 1728, 1708 (C=O); UV (C₂H₅OH) λ_max (log ε) 291 (12342), 239 (22237), 222 (24678).

2.2.2.7 3-p-Chlorobenzyl-4-[2-(2-methylbenzoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-one (3g)

Yield 99.33%. Mp: 228°C. IR (cm⁻¹) 3163 (νNH), 1737, 1702 (νC=O), 1595 (νC=O), 1231 (νC=O). ¹H NMR (400 MHz, DMSO-d₆) δ 2.60 (s, 3H, PhCH₃), 7.32 (d, 2H, ArH; J=8.40 Hz), 7.38 (d, 2H, ArH; J=8.40 Hz), 7.39-7.49 (m, 4H, ArH), 7.58-7.67 (m, 2H, Ar-H), 8.00-8.03 (m, 1H, Ar-H), 8.16-8.18 (m, 1H, Ar-H), 9.97 (s, 1H, N=CH), 11.98 (s, 1H, NH). ¹³C NMR (100 MHz, DMSO-d₆) δ 21.12 (PhCH₃), 30.20 (CH₃Ph), 123.68, 126.12, 126.23, 126.52, 126.69, 127.74, 128.35 (2C), 130.69 (2C), 130.86, 131.45, 131.87, 132.60, 133.19, 134.60, 140.38, 150.05 (Ar-C), 145.81 (triazole C₅), 148.01 (N=CH), 151.21 (triazole C₅), 165.11 (C=O); UV (C₂H₅OH) λ_max (log ε) 260 (13116), 234 (16566).

2.3 Antioxidant Activity

2.3.1 Chemicals

Butylated hydroxytoluene (BHT) was obtained from E. Merck. Ferrous chloride, α-tocopherol, DPPH, 3-(2-pyridyl)-5,6-bis(phenylsulfonic acid)-1,2,4-triazine (ferrozine), butylated hydroxyanisole (BHA), ethylene-diaminetetraacetic acid (EDTA) and trichloroacetic acid (TCA) were obtained from Sigma.

2.3.2 Reducing power

The reducing power of the synthesized new compounds was determined according to the method of Oyaizu [12]. Different concentrations of the samples (50-250 μg/mL) in DMSO (1 mL) were mixed with phosphate buffer (2.5 mL, 0.2 M, pH = 6.6) and potassium ferricyanide (2.5 mL, 1%). The mixture was incubated at 50°C for 20 min, after which a portion (2.5 mL) of trichloroacetic acid (10%) was added to the mixture, which was then centrifuged for 10 min at 1000 x g. The upper layer of solution (2.5 mL) was mixed with distilled water (2.5 mL) and FeCl₃ (0.5 mL, 0.1%), and then the absorbance was measured at 700 nm in a spectrophotometer. Higher absorbance of the reaction mixture indicated greater reducing power.

2.3.3 Free radical scavenging activity

Free radical scavenging activity of the new compounds was measured by DPPH, using the method of Blois [13]. Briefly, 0.1 mM solution of
DPPH$^-$ in ethanol was prepared, and this solution (1 mL) was added to sample solutions in DMSO (3 mL) at different concentrations (50-250 µg/mL). The mixture was shaken vigorously and allowed to stand at room temperature for 30 min. Then the absorbance was measured at 517 nm in a spectrophotometer. Lower absorbance of the reaction mixture indicated higher free radical scavenging activity. The DPPH concentration (mM) in the reaction medium was calculated from the following calibration curve and determined by linear regression (R: 0.997):

\[
\text{Absorbance} = 0.0003 \times \text{DPPH}^+ - 0.0174
\]

The capability to scavenge the DPPH radical was calculated using the following equation:

\[
\text{DPPH}^+ \text{ scavenging effect (\%)} = \left( \frac{A_0 - A_t}{A_0} \right) \times 100
\]

where $A_0$ is the absorbance of the control reaction and $A_t$ is the absorbance in the presence of the samples or standards.

### 2.3.4 Metal Chelating Activity

The chelation of ferrous ions by the synthesized new compounds and standards were estimated by the method of Dinis et al. [14]. Briefly, the synthesized compounds (30-90 µg/mL) were added to a 2 mM solution of FeCl$_2$·4H$_2$O (0.05 mL). The reaction was initiated by the addition of 5 mM ferrozine (0.2 mL), and the mixture was shaken vigorously and left standing at the room temperature for 10 min. After the mixture had reached equilibrium, the absorbance of the solution was measured at 562 nm in a spectrophotometer. The percentage inhibition of ferrozine-Fe$^{2+}$ complex formation was given by the formula: % Inhibition = \left( \frac{A_0 - A_t}{A_0} \right) \times 100,

### 3. RESULTS AND DISCUSSION

#### 3.1 Chemistry

In this study the 3-alkyl(aryl)-4-[2-(2-methylbenzoxoxy)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-ones 3a-i were prepared. The starting compounds 1a-i were prepared as described in the literature [15,16]. Compounds 3a-i were synthesized from the reactions of compounds 1a-i with 2-(2-methylbenzoxoxy)benzaldehyde (2) which had also been synthesized by the reactions of 2-hydroxybenzaldehyde with 2-methylbenzoyl chloride by using triethylamine (Scheme 1).

The structures of nine new 3-alkyl(aryl)-4-[2-(2-methylbenzoyl)-benzylidenamino]-4,5-dihydro-1H-1,2,4-triazol-5-ones 3a-i were characterized by using IR, $^1$H-NMR, $^{13}$C-NMR and UV spectral data.

#### 3.2 Antioxidant Activity

The antioxidant activity of nine new compounds 3a-i was determined. Several methods have been used to determine antioxidant activities and the methods used in the study are given below:

##### 3.2.1 Total reductive capability using the potassium ferricyanide reduction method

The reductive capabilities of compounds were assessed by the extent of conversion of the Fe$^{3+}$

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![Scheme 1. Synthetic route of compounds 3a-i](image-url)
ferricyanide complex to the Fe$^{2+}$/ferrous form. The reducing powers of the compounds were observed at different concentrations, and results were compared with BHA, BHT and α-tocopherol. It has been observed that the reducing capacity of a compound may serve as a significant indicator of its potential antioxidant activity [17]. The antioxidant activity of putative antioxidant has been attributed to various mechanisms, among which are prevention chain initiation, binding of transition metal ion catalyst, decomposition of peroxides, prevention of continued hydrogen abstraction, reductive capacity and radical scavenging [18].

In this study, all the amount of the compounds showed lower absorbance than standard antioxidants. Hence, no activities were observed to reduce metal ions complexes to their lower oxidation state or to take part in any electron transfer reaction. In other words, synthesized compounds did not show the reductive activities.

### 3.2.2 DPPH radical scavenging activity

The scavenging the stable DPPH radical model is a widely used method to evaluate antioxidant activities in a relatively short time compared with other methods. The effect of antioxidants on DPPH radical scavenging was thought to be due to their hydrogen donating ability [19]. DPPH is a stable free radical and accepts an electron or hydrogen radical to become a stable diamagnetic molecule [20]. The reduction capability of DPPH radicals was determined by decrease in its absorbance at 517 nm induced by antioxidants. The absorption maximum of a stable DPPH radical in ethanol was at 517 nm. The decrease in absorbance of DPPH radical was caused by antioxidants because of reaction between antioxidant molecules and radical, progresses, which resulted in the scavenging of the radical by hydrogen donation. It is visually noticeable as a discoloration from purple to yellow. Hence, DPPH is usually used as a substrate to evaluate antioxidative activity of antioxidants [21]. In the study, antiradical activities of compounds and standard antioxidants such as BHA and α-tocopherol were determined by using DPPH method. Scavenging effect values of compounds 3a-i, BHA and α-tocopherol at different concentrations are given Fig. 1. The newly synthesized compounds did not show a significant activity as a radical scavenger, but compound 3a showed higher activities than the other compounds and its reductive ability was concentration-dependent as seen in Fig. 1.

### 3.2.3 Ferrous ion chelating activity

The chelating effect towards ferrous ions by the compounds and standards was determined. Ferrozine can quantitatively form complexes with...
Fe$^{2+}$. In the presence of chelating agents, the complex formation is disrupted with the result that the red colour of the complex is decreased. Measurement of colour reduction therefore allows estimation of the chelating activity of the coexisting chelator [22]. Transition metals have significant role in the generation oxygen free radicals in living organism. The ferric iron (Fe$^{3+}$) is the relatively biologically inactive form of iron. However, it can be reduced to the active Fe$^{2+}$, depending on condition, particularly pH [23] and oxidized back through Fenton type reactions with the production of hydroxyl radical or Haber-Weiss reactions with superoxide anions. The production of these radicals may lead to lipid peroxidation, protein modification and DNA damage. Chelating agents may not activate metal ions and potentially inhibit the metal-dependent processes [24]. Also, the production of highly active ROS such as O$_2^\cdot$, H$_2$O$_2$ and OH$^\cdot$ is also catalyzed by free iron though Haber-Weiss reactions:

$$\text{O}_2 + \text{H}_2\text{O}_2 \rightarrow \text{O}_2 + \text{OH}^\cdot + \text{OH}^\cdot$$

Among the transition metals, iron is known as the most important lipid oxidation pro-oxidant due to its high reactivity. The ferrous state of iron accelerates lipid oxidation by breaking down the hydrogen and lipid peroxides to reactive free radicals via the Fenton reactions:

$$\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{OH}^\cdot + \text{OH}^\cdot$$

Fe$^{3+}$ ion also produces radicals from peroxides, even though the rate is tenfold less than that of Fe$^{2+}$ ion, which is the most powerful pro-oxidant among the various types of metal ions [25]. Ferrous ion chelating activities of compounds 3a-i and standard antioxidants are shown in Fig. 2. It was reported that chelating agents that form $\sigma$-bonds with a metal are effective as secondary antioxidants because they reduce the redox potential thereby stabilizing the oxidized form of metal ion [26].

Low absorbance at 562 nm indicates high metal chelating activity. The data obtained from Fig. 2 reveal that the metal chelating effects of the compounds were not concentration-dependent. At the lowest concentration, all of the compounds showed better concentrations than $\alpha$-tocopherol.

4. CONCLUSION

In this study, the structures of nine new 4,5-dihydro-1H-1,2,4-triazol-5-one derivatives synthesized from the reactions of 1 type compounds with a benzaldehyde derivative were identified using IR, $^1$H NMR, $^{13}$C NMR, and UV spectral data. The newly synthesized compounds were screened for their antioxidant activities.

COMPETING INTERESTS

Authors have declared that no competing interests exist.
REFERENCES


**APPENDIX**

**Fig. 1.** IR spectrum of compound 2

**Fig. 2.** $^1$H-NMR spectrum of compound 2
Fig. 3. $^{13}$C-NMR spectrum of compound 2

Fig. 4. UV spectrum of compound 2
Fig. 5. IR Spectrum of compound 3a

Fig. 6. $^1$H-NMR Spectrum of Compound 3a
Fig. 7. $^{13}$C-NMR spectrum of compound 3a

Fig. 8. UV spectrum of compound 3a
Fig. 9. IR spectrum of compound 3b

Fig. 10. $^1$H-NMR spectrum of compound 3b
Fig. 11. $^{13}$C-NMR Spectrum of Compound 3b

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Fig. 12. UV spectrum of compound 3b
Fig. 13. IR spectrum of compound 3c

Fig. 14. $^1$H-NMR spectrum of compound 3c
Fig. 15. $^{13}$C-NMR Spectrum of Compound 3c

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Fig. 16. UV spectrum of compound 3c
Fig. 17. IR spectrum of compound 3d

![IR spectrum of compound 3d](image)

Fig. 18. $^1$H-NMR spectrum of compound 3d

![$^1$H-NMR spectrum of compound 3d](image)
Fig. 19. $^{13}$C-NMR Spectrum of Compound 3d

Fig. 20. UV spectrum of compound 3d
Fig. 21. IR Spectrum of Compound 3e

Fig. 22. $^1$H-NMR spectrum of compound 3e
Fig. 23. $^{13}$C-NMR Spectrum of Compound 3e

Fig. 24. UV spectrum of compound 3e
**Fig. 25.** IR spectrum of compound 3f

**Fig. 26.** $^1$H-NMR spectrum of compound 3f
**Fig. 27.** $^{13}$C-NMR spectrum of compound 3f

**Fig. 28.** UV spectrum of compound 3f
Fig. 29. IR spectrum of compound 3g

Fig. 30. $^1$H-NMR spectrum of compound 3g
Fig. 31. $^{13}$C-NMR spectrum of compound 3g

![C-NMR spectrum of compound 3g](image)

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Fig. 32. UV spectrum of compound 3g

![UV spectrum of compound 3g](image)
Fig. 33. IR spectrum of compound 3h

Fig. 34. ¹H-NMR spectrum of compound 3h
Fig. 35. $^{13}$C-NMR spectrum of compound 3h

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Fig. 36. UV spectrum of compound 3h
Fig. 37. IR spectrum of compound 3i

Fig. 38. $^1$H-NMR spectrum of compound 3i
Fig. 39. $^{13}$C-NMR spectrum of compound 3i

![C-NMR spectrum of compound 3i](image)

<table>
<thead>
<tr>
<th>No.</th>
<th>P/V</th>
<th>Wavelength(nm)</th>
<th>Abs</th>
<th>Comment</th>
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<td>1</td>
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<td>1.988</td>
<td>14232</td>
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<tr>
<td>2</td>
<td>Peak</td>
<td>234.00</td>
<td>1.574</td>
<td>32223</td>
</tr>
</tbody>
</table>

Fig. 40. UV spectrum of compound 3i

![UV spectrum of compound 3i](image)

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