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Benzoil Ester Grubu İçeren 2,4-Dinitrofenilhidrazin Temelli Bazı Yeni Hidrazon Bileşiklerinin Sentezi ve Yapısal Karakterizasyonu

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Özet

Hidrazon bileşikleri, ilaç tasarımı çalışmalarında aday bileşikleri elde etmek için kullanılan önemli öncülerdir. Bu çalışmada, başlangıç materyali olarak 4-(dietilamino) salisilaldehitten türetilen benzoil ester türevleri (**1-5**) sübstitüe benzoil klorür türevleri (benzoil klorür, 2-nitrobenzoil klorür, 3-nitrobenzoil klorür, 4-nitrobenzoil klorür ve 3,5-dinitrobenzoil klorür) ile çözücü olarak piridin ortamında 1:1 mol oranında reaksiyona girmesiyle sentezlendi. Elde edilen benzoil esterler ile 2,4-dinitrofenilhidrazinin kondenzasyon reaksiyonu ile yeni bir dizi hidrazon bileşikleri (**6-10**) sentezlendi ve bu bileşikler yapısal karakterizasyonu FT-IR, ^{1}H NMR, ^{13}C NMR ve element analizi aydınlatıldı. Sonuç olarak, bu bileşiklerin biyolojik aktiviteler gösterebileceği düşünülmektedir.

Synthesis and Structural Characterization of Some Novel Hydrazone Compounds Based on 2,4-Dinitrophenylhydrazine Containing Benzoyl Ester Group

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ABSTRACT

Hydrazone compounds are important precursors employed to obtain candidate compounds in drug design studies. In the present study, benzoyl ester derivatives (**1-5**) derived from 4-(diethylamino) salicylaldehyde as a starting material were synthesized by reacting with substituted benzoyl chloride derivatives (benzoyl chloride, 2-nitrobenzoyl chloride, 3-

Keywords:
Synthesis Benzoyl Ester, Hydrazone, Characterization

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nitrobenzoyl chloride, 4-nitrobenzoyl chloride and 3,5-dinitrobenzoyl chloride) in a 1:1 mole ratio in pyridine as a solvent. A new series of hydrazone compounds (**6-10**) were synthesized by the condensation reaction of 2,4-dinitrophenylhydrazine with the obtained benzoyl esters, and the structural characterization of these compounds was clarified by FT-IR, ^{1}H NMR, ^{13}C NMR and elemental analysis. As a result, it is thought that these compounds may show biological activities.

1. INTRODUCTION

Nowadays, it is necessary to design and develop new drug candidates, since many drugs used in clinical practice are insufficient against some diseases, especially cancer, Alzheimer and epilepsy. In addition, new drug candidates are needed for use in the treatment of emerging diseases such as Covid 19. The goal of drug design is to discover less toxic and side-effects but more potent therapeutic agents. In line with this purpose, studies are constantly being carried out to design and synthesize new bioactive molecules that can be new drug candidates (Naveen Kumar et al., 2014; Angelova et al., 2016; Haghighijoo et al., 2017; Bingul et al., 2020; Bozkurt et al., 2020; Aktar et al., 2022; Kamalı et al., 2022).

Hydrazone compounds, which are organic molecules with the general formula -CO-NHN=CH- containing an azomethine –NHN=CH– group in their structure, constitute one of the important classes of organic compounds in medicinal chemistry. Hydrazones are obtained by the reaction of hydrazine or hydrazides with aldehydes and ketones (Başaran et al., 2022; Çakmak et al., 2022). These compounds occur as intermediates in the Wolff-Kishner reaction (Ul et al., 2022). Also, hydrazones can be synthesized from β-keto acids or β-keto esters and aryldiazonium salts by the Japp-Klingemann reaction (Wang et al., 2022).

In recent years, hydrazone functional groups are involved in the structure of many bioactive compounds (Rollas and Küçükgüzel, 2006; Asif and Husain, 2013). In addition, it is known that many active pharmaceutical components contain a hydrazone functional group. Due to these properties, this class of compounds has attracted great interest in recent years. Therefore, many medicinal chemists are synthesizing hydrazone-bearing compounds and trying to determine their biological significance by in vitro studies. As a result of these studies, it has been determined that these compounds show important pharmacological activities such as analgesic, anti-inflammatory, antituberculosis, anticancer, anticonvulsant, antihypertensive, anti-HIV, antibacterial and antifungal according to the difference in the substituents they carry (Rollas and Küçükgüzel, 2006; Singh et al., 2016; Shirinzadeh et al., 2011; Surov et al., 2016; Sıcak et al., 2019).

The goal of this research was to synthesis and characterize new hydrazone compounds with various bioactivities. For this purpose, benzoyl esters (**1-5**) from 4-(diethylamino) salicylaldehyde was synthesized in our previous study (Çakmak et al., 2021) were used as starting material. Then, these

benzoyl esters were reacted with 2,4-dinitrophenylhydrazine to obtain target molecules (**6-10**). The characterization of the synthesized target molecules was done by elemental analysis and some spectroscopic methods.

2. EXPERIMENTAL

2.1. Chemistry

All chemicals were procured from Sigma-Aldrich or Merck and used without any additional purification. Thin-layer chromatography (TLC) was employed to monitor the progress of the reaction. Melting points of newly synthesized molecules were measured on a Barnstead IA9100 Electrothermal Digital Melting Points Apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE III 400 MHz spectrometer. The FT-IR spectra were recorded on a Cary 630 FTIR spectrometer with the diamond ATR module at a scan range of 4000–600 cm^{-1}. Elemental analysis was performed on a Thermo Scientific Flash 2000 elemental analyzer.

2.2. The preparation of the benzoyl esters (1-5)

The intermediate compounds (**1-5**) in this study were synthesized in our previous study. These compounds were synthesized according to the general procedure given below. In summary, **1-5** was obtained by the reaction of 4-(diethylamino) salicylaldehyde (5 mmol) with the suitable benzoyl chloride derivatives (5 mmol) under reflux for 1 h. After the reflux is complete, the reaction mixture was left to cool down and then poured onto ice-cold water. After these procedures, the formed precipitate was filtered, and then washed with distilled water. At last, the residue was recrystallized from ethanol to give the aryl ester (Çakmak et al., 2021; Çınar et al., 2021).

2.3. The synthesis of the target molecules (6-10)

A solution of 2,4-dinitrophenylhydrazine (3 mmol) and a benzoyl ester derivative (3 mmol) in absolute ethanol (15 mL) was heated under reflux for 2 h. When the reaction was completed, the reaction mixture was allowed to cool. The crude product was collected by filtration, washed with petroleum ether, and then dried. At last, the residue was recrystallized from ethanol to afford the target molecule (Başaran et al., 2022; Çakmak et al., 2022).

3. RESULT AND DISCUSSION

3.1. Synthesis and characterization

The synthesis method of the target molecules (**6-10**) is illustrated in Scheme 1. These compounds were synthesized in two steps and with high purity. Firstly, intermediates (**1-5**) were facilely obtained by reacting 4-(diethylamino) salicylaldehyde and some benzoyl chloride derivatives in a pyridine medium. The synthesis and characterization of intermediates were discussed in our previous study (Çakmak et al., 2021). In the synthesis step of the target molecules, 4-(diethylamino)salicylaldehyde ester derivatives were reacted with 2,4-dinitrophenylhydrazine in an ethanol medium. As a result, five

new hydrazone derivatives have been synthesized for the first time. The structures of these molecules were characterized by elemental analysis, FT-IR, ^{1}H NMR and ^{13}C NMR.

Scheme 1. Synthetic route for the synthesis of hydrazone compounds

3.2. The characterization of target molecules

In this study, five new hydrazone compounds; 5-(diethylamino)-2-((2-(2,4-dinitrophenyl)hydrazono)methyl)phenyl benzoate (**6**), 5-(diethylamino)-2-((2-(2,4-dinitrophenyl)hydrazono)methyl)phenyl 2-nitrobenzoate (**7**), 5-(diethylamino)-2-((2-(2,4-dinitrophenyl)hydrazono)methyl)phenyl 3-nitrobenzoate (**8**), 5-(diethylamino)-2-((2-(2,4-dinitrophenyl)hydrazono)methyl)phenyl 4-nitrobenzoate (**9**), 5-(diethylamino)-2-((2-(2,4-dinitrophenyl)hydrazono)methyl)phenyl 3,5-dinitrobenzoate (**10**) were obtained as a result of the reaction of 2,4-dinitrophenylhydrazine compounds with benzoyl ester derivatives. All of the compounds were obtained in solid form with a reaction yield of 69-78%. The melting points for the hydrazones (**6-10**) were determined to be between 231-274 °C. In addition, the elemental analysis data of the targeted compounds were compatible with the theoretical data (Table 1).

Table 1. Physical properties and elemental analysis data of synthesized compounds

FT-IR Spectroscopy

FT-IR spectra of all synthesized hydrazones observed strong absorption peaks in the 1735–1747 cm^{-1} range, representing the presence of the carbonyl (C=O), while the C=N stretching band of the imino group was established at 1596–1598 cm^{-1}. The aromatic stretching bands were shown at 3104-2983 cm^{-1}, while N-H stretching bands at 3259–3293 cm^{-1} were observed. Asymmetrical and symmetrical stretching bands of the NO_2 were also detected at 1533–1542 cm^{-1} and 1317–1329 cm^{-1}, respectively (Table 2). When the FT-IR spectrum of the compound **10**, which we have chosen as an example is examined, the N-H stretching band was observed at 3285 cm^{-1}; aromatic asymmetric and symmetrical C-H stretching bands were determined at 3091 cm^{-1} and 2983 cm^{-1}, respectively; C=O stretching band was observed at1745 cm^{-1}; C=N stretching band was detected at 1541 cm^{-1}; Asymmetrical and symmetrical NO_2 stretching band were determined at 1541 cm^{-1} and 1335 cm^{-1}, respectively (Figure 1).

Figure 1. FT-IR spectrum of compound **10**

Table 2. FT-IR data of hydrazone compounds

^{1}H NMR Spectroscopy

Considering the ^{1}H NMR spectra of the hydrazone compounds (**6-10**), the -NH_2 peak of 2,4-dinitrophenylhydrazine between 4-5 ppm disappeared. The NH proton between 11.14-10.96 ppm and the CH=N proton at 6.00 ppm is the most important proofs of the existence of the hydrazone skeleton. The aromatic protons were determined between 6.41-9.42 ppm for targeted compounds. Also, the methyl protons of the diethylamino group resonate as triplet peaks at 1.26-1.27 ppm, while the methylene protons resonate at 3.45-3.47 ppm. also resonated as quartet peaks (Table 3). When the ^{1}H NMR spectrum of the compound **10** we selected as an example was examined, it was determined that the NH proton resonated at 11.20 ppm, while the CH=N proton resonated at 8.01 ppm. The protons of the phenyl rings in the compound were found to have resonance between 6.41 and 9.42 ppm (Figure 2).

Figure 2. ^{1}H NMR spectrum of compound **10**

Table 3. ^{1}H NMR data of hydrazone compounds

^{13}C NMR Spectroscopy

In the ^{13}C NMR spectra, C=N carbon of targeted compounds (**6-10**), which was significant, resonated at 152.15-159.00 ppm. The observation of this peak is another definitive proof of the hydrazone skeleton. For compounds **6-10**, carbon of carbonyl (C=O) was observed at 162.06-164.94 ppm. Carbons of aromatic rings were observed between 99.09-159.41 ppm. Furthermore, methyl carbons and methylene carbons were detected at 12.52-12.88 and 44.46-44.89 ppm, respectively (Table 4). When we examined the ^{13}C NMR spectrum of compound **10**, which we selected among the synthesized compounds, it was observed that the carbon of C=O resonated at 162.06 ppm, while the carbon of C=N resonated at 151.38 ppm. The carbons of the phenyl rings in the structure of this compound resonated between 150.79 and 105.01 ppm. In addition, methyl and methylene carbons belonging to the dimethylamino group in the structure were found to resonate at 12.88 and 44.46 ppm, respectively (Figure 3).

Figure 3. ^{13}C NMR spectrum of compound **10**

Table 4. ^{13}C NMR data of hydrazone compounds

4. CONCLUSION

In this research, we reported the synthesis and characterization of 4-(diethylamino) salicylaldehyde-based hydrazone compounds (**6-10**) are a remarkable class of compounds with diverse biological activities. These hydrazone derivatives as the target compounds were synthesized for the

first time and their structures were elucidated by elemental analysis and some spectroscopic techniques. To determine the biological importance of these compounds, it is planned to examine their antioxidant, anticancer and antibacterial activities as well as their enzyme inhibition activities against some metabolic enzymes (acetylcholinesterase, butyrylcholinesterase and human carbonic anhydrase isoenzymes) in further studies.

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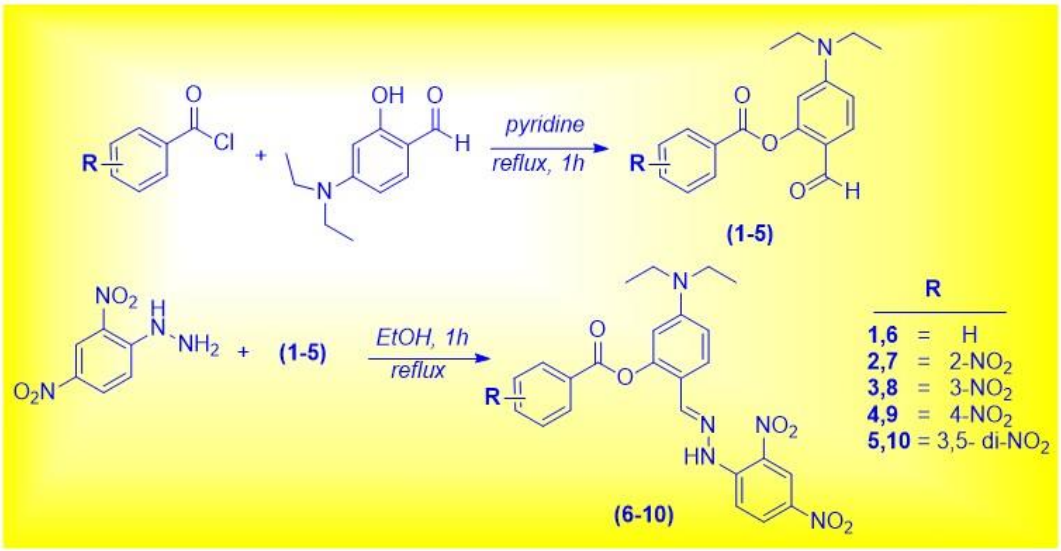
6. FIGURES AND TABLES



Scheme 1. Synthetic route for the synthesis of hydrazone compounds

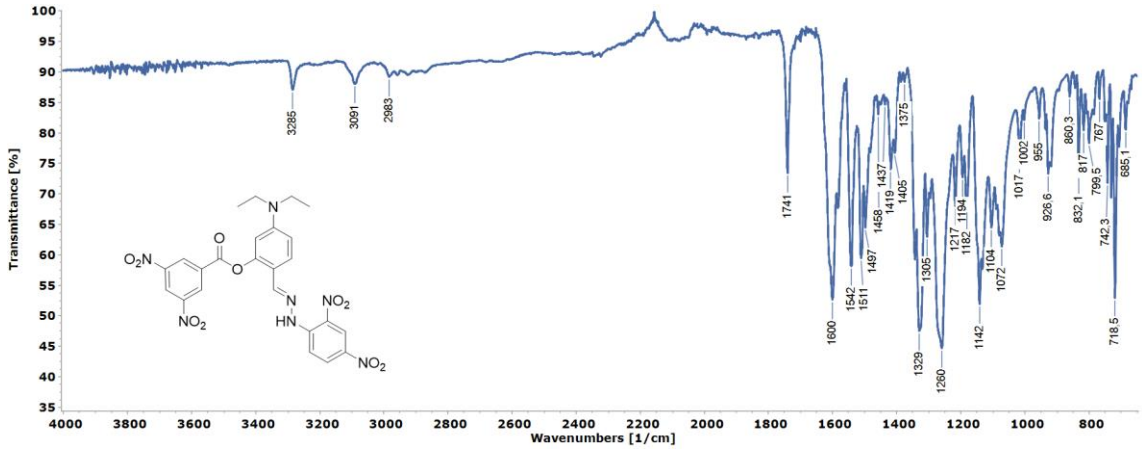


Figure 1. FT-IR spectrum of compound **10**

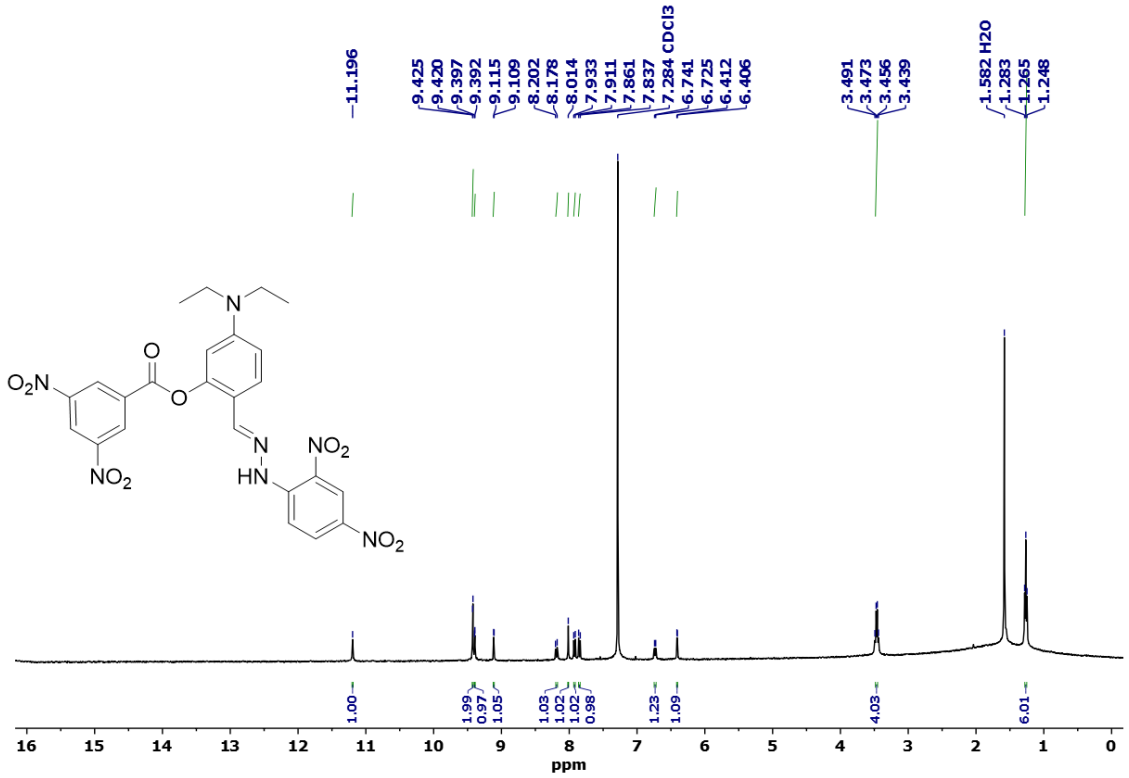


Figure 2. [1]H NMR spectrum of compound **10**

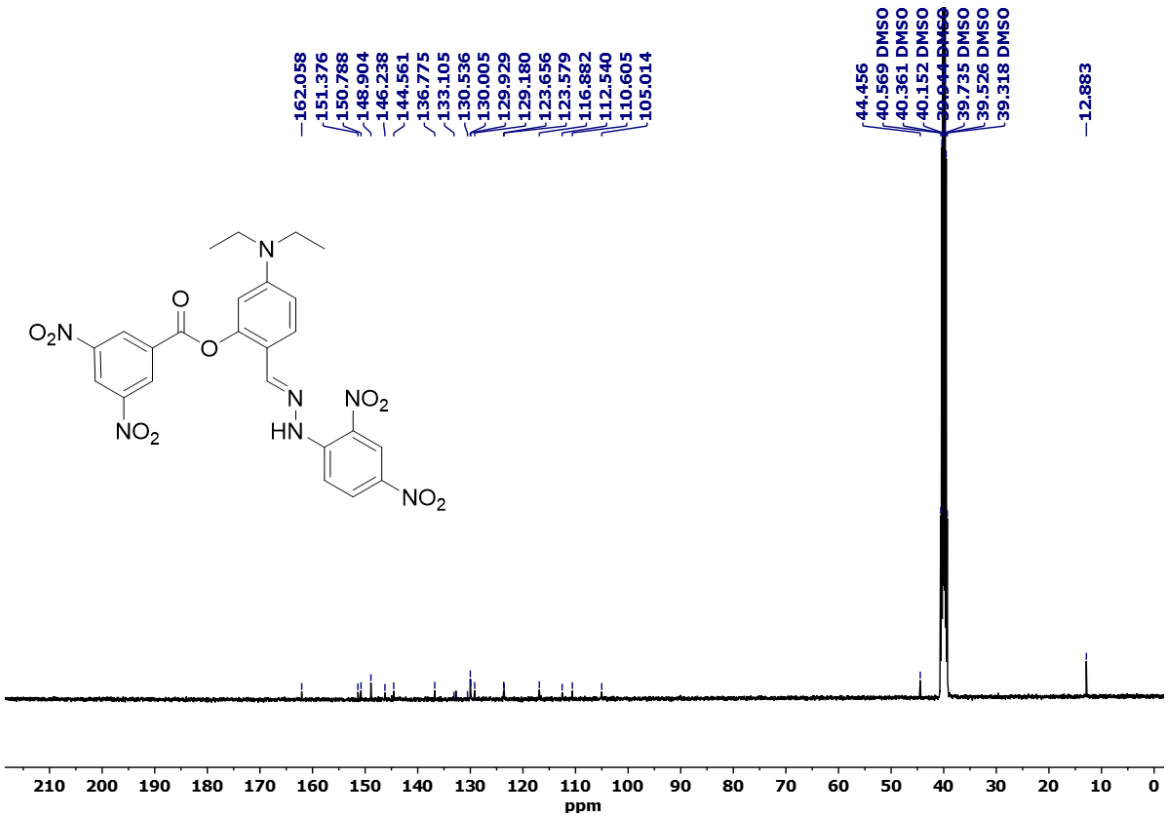


Figure 3. [13]C NMR spectrum of compound **10**

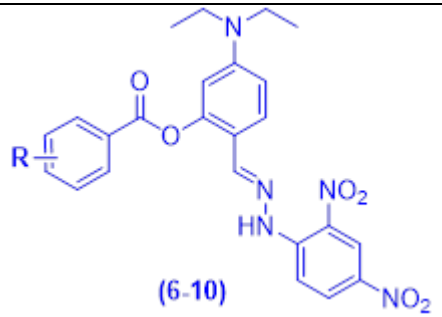
Table 1. Physical properties and elemental analysis data of synthesized compounds

Comp.	Molecular Formula (g/mol)	Color	Elemental Analysis % Calculated - (% Found)			Yield (%)	M.p. (ºC)
			C	***H***	***N***		
6	$C_{24}H_{23}N_5O_6$ (477.48)	Purple	60.37 - (60.49)	4.86 - (4.77)	14.67 - (14.72)	75	241-243
7	$C_{24}H_{22}N_6O_8$ (522.47)	Purple	55.17 - (55.28)	4.24 - (4.30)	16.09 - (16.14)	71	231-232
8	$C_{24}H_{22}N_6O_8$ (522.47)	Dark brown	55.17 - (55.09)	4.24 - (4.26)	16.09 - (16.05)	74	254-256
9	$C_{24}H_{22}N_6O_8$ (522.47)	Dark brown	55.17 - (55.22)	4.24 - (4.17)	16.09 - (16.14)	69	273-274
10	$C_{24}H_{21}N_7O_{10}$ (567.47)	Dark Red	50.80 - (50.93)	3.73 - (3.75)	17.28 - (17.32)	78	258-259

Table 2. FT-IR data of hydrazone compounds

Compound	N-H str. (cm^{-1})	Aromatic C-H str. (cm^{-1})	C=O str. (cm^{-1})	C=N str. (cm^{-1})	NO_2 asymmetric str. (cm^{-1})	NO_2 symmetric str. (cm^{-1})
6	3259	3104, 2970	1735	1598	1541	1324
7	3271	3106, 2966	1741	1598	1533	1327
8	3276	3088, 2974	1733	1596	1541	1321
9	3293	3098, 2971	1747	1598	1542	1317
10	3285	3091, 2983	1741	1600	1542	1329

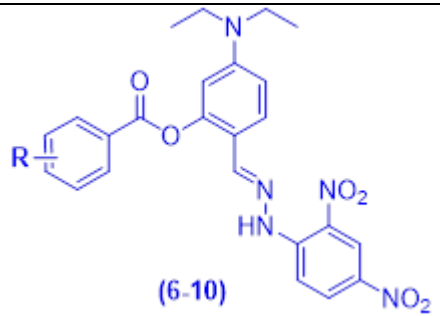
Table 3. ^{1}H NMR data of hydrazone compounds



(6-10)

Comp.	R	-N*H*- (ppm)	-C*H*=N- (ppm)	Aromatic protons (Ar*H*) (ppm)	–N(C*H*$_2$CH$_3$)$_2$ (ppm)	–N(CH$_2$C*H*$_3$)$_2$ (ppm)
6	H	11.14 (s, 1H)	8.02 (s, 1H)	9.07 (d, *J* = 2.5 Hz, 1H), 8.32 (d, *J* = 7.3 Hz, 2H), 7.89 (dd, *J* = 9.6, 2.5 Hz, 1H), 7.77 (t, *J* = 7.5 Hz, 1H), 7.71 (d, *J* = 8.9 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.51 (d, *J* = 9.6 Hz, 1H), 6.66 (dd, *J* = 8.9, 2.3 Hz, 1H), 6.45 (d, *J* = 2.3 Hz, 1H)	3.45 (q, *J* = 7.0 Hz, 4H)	1.27 (t, *J* = 7.0 Hz, 6H)
7	2-NO$_2$	11.29 (s, 1H)	8.12 (s, 1H)	9.14 (d, *J* = 2.5 Hz, 1H), 8.20 (dd, *J* = 9.6, 2.6 Hz, 1H), 8.09 – 8.05 (m, 1H), 8.04 – 7.99 (m, 1H), 7.91 – 7.86 (m, 2H), 7.85 – 7.79 (m, 2H), 6.67 (d, *J* = 9.0 Hz, 1H), 6.56 (d, *J* = 2.3 Hz, 1H)	3.47 (q, *J* = 7.0 Hz, 4H)	1.27 (t, *J* = 7.0 Hz, 6H)
8	3-NO$_2$	11.18 (s, 1H)	8.02 (s, 1H)	9.19 (d, *J* = 2.5 Hz, 1H), 9.15 (s, 1H), 9.10 (d, *J* = 2.5 Hz, 1H), 8.62 (t, *J* = 8.1 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 9.7 Hz, 1H), 7.62 (d, *J* = 9.7 Hz, 1H), 6.43 (d, *J* = 2.4 Hz, 1H)	3.46 (q, *J* = 7.0 Hz, 4H)	1.26 (t, *J* = 7.0 Hz, 6H)
9	4-NO$_2$	11.18 (s, 1H)	8.02 (s, 1H)	9.11 (d, *J* = 2.4 Hz, 1H), 8.51 – 8.45 (m, 4H), 8.10 (d, *J* = 7.1 Hz, 1H), 7.87 (d, *J* = 9.0 Hz, 1H), 7.75 (d, *J* = 9.2 Hz, 1H), 6.71 (d, *J* = 9.6 Hz, 1H), 6.43 (br.s, 1H)	3.46 (q, *J* = 7.0 Hz, 4H)	1.26 (t, *J* = 7.0 Hz, 6H)
10	3,5-di-NO$_2$	11.20 (s, 1H)	8.01 (s, 1H)	9.42 (d, *J* = 2.0 Hz, 2H), 9.39 (d, *J* = 2.0 Hz, 1H), 9.11 (d, *J* = 2.5 Hz, 1H), 8.19 (d, *J* = 9.6 Hz, 1H), 7.92 (d, *J* = 9.0 Hz, 1H), 7.85 (d, *J* = 9.5 Hz, 1H), 6.73 (d, *J* = 6.7 Hz, 1H), 6.41 (d, *J* = 2.4 Hz, 1H)	3.46 (q, *J* = 7.0 Hz, 4H)	1.27 (t, *J* = 7.0 Hz, 6H)

Table 4. ^{13}C NMR data of hydrazone compounds



(6-10)

Comp.	R	*C*=O (ppm)	*C*=N (ppm)	Aromatic carbons (Ar*C*) (ppm)	–N(*C*H$_2$CH$_3$)$_2$ (ppm)	–N(CH$_2$*C*H$_3$)$_2$ (ppm)
6	H	164.94	151.26	150.75, 145.21, 144.38, 137.29, 134.07, 133.04, 130.92, 130.48, 129.40, 128.90, 128.59, 123.58, 116.53, 112.14, 109.59, 105.26	44.72	12.56
7	2-NO_2	163.34	151.13	159.41, 150.66, 149.04, 145.86, 144.65, 136.76, 133.98, 131.51, 129.79, 129.26, 124.75, 123.63, 116.99, 112.64, 110.62, 107.60, 104.33, 97.50	44.53	12.82
8	3-NO_2	164.21	151.43	154.20, 148.35, 145.27, 145.11, 137.89, 135.24, 133.80, 129.86, 128.96, 127.71, 127.34, 124.55, 123.39, 116.32, 110.35, 103.59, 99.09	44.89	12.54
9	4-NO_2	163.75	151.15	154.19, 150.66, 145.21, 145.13, 137.83, 133.88, 130.64, 129.83, 128.86, 128.15, 123.49, 123.29, 116.59, 109.80, 103.78, 99.45	44.87	12.52
10	3,5-di-NO_2	162.06	151.38	150.79, 148.90, 146.24, 144.56, 136.77, 133.10, 130.54, 130.00, 129.93, 129.18, 123.66, 123.58, 116.88, 112.54, 110.61, 105.01	44.46	12.88