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SYNTHESIS OF SOME NOVEL NITRONES AND THEIR APPLICATIONS IN ORGANIC SYNTHESES

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IN
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AMJED AHMED ABED

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SYNTHESIS OF SOME NOVEL NITRONES AND THEIR APPLICATIONS IN ORGANIC SYNTHESES

By Amjed Ahmed ABED

February 2022

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science

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ABSTRACT

SYNTHESIS OF SOME NOVEL NITRONES AND THEIR APPLICATIONS IN ORGANIC SYNTHESES

Amjed Ahmed ABED

Master of Science in Chemistry

Advisor: Prof. Dr. Zeynep GÜLTEKİN

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February 2022

Nitrones are reactive 1,3-dipoles which readily form isoxazolidines and isoxazolines with olefinic and acetylenic dipolarophiles. In this study, a novel nitrones (56,57) were prepared in two steps in good yields. In the first step, *N*-phenylhydroxylamine was prepared then followed by a condensation reaction with some selected aldehydes. All nitrones were obtained in good yield (60-93%). In the second stage of the study, due to the importance of isoxazolidines in biologically active compounds, we aimed to extend our project by employing 3+2-cycloaddition reactions by using nitrones. After the success in the preparation of nitrones (9, 53-57), we investigated the synthesis of some novel isoxazolidine derivatives by 3+2 cycloaddition reaction. As a result of the reaction of nitrones with styrene, the desired isoxazolidine derivatives (58-63) were obtained in good yield and regioselectivity. All nitrones and cycloadducts were characterized by ^{1}H, ^{13}C, and other NMR techniques (DEPT, HETCOR, COSY). The stereochemistry of the major cycloadduct 61 was determined by X-ray crystallography.

2022, **99 Pages**

Keywords: Aldehydes, N-hydroxylamines, Nitrone, 1.3-dipolar cycloaddition, Isoxazolidines.

ÖZET

BAZI YENİ NİTRONLARIN SENTEZİ VE ORGANİK SENTEZDE UYGULAMALARI

Amjed Ahmed ABED
Kimya, Yüksek Lisans
Tez Danışmanı: Prof. Dr. Zeynep GÜLTEKİN
Eş Danışman: Dr. Akram Noori Mohammed QADDO
Şubat 2022

Nitronlar, olefinik ve asetilenik dipolarofillerle kolayca izoksazolidin ve izoksazolinleri oluşturan reaktif 1,3-dipollerdir.Bu çalışmada, iki adımda iyi verimle yeni nitronlar (56,57) hazırlandı, ardından seçilen bazı aldehitlerle kondenzasyon reaksiyonu incelendi. Sentezlenen nitronlar iyi verimle (% 60-93) elde edildi. Çalışmanın ikinci aşamasında izoksazolinlerin biyolojik olarak aktif bileşiklerdeki öneminden dolayı, hazırlanan nitronları kullanarak 3+2 halka katılma reaksiyonları ile projemizi genişletmeyi amaçladık. Nitronların (9, 53-57) hazırlanmasındaki başarının ardından 3+2-halka katılma reaksiyonu ile bazı yeni izoksazolidin türevlerinin sentezini araştırdık. Nitronların stiren ile reaksiyonu sonucu istenen izoksazolidin türevleri (58-63) iyi verim ve regioselektivitede ele geçti. Tüm nitronlar ve halka katılma ürünleri ^{1}H, ^{13}C ve diğer NMR teknikleri (DEPT, HETCOR, COSY) yardımıyla yapıları aydınlatıldı. Ana ürün 61'in stereokimyası X-ışınları yoluyla aydınlatıldı.

2022, 99 sayfa

Anahtar Kelimeler: Aldehitler, N-hidroksilaminler, Nitron, 1.3-dipolar halka katılma, İzoksazolidinler.

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I would like to thank Çankırı Karatekin University for spectroscopic analysis (^{1}H, ^{13}C, COSY, DEPT, HSQC, and HETCOR), especially to Yaşar İPEK. I would like to thank Sinop University's scientific and technology research application and research center, Turkey, for the usage of the Bruker D8-QUEST diffractometer. And especially thank Assoc. Prof. Dr. Onur ŞAHİN for measurement XRD-3 analysis of cycloadduct 61.

I would like to thank my parents whose love and guidance are with me in whatever i pursue and all my brothers, for their continued support. And thanks to my wife for supporting me at all times she is the one who encouraged me to complete my master's, she was and still supports me, i thank my family for being patient and supporting me during this stage.

Amjed Ahmed ABED

Çankırı- 2022

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LIST OF SYMBOLS

^{o}C	Celsius Temperature
D	Doublet
HR	Hour
J	Spin-spin coupling
M	Multiplet
MIN	Minute
ML	Milliliter
MMOL	Milimol
MP	Melting point
RT	Room temperature
S	Singlet

LIST OF ABBREVIATIONS

AR	Aryl
BINOL	1,1'-binaphthalene-2,2'-diol
COSY	Correlation spectroscopy
DCM	Dichloromethane
DEPT	Distortionless enhancement by polarization transfer
ET	Ethyl
HETCOR	Heteronuclear correlation spectroscopy
HSQC	Heteronuclear single quantum coherence
IR	Infrared spectroscopy
ME	Methyl
NMR	Nuclear magnetic resonance
NU	Nucleophile
PBN	α-phenyl-tert-butyl nitrone
PH	Phenyl
PT	Platinum
PTC	Phase transfer catalyst
TLC	Thin layer chromatography
UHP	Urea complex with hydrogen peroxide
Y-CD	Y-cyclodextrin

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1. INTRODUCTION

Nitrone nomenclature derived from a shortening of ‘‘nitrogen–ketones’’ due to highlight their resemblance to ketones (Figure 1.1) originally synthesized by Beckmann in 1890 (Beckmann 1890, Hamer and Macaluso 1964).

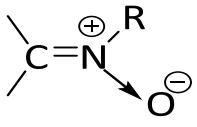


Figure 1.1 General structure of nitrone

Nitrones are very important in academia and industry (Martin and Jones 2002). They started to be utilized in analytical chemistry applications in the late 1960s. Nitrones will react with “trap” and stabilize free radical intermediates (Figure 1.2) (Floyd *et al.* 2008).

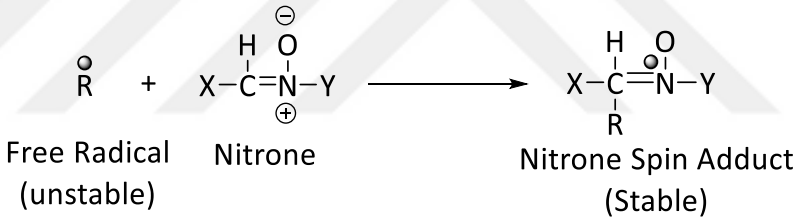


Figure 1.2 Properties of nitrones

For example, One of its most generally recognized nitrones is PBN α-phenyl-tert-butyl nitrone 1 and its derivatives have been reported as spin traps (Figure 1.3) (Floyd *et al.* 2002).

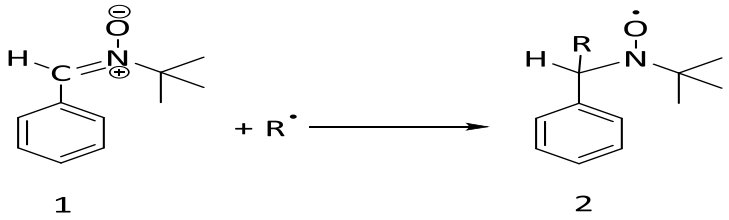
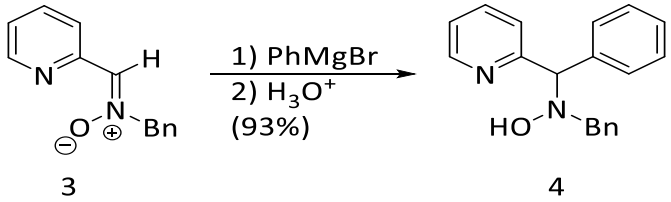


Figure 1.3 PBN spin trap

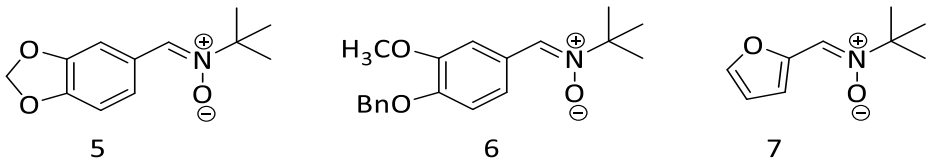
Nitrone chemistry was received extensive attention since its discovery. The electrophilicity of the C=N of nitrones was got great interest for use in reactions with nucleophilic reagents (Yang 2012, Arakawa *et al.* 2020). For example, adding the phenylmagnesium bromide to nitrone as nucleophilic addition reagent gives *N*-hydroxyl amine 4, Scheme (1.1) (Merino *et al*. 2000).



3 4 (1.1)

Nucleophilic addition to nitrone Scheme (1.1)

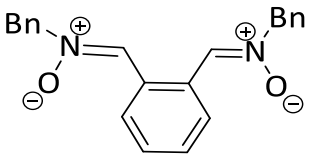
The importance of nitrones as valuable synthetic intermediates have been widely established, and various techniques for producing nitrones. They are generally recognized as essential useful biological activity agents and are utilized as medicines (Chiacchio *et al.* 1994). Furthermore, three compounds synthesis from furfural, piperonal, and *O*-benzyl vanillin (Figure 1.4) demonstrated protection against microvascular damages and were the most powerful of the series competing with PBN (Rosselin *et al*. 2017).



5 6 7

Figure 1.4 Nitrones have biological effects

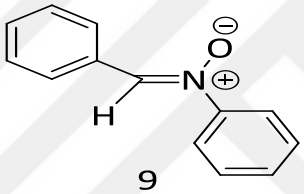
And also the high protective effect of HBN6 (Figure 1.5) as an agent having powerful biological activities (Chamorro *et al*. 2020).



8

Figure 1.5 Structures HBN6

In addition, nitrones have received significant interest as possible therapeutic agents for treating various reactive oxygen species diseases (Marano *et al.* 2021). And nitrone may also be applied as an inhibitor of corrosion for mild steel in organic acid, which was expected to be an efficient inhibitor against acidic and microbiological corrosion as α, N-diphenyl nitrone (Figure1.6) (Thirumalaikumar and Jegannathan 2011).



9

Figure 1.6 α, N-diphenyl nitrone

Nitrones are greatly employed in the preparation of different nitrogen-heterocycle compounds biologically active, antibiotics, alkaloids, amino sugars (Murahashi *et al.*1990).

1.1 Previous Syntheses of Nitrones

Nitrones are easily produced via oxidation reactions, most of the known oxidation techniques of nitrone production are based upon oxidation of imines, secondary amine, hydroxylamine, or isoxazolidines (Figure 1.7) (Mutlaq *et al.* 2021).

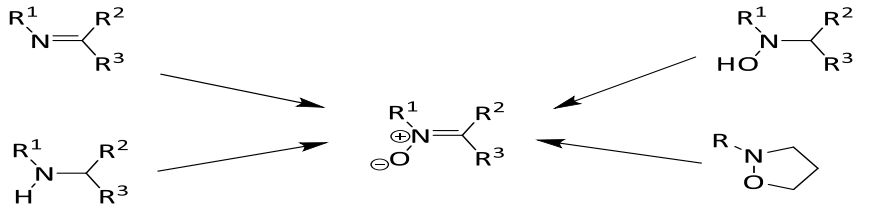
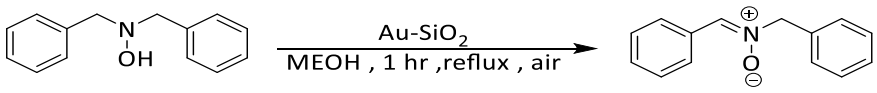


Figure 1.7 Some methods of nitrone synthesis.

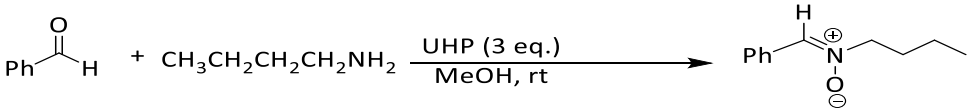
Nitrones can be prepared by using a range of oxidizing reagents using *N*,*N*-disubstituted hydroxyl amines as a starting materials. For example *N*,*N*-dibenzylhydroxylamine 10 was oxidized to nitrone 11 using the gold catalyst supported on silica Scheme (1.2) (D'Adamio *et al.* 2015).



10 11 (1.2)

Hydroxylamine oxidation via gold Scheme (1.2)

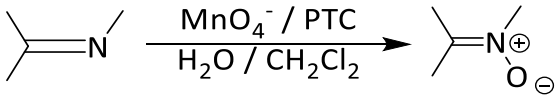
Additionally, a new method to nitrones synthesis from aromatic aldehydes and amines using UHP as an oxidant in the presence of methyl tri oxorhenium (MTO) Scheme (1.3) (Cardona *et al*. 2008).



12 13 14 (1.3)

Synthesis of nitrones from aldehydes and primary amines Scheme (1.3)

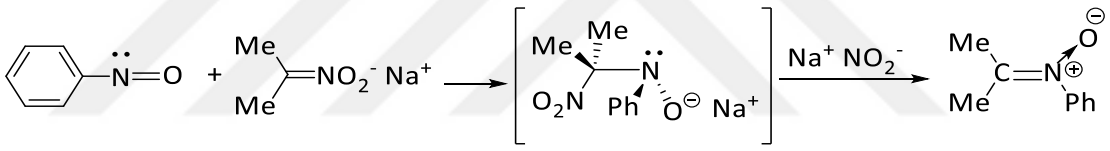
Furthermore the permanganate ion, MnO_4^-, is one of the classic oxidation reagents in organic and inorganic chemistry. Christensen and Jørgensen (1989) was reported oxidation of imines to nitrones by using permanganate ion under phase transfer catalyst (PTC) conditions Scheme (1.4) (Christensen and Jørgensen 1989).



(1.4)

Oxidation of imines Scheme (1.4)

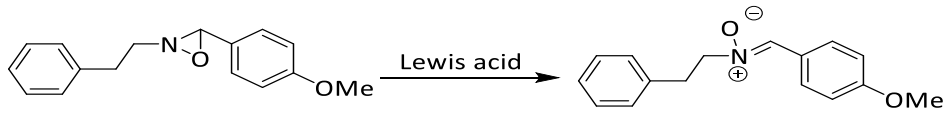
In addition, nitrones were synthesized according to a recognized generic technique, nitroso compounds with carbanions. This strategy included the addition of carbanion, stabilized with a leaving group to the nitrogen of nitroso group, then a leaving group was removed Scheme (1.5) (Lyapkalo *et al.*1996).



(1.5)

Interaction of nitroso compounds with carbanions Scheme (1.5)

Xing and co.workers have described an efficient process to the opening ring for 3-aryloxaziridines as a chemoselective rearrangement of to give nitrones at 70ºC after 3 hr using $AlCl_3$ as a catalyst Scheme (1.6) (Xing *et al.* 2009).



15 16

(1.6)

Lewis acid catalyzed rearrangement of oxaziridine to nitrone Scheme (1.6)

Moreover, oximes analogous can be utilized in the preparation of nitrones. This methodology includes the production of oximes from aldehydes and ketones by their

interaction with hydroxylamine, then reaction with *N*-substituted hydroxylamines to produce desired nitrones (Figure 1.8) (see as a review: Delpierre and Lamchen 1965).

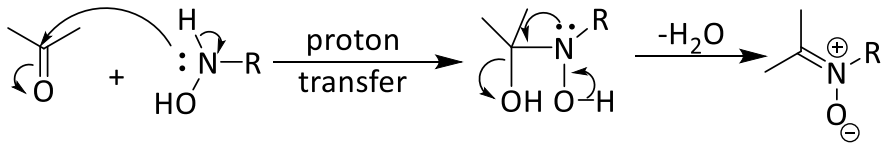
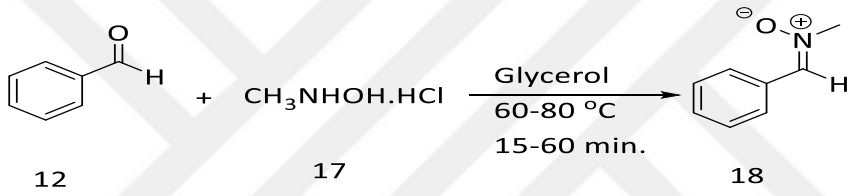


Figure 1.8 Synthesis of nitrones from *N*-substituted hydroxylamines

A range of nitrones was synthesized efficiently by condensation of *N*-substituted hydroxylamines and aldehydes in a green solvent Scheme (1.7) (Shariatipour *et al*. 2019).



(1.7)

Condensation of *N*-substituted hydroxylamines and aldehydes Scheme (1.7)

1.2 Applications of Nitrones

Nitrones are valuable synthetic intermediates for organic synthesis (Murahashi and Imada *et al.* 2018). Nucleophilic addition to nitrones give hydroxylamines (Figure 1.9) (Merino 2005). Hydroxyl amines are useful intermediate in organic chemistry (Cicchi *et al*. 2003). And several biologically active compounds containig hydroxylamine groups (Shariatipour *et al*. 2019).

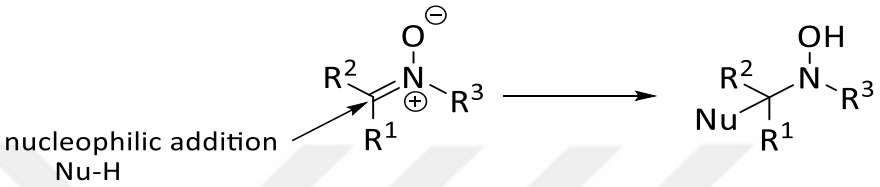
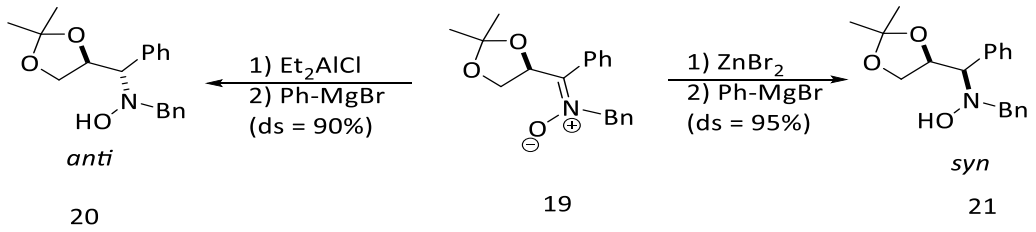


Figure 1.9 Nucleophilic addition to nitrones

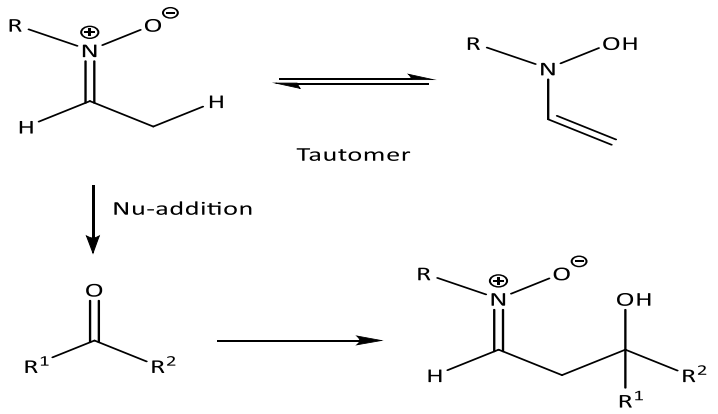
Merino carried out nucleophilic addition to nitrone 19 in the presence of lewis acid Et_2AlCl and $ZnBr_2$. The syn and anti product 20 and 21 obtained in good yield and selectivity Scheme (1.8) (Merino 2005).



(1.8)

The addition of Grignard reagents to nitrones Scheme (1.8)

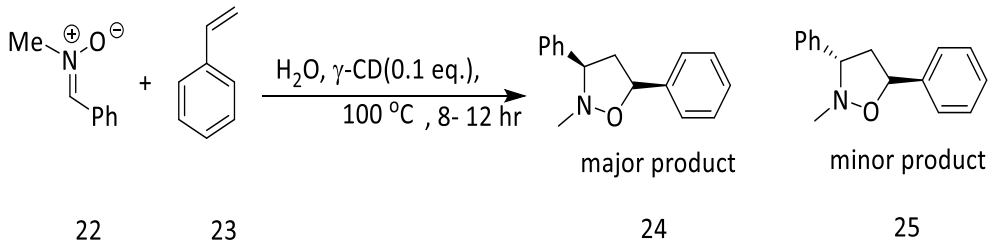
Moreover, Nitrone was converted to β-hydroxynitrone by Arnó and this reaction was accepted by Arnó as a the aldol reaction type reaction Scheme (1.9) (Arnó *et al*. 2004).



(1.9)

Nitrone was converted to β-hydroxynitrone Scheme (1.9)

Nitrogen containing compounds have attracted enormous interest are common in naturally occurring compounds and in important biologically active molecules (Murahashi and Imada 2018). Finally, nitrones are important dipoles in 1,3-dipolar cycloaddition reactions for synthesise of isoxazolidine rings. Martine investigated 1,3-dipolar cyloaddition reaction with nitrone 22 and styrene by using a catalytic quantity of γ-cyclodextrin (γ-CD) and obtained isoxazolidine 24 and 25 after 8 hours Scheme (1.10) (Martina *et al.* 2019).



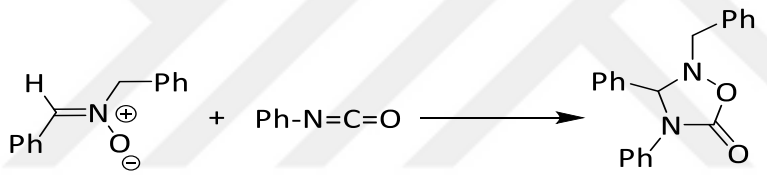
(1.10)

1,3-dipolar cyloaddition reaction with nitrone 22 and styrene Scheme (1.10)

2. LITERATURE REVIEV

2.1 1,3-Dipolar Cycloadditions with Nitrones

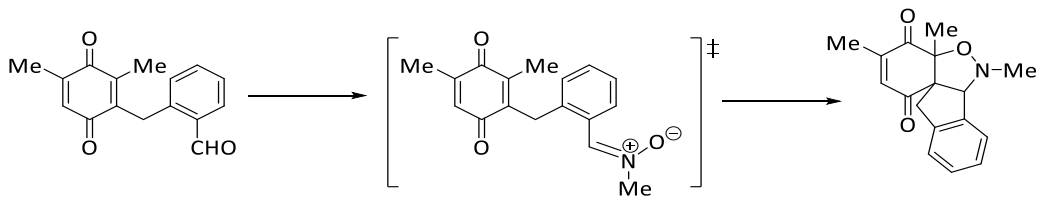
The 1,3-dipolar cycloaddition process is one of the most effective methods for the production of heterocyclic compounds (Nguyen *et al*. 2010, Anderson 2016). This approach occurs quickly and in high yields. Nitrones may react as 1,3-dipolar species with a wide range of dipolarphiles to produce various products. One of the most synthetic uses of nitrones is their usage as 1,3-dipoles in cycloaddition reactions to olefins for the synthesis of isoxazolidines (Salman and Majeed 2013). One of the earliest instances of 1,3-dipolar cycloadditions was published by Beckmann. 1,3-dipolar cycloaddition of Nitrone 14 with phenyl isocyanate 26 gives cycloadduct 27 Scheme (2.1) (Beckmann 1890).



14 26 27 (2.1)

Ernst Beckmann's procedure Scheme (2.1)

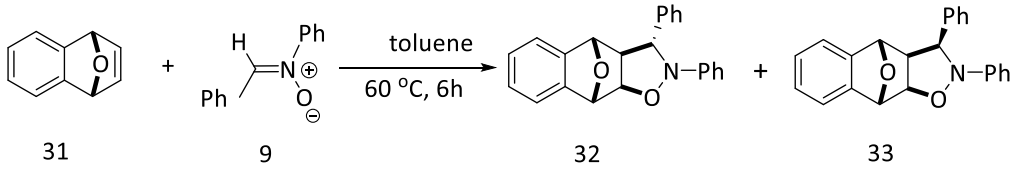
The intramolecular nitrone to olefin cycloaddition offers a flexible technique for the production of the isoxazolidine ring system. For example, Schultz and coworkers has investigated intramolecular cycloaddition of 29, the cycloadduct 30 obtained in 65% yield Scheme (2.2) (Schultz *et al*. 1987).



28 29 30 (2.2)

Intramolecular nitrone olefin cycloaddition Scheme (2.2)

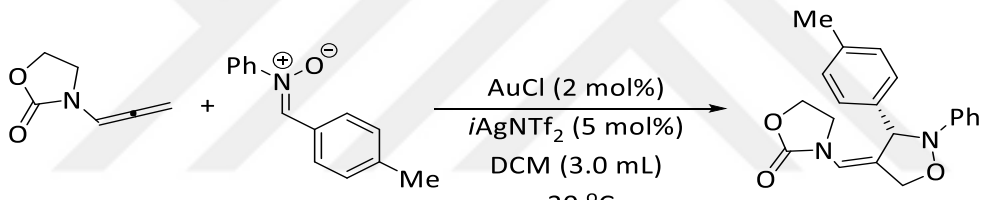
1,3-dipolar cycloaddition of oxa(aza) bicyclic alkene 31 and nitrone 9 was investigated by yao and co workers. The exo-32 and endo-33 was produced at 60^{o}C in toluene in high yield 99% and with 6:1 diastereoselectivity Scheme (2.3) (Yao *et al*. 2019).



31 9 32 33 (2.3)

1,3-dipolar cycloaddition of nitrone with oxa(aza) bicyclic alkene Scheme (2.3)

And the reaction of *N*-allenylamide 34 with nitrone 35 in a gold catalyst in the presence of BINOL as a chiral ligand. The cycloaddition product 36 obtained in 99% yield and excellent enantioselectivity (97% ee) Scheme (2.4) (Li *et al*. 2013).



34 35 36 (2.4)

Enantioselective 1,3-dipolar cycloaddition of nitrone and *N*-allenyl amide Scheme (2.4)

1,3-dipolar cycloaddition of nitrone and olefin results 5- or 4-substitued isoxazolidines (Figure 2.1) (Li *et al*. 2021).

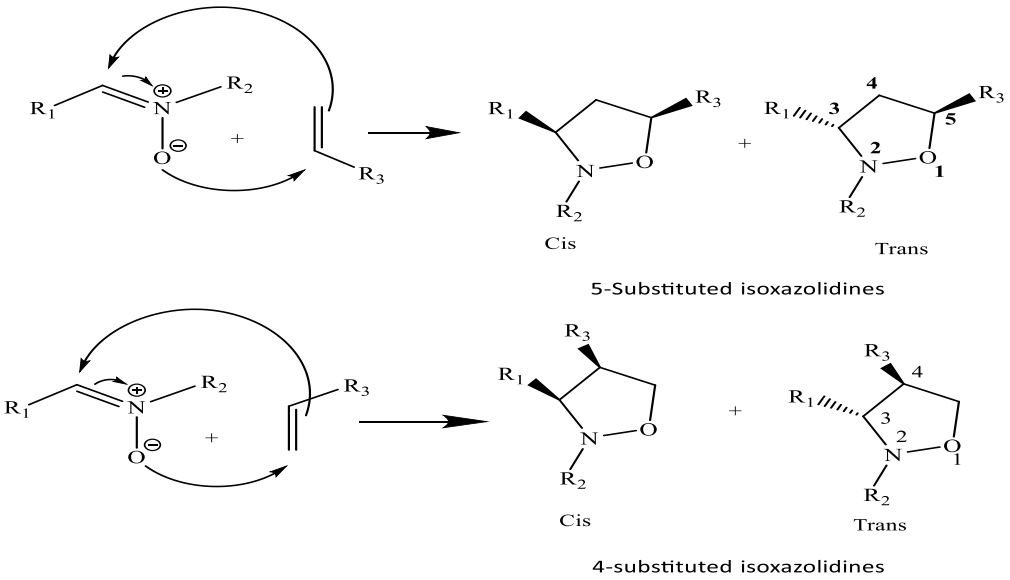
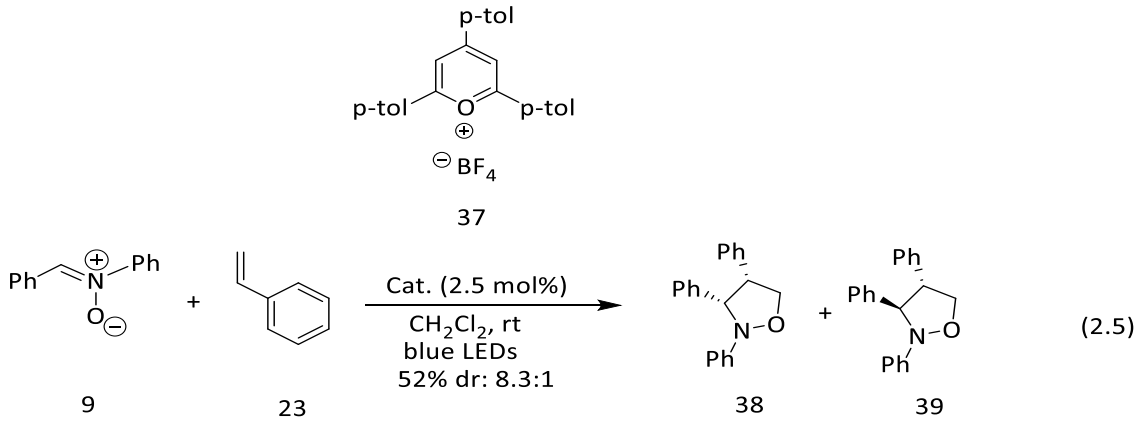


Figure 2.1 Nitrone olefin cycloadditions

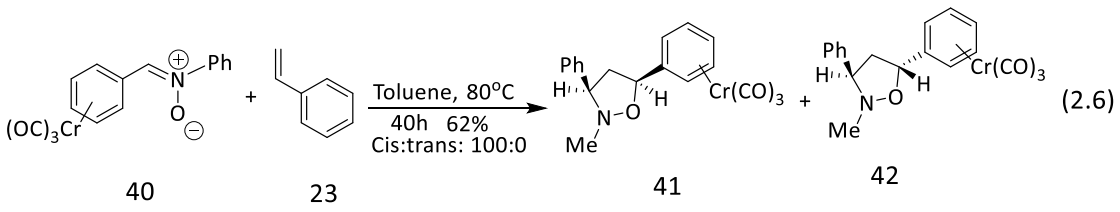
Styrene has been used as a dipolorophile in 3+2 cycloaddition reactions by many researchers (Rao *et al*. 2017, Kuban *et al*. 1999, Li *et al*. 2021). For example, cycloaddition of nitrone 9 with styrene under irridation with blue LEDs, which give exclusively 4-substituted isoxazolidines 38 and 39 in 8.3:1 regio Scheme (2.5) (Zheng *et al*. 2017).



Cycloaddition of nitrone with styrene Scheme (2.5)

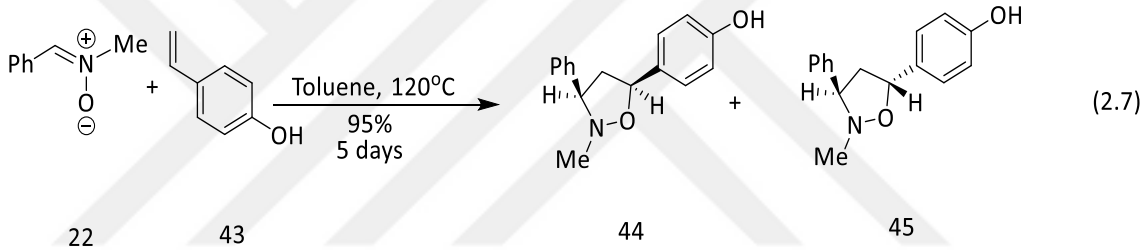
And also, 1,3-dipolar cycloaddition reaction between nitrone 40 and styrene was investigated by Artemov. Stereoselectivity of cycloaddition reaction was improved

using nitrone 40 containig $Cr(CO)_3$ group in (cis:trans= 100:0) Scheme (2.6) (Artemov *et al*. 2013).



1,3-dipolar cycloaddition of nitrone Scheme (2.6)

Subsituted styrene can also be used such as, nitrone 22 reacts with Subsituted styrene gives mixture of 5-substituted isoxazolidines cis 44 and trans 45 (ratio: 66:34) in 94% yield Scheme (2.7) (Chiacchio *et al.* 1994).



3+2 cycloaddition reactions Scheme (2.7)

2.2 Importance of Isoxazolidine rings

Isoxazolidines are saturated five-membered heterocycles containing nearby nitrogen and oxygen atoms, which are the result of 1,3-dipolar cycloaddition reactions involving nitrones and alkenes. Due to the instability of the N-O bond under mild reduction conditions, isoxazolidines have long been considered as synthetically important species and have been used as in particular amino acids, amino-sugars, and alkaloids (Figure 2.2) (Frederickson 1997).

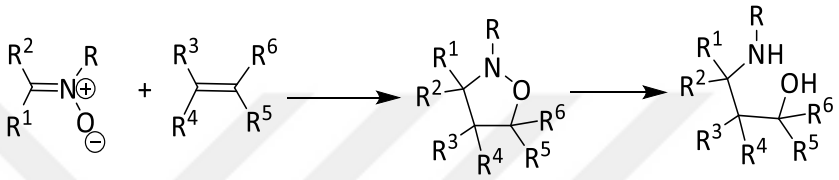


Figure 2.2 The 1,3-dipolar cycloaddition reaction and clavage of N-O bond

The some of isoxazolidines have naturally occurring molecules with distinct biological characteristics. For example, spiroisoxazolidine derivatives shown anti-mycobacterial and anti-invasive activities (Figure 2.3) (Arumugam *et al*. 2010).

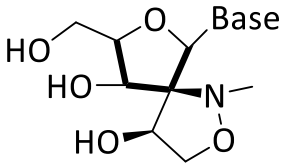


Figure 2.3 Spiroisoxazolidine compound

And also isoxazolidines are important synthons for the creation of simple and complicated compounds. This is because the ring can be opened by breaking N-O bond (Figure 2.4) (Iannazzo *et al*. 2010).

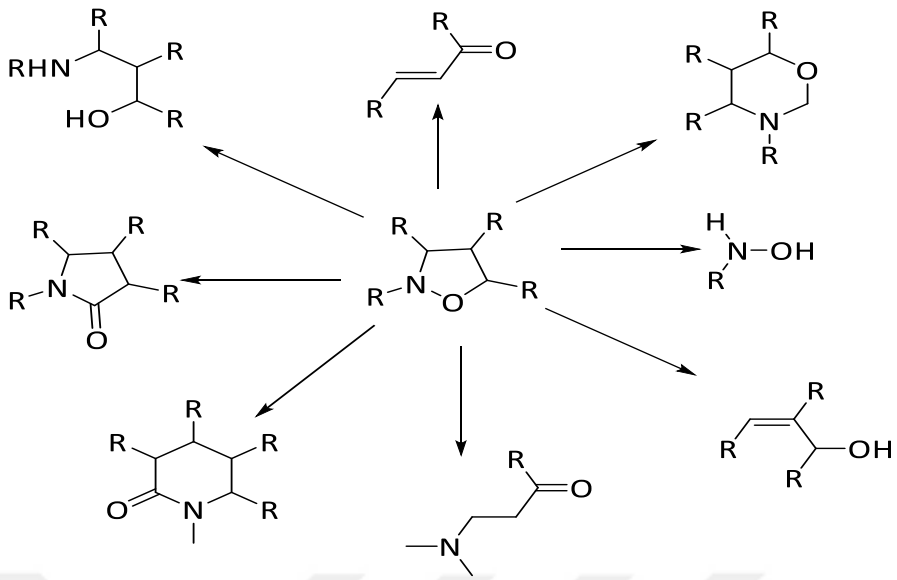
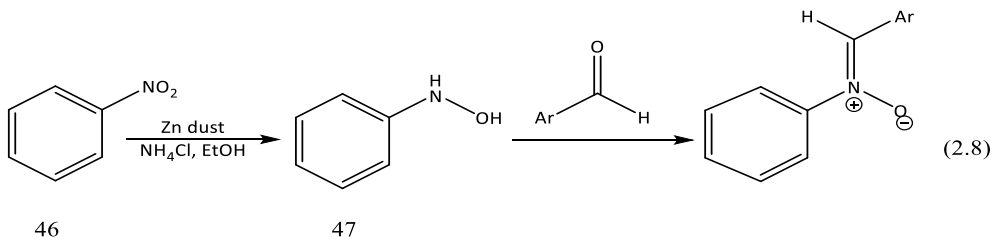


Figure 2.4 Ring opening reactions of isoxazolidines

2.3 Project Aims

The first purpose of the project was to develop the strategies for the preparation of some new nitrone derivatives. To begin with, nitrones will be targeted starting from nitrobenzene. The first step in the synthesis is the reduction of nitrobenzene using zinc metal under mild conditions to produce *N*-phenylhydroxylamine, followed by a condensation reaction with some selected aldehydes. We followed three different procedures to make a comparison among these procedures. All these procedures would generate the target molecules Scheme (2.8).

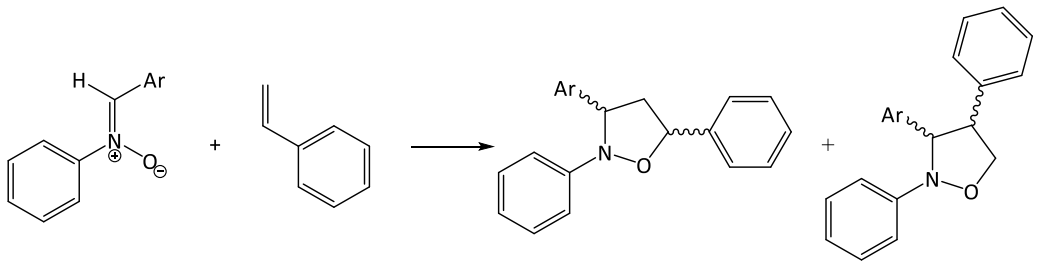


(2.8)

46 47

Proposed synthesis of nitrones Scheme (2.8)

In addition, we targeted to evolve the procedure for the synthesis of some new isoxazolidine derivatives. The 1,3-dipolar cycloaddition reaction of nitrones to styrene under thermal conditions Scheme (2.9) was the key step that must reach the desired compounds.



(2.9)

1,3-dipolar cycloaddition of nitrones Scheme (2.9)

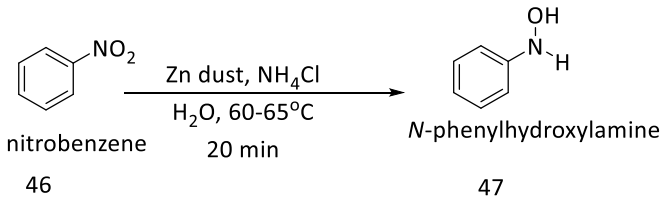
3. MATERIALS AND METHODS

3.1 Chemical Used in Material Synthesis

The chemicals used were purchased from sources without additional purification. A Büchi rotary evaporator coupled to a pump low-pressure was used for solvent evaporation. TLC plates wear shown using $KMnO_4$ (aqueous solution made with NaOH). The spectra (NMR) was measured on a Agilent 600 MHz spectrometer in $CDCl_3$. The multiplicities are defined as s = singlet, d = doublet, m = multiplet. The chemical shifts were recorded on the δ scale. All coupling constants are measured in Hertz. Infra-red spectra were recorded on a Shimadzu FTIR spectrophotometer. Melting points were determined using a electrothermal IA9200 digital melting point apparatus and are uncorrected. Nitrobenzene, NH_4Cl, Zn, Toluene , EtOH, MeOH were purchased from Biochem Chemopharma and Scharlau and Sigma-Aldrich. Toulene, EtOH, and MeOH were used without purification.

3.2 General procedure for synthesis of nitrone and cycloadditions

Starting material *N*-phenylhydroxylamine 47 was synthesized according to the lirerature method (Furniss *et al.* 1989. see also: Salman and Majeed 2013, Mahieddine *et al*. 2016, Salman *et al.* 2020).



Ammonium chloride (0.117 mol, 6.2 g) in 200 mL of water placed in a 500 mL round bottom flask, fitted with a magnetic stirrer. Freshly distilled nitrobenzene (0.102 mol, 12.5 g) was added, then the mixture was stirred vigorously. Zinc powder (0.2 mol, 15 g) was added for 15 minutes. At the same time the temperature was raised to 60^{o}C until finish all zinc addition, then stirring was continued for extra 15 minutes after the finish of zinc powder addition. After completion of the reaction, the reaction mixture was filtered to remove zinc oxide washed with 60 mL of hot water. The filtrate solution was placed in a beaker and saturated with NaCl (about 100 gr), cooled to 0^{o}C. *N*-phenylhydroxylamine products have been started growing as yellow needles, the product needles are separated by pouring solution into another beaker. The crude product was diluted with water (30 ml) and extracted with diethyl ether (3 × 50 mL), the combined organic extracts were collected and evaporated to give the title compound in 75 % yield. literature m.p = 81^{o}C (Furniss *et al*, 1989).

All the nitrones was prepared by the same method but different reaction conditions as shown down.

General Procedure A: (Shariatipour *et al*. 2019).

An aldehyde (10 mmol, 1.0 eq.) was added to a stirred solution *N*-phenylhydroxylamine (10 mmol, 1.0 eq.) in methanol (10 mL). The reaction mixture was heated at 60^{o}C until the disappearance of the starting material, monitored by TLC. Then, the solvent was removed under reduced pressure to yield the crude product. The pure nitrone was obtained by recrystallization of the crude product from hot ethanol, which was stored in a dark and cold place.

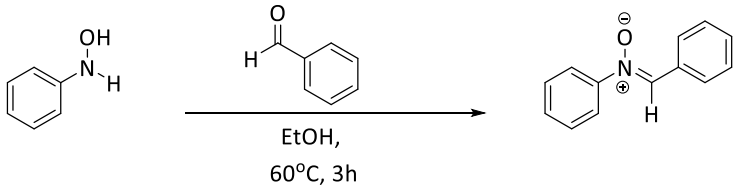
General Procedure B: (Salman *et al*. 2020).

An aldehyde (10 mmol, 1.0 eq.) was added to a stirred solution *N*-phenylhydroxylamine (10 mmol, 1.0 eq.) in ethanol (10 mL). The reaction mixture was stirred at room temperature for the required time till the disappearance of the starting material, followed by TLC. Thereafter, the solvent was removed under reduced pressure to yield the crude product. The crude product was recrystallized from hot ethanol to yield pure nitrone, which was stored in a dark and cold place.

General Procedure C: (Mahieddine *et al*. 2016).

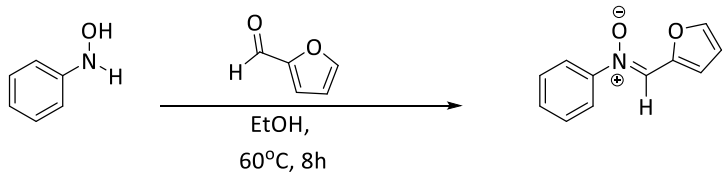
An aldehyde (10 mmol, 1.0 eq.) was added to a stirred solution *N*-phenylhydroxylamine (10 mmol, 1.0 eq.) in ethanol (10 mL). Then the reaction mixture was heated at 60 ^{o}C until the starting material was consumed monitoring by TLC. Thereafter, the solvent was evaporated under reduced pressure to yield the crude product. The crude product was recrystallized from hot ethanol to yield pure nitrone, which was stored in a dark and cold place.

(*Z*)-*N*,1-diphenylmethanimine oxide (9)



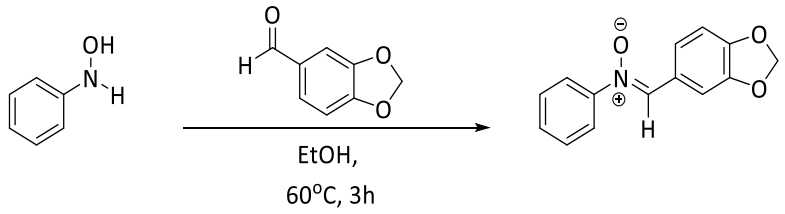
The title compound was prepared following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and benzaldehyde 12 (0.96 g, 10 mmol, 1.0 eq.) to give (*Z*)-*N*,1-diphenylmethanimine oxide 9 as a yellow solid (1.75 g, 89%) m.p. = 99 - 100 ^{o}C, IR (KBr) ν_{max} /cm^{-1} : 3103, 3059, 1591 (C=N), 1546, 1483, 1460, 1444, 1396 (C-N), 1298, 1068 (N-O); ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 8.45 – 8.37 (2H, m, N-C-*o*-Ar*H*), 7.92 (1H, s, N=C*H*), 7.76 – 7.77 (2H, d, *J* = 7.3 Hz, N-C-*o*-Ar*H*), 7.52 – 7.42 (6H, m, Ar*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 149.07 (N=*C*H), 134.67 (N-*C*), 130.97 (N=CH-*C*), 129.95 (ArCH), 129.85 (ArCH), 129.17 (ArCH), 129.05 (2xArCH), 129.04 (ArCH), 128.95 (ArCH), 128.66 (ArCH), 121.77 (2xArCH). (literature m.p = 113-115^{o}C) (Salehzadeh and Mashhadizadeh 2019).

(*Z*)-1-(furan-2-yl)-*N*-phenylmethanimine oxide (53)



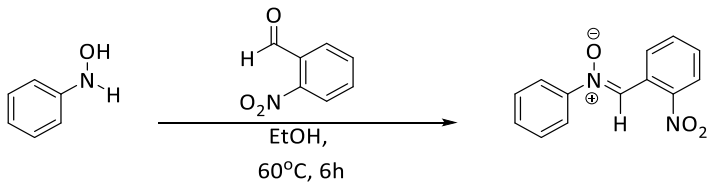
The title compound was prepared following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and furfural 48 (0.96 g, 10 mmol, 1.0 eq.) to yield (*Z*)-1-(furan-2-yl)-*N*-phenylmethanimine oxide 53 as a light brown solid (1.72 g, 92%). m.p.= 88-90 ºC, IR (KBr) ν_{max} /cm^{-1}: 3184, 3099, 3057, 1593 (C=N), 1570, 1544, 1388 (C-N), 1365, 1240, 1068 (N-O$^-$); ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm 8.14 (1H, s, OCH), 7.99 – 7.98 (1H, d, *J* = 2.4 Hz, O-CHC*H*), 7.78 – 7.76 (2H, d, *J* = 7.6 Hz, Ar*H*), 7.55 (1H, s, N=C*H*), 7.45 – 7.41 (3H, m, Ar*H*), 6.61 (1H, s, O-CCH); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 147.50 (N=*C*H), 147.22 (N-*C*), 144.69 (N=CH-*C* and *OCH*), 129.95 (ArCH), 129.18 (2xArCH), 124.36 (ArCH), 121.02 (ArCH), 116.52 (O-*C*H), 112.72 (O-CH*C*H). Nitrone 53 was prepared as brown solid in yield 67% using MeOH as a solvent at rt. in 3 h using catalytic proline (3 equiv) (Choi *et al*. 2018).

(*Z*)-1-(benzo[d][1,3]dioxol-5-yl)-*N*-phenylmethanimine oxide (54)



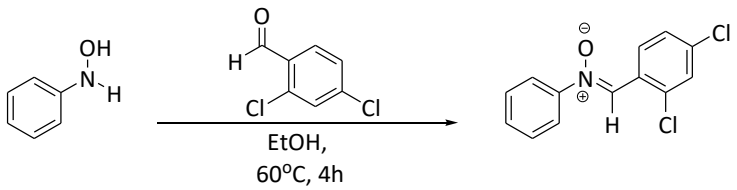
The title compound was prepared following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and piperonal 49 (1.5 g, 10 mmol, 1.0 eq.) to yield (*Z*)-1-(benzo[d][1,3]dioxol-5-yl)-*N*-phenylmethanimine oxide 54 as a light brown solid (2.24 g, 93%). m.p. = 130-132 ºC; IR (KBr) ν_{max} /cm^{-1}: 3057, 2895, 1618 (C=N), 1598, 1489, 1452, 1392 (C-N), 1267, 1192 (N-O$^-$), 1056; ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 8.30 (1H, s, OCC*H*), 7.82 (1H, s, N=C*H*), 7.75 – 7.74 (2H, d, *J* = 7.7 Hz, O-CC*H*CH +Ar*H*), 7.70 – 7.68 (1H, d, *J* = 7.6 Hz, O-CCHC*H*), 7.47 - 7.41 (3H, m, Ar*H*), 6.91 – 6.89 (1H, d, *J* = 8.2 Hz,Ar-H), 6.04 (2H, s, OCH_2O); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 149.6 (O-*C*), 149.71 (O-*C*), 148.89 (N-*C*), 147.71 (N=CH-*C*), 134.33 (N=*C*H), 129.74 (ArCH), 129.14 (ArCH), 128.72 (ArCH), 125.31 (ArCH), 125.22 (ArCH), 121.64 (ArCH), 108.64 (OC*C*H), 108.51 (OC*C*H), 101.66 (O-*C*H_2). Nitrone 54 was prepared as brown solid in yield 45% using MeOH as a solvent at rt. in 3 h using catalytic proline (3 equiv) (Choi *et al*. 2018).

(*Z*)-1-(2-nitrophenyl)-*N*-phenylmethanimine oxide (55)



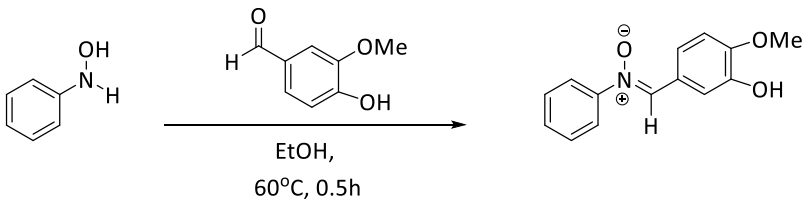
The title compound was prepared following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and 2-nitrobenzaldehyde 50 (1.51 g, 10 mmol, 1.0 eq.) to give (*Z*)-1-(2-nitrophenyl)-*N*-phenylmethanimine oxide 55 as a yellow solid (2.25 g, 92%). m.p. = 84-85 ^{o}C; IR (KBr) ν_{max} /cm^{-1}: 3072, 1589 (C=N), 1570, 1521, 1467, 1342 (C-N), 1193, 1091 (N-O^{-}), 1070; ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 9.36 – 9.30 (1H, d, *J* = 8.1 Hz,O_2NCC*H*), 8.58 (1H, s, 1H, s, N=C*H*), 8.13 – 8.07 (1H, d, *J* = 8.2 Hz, O_2NCC*H*), 7.83 - 7.71 (3H, m, N-C-*o*-Ar*H*), 7.57 - 7.51 (1H, t, *J* = 7.8 Hz, O_2NCCHC*H*), 7.52 –7.48 (3H, m, Ar*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 149.21 (*C*-NO_2), 147.53 (N=*C*H), 133.58 (N-*C*),130.6 (N=CH-*C*), 130.4 (ArCH), 129.45 (ArCH), 129.35 (2xArCH), 128.47 (ArCH), 125.06 (ArCH), 124.54 (ArCH), 121.82 (2xArCH). Nitrone 55 was prepared in literature by using MeOH and surfactant (Chatterjee *et al*. 2003).

(*Z*)-1-(2,4-dichlorophenyl)-*N*-phenylmethanimine oxide (56)



The title compound was synthesised following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and 2,4-dichlorobenzaldehyde 51 (1.74 g, 10 mmol, 1.0 eq.) to give (*Z*)-1-(2,4-dichlorophenyl)-*N*-phenylmethanimine oxide 56 as a yellow solid (2.45 g, 92%). m.p. = 79-80 ºC; IR (KBr) ν_{max} /cm^{-1}: 3072, 1577 (C=N), 1539, 1483, 1460, 1384 (C-N), 1186 (N-O$^-$), 1087, 1051; ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 9.54 -9.52 (1H, d, *J* = 8.7 Hz, ClCC*H*CH), 8.37 (1H, s, N=C*H*), 7.50 – 7.21 (7H, m, ArCH); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 149.30 (N-*C*), 136.29 (*C*-Cl), 134.14 (*C*-Cl), 130.37 (N=*C*H), 129.78 (N=CH-*C*), 129.46 (ArCH), 129.30 (ArCH), 129.24 (ArCH), 127.66 (ArCH), 127.54 (ArCH), 126.97 (ArCH), 126.63 (ArCH), 121.76 (ArCH).

(*Z*)-1-(3-hydroxy-4-methoxyphenyl)-*N*-phenylmethanimine oxide (57)

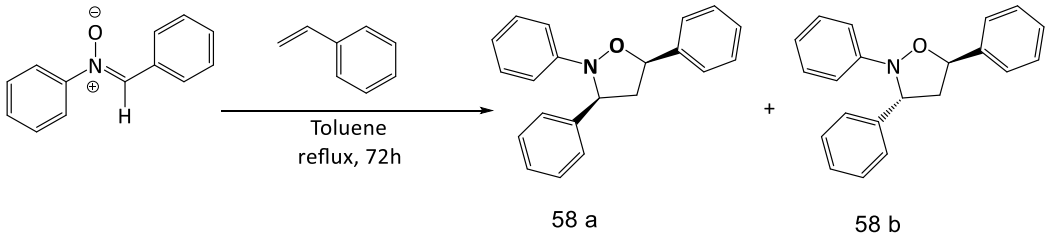


The title compound was prepared following general procedure C, (1.09 g, 10 mmol, 1.0 eq.) of *N*-phenylhydroxylamine 47 and vanillin 52 (1.52 g, 10 mmol, 1.0 eq.) to preduce (*Z*)-1-(3-hydroxy-4-methoxyphenyl)-*N*-phenylmethanimine oxide 57 as a yellow solid (2.26 g, 93%). m.p. = 198-199 ^{o}C; IR (KBr) ν_{max} /cm^{-1}: 3078, 2943, 1654, 1585 (C=N), 1571, 1517, 1456, 1390 (C-N), 1276, 1051 (N-O^{-}); ^{1}H NMR (600 MHz, DMSO) δ ppm: 9.79 (1H, s, O*H*), 8.35 – 8.34 (2H, m, N-C-*o*-Ar*H*), 7.88–7.84 (3H, m, N=C*H* +ArCH), 7.52 –7.47 (3H, m,ArCH), 7.87 -7.83 (1H, m, ArCH), 6.87 – 6.86 (1H, d, *J* = 8.2 Hz, ArCH), 3.79 (3H, s, OC*H*$_3$); ^{13}C NMR(151 MHz, DMSO) δ ppm: 144.06 (*C*OCH$_3$), 143.56 (*C*-OH), 141.34 (N=*C*H), 130.05 (N-*C*), 124.95 (N=CH-*C*), 124.41 (ArCH), 120.23 (2xArCH), 118.87 (ArCH), 116.84 (2xArCH), 109.68 (ArCH), 105.80, (ArCH), 51.30 (O*C*H$_3$).

General Procedure D: (Huisgen *et al*. 1968, Rao *et al*. 2017).

A 50 mL one-necked round bottom flask, charged with a magnetic follower, a reflux condenser. The flask was charged with a solution of nitrone (10 mmol, 1.0 eq.) in different solvents (10 mL) styrene (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) was added to the flask at room temperature. After that the reaction mixture was heated at reflux for the desired time till a reactants disappeared, which was confirmed by TLC. After cooling to room temperature the solvent was removed under reduced pressure to yield the crude product. The crude product was recrystallized from hot ethanol to afford pure desired isoxazolidines.

(*3SR,5RS*)-2,3,5-triphenylisoxazolidine (58)



Following the general procedure D: To (*Z*)-*N*,1-diphenylmethanimine oxide 9 (1.97 g, 10 mmol, 1.0 eq.) and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using toluene as a solvent then the reaction mixture was heated at approximately 110ºC for 3 days. After 3 days the reaction mixture was cooled to room temperature then the solvent evaporated gave the title compounds as a mixture of inseparable diastereoisomers (58a and 58b**)** as a pale yellow solid (2.31 g, 77% overall yield). The ratio of the crude products were determined by ^{1}H NMR spectroscopy and found in (90:10) **(**cis (58a) and trans(58b**)** as mixture of two diastereoisomers. Literature m.p. of cis isomer: 99-100ºC, Trans isomer: 101.5-102.5 (Huisgen *et al*. 1968).

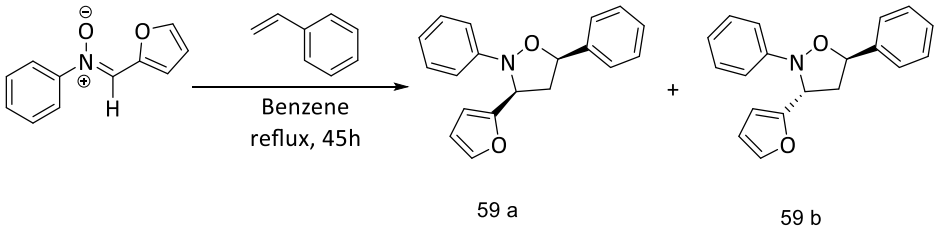
Cis (*3SR,5RS*)-2,3,5-triphenylisoxazolidine(58a)

^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.59 – 6.95 (15H, m,Ar-H), 5.19-5.18 (1H, dd, J = 9.9, 5.7 Hz, OC*H*Ph), 4.93-4.95 (1H, t, J = 7.8 Hz, NC*H*Ar), 3.23 – 3.17 (1H, m, NCHC*H*H), 2.52-2.47 (1H, dd, J = 19.3, 10.8 Hz, NCHCH*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 152.54 (N-*C*),142.89 (N-CH-*C*), 137.76 (O-CH-*C*), 128.99 (2xArCH), 128.84 (2xArCH), 128.58 (2xArCH), 128.39 (ArCH), 127.34 (ArCH), 126.88 (2xArCH), 126.27 (2xArCH), 121.36 (ArCH), 113.90 (2xArCH), 80.60 (O-*C*), 71.57 (N-CH), 48.76 (N-CH-CH_2).

Trans (*3SR,5RS*)-2,3,5-triphenylisoxazolidine(58b)

1H NMR (600 MHz, CDCl3) δ ppm: 7.59 – 6.95 (15H, m,Ar-H), 5.36-5.38 (1H, m, OC*H*Ph), 4.69-4.71 (1H, m, NC*H*Ar), 2.76-2.82 (1H, m, NCHC*H*H), 2.70-2.65 (1H, m, NCHCH*H*).

(*3SR,5RS*)-3-(furan-2-yl)-2,5-diphenylisoxazolidine (59)



Following the general procedure D, (1.87 g, 10 mmol, 1.0 eq.) of (*Z*)-1-(furan-2-yl)-*N*-phenylmethanimine oxide 53 and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using benzene as a solvent and the reaction mixture was refluxed for 45 hours to yield a combination of two inseparable diastereoisomers 59a and 59b (2.00 g, 69% overall yield) as a light brown oil. ^{1}HNMR spectroscopy of crude products showed two diastereoisomers, and the ratio of cis 59a and trans 59b were 95:5.

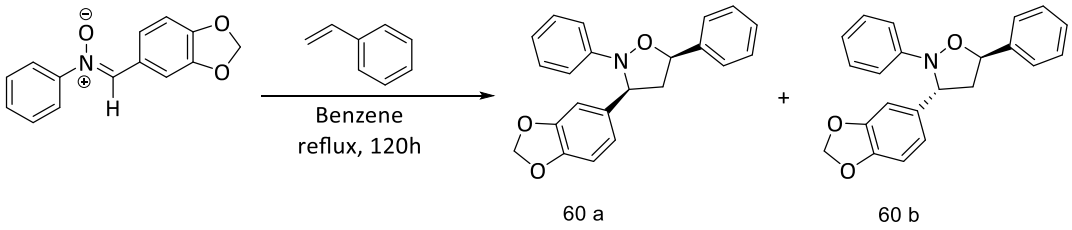
Cis *3SR,5RS*3-(furan-2-yl)-2,5-diphenylisoxazolidine (59a)

^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.47 -7.46 (1H, d, J = 8.0 Hz, OC*H*CH), 7.41– 6.99 (10H, m, Ar-H), 6.39-6.41 (1H, d, J = 12 Hz, OCHCHC*H*), 5.19-5.16 (1H, t, J = 7.1 Hz, OC*H*Ph), 5.04 -5.02 (1H, t, J = 7.7 Hz, NC*H*Ar), 3.08 – 3.03 (1H, m, NCHC*H*H), 2.69 – 2.65 (1H, m, NCHCH*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 154.56 (O-*C*-furan), 151.85 (N-*C*), 142.49 (O-*C*H-furan), 137.86 (O-CH-*C*), 129.08 (2xArCH), 128.61 (2xArCH), 128.41 (ArCH), 126.90 (2xArCH), 121.87 (ArCH), 114.33 (2xArCH), 110.42 (O-C-*C*H-furan), 106.61 (O-CH-*C*H-furan), 80.24 (O-*C*), 65.31 (N-*CH*), 43.57 (N-CH-*C*H_2).

Trans *3SR,5RS*3-(furan-2-yl)-2,5-diphenylisoxazolidine (59b)

^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.47 -7.46 (1H, d, J = 8.0 Hz, OC*H*CH), 7.41– 6.99 (10H, m, Ar-H), 6.39-6.41 (2H, d, J = 12 Hz, OCHCHC*H*), 5.40-5.42 (1H, t, J = 9 Hz, OC*H*Ph), 4.76 -4.78 (1H, dd, J = 9 and 3 Hz, NC*H*Ar), 2.84 – 2.88 (1H, m, NCHC*H*H), 2.62 – 2.67 (1H, m, NCHCH*H*).

(*3SR,5RS*)-3-(benzo[d][1,3]dioxol-5-yl)-2,5-diphenylisoxazolidine (60)



Following the general procedure D, using (2.41g, 10 mmol, 1.0 eq.) of (*Z*)-1-(benzo[d][1,3]dioxol-5-yl)-*N*-phenylmethanimine oxide 54 and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using benzene as a solvent and the reaction mixture was refluxed for 5 days to yield a combination of two inseparable diastereoisomers 60a and 60b (2.59 g, 75% overall yield) as a yellow solid. ^{1}HNMR spectroscopy of crude products showed two diastereoisomers, and the ratio of cis 60a and trans 60b were 90:10.

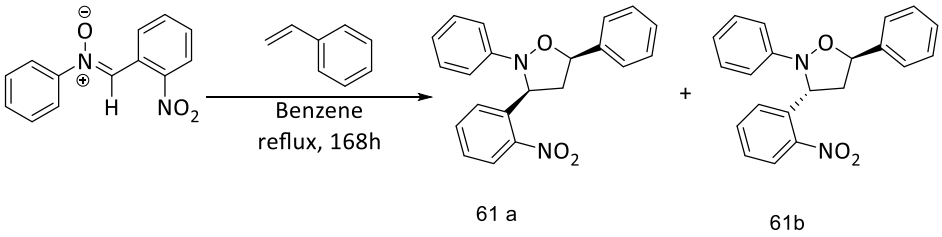
Cis (*3SR,5RS*)-3-(benzo[d][1,3]dioxol-5-yl)-2,5-diphenylisoxazolidine (60a)

Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.79–6.48 (13H, m, Ar-H), 5.98-5.97 (2H, d, J = 2.5 Hz, OC*H*$_2$O), 5.18-5.15 (1H, t, J = 7.1 Hz, OC*H*Ph), 4.85-4.83 (1H, t, J = 7.7 Hz, NC*H*Ar), 3.17-3.13 (1H, m, NCHC*H*H), 2.46-2.42 (1H, m, NCHCH*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 152.44 (N-*C*), 148.17 (O-*C*), 146.81 (O-*C*), 137.79 (N-CH-*C*), 136.86 (O-CH-*C*), 128.98 (2xArCH), 128.60 (2xArCH), 128.40 (ArCH), 126.83 (2xArCH), 121.40 (ArCH), 119.37 (ArCH), 113.94 (2xArCH), 108.32 (ArCH), 106.85 (ArCH), 101.08 (O-*C*H$_2$-O), 80.55 (O-*C*), 71.46 (N-*C*H), 48.96 (N-CH-*C*H$_2$).

Trans (*3SR,5RS*)-3-(benzo[d][1,3]dioxol-5-yl)-2,5-diphenylisoxazolidine (60b)

Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.49–6.82 (13H, m, Ar-H), 5.98-5.97 (2H, d, J = 2.5 Hz, OC*H*$_2$O), 5.38-5.35 (1H, t, J = 8.0 Hz, OC*H*Ph), 4.61-4.58 (1H, m, NC*H*Ar), 2.77–2.74 (1H, m, NCHC*H*H), 2.64-2.62 (1H, m, NCHCH*H*).

(*3SR,5RS*)-3-(2-nitrophenyl)-2,5-diphenylisoxazolidine (61)

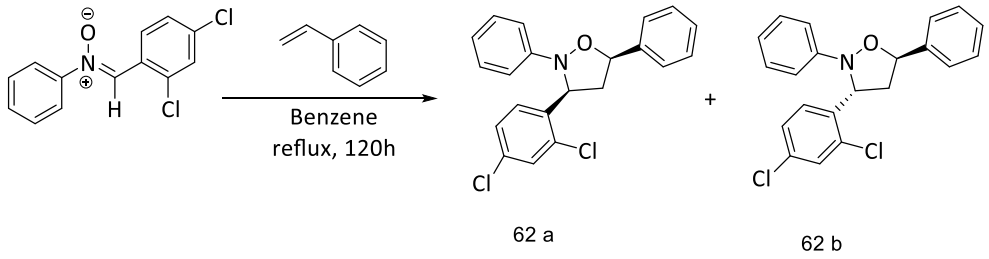


Following the general procedure D, (2.42 g, 10 mmol, 1.0 eq.) of (*Z*)-1-(2-nitrophenyl)-*N*-phenylmethanimine oxide 55 and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using benzene as a solvent and the reaction mixture was refluxed for 7 days to yield a combination of two inseparable diastereoisomers 61a and 61b (2.35 g, 68% yield). ^{1}HNMR spectroscopy of crude products showed two diastereoisomers, and the ratio of cis 61a and trans 61b were 95:5. X-ray crystallography confirmed the primary diastereoisomer's arrangement as cis-diastereoisomer.

(*3SR,5RS*)-3-(2-nitrophenyl)-2,5-diphenylisoxazolidine (61a)

Yellow crystal: ^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 8.29-8.28 (1H, d, J = 7.9 Hz, O_2NCCH), 8.09 -8.08 (1H, d, J = 8.1 Hz, O_2NCCCH), 7.73-7.71 (1H, t, J = 7.5 Hz, $O_2NCCHCH$), 7.49-7.47 (1H, t, J = 7.7 Hz, O_2NCCCH), 7.40-7.23 (7H, m, ArCH), 7.03-6.98 (3H, m, ArCH), 5.73 -5.71 (1H, t, J = 7.1 Hz, OC*H*Ph), 5.25-5.22 (1H, t, J = 7.8 Hz, NCHAr), 3.56-3.49 (1H, m, NCHCHH), 2.30-2.25 (1H, m, NCHCHH); ^{13}C NMR (151 MHz, CDCl3) δ ppm: 151.3 (O_2N-C), 147.1 (N-C), 139.2 (N-CH-C),137.3 (O-CH-C), 134.1 (ArCH), 129.2 (2xArCH), 129.0 (ArCH), 128.6 (2xArCH), 128.5 (ArCH), 128.2 (ArCH), 126.8 (2xArCH), 124.8 (ArCH), 121.7 (ArCH), 113.6 (2xArCH), 80.52 (O-C), 68.08 (N-CH), 47.75 (N-CH-CH2).

(*3SR,5RS*)-3-(3,5-dichlorophenyl)-2,5-diphenylisoxazolidine(62)



62 a 62 b

Following the general procedure D, (2.66 g, 10 mmol, 1.0 eq.) of (*Z*)-1-(2,4-dichlorophenyl)-*N*-phenylmethanimine oxide 56 and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using benzene as a solvent and the reaction mixture was refluxed for 5 days to yield a combination of two inseparable diastereoisomers 62a and 62b (2.47 g, 67% overall yield) as a dark black oil. ^{1}HNMR spectroscopy of crude products showed two diastereoisomers, and the ratio of cis 62a and trans 62b were 88:12.

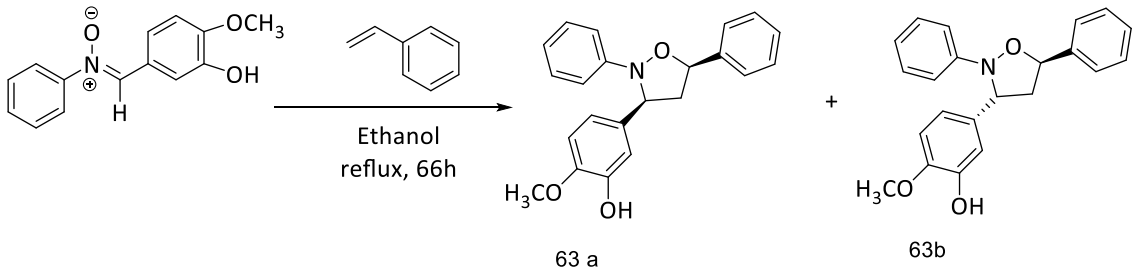
Cis (3SR,5RS)-3-(3,5-dichlorophenyl)-2,5-diphenylisoxazolidine(62a)

^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.91 (1H, d, J = 8.3 Hz, ClC*H*Cl), 7.60 – 7.26 (9H, m, ArCH), 7.08 – 6.93 (3H, m, ArCH), 5.30-5.28 (1H, t, J = 7.1 Hz, OC*H*Ph), 5.22-5.19 (1H, t, J = 7.8 Hz, NC*H*Ar), 3.40 -3.36 (1H, m, NCHCH*H*), 2.27-2.22 (1H, m, NCHC*H*H); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 151.61 (N-*C*), 139.34 (N-CH-*C*), 137.39 (Cl-*C*), 133.50 (Cl-*C*), 132.39 (O-CH-*C*), 129.2 (2xArCH), 129.1 (ArCH), 128.9 (ArCH), 128.6 (2xArCH), 128.5 (ArCH), 127.8 (ArCH), 126.9 (2xArCH), 121.6 (ArCH), 113.3 (2xArCH), 80.62 (O-*C*),68.2 (N-*C*H), 46.8 (N-CH-*C*H_2).

Trans (3SR,5RS)-3-(3,5-dichlorophenyl)-2,5-diphenylisoxazolidine(62b)

^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.86-7.84 (1H, d, J = 8.3 Hz, ClC*H*Cl), 7.60 – 7.26 (9H, m, ArCH), 7.08 – 6.93 (3H, m, ArCH), 5.09-5.08 (1H, d, J = 9.2 Hz, OC*H*Ph), 3.50-3.47 (1H, m, NC*H*Ar), 2.92-2.86 (1H, m, NCHCH*H*), 2.55-2.52 (1H, m, NCHC*H*H).

5-(*3SR,5RS*)-2,5-diphenylisoxazolidin-3-yl)-2-methoxyphenol (63)



Following the general procedure D, (2.43 g, 10 mmol, 1.0 eq.) of (*Z*)-1-(3-hydroxy-4methoxyphenyl)-*N*-phenylmethanimine oxide 57 and styrene 23 (1.04 g, 1.14 mL, 10 mmol, 1.0 eq.) using ethanol 60% as a solvent and the reaction mixture was refluxed for 66 hrs to yield a combination of two inseparable diastereoisomers 63a and 63b (2.32 g, 67% overall yield) as a yellow solid. ^{1}HNMR spectroscopy of crude products showed two diastereoisomers, and the ratio of cis 63a and trans 63b were 88:12.

Cis 5-(*3SR,5RS*)-2,5-diphenylisoxazolidin-3-yl)-2 methoxyphenol (63a)

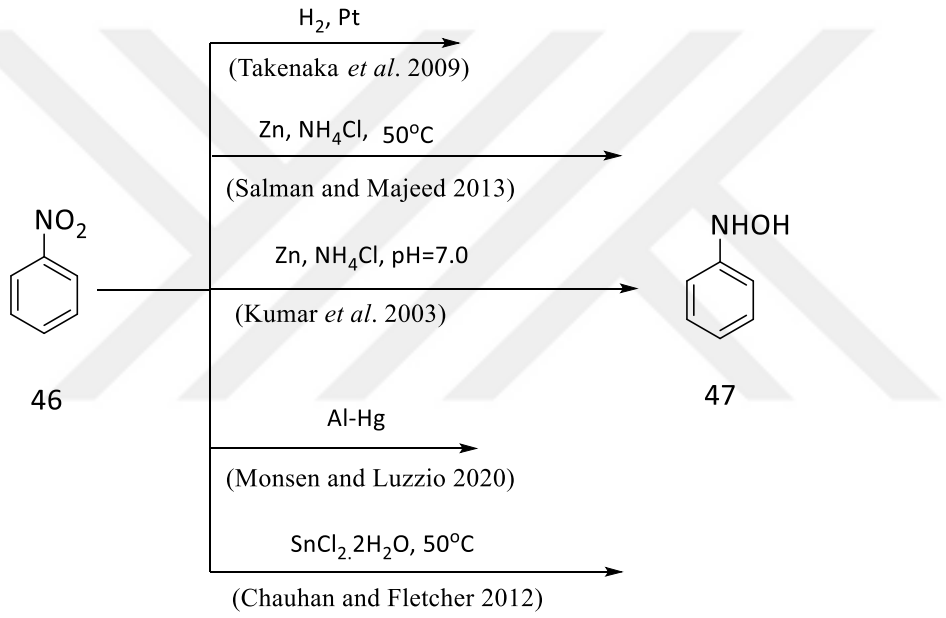
^{1}H NMR (600 MHz, $CDCl_3$) δ ppm: 7.47-6.92 (13H, m, Ar-H), 5.61 (1H, s, O*H*), 5.21-5.18 (1H, t, J = 7.1 Hz, OC*H*Ph), 4.87-4.85 (1H, t, J = 7.8 Hz, NC*H*Ar), 3.88 (3H, s, OCH_3), 3.18-3.15 (1H, m, NCHCH*H*), 2.50-2.45 (1H, dd, J = 20.2, 10.0 Hz, NCHCH*H*); ^{13}C NMR (151 MHz, $CDCl_3$) δ ppm: 152.52 (HO-*C*), 146.95 (MeO-*C*), 144.80 (N-*C*), 138.00 (N-CH-*C*),134.83 (O-CH-*C*), 128.98 (2xArCH), 128.58 (ArCH), 128.33 (ArCH), 126.82 (2xArCH), 121.38 (ArCH), 119.05 (ArCH), 115.92 (ArCH), 114.29 (ArCH), 113.98 (2xArCH), 108.50 (ArCH), 80.39 (O-*C*), 71.36 (N-*C*H), 55.98 (OCH_3), 48.69 (N-CH-CH_2).

Trans 5-(*3SR,5RS*)-2,5-diphenylisoxazolidin-3-yl)-2 methoxyphenol (63b)

1H NMR (600 MHz, CDCl3) δ ppm: 7.47-6.92 (13H, m, Ar-H), 5.61 (1H, s, O*H*), 5.38-5.36 (1H, t, J = 7.1 Hz, OC*H*Ph), 4.63-4.58 (1H, m, NC*H*Ar), 3.91 (3H, s, OCH_3), 2.78-2.74 (1H, m, NCHCH*H*), 2.68-2.65 (1H, m, NCHC*H*H).

4. RESULTS AND DISCUSSION

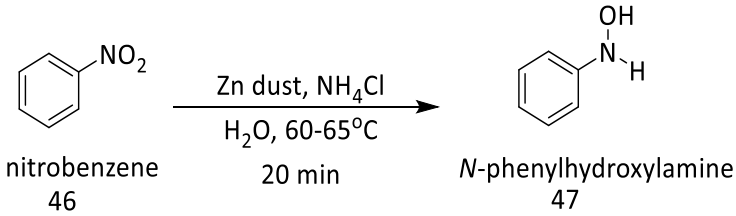
Our approach to the synthesis of a series of new nitrones involves the condensation strategy of *N*-phenylhydroxylamine with a range of different aldehydes. *N*-phenylhydroxylamine can easily be prepared by aromatic nitro compound. Several metals and reducing agents were used to reduce nitro group to hydroxylamines for example, Zn (Ung *et al*. 2005, Kumar *et al*. 2003), Al-Hg (Monsen and Luzzio 2020), $SnCl_2$ (Kazemi *et al*. 2019), $NaBH_4$ (Aliyan *et al*. 2012), catalytic hydrogenation (Takenaka *et al*. 2009) Scheme (4.1).



(4.1)

Reduce nitro group to hydroxylamines Scheme (4.1)

For preparation of nitrones we decided to prepare aromatic *N*-phenylhydroxylamine. This compound was prepared by several authors such as (Salman and Majeed 2013, Mahieddine *et al*. 2016, Salman *et al.* 2020). *N*-phenylhydroxylamine was prepared from heating nitrobenzene using zn and ammonium chloride at 60^0C Scheme (4.2) (Furniss *et al.* 1989)



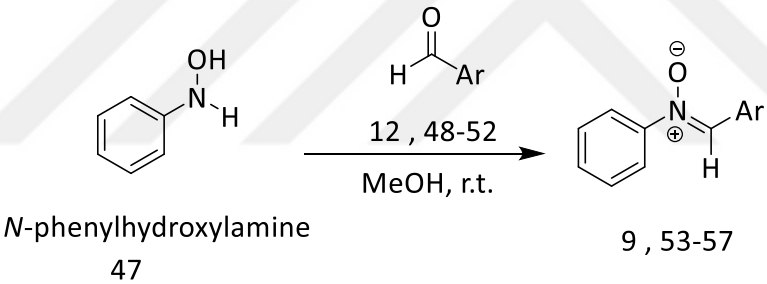
nitrobenzene
46

N-phenylhydroxylamine
47

(4.2)

Preparation *N*-phenylhydroxylamine Scheme (4.2)

After preparation of *N*-phenylhydroxylamine, employed in the synthesis of nitrones. The optimization was applied to the reaction conditions, we were accomplished three different procedures. The first procedure (A), was included the condensation reaction of *N*-phenylhydroxylamine (47) with different aldehydes 12 and (48-52) separately using methanol as solvent.The stirring of the reaction mixture at room temperature for the desired times furnished to the demanded products but in moderate yields 60-64% Scheme (4.3), (Table 4.1)



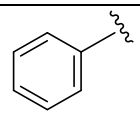
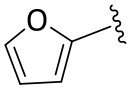
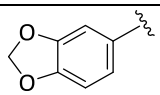
N-phenylhydroxylamine
47

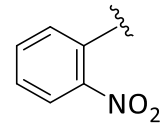
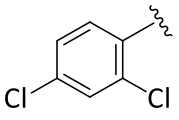
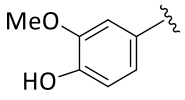
9 , 53-57

(4.3)

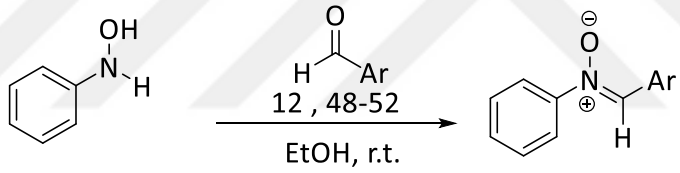
Procedure A for the synthesis of nitrones Scheme (4.3)

Table 4.1 Nitrones produced in procedure A

AR	NITRONE NO.	REACTION TIME / HR.	YIELD %
 12	9	3.0	64
 48	53	8.0	60
 49	54	3.0	60

 50	55	8.0	60
 51	56	8.0	6.3
 52	57	3.0	60

The second procedure (B), was begun by the condensation reaction of *N*-phenylhydroxylamine (47) with a series of different aldehydes 12 and (48-52) using ethanol as solvent. Then the reaction mixture was stirred at room temperature for the indicated times producing the desired molecules in slightly better yields 73-78%, but this procedure has taken a longer time than the previous one Scheme (4.4), (Table 4.2).



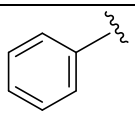
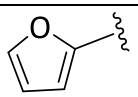
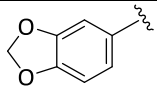
(4.4)

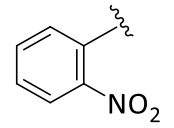
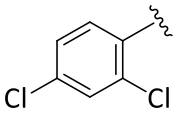
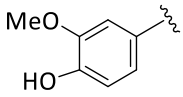
N-phenylhydroxylamine
47

9 , 53-57

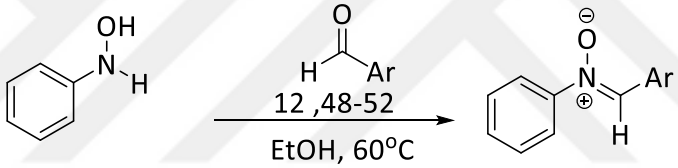
Procedure B for the synthesis of nitrones Scheme (4.4)

Table 4.2 Nitrones produced in procedure B

AR	NITRONE NO.	REACTION TIME / HR.	YIELD %
 12	9	5.0	73
 48	53	17.0	78
 49	54	4.0	78

 50	55	12.0	77
 51	56	44.0	75
 52	57	5.0	77

Following by modifying the reaction conditions, the optimal conditions for the reaction were reached by accomplishing it in ethanol as a reaction solvent, followed by heating at giving the desired product. As a result, the best nitrone yield was achieved at 60°C and shorter time as indicated below Scheme (4.5), (Table 4.3).



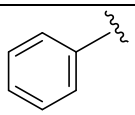
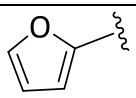
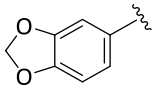
N-phenylhydroxylamine
47

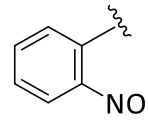
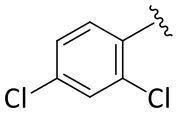
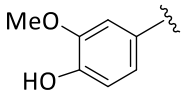
9 , 53-57

(4.5)

Procedure C for the synthesis of nitrones Scheme (4.5)

Table 4.3 Nitrones produced in procedure C

AR	NITRONE NO	REACTION TIME / HR	YIELD %
 12	9	2.5	89
 48	53	6.0	92
 49	54	2.0	93

 50	55	6.0	93
 51	56	4.0	92
 52	57	0.5	93

All IR spectra for the synthesized nitrones illustrated the evanescence of (C=O) of aldehyde's carbonyl stretching band within the region (1740-1720) cm^{-1} and the appearance of (C=N), (C-N), and (N-O) bands within the regions (1618-1577) cm^{-1}, (1396-1342) cm^{-1}, and (119-1051) cm^{-1} respectively. For example, compound 53 as a representative compound showed the IR spectrum below (Figure 4.1).

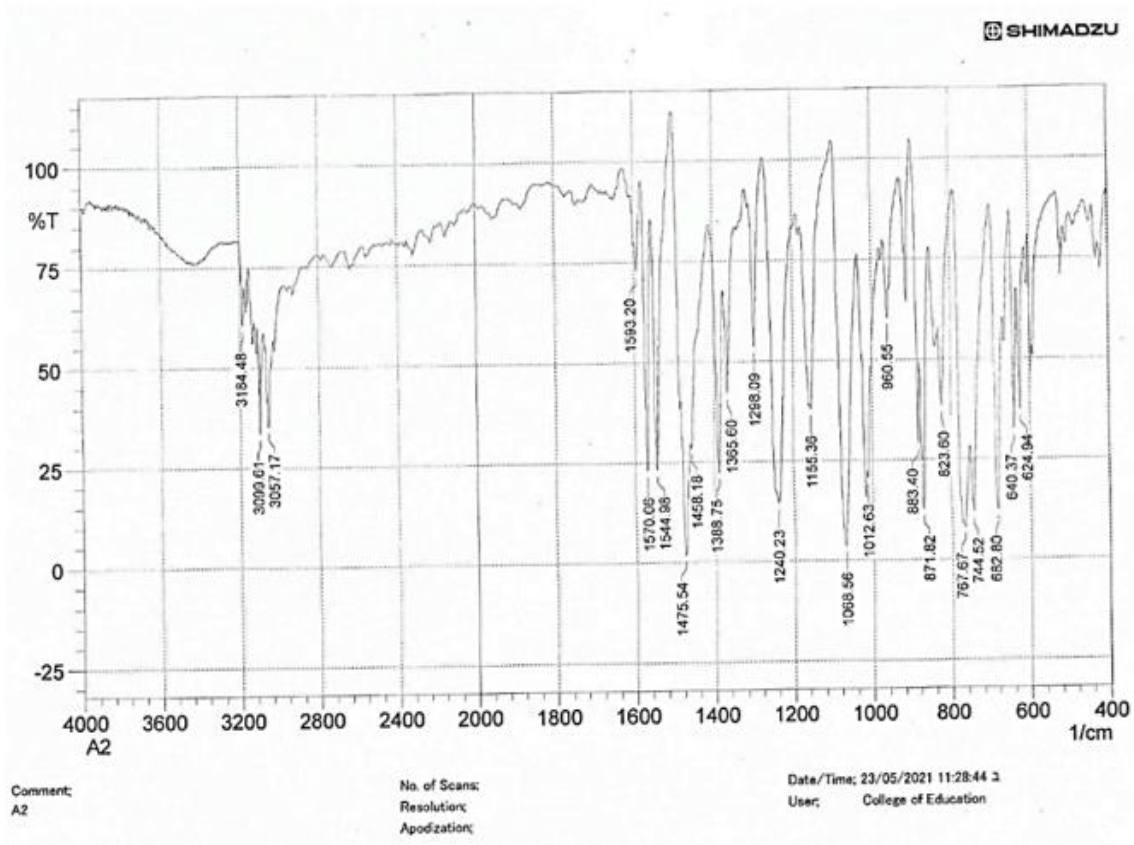
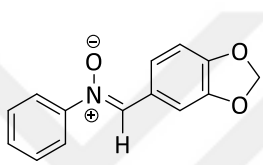
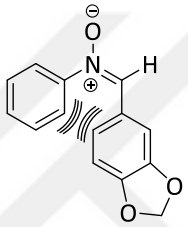


Figure 4.1 IR spectrum for compouned 53

Configurational determination of compounds 53 and 54: Inouye has concluded a solvent effect directly on the configuration of the nitrones. This study has Interestingly explained that the *E*-nitrones will be the major product in non-polar solvents whilst in polar solvents, *Z*-nitrones are the major products. Moreover, he also suggested that the *Z*-isomer nitrones exist as crystalline only. Additionally, the steric hindrance effect in the *Z*-isomer nitrones will be less than in the *E*-isomer, which led to being more stability in the *Z*-isomer (Inouye *et al*. 1980) This study is strongly agreed with our finding (Figure 4.2).



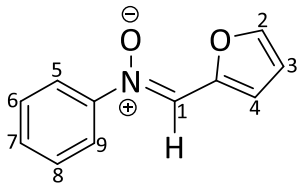
(*Z*)-1-(benzo[*d*][1,3]dioxol-5-yl)-*N*-phenylmethanimine oxide



(*E*)-1-(benzo[*d*][1,3]dioxol-5-yl)-*N*-phenylmethanimine oxide

Figure 4.2 *Z*-isomer and *E*-isomer nitrones Compound 54

Determination of configuration of 53 by ^{1}H NMR:



The structure of 53 was determined by the use of ^{1}H and Correlated spectroscopy (COSY) NMR spectroscopy. It was found that H at C-1 appears as singlet at 7.55 ppm, H at C-2 appears as singlet at 8.1 ppm, H at C-4 appears as singlet at 6.6 ppm, which coupled with C-3 H, C-2 H and C-1 H (Figure 4.3).

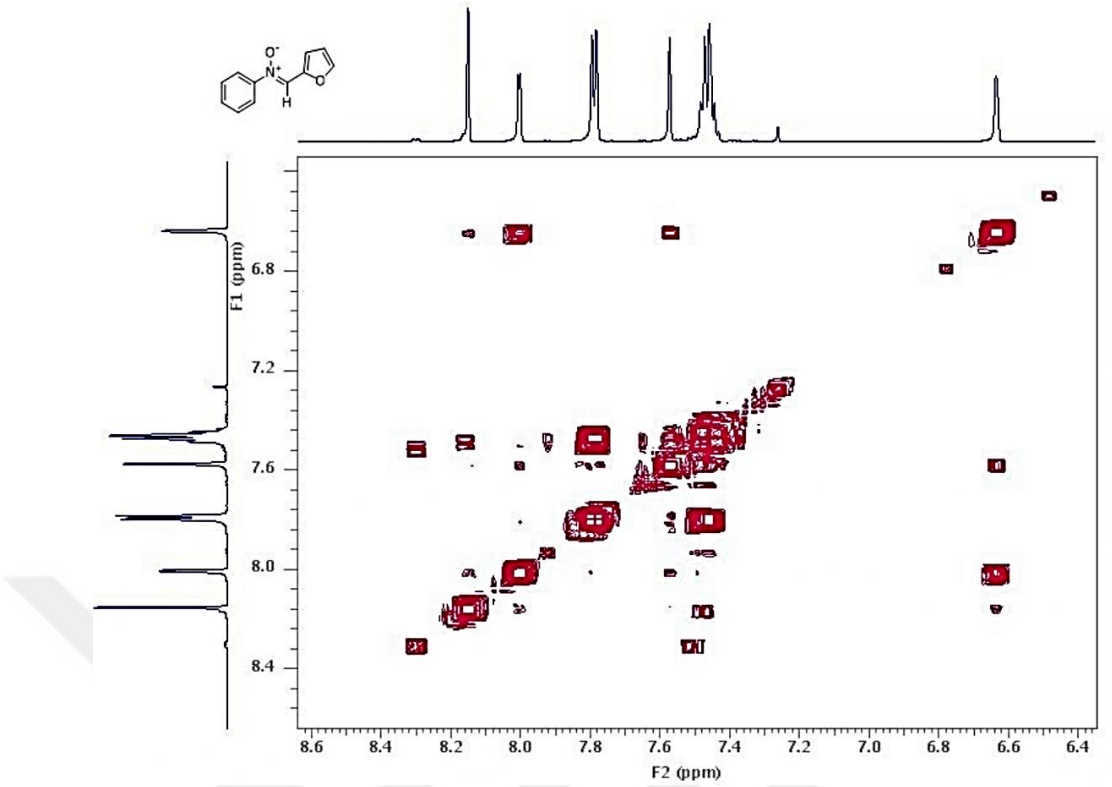


Figure 4.3 COSY spectrum of 53

Moreover, Heteronuclear Single-Quantum Correlation Spectroscopy (HSQC) NMR spectroscopy was used in the determination of the structure of 53. ^{13}C NMR of this compound showed these results measured by ppm: 147.50 (N=*C*H), 147.22 (N-*C*), 144.69 (N=CH-*C*), 129.95 (Arquat.C), 129.18 (ArCH), 124.36 (ArCH), 121.02 (ArCH), 116.52 (O-*C*H), 112.72 (O-CH*C*H). As a result of (HSQC) spectroscopy, we can strongly confirm the right structure of the nitrone 53 (Figure 4.4). All data are compatible with the literature (Choi *et al*. 2018).

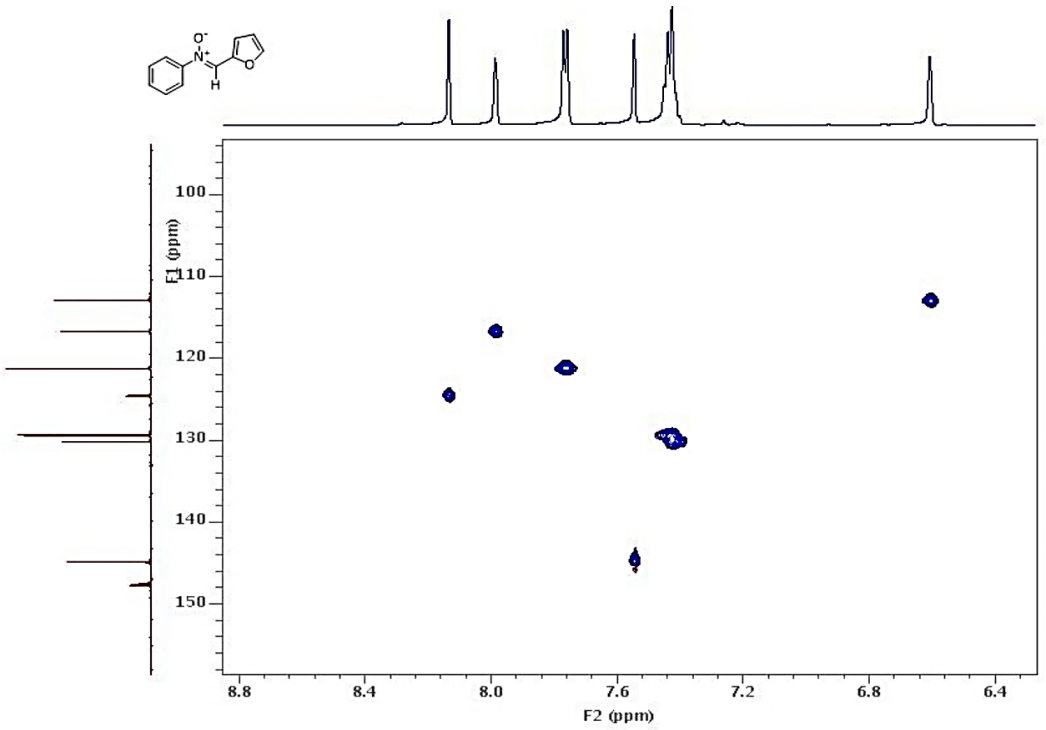
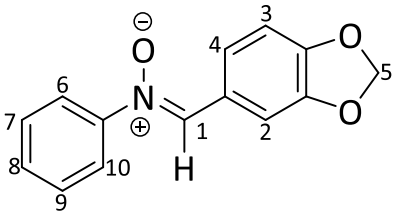


Figure 4.4 HSQC spectrum of 53

Determination of configuration of 54 by ^{1}H NMR:



The structure of 54 was determined by the use of ^{1}H and Heteronuclear Single-Quantum Correlation Spectroscopy (HSQC) NMR spectroscopy. It was found that H at C-1 appears as singlet at 7.82 ppm, H at C-2 appears as singlet at 8.30 ppm, two protons at C-5 appear as singlet at 6.04 ppm. ^{13}C NMR of this compound illustrated these results measured by ppm: 190.34 (O-*C*), 149.71 (O-*C*), 148.89 (N-*C*), 147.71 (N=CH-*C*), 134.33 (N=*C*H), 129.74 (Arquat.C), 129.14 (ArCH), 128.72 (ArCH), 125.31 (ArCH), 125.22 (ArCH), 121.64 (ArCH), 108.64 (OC*C*H), 108.51 (OC*C*H), 108.35 (ArCH), 102.12 (ArCH), 101.66 (O-*C*H_2) (Figure 4.5). All data are compatible with the literature (Choi *et al*. 2018).

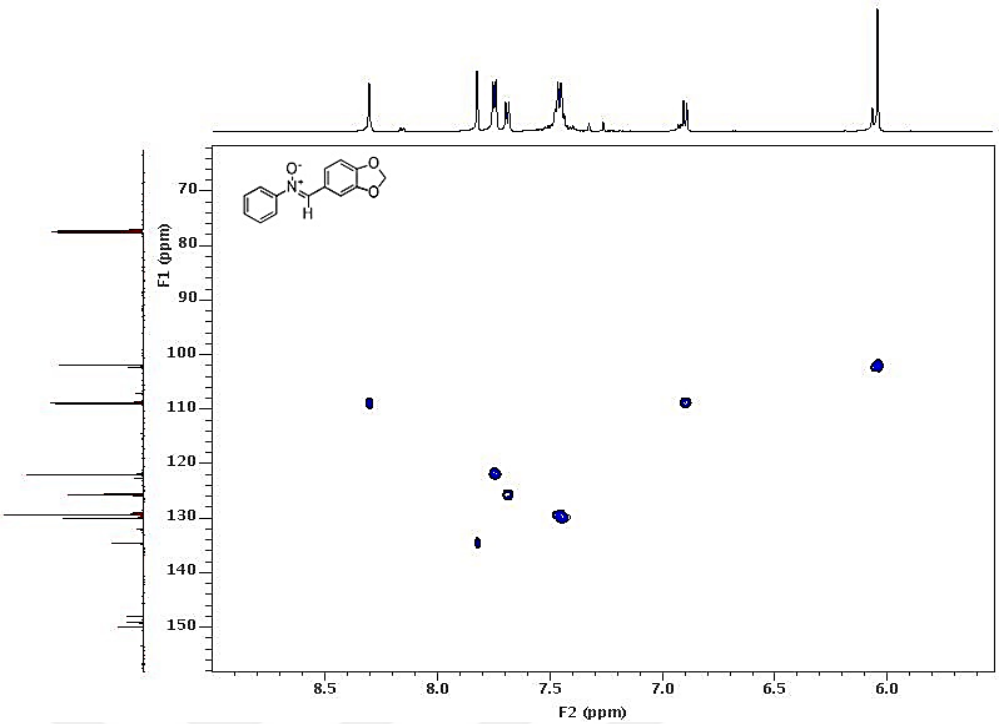
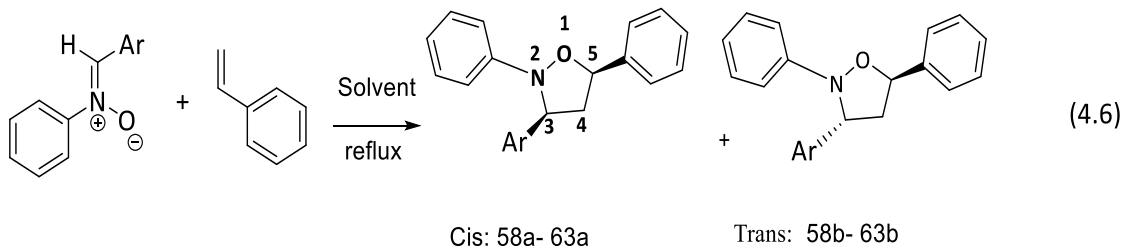


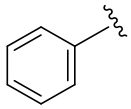
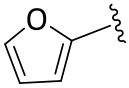
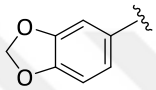
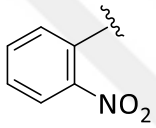
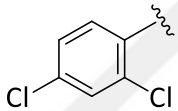
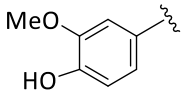
Figure 4.5 HSQC spectrum of 54

The configuration of the compounds 53 and 54 were established. Therefor, this would concider as a base for establishing the formation of the nitrones remaining by ^{1}HNMR only as shown in the appendix. The achieve success in the synthesis of nitrones, we considered that the protocol could be extended to the synthesis of isoxazolidine using 1,3-dipolar cycloaddition reaction. The cyclization strategy was involved heating at reflux for nitrones with styrene in the different solvents, each product was obtained at indicated time below Scheme (4.6), (Table 4.4).



Scheme (4.6)

Table 4.4 Isoxazolidine produced in procedure D

AR	ISOXAZOLIDINE	REACTION TIME / HR	RATIO OF DIASTEREOISOMERS (CIS: 58A-63A : *TRANS: 58B-63B)*	YIELD %	PHYSICAL STATUS
	58	72	90:10	77	Light yellow solid
	59	45	95:5	69	Light brown oil
	60	120	90:10	75	Yellow solid
	61	168	95:5	68	Yellow crystal
	62	120	88:12	67	Dark black oil
	63	66	88:12	67	Yellow solid

In the order to establish the stereochemistry of the isoxazolidine. The structure of compound 61a was confirmed by use single-crystal X-ray, that showed this compound is a cis-diastereoisomer. The substituents at C-3 and C-5 were (unsurprisingly) are in the sameface (Figure 4.6).

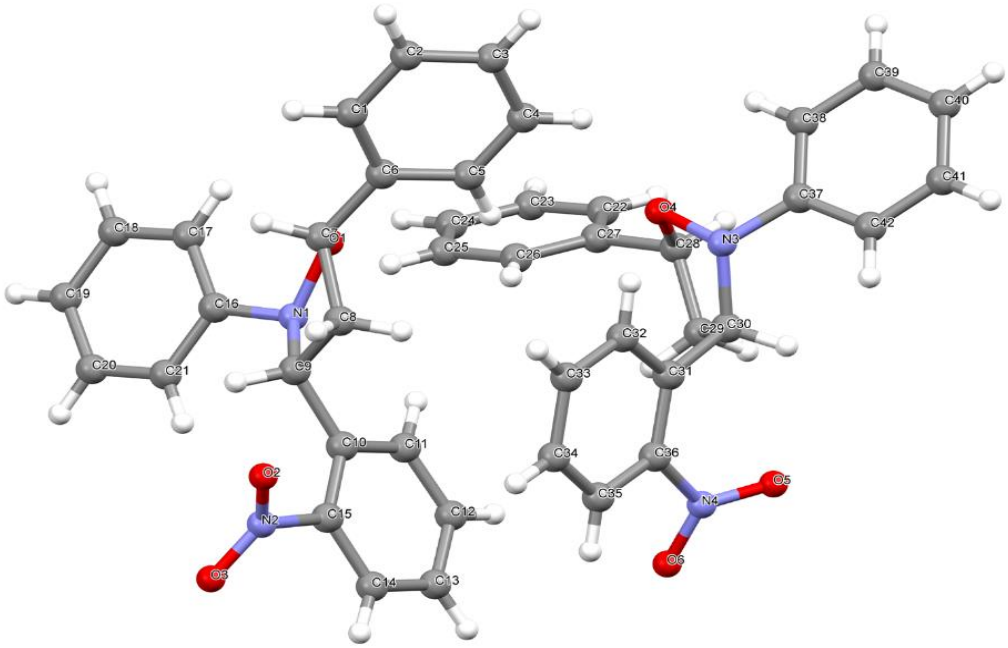


Figure 4.6 Crystal structure of the major product 61a which confirms *cis*-configuration

Moreover, the spectrum ^{1}H NMR for compound 61a showed a triplet for proton at C-3 (5.23 ppm), with a coupling constant (7.8 Hz), the proton at C-5 showed a triplet (5.72) with a coupling constant (7.1 Hz). Furthermore, the protons at C-4 showed two multiplet signals at 3.56 – 3.46 and 2.31 – 2.25. Spectroscopic data for the rest of the products are identical and closely matched with compound 61, which allows us to determine the stereochemistry (Figure 4.7).

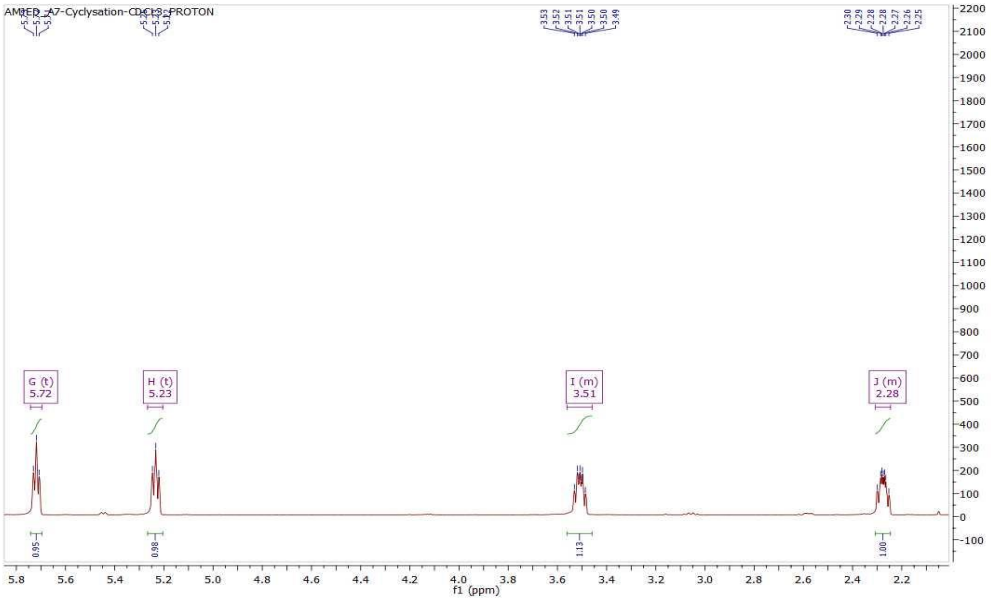


Figure 4.7 Proton [1]H NMR signals at C-3, C-5, and C-4 for compound 61

Furthermore, the physical and spectroscopic properties of compound 58a were found to be quite comparable to those described in the literature (Huisgen *et al.* 1968). Moreover, Zheng and co-workers have been synthesized the compound (*3R,4R*)-2,3,4-triphenylisoxazolidine, which shows chemical shifts that differ from our results in [1]HNMR analysis (Figure 4.8) (Zheng *et al.* 2017). We can clearly establish the absolute configuration of our products by comparing our findings to Zheng's results (Figure 4.9).

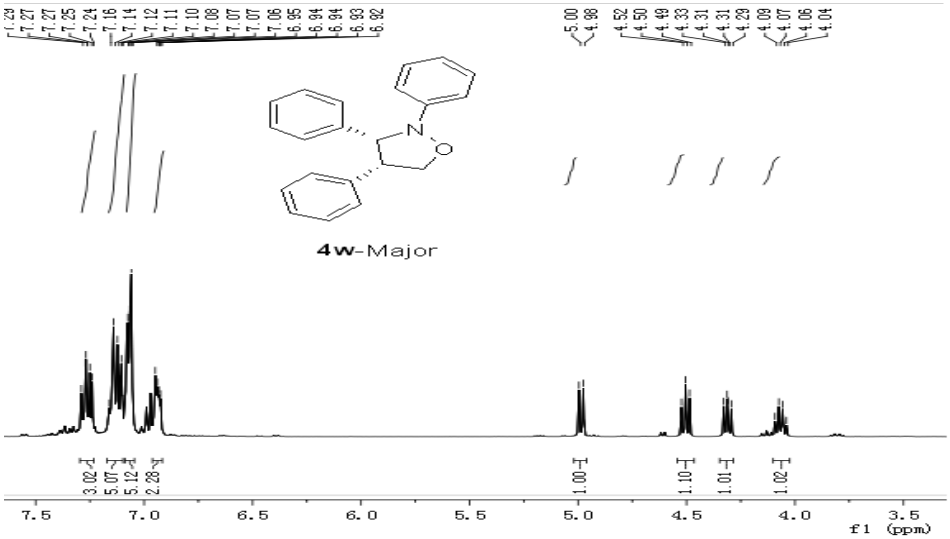


Figure 4.8 [1]H NMR spectrum (Zheng *et al.* 2017).

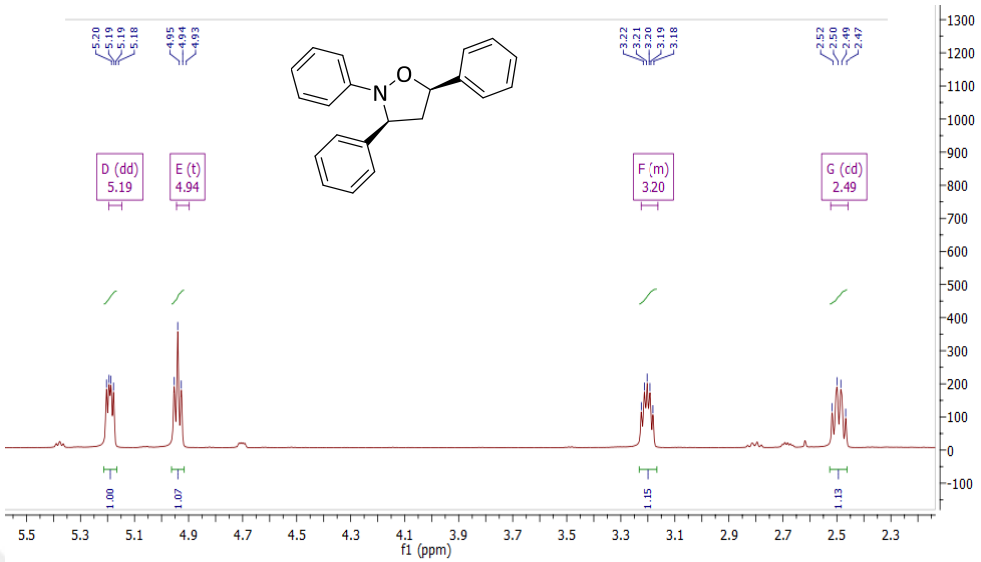


Figure 4.9 Compound 58a ^{1}H NMR spectrum

5. CONCLUSIONS AND RECOMMENDATION

To summarize, we have successfully developed a new and efficient methodology for producing a variety of some novel nitrones from commercially available starting materials. In the synthesis of nitron, we utilized three different approaches, beginning with *N*-phenylhydroxylamine, followed by a condensation reaction with some specified aldehydes. We have found that the best conditions for the reaction were achieved by performing it in refluxed ethanol as a reaction solvent to produce the desired product. The 1,3-dipolar cycloaddition technology was expanded in the production of new isoxazolidine derivatives. These nitrones have been used in the synthesis of a number of novel isoxazolidines. The required compounds were created by the addition of nitrones to styrene under catalyst-free conditions.

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APPENDICES

APPENDIX 1. Crystal structure data (Compound 61)

APPENDIX 2. ^{1}H NMR Spectra for the compounds 9 and 53-63.

APPENDIX 3. ^{13}C NMR Spectra for the compounds 9 and 54-64

APPENDIX 1. Crystal structure data compound 61

Introduction: The X-ray crystallography data shown in the appendix were obtained by Assoc. Prof. Dr. Onur ŞAHİN at the University of Sinop, Turkey.

X-Ray diffraction analysis

Suitable crystal of **gultekin1 (61-Cy)** was selected for data collection which was performed on a D8-QUEST diffractometer equipped with a graphite-monochromatic Mo-K_α radiation at 296 K. The structure was solved by direct methods using SHELXS-2013 [1] and refined by full-matrix least-squares methods on F^2 using SHELXL-2013 [2]. The H atoms were located from different maps and then treated as riding atoms with C-H distances of 0.93-0.98Å.The following procedures were implemented in our analysis: data collection: Bruker APEX2 [3]; program used for molecular graphics were as follow: MERCURY programs [4]; software used to prepare material for publication: WinGX [5].

References

[1] Sheldrick, G.M. Acta Cryst. 2008, A64, 112.

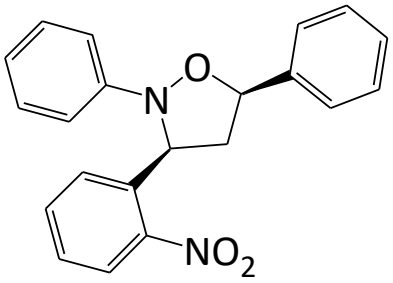
[2] Sheldrick, G. M. Acta Cryst. 2015, C71, 3.

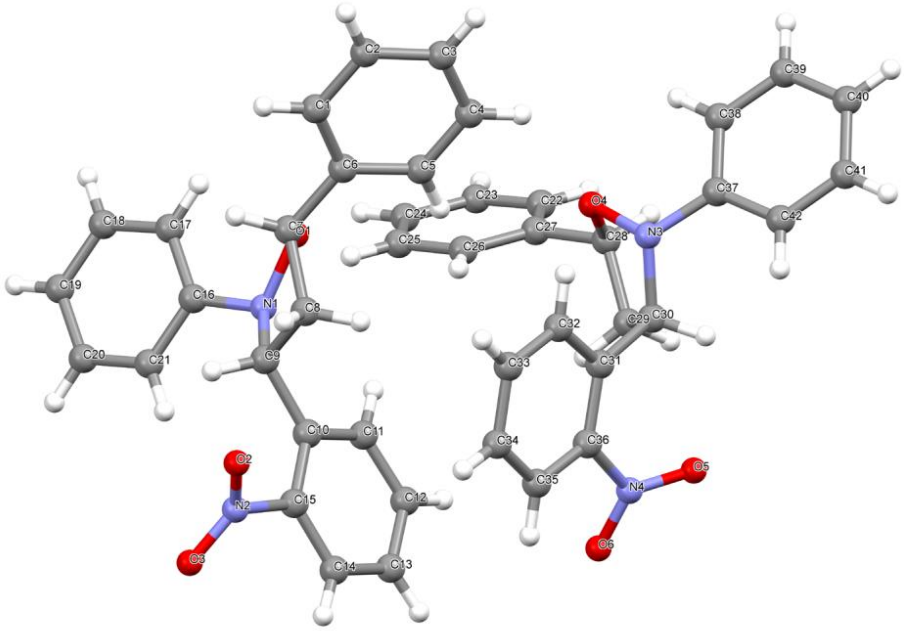
[3] APEX2, Bruker AXS Inc. Madison Wisconsin USA (2013).

[4] Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). J. Appl. Cryst. 41, 466–470.

[5] Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849–854.

Crystal data and structure refinement for the compound 61.





Title

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Abstract

Table 1

Experimental details

Crystal data	
Chemical formula	$C_{21}H_{18}N_2O_3$
M_r	346.37
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	9.5280 (6), 13.5985 (10), 14.7652 (11)
α, β, γ (°)	100.134 (2), 98.918 (3), 106.949 (2)
V (Å^3)	1757.7 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.19 × 0.18 × 0.15
Data collection	
Diffractometer	Bruker *APEX3* CCD
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	67393, 8747, 6440
R_{int}	0.049
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.669
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.130, 1.06
No. of reflections	8747
No. of parameters	469
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.20

Computer programs: *APEX3* (Bruker, 2013), *SAINT*(Bruker, 2013), Bruker *SAINT*, *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *Mercury* (Macrae, 2008), *WinGX* (Farrugia, 2012).

Acknowledgements

Funding information

References

Figure 1

supporting information

Title

Computing details

Data collection: *APEX3* (Bruker, 2013); cell refinement: *SAINT*(Bruker, 2013); data reduction: Bruker *SAINT*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

(shelx)

Crystal data

$C_{21}H_{18}N_2O_3$
$M_r = 346.37$
Triclinic, $P\overline{1}$
$a = 9.5280\ (6)$ Å
$b = 13.5985\ (10)$ Å
$c = 14.7652\ (11)$ Å
$\alpha = 100.134\ (2)°$
$\beta = 98.918\ (3)°$
$\gamma = 106.949\ (2)°$
$V = 1757.7\ (2)$ Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9985 reflections
$\theta = 2.3–27.8°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Block, yellow
0.19 × 0.18 × 0.15 mm

Data collection

Bruker APEX-3 CCD diffractometer
φ and ω scans
67393 measured reflections
8747 independent reflections
6440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.9°$
$h = -12\rightarrow12$
$k = -18\rightarrow18$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.130$
$S = 1.06$
8747 reflections
469 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.7014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6219 (2)	0.53929 (15)	0.67168 (14)	0.0493 (4)
H1	0.6137	0.5658	0.6179	0.059*
C2	0.7506 (2)	0.58421 (16)	0.74217 (17)	0.0600 (5)
H2	0.8285	0.6406	0.7355	0.072*
C3	0.7641 (2)	0.54619 (17)	0.82178 (16)	0.0600 (5)
H3	0.8501	0.5774	0.8698	0.072*
C4	0.6498 (2)	0.46146 (17)	0.83051 (14)	0.0584 (5)
H4	0.6595	0.4345	0.8840	0.070*
C5	0.5216 (2)	0.41664 (15)	0.76060 (13)	0.0493 (4)
H5	0.4448	0.3596	0.7673	0.059*
C6	0.50487 (18)	0.45519 (13)	0.68013 (12)	0.0399 (4)
C7	0.36412 (19)	0.40831 (13)	0.60337 (12)	0.0421 (4)
H7	0.3779	0.4403	0.5495	0.050*
C8	0.21871 (19)	0.41212 (13)	0.63004 (14)	0.0456 (4)
H8A	0.2049	0.4797	0.6280	0.055*
H8B	0.2159	0.3999	0.6926	0.055*
C9	0.09969 (17)	0.32250 (12)	0.55481 (11)	0.0357 (3)
H9	0.0656	0.3498	0.5014	0.043*
C10	−0.03410 (17)	0.26683 (12)	0.59226 (11)	0.0348 (3)
C11	−0.0341 (2)	0.18068 (14)	0.63077 (13)	0.0451 (4)
H11	0.0473	0.1561	0.6314	0.054*
C12	−0.1522 (2)	0.13050 (16)	0.66818 (14)	0.0545 (5)
H12	−0.1479	0.0740	0.6948	0.065*
C13	−0.2754 (2)	0.16321 (17)	0.66644 (14)	0.0578 (5)
H13	−0.3540	0.1294	0.6922	0.069*
C14	−0.2826 (2)	0.24597 (17)	0.62663 (13)	0.0519 (5)
H14	−0.3668	0.2677	0.6236	0.062*
C15	−0.16235 (19)	0.29657 (14)	0.59107 (11)	0.0415 (4)
C16	0.16846 (16)	0.21167 (11)	0.42749 (11)	0.0313 (3)
C17	0.28864 (18)	0.19818 (13)	0.39031 (12)	0.0390 (4)
H17	0.3840	0.2190	0.4291	0.047*
C18	0.2656 (2)	0.15356 (15)	0.29521 (13)	0.0479 (4)
H18	0.3468	0.1455	0.2706	0.057*
C19	0.1260 (2)	0.12092 (14)	0.23638 (12)	0.0491 (4)
H19	0.1122	0.0908	0.1727	0.059*
C20	0.0066 (2)	0.13353 (14)	0.27347 (13)	0.0496 (4)
H20	−0.0887	0.1117	0.2344	0.059*
C21	0.02693 (18)	0.17818 (13)	0.36784 (13)	0.0431 (4)
H21	−0.0548	0.1860	0.3919	0.052*
C22	0.41362 (19)	−0.03205 (14)	0.75724 (13)	0.0447 (4)
H22	0.4499	−0.0624	0.8034	0.054*
C23	0.3937 (2)	−0.07912 (15)	0.66297 (14)	0.0512 (5)
H23	0.4141	−0.1419	0.6461	0.061*
C24	0.3438 (2)	−0.03344 (16)	0.59437 (14)	0.0531 (5)
H24	0.3325	−0.0643	0.5311	0.064*
C25	0.3106 (2)	0.05811 (16)	0.61930 (14)	0.0564 (5)
H25	0.2764	0.0890	0.5729	0.068*
C26	0.3276 (2)	0.10378 (14)	0.71243 (14)	0.0494 (4)
H26	0.3036	0.1652	0.7285	0.059*

C27	0.38004 (17)	0.05995 (13)	0.78327 (12)	0.0378 (4)
C28	0.39477 (19)	0.10894 (13)	0.88505 (12)	0.0416 (4)
H28	0.4504	0.0762	0.9251	0.050*
C29	0.2480 (2)	0.10626 (14)	0.91564 (15)	0.0494 (4)
H29A	0.2180	0.0508	0.9490	0.059*
H29B	0.1678	0.0946	0.8616	0.059*
C30	0.28511 (18)	0.21676 (13)	0.98161 (12)	0.0377 (4)
H30	0.2680	0.2088	1.0441	0.045*
C31	0.19428 (18)	0.28219 (13)	0.94367 (11)	0.0372 (4)
C32	0.2670 (2)	0.37820 (14)	0.92387 (13)	0.0480 (4)
H32	0.3715	0.4027	0.9362	0.058*
C33	0.1888 (3)	0.43829 (17)	0.88641 (15)	0.0626 (5)
H33	0.2412	0.5023	0.8745	0.075*
C34	0.0346 (3)	0.40417 (19)	0.86668 (17)	0.0688 (6)
H34	−0.0175	0.4444	0.8408	0.083*
C35	−0.0425 (2)	0.31026 (18)	0.88539 (14)	0.0584 (5)
H35	−0.1470	0.2864	0.8723	0.070*
C36	0.03717 (19)	0.25158 (14)	0.92399 (11)	0.0418 (4)
C37	0.55148 (18)	0.27291 (12)	1.06952 (12)	0.0377 (4)
C38	0.6870 (2)	0.25635 (15)	1.06228 (14)	0.0504 (4)
H38	0.7053	0.2343	1.0033	0.060*
C39	0.7943 (2)	0.27273 (17)	1.14269 (16)	0.0592 (5)
H39	0.8849	0.2619	1.1372	0.071*
C40	0.7702 (2)	0.30462 (16)	1.23049 (15)	0.0565 (5)
H40	0.8436	0.3158	1.2842	0.068*
C41	0.6349 (2)	0.31997 (14)	1.23794 (13)	0.0489 (4)
H41	0.6167	0.3407	1.2973	0.059*
C42	0.5267 (2)	0.30498 (13)	1.15866 (12)	0.0434 (4)
H42	0.4367	0.3164	1.1647	0.052*
N1	0.18226 (14)	0.24919 (10)	0.52514 (9)	0.0361 (3)
N2	−0.1767 (2)	0.38528 (16)	0.55047 (13)	0.0584 (4)
N3	0.44640 (15)	0.26936 (11)	0.98835 (10)	0.0400 (3)
N4	−0.05408 (17)	0.15356 (13)	0.94468 (11)	0.0489 (4)
O1	0.33662 (13)	0.29623 (9)	0.57485 (8)	0.0453 (3)
O2	−0.0748 (2)	0.46910 (15)	0.57717 (16)	0.0905 (6)
O3	−0.2933 (2)	0.37166 (17)	0.49481 (14)	0.1005 (6)
O4	0.47673 (13)	0.22150 (9)	0.90277 (8)	0.0433 (3)
O5	−0.00113 (16)	0.12387 (14)	1.01053 (12)	0.0738 (5)
O6	−0.18003 (17)	0.10802 (13)	0.89615 (12)	0.0755 (5)

Atomic displacement parameters (Å²)

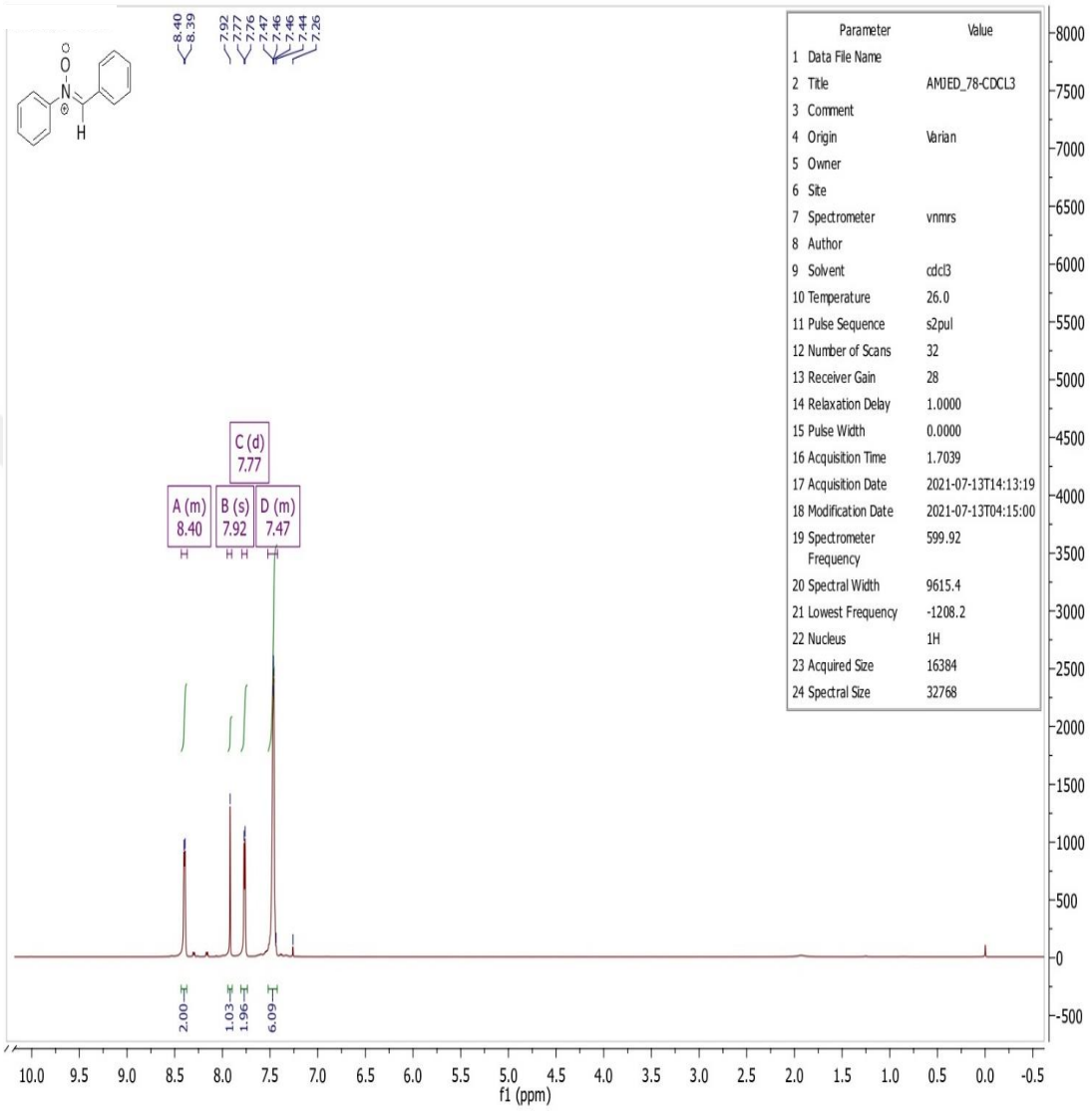
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0509 (11)	0.0470 (10)	0.0542 (11)	0.0152 (8)	0.0231 (9)	0.0137 (8)
C2	0.0406 (10)	0.0468 (11)	0.0839 (16)	0.0047 (8)	0.0215 (10)	0.0023 (10)
C3	0.0432 (11)	0.0575 (12)	0.0677 (14)	0.0203 (9)	−0.0005 (9)	−0.0100 (10)
C4	0.0666 (13)	0.0596 (12)	0.0484 (11)	0.0255 (11)	0.0062 (10)	0.0084 (9)
C5	0.0486 (10)	0.0461 (10)	0.0493 (10)	0.0085 (8)	0.0144 (8)	0.0103 (8)
C6	0.0378 (9)	0.0388 (9)	0.0422 (9)	0.0130 (7)	0.0135 (7)	0.0022 (7)
C7	0.0417 (9)	0.0427 (9)	0.0443 (9)	0.0130 (7)	0.0146 (7)	0.0133 (7)
C8	0.0409 (9)	0.0346 (9)	0.0587 (11)	0.0111 (7)	0.0175 (8)	0.0008 (8)
C9	0.0369 (8)	0.0353 (8)	0.0401 (8)	0.0158 (7)	0.0155 (7)	0.0094 (7)

C10	0.0331 (8)	0.0373 (8)	0.0318 (8)	0.0093 (6)	0.0076 (6)	0.0052 (6)
C11	0.0392 (9)	0.0486 (10)	0.0486 (10)	0.0128 (8)	0.0091 (8)	0.0175 (8)
C12	0.0535 (11)	0.0559 (11)	0.0507 (11)	0.0070 (9)	0.0120 (9)	0.0225 (9)
C13	0.0431 (11)	0.0717 (14)	0.0484 (11)	0.0000 (9)	0.0170 (9)	0.0139 (10)
C14	0.0372 (9)	0.0725 (13)	0.0453 (10)	0.0184 (9)	0.0151 (8)	0.0056 (9)
C15	0.0429 (9)	0.0484 (10)	0.0361 (9)	0.0190 (8)	0.0132 (7)	0.0067 (7)
C16	0.0329 (8)	0.0250 (7)	0.0365 (8)	0.0090 (6)	0.0095 (6)	0.0075 (6)
C17	0.0326 (8)	0.0446 (9)	0.0406 (9)	0.0150 (7)	0.0080 (7)	0.0082 (7)
C18	0.0499 (10)	0.0578 (11)	0.0450 (10)	0.0270 (9)	0.0192 (8)	0.0116 (8)
C19	0.0649 (12)	0.0462 (10)	0.0349 (9)	0.0231 (9)	0.0050 (8)	0.0031 (7)
C20	0.0420 (10)	0.0425 (10)	0.0503 (11)	0.0087 (8)	−0.0066 (8)	−0.0011 (8)
C21	0.0293 (8)	0.0416 (9)	0.0528 (10)	0.0076 (7)	0.0082 (7)	0.0043 (8)
C22	0.0432 (10)	0.0481 (10)	0.0496 (10)	0.0231 (8)	0.0105 (8)	0.0145 (8)
C23	0.0461 (10)	0.0474 (10)	0.0588 (12)	0.0202 (8)	0.0103 (9)	0.0016 (9)
C24	0.0454 (10)	0.0594 (12)	0.0440 (10)	0.0095 (9)	0.0051 (8)	0.0027 (9)
C25	0.0589 (12)	0.0545 (12)	0.0495 (11)	0.0136 (10)	−0.0027 (9)	0.0170 (9)
C26	0.0508 (11)	0.0403 (9)	0.0583 (11)	0.0192 (8)	0.0061 (9)	0.0125 (8)
C27	0.0311 (8)	0.0392 (9)	0.0445 (9)	0.0115 (7)	0.0111 (7)	0.0107 (7)
C28	0.0421 (9)	0.0406 (9)	0.0481 (10)	0.0175 (7)	0.0158 (8)	0.0133 (7)
C29	0.0446 (10)	0.0371 (9)	0.0681 (12)	0.0103 (8)	0.0264 (9)	0.0092 (8)
C30	0.0356 (8)	0.0402 (9)	0.0389 (9)	0.0117 (7)	0.0136 (7)	0.0097 (7)
C31	0.0436 (9)	0.0406 (9)	0.0291 (8)	0.0172 (7)	0.0113 (7)	0.0036 (6)
C32	0.0535 (11)	0.0447 (10)	0.0470 (10)	0.0157 (8)	0.0146 (8)	0.0110 (8)
C33	0.0825 (16)	0.0496 (11)	0.0648 (13)	0.0280 (11)	0.0214 (12)	0.0206 (10)
C34	0.0826 (17)	0.0720 (15)	0.0720 (15)	0.0491 (13)	0.0166 (12)	0.0278 (12)
C35	0.0559 (12)	0.0716 (14)	0.0548 (12)	0.0346 (11)	0.0098 (9)	0.0118 (10)
C36	0.0442 (9)	0.0489 (10)	0.0349 (8)	0.0202 (8)	0.0117 (7)	0.0054 (7)
C37	0.0348 (8)	0.0304 (8)	0.0456 (9)	0.0082 (6)	0.0104 (7)	0.0061 (7)
C38	0.0414 (10)	0.0571 (11)	0.0530 (11)	0.0178 (8)	0.0160 (8)	0.0058 (9)
C39	0.0408 (10)	0.0676 (13)	0.0703 (14)	0.0241 (9)	0.0099 (9)	0.0102 (11)
C40	0.0526 (11)	0.0525 (11)	0.0572 (12)	0.0170 (9)	−0.0028 (9)	0.0078 (9)
C41	0.0569 (11)	0.0400 (9)	0.0443 (10)	0.0143 (8)	0.0080 (8)	0.0013 (8)
C42	0.0409 (9)	0.0386 (9)	0.0497 (10)	0.0147 (7)	0.0120 (8)	0.0035 (7)
N1	0.0344 (7)	0.0372 (7)	0.0380 (7)	0.0148 (6)	0.0102 (6)	0.0053 (6)
N2	0.0677 (12)	0.0672 (12)	0.0647 (11)	0.0448 (10)	0.0325 (9)	0.0227 (9)
N3	0.0361 (7)	0.0431 (8)	0.0413 (8)	0.0119 (6)	0.0135 (6)	0.0083 (6)
N4	0.0410 (8)	0.0599 (10)	0.0493 (9)	0.0197 (8)	0.0190 (7)	0.0080 (8)
O1	0.0409 (6)	0.0479 (7)	0.0424 (6)	0.0222 (5)	−0.0019 (5)	−0.0048 (5)
O2	0.0831 (12)	0.0671 (11)	0.1532 (18)	0.0433 (10)	0.0517 (12)	0.0520 (12)
O3	0.1167 (16)	0.1206 (16)	0.0834 (13)	0.0735 (13)	−0.0010 (11)	0.0335 (11)
O4	0.0411 (6)	0.0443 (7)	0.0426 (6)	0.0079 (5)	0.0173 (5)	0.0086 (5)
O5	0.0532 (9)	0.0948 (12)	0.0833 (11)	0.0180 (8)	0.0206 (8)	0.0517 (10)
O6	0.0473 (9)	0.0818 (11)	0.0790 (11)	0.0045 (8)	0.0050 (8)	0.0083 (9)

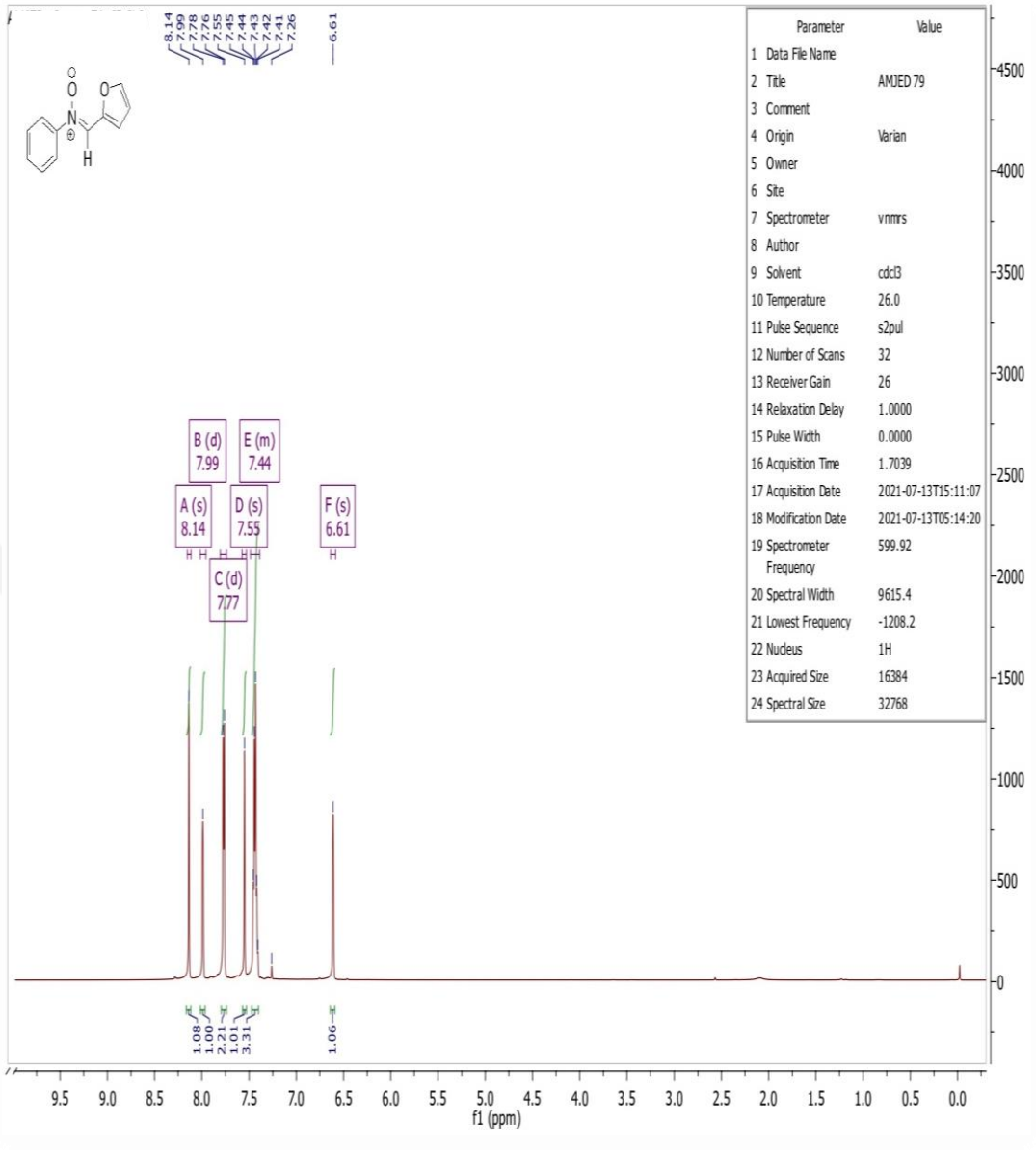
Geometric parameters (Å, º)

C1—C2	1.380 (3)	C23—C24	1.372 (3)
C1—C6	1.385 (2)	C23—H23	0.9300
C1—H1	0.9300	C24—C25	1.375 (3)
C2—C3	1.368 (3)	C24—H24	0.9300
C2—H2	0.9300	C25—C26	1.369 (3)
C3—C4	1.376 (3)	C25—H25	0.9300

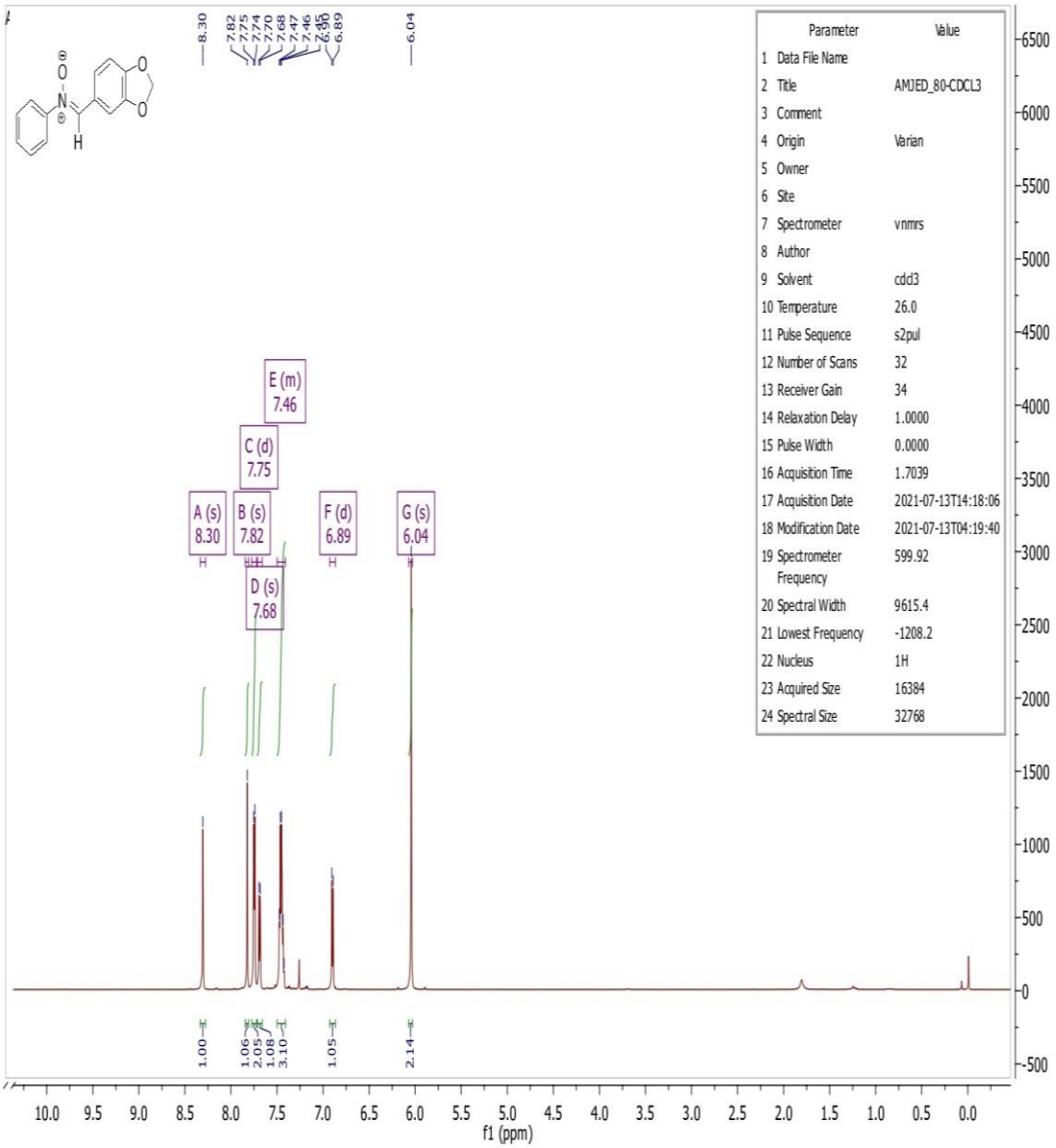
APPENDIX 2. ^{1}H NMR Spectra for the compounds 9 and 53-63.



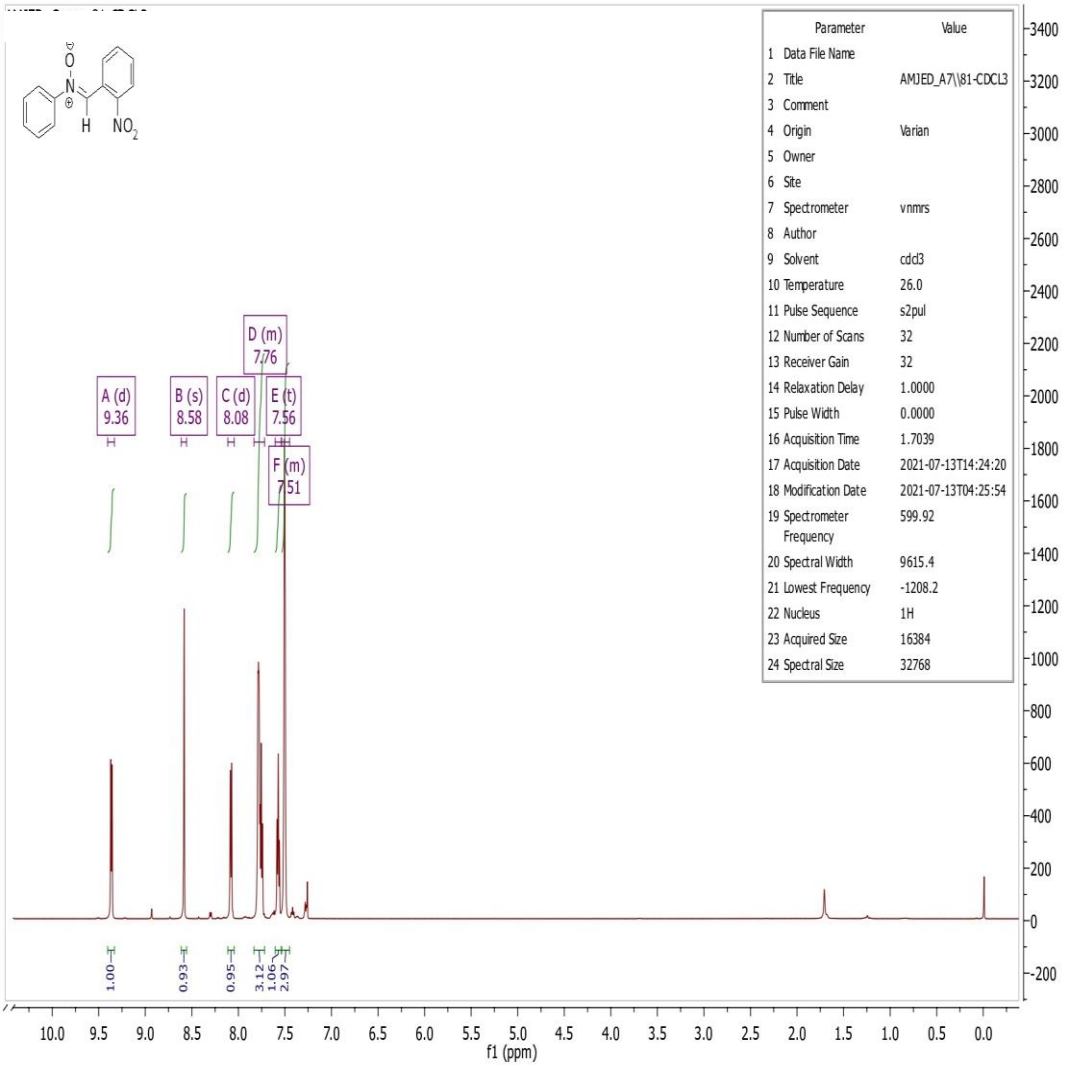
^{1}H NMR spectrum for the compound 9



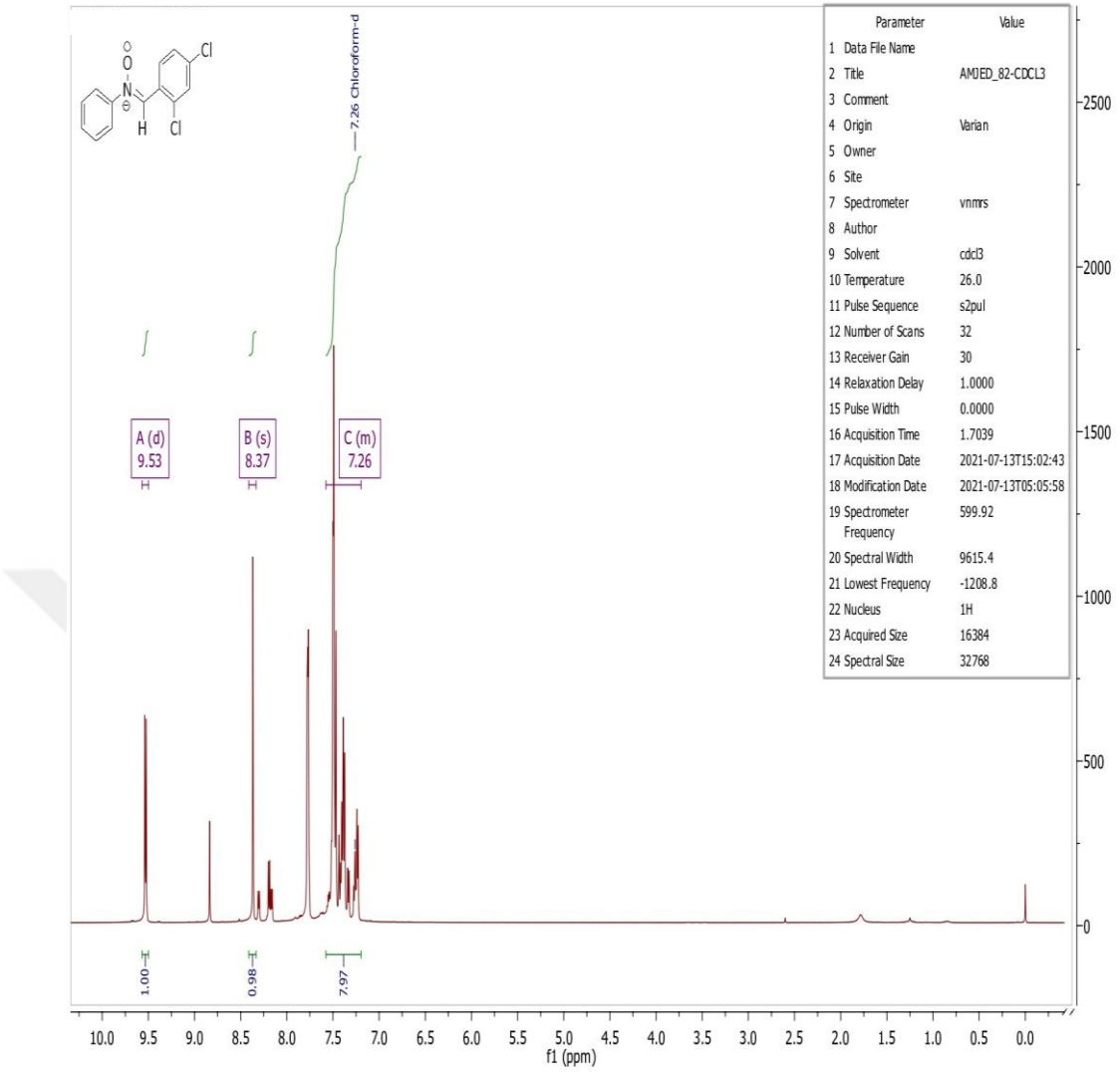
^{1}H NMR spectrum for the compound 53



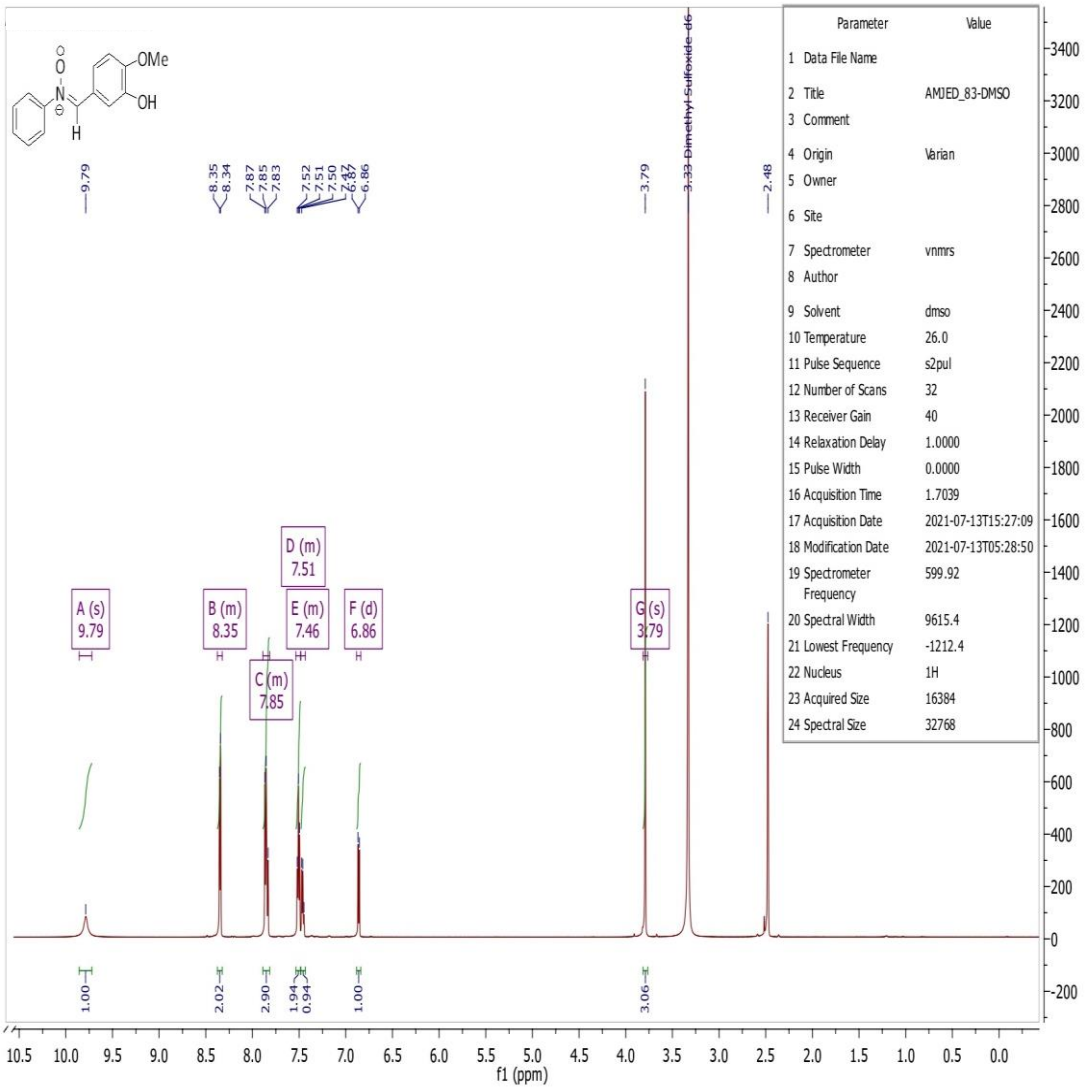
^{1}H NMR spectrum for the compound 54



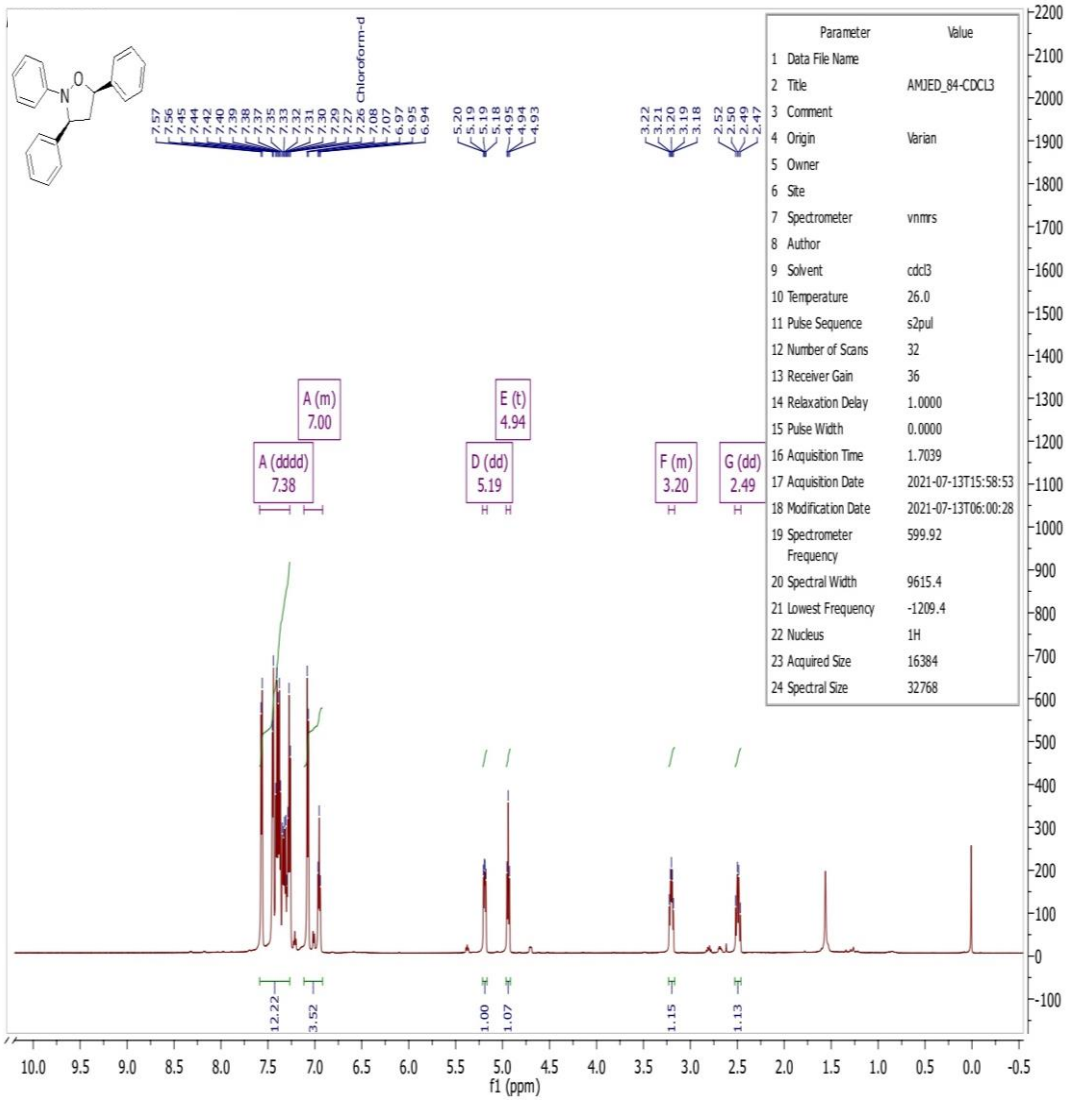
^{1}H NMR spectrum for the compound 55



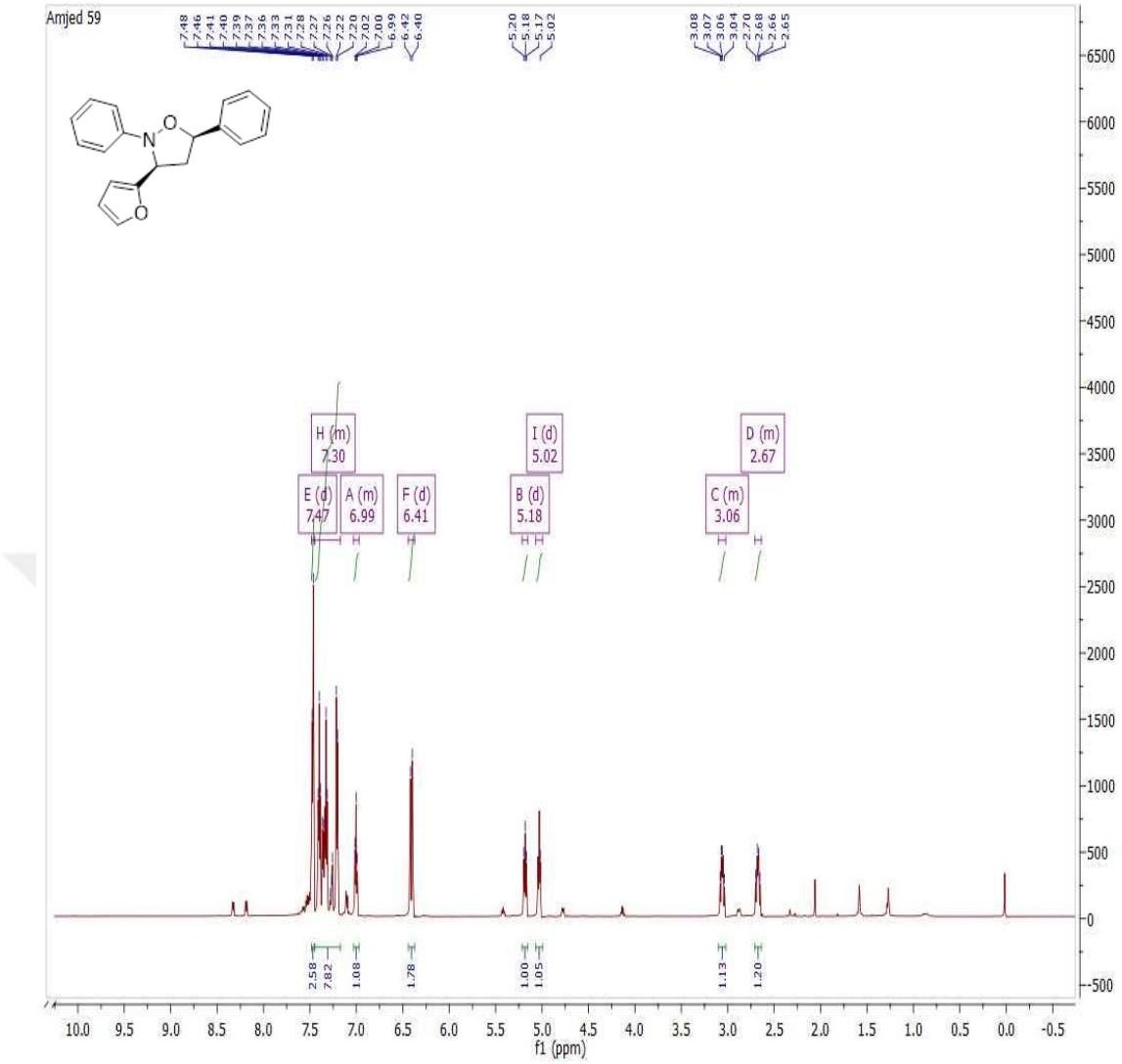
^{1}H NMR spectrum for the compound 56



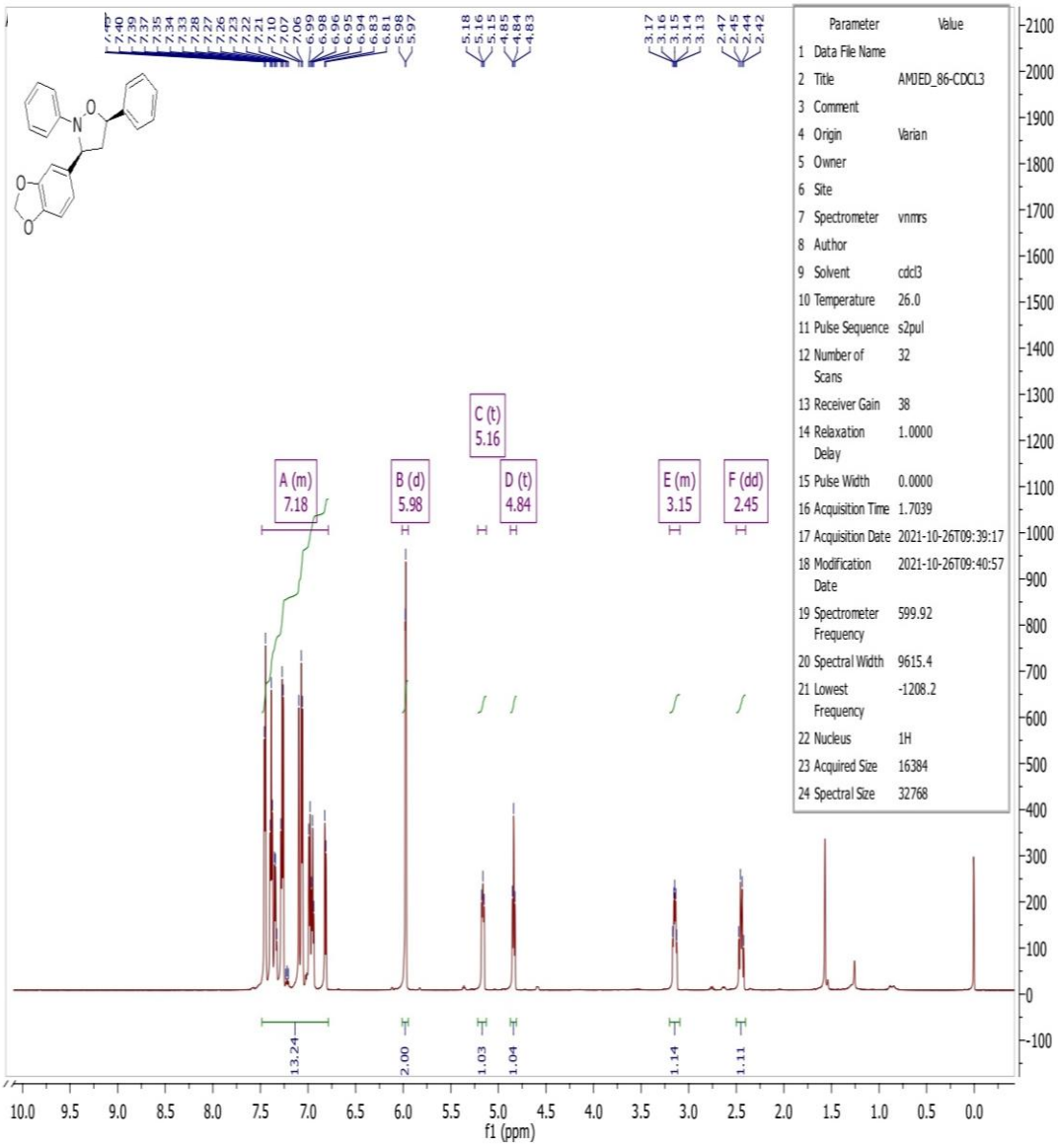
^{1}H NMR spectrum for the compound 57



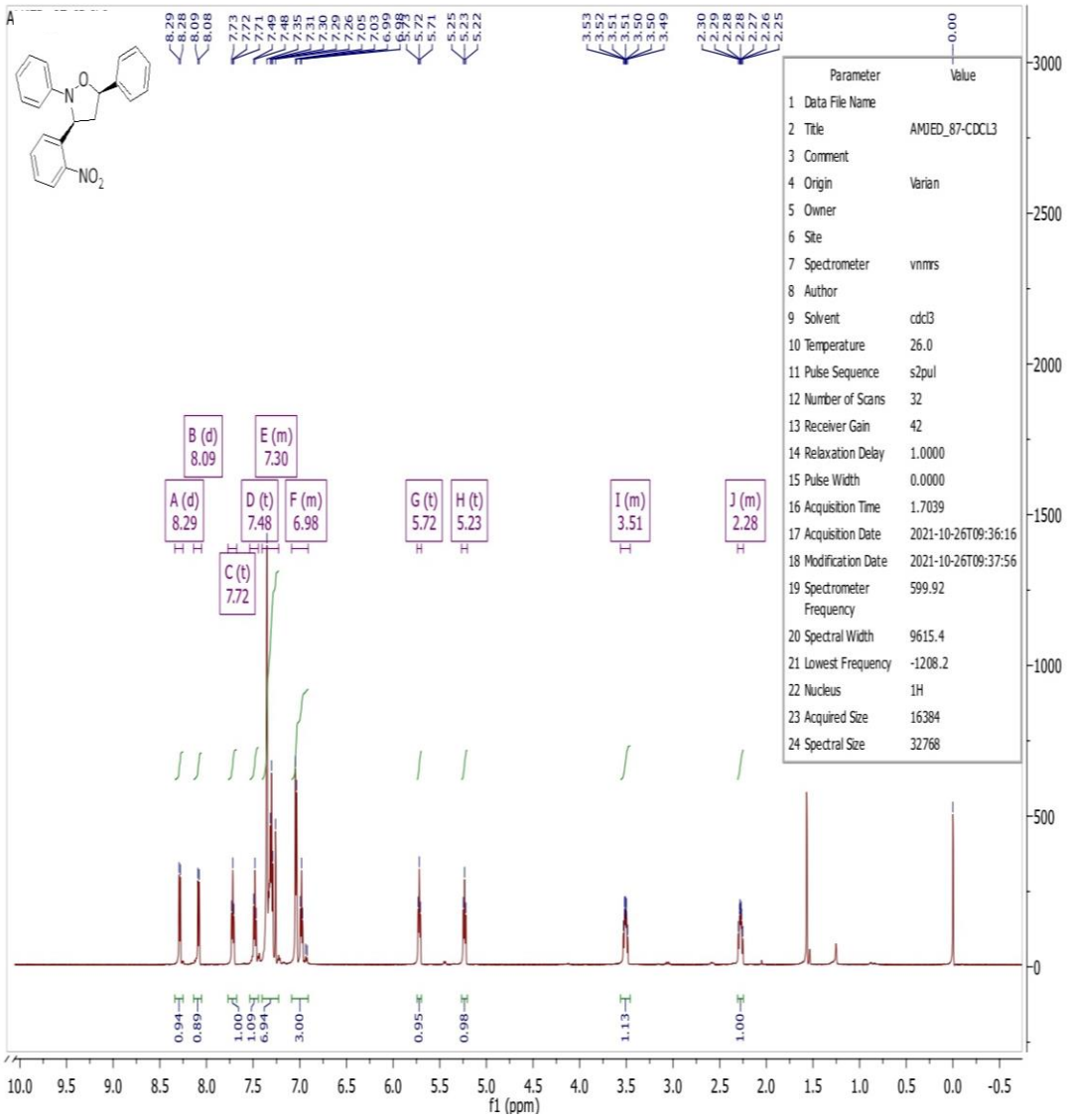
^{1}H NMR spectrum for the compound 58



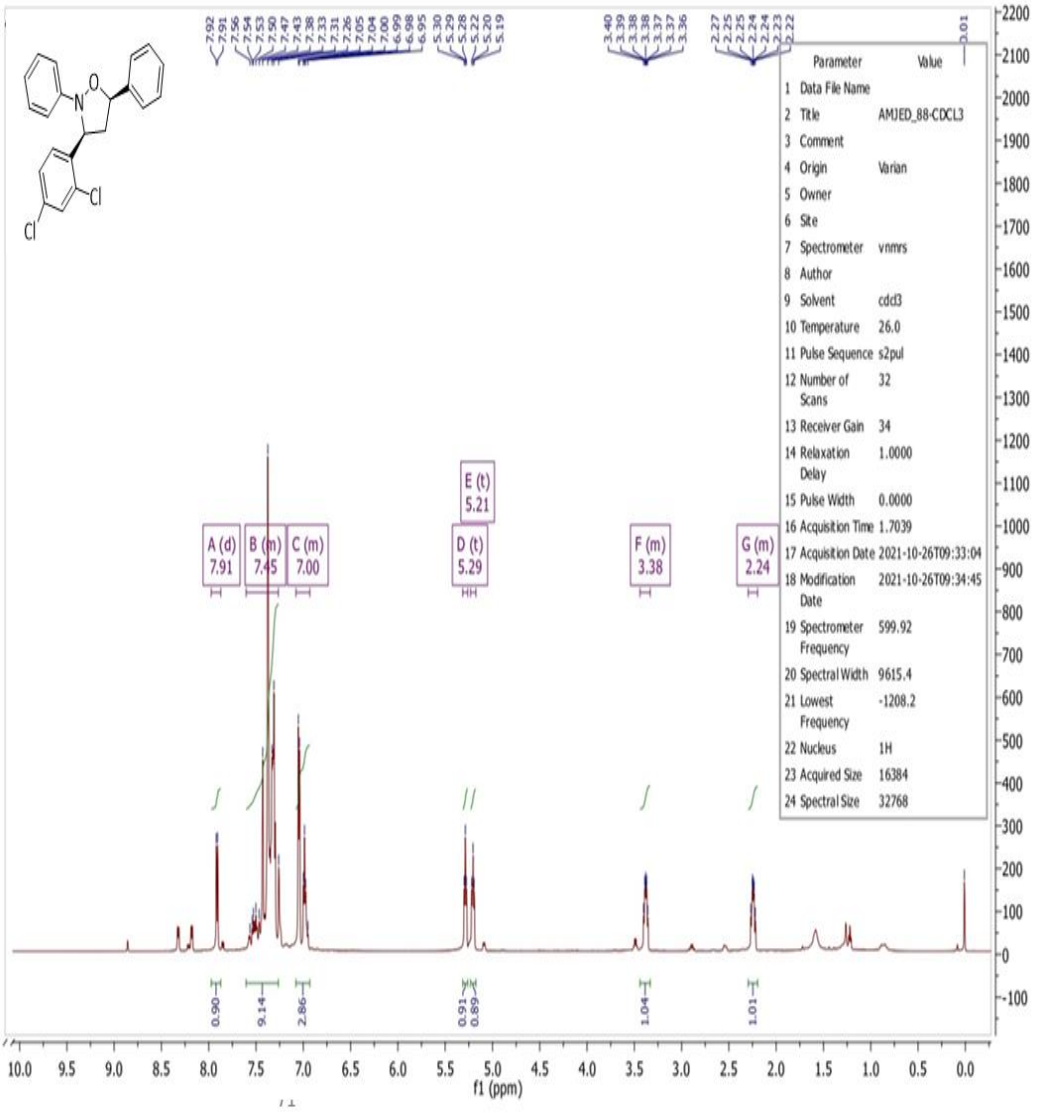
^{1}H NMR spectrum for the compound 59



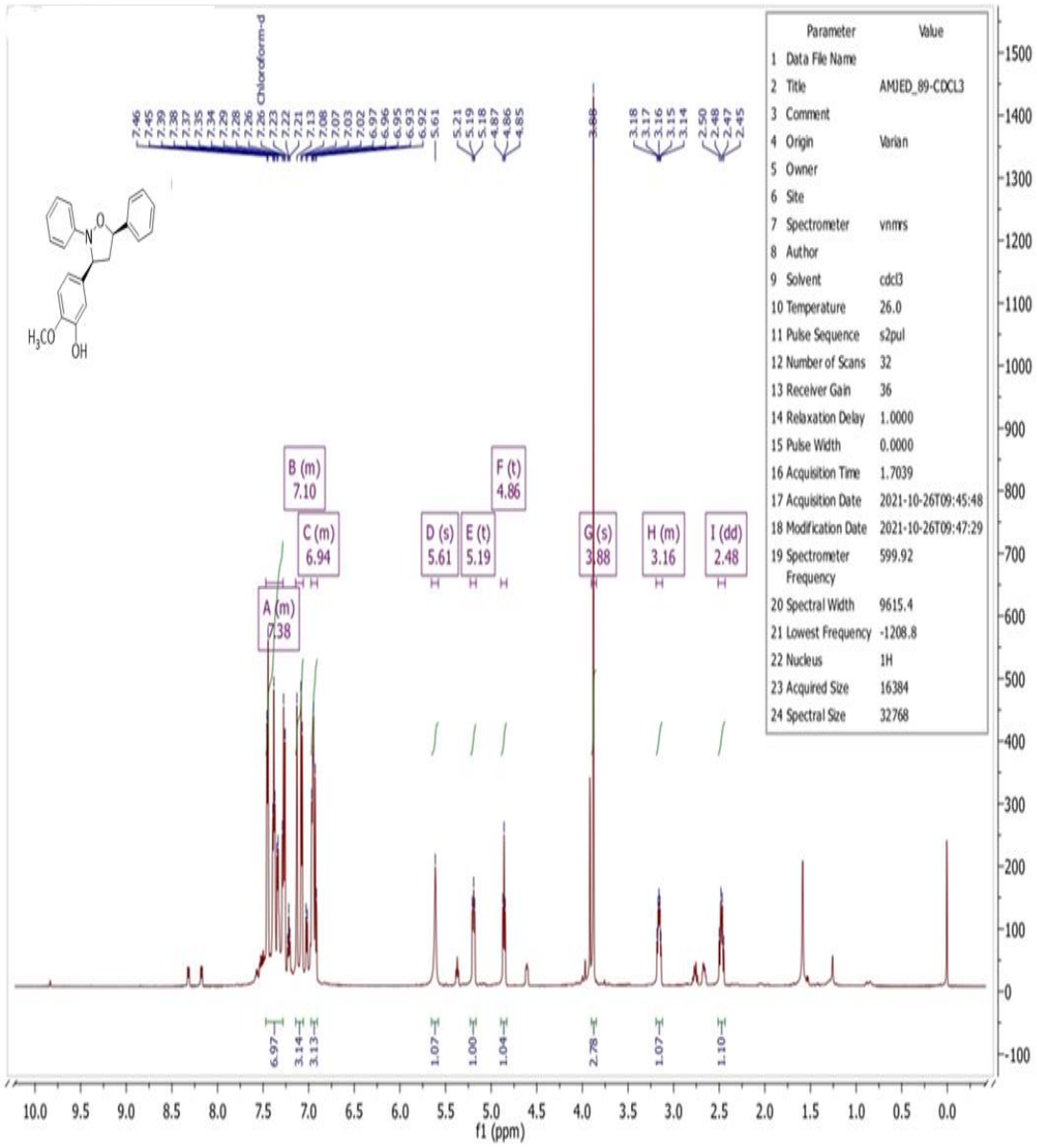
^{1}H NMR spectrum for the compound 60



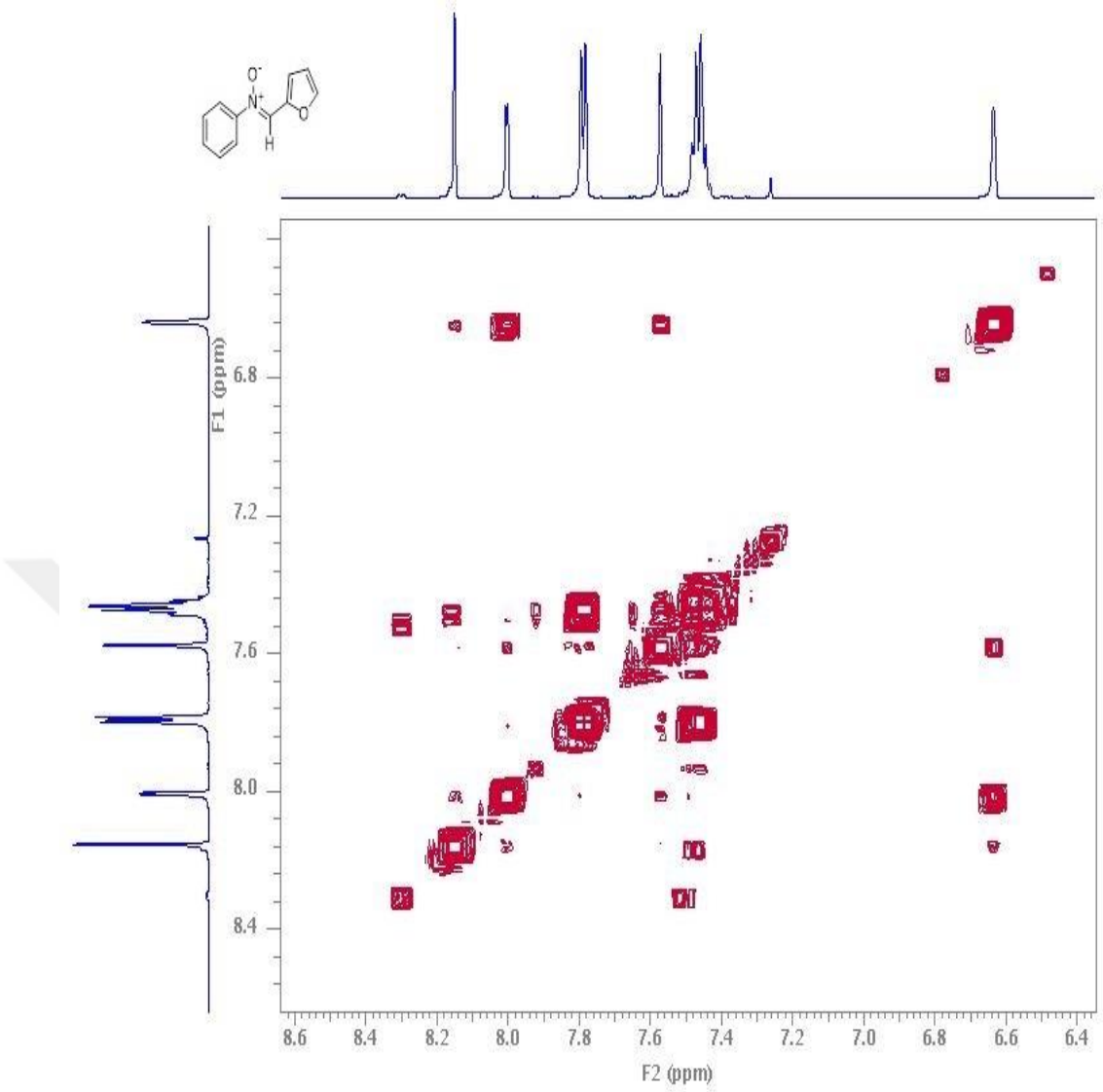
^{1}H NMR spectrum for the compound 61



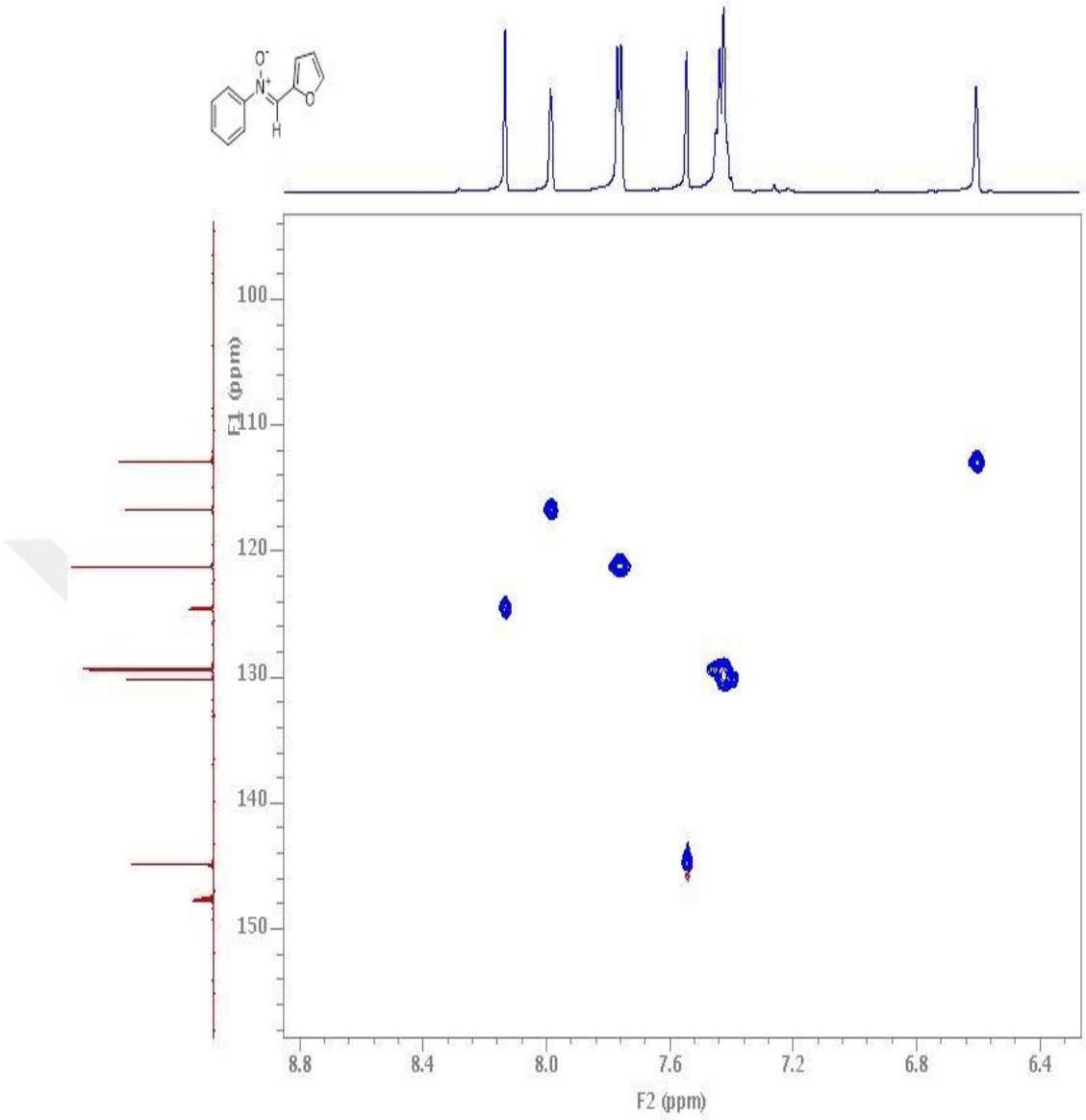
^{1}H NMR spectrum for the compound 62



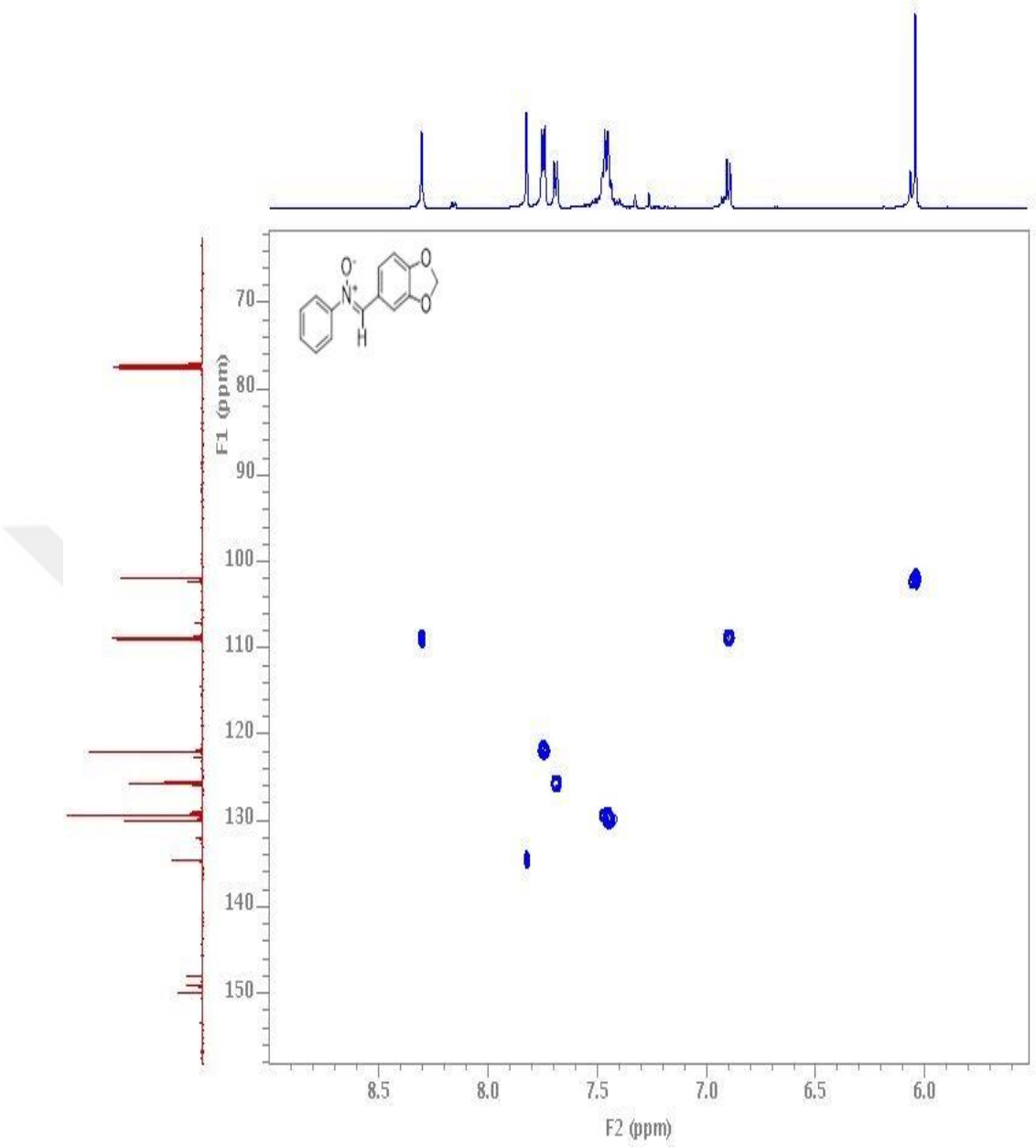
^{1}H NMR spectrum for the compound 63



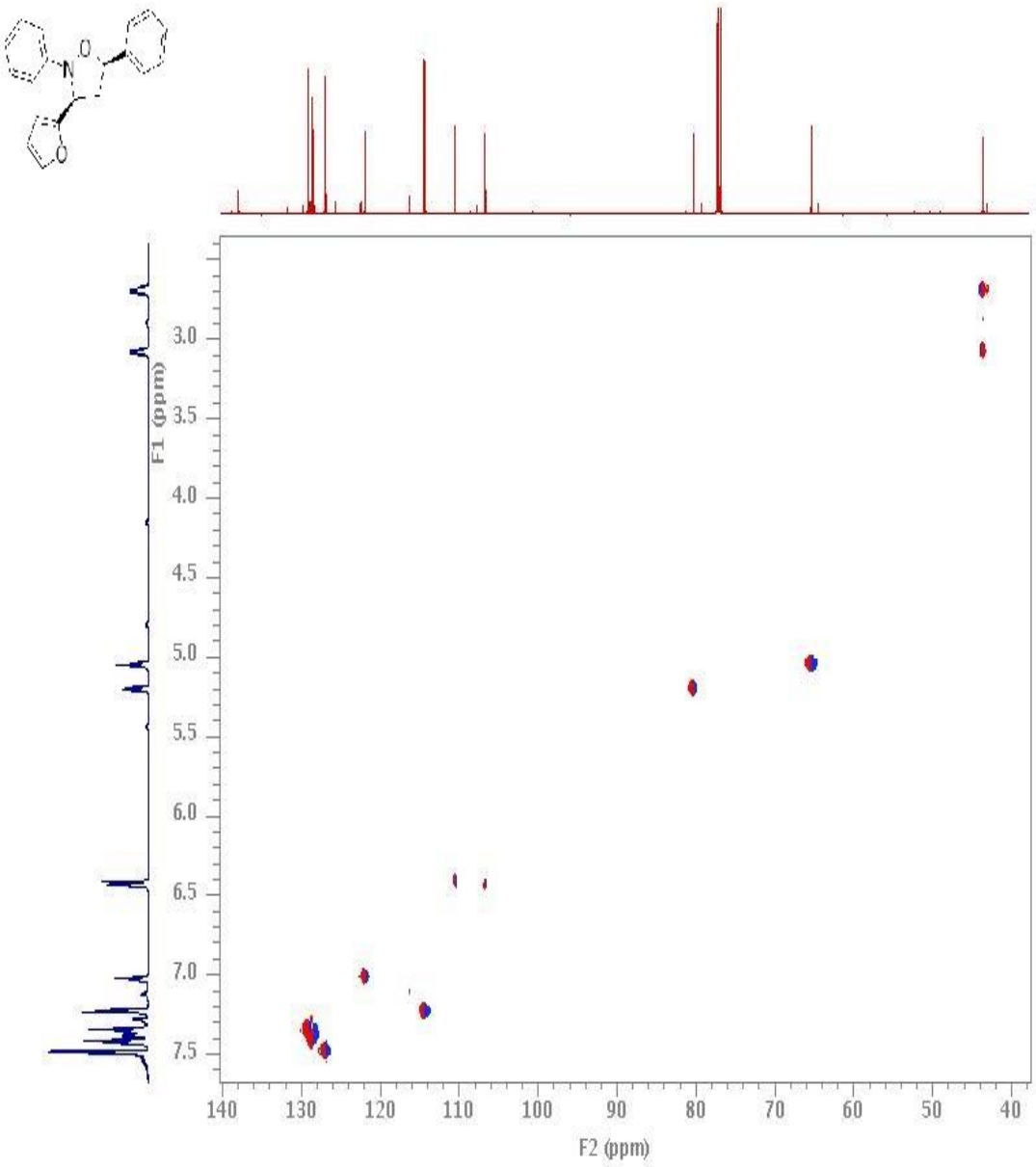
^{1}H NMR COSYspectrum for the compound 53



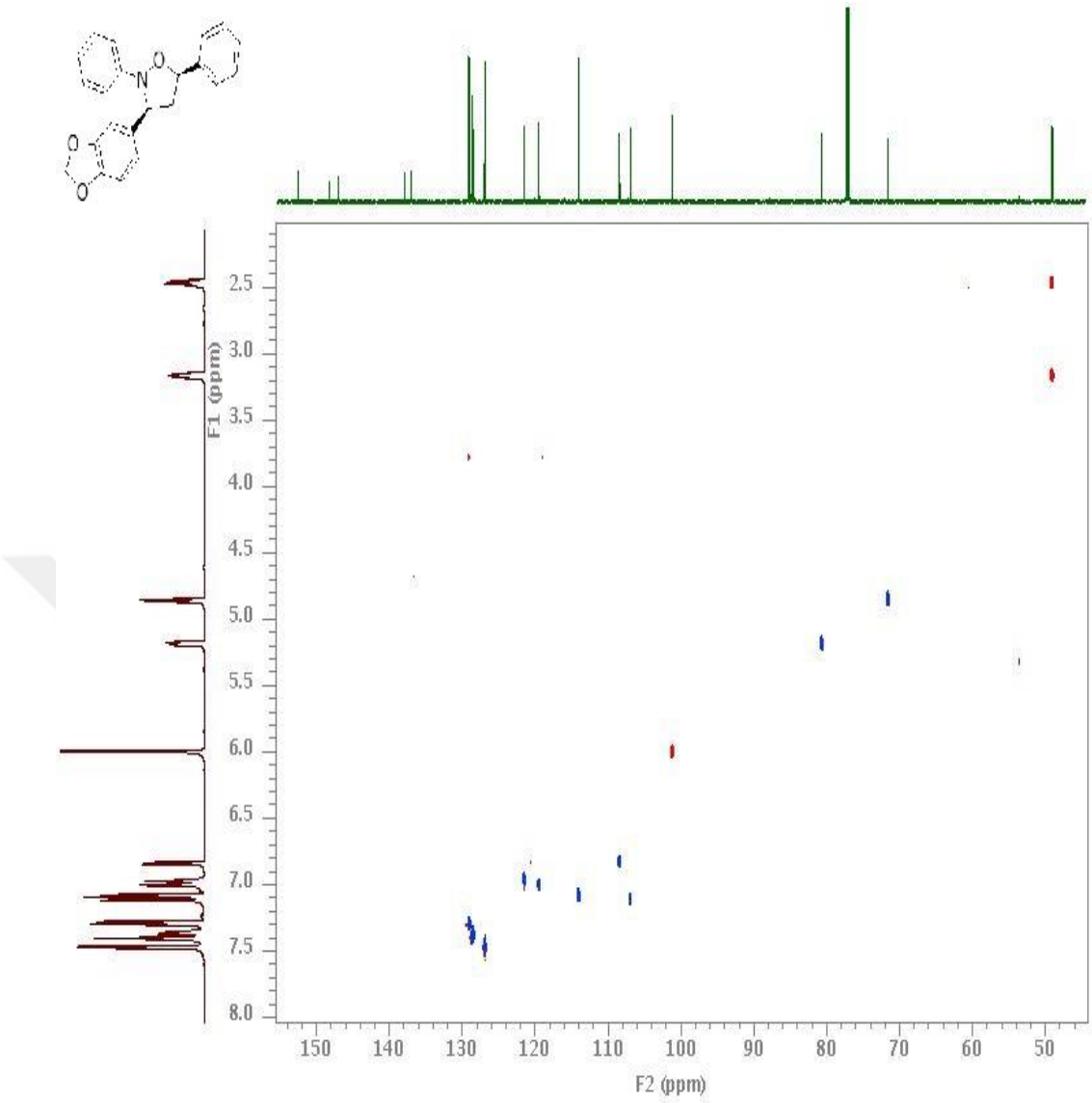
HSQC spectrum for the compound 53



HSQC spectrum for the compound 54

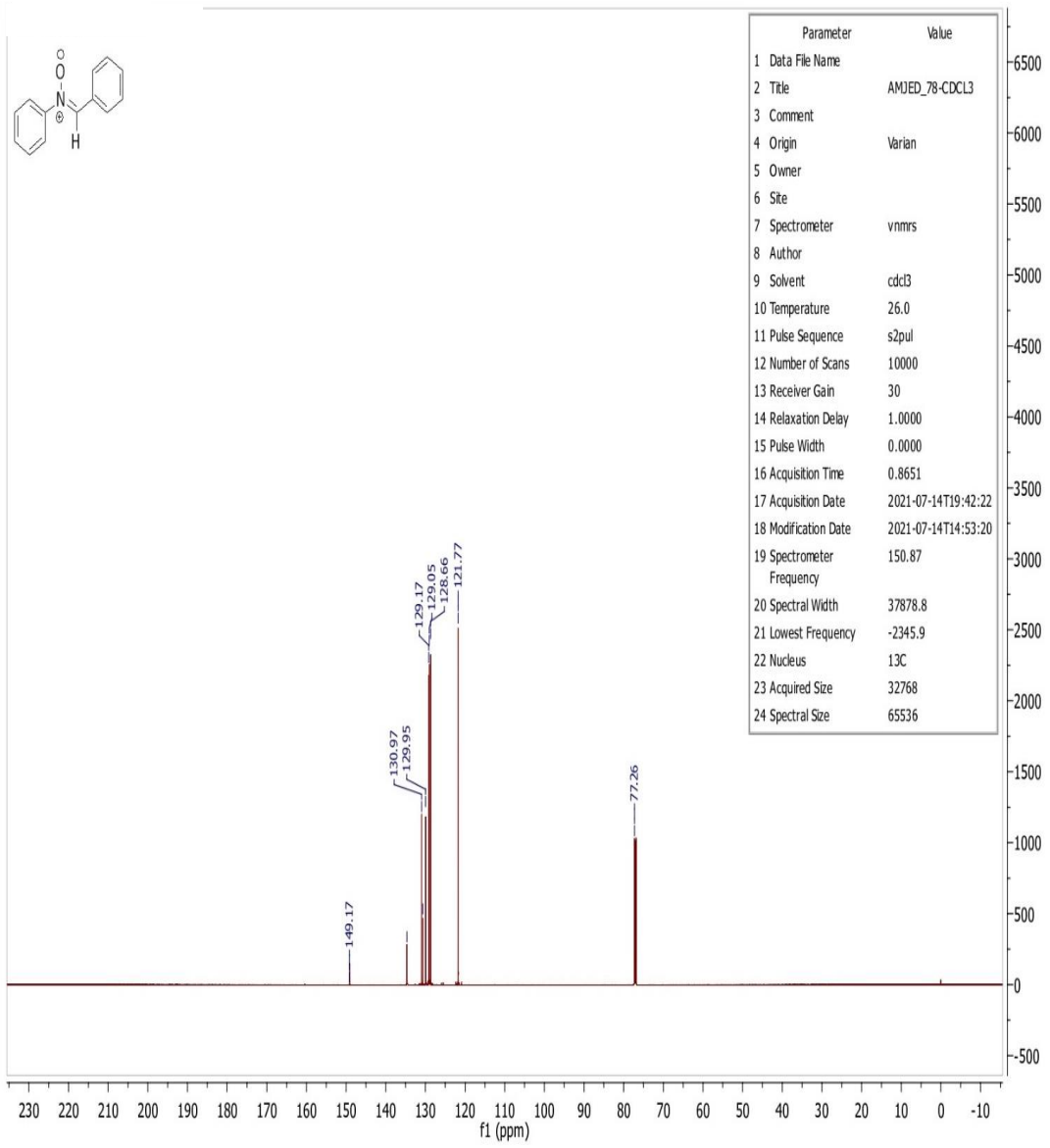


HETCOR spectrum for the compound 59

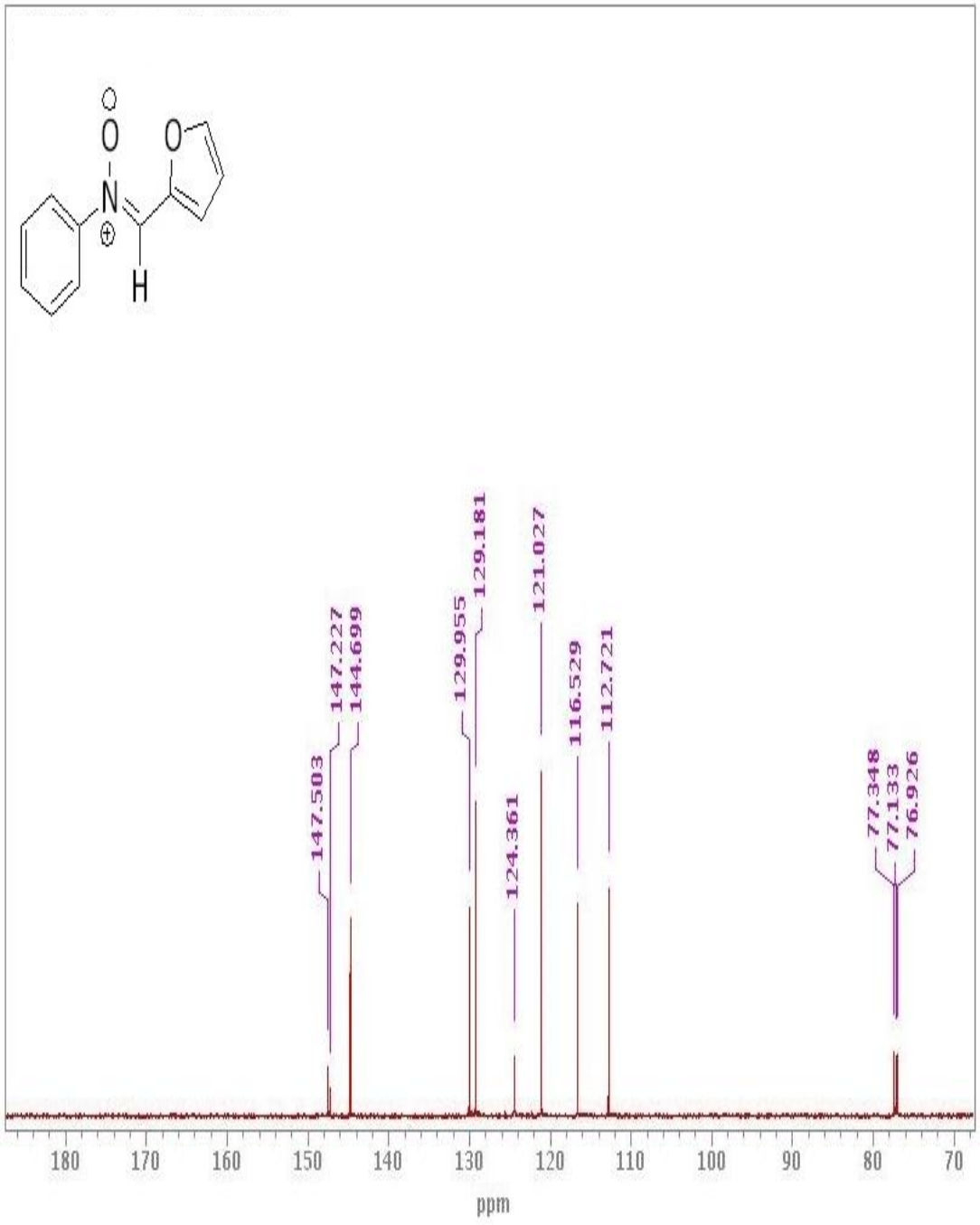


HETCOR spectrum for the compound 60

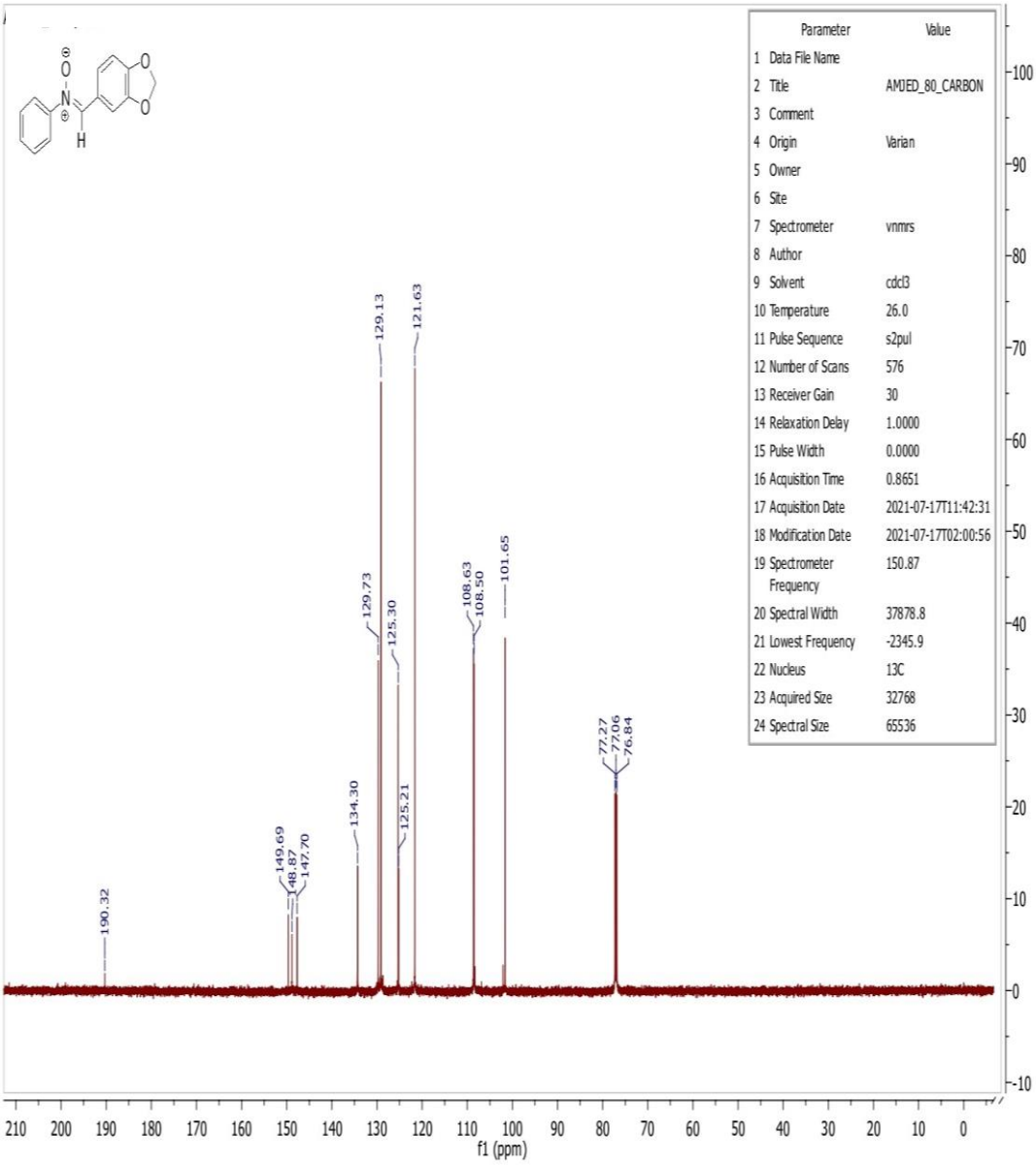
APPENDIX 3. ^{13}C NMR spectra for the compounds 9 and 54-64



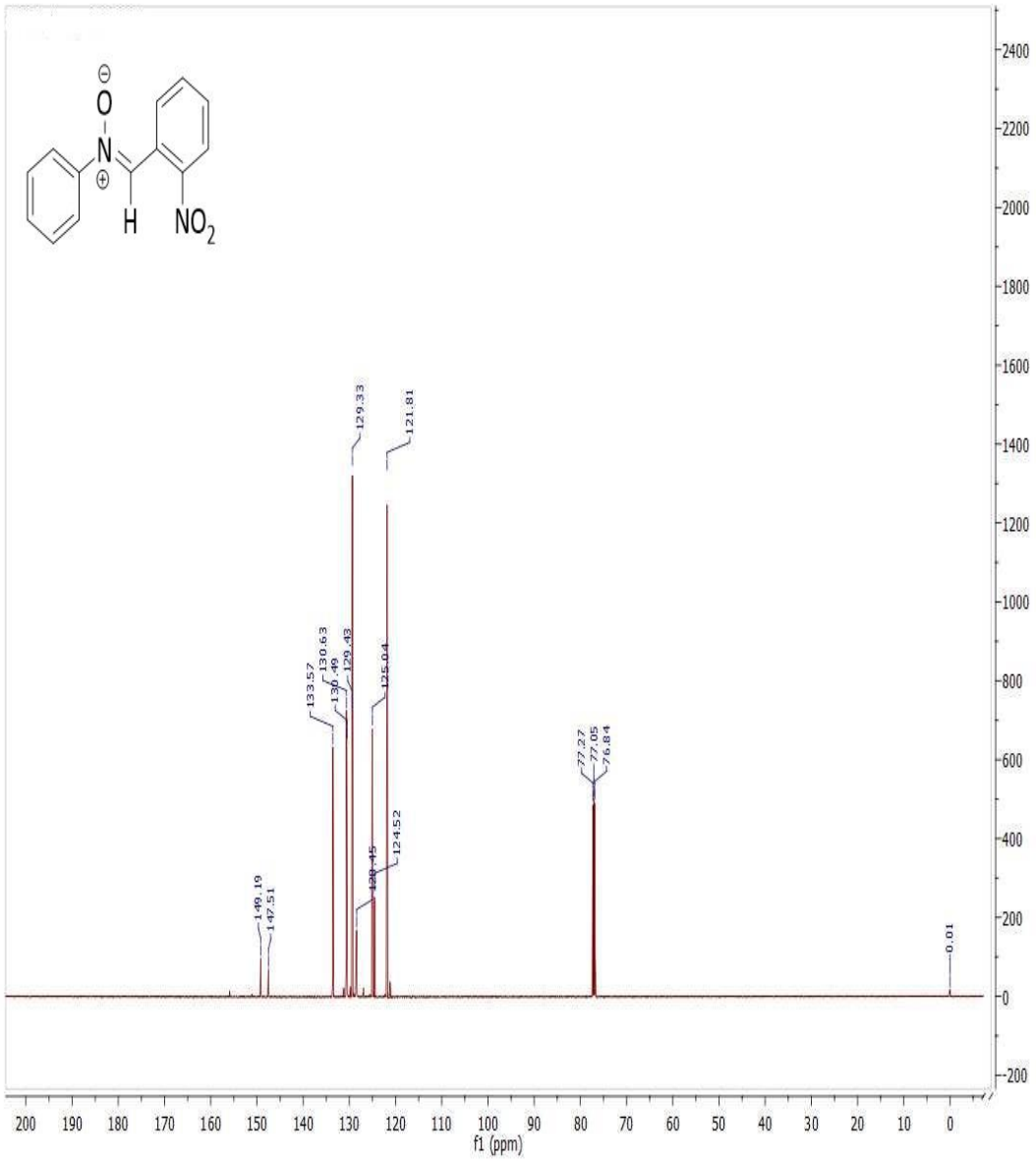
^{13}C NMR spectrum for the compound 9



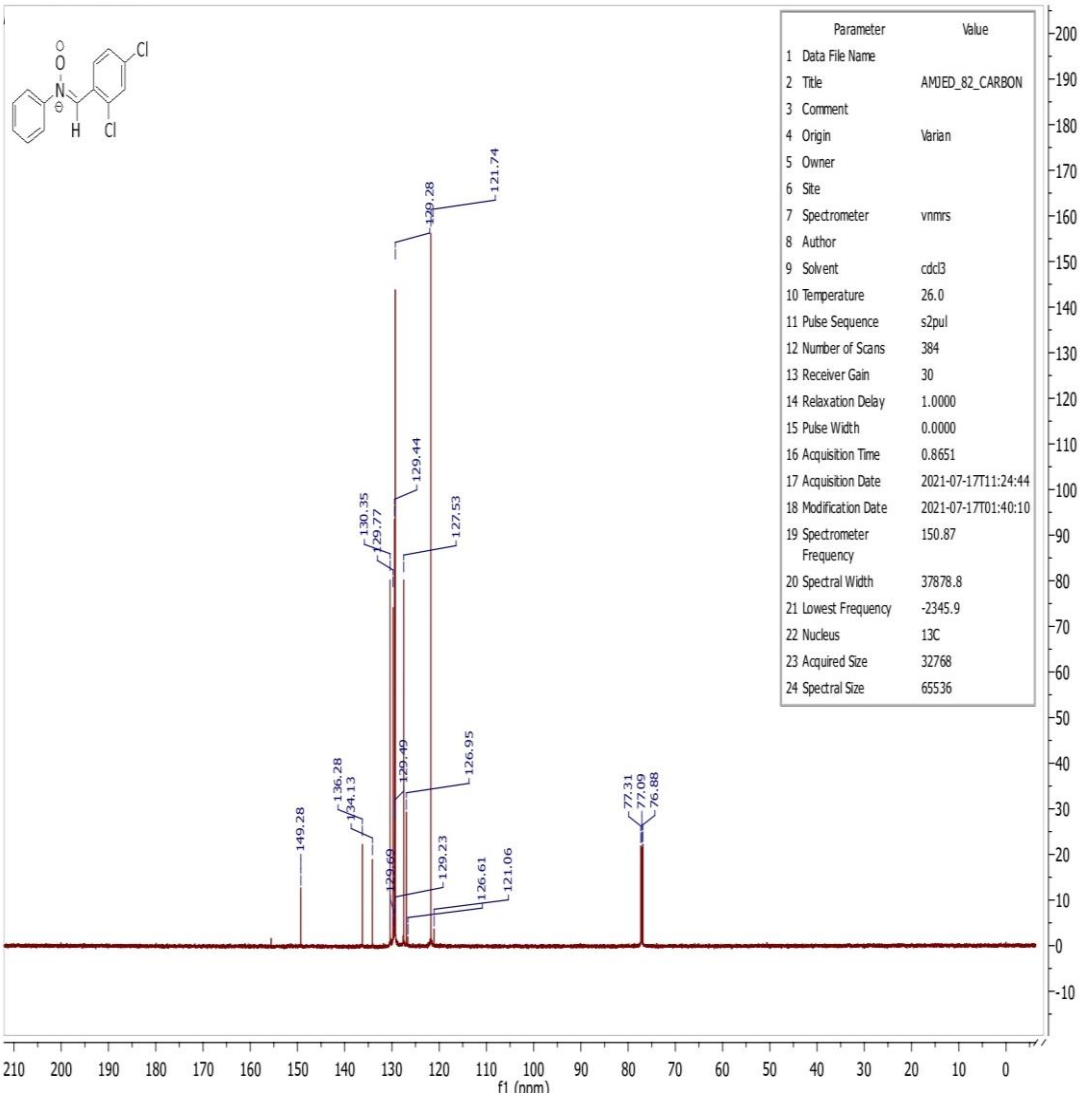
^{13}C NMRspectrum for the compound 53



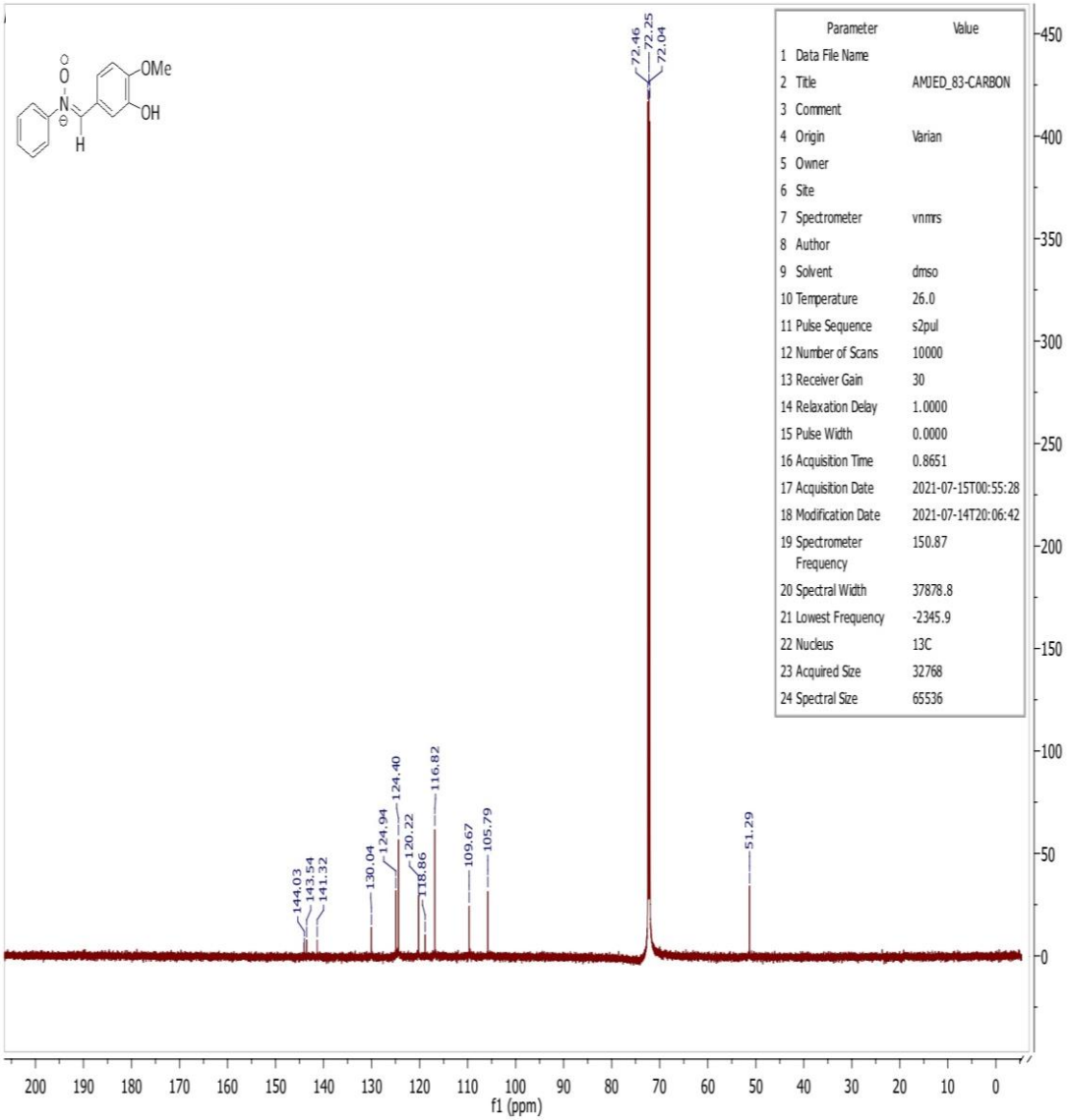
^{13}C NMR spectrum for the compound 54



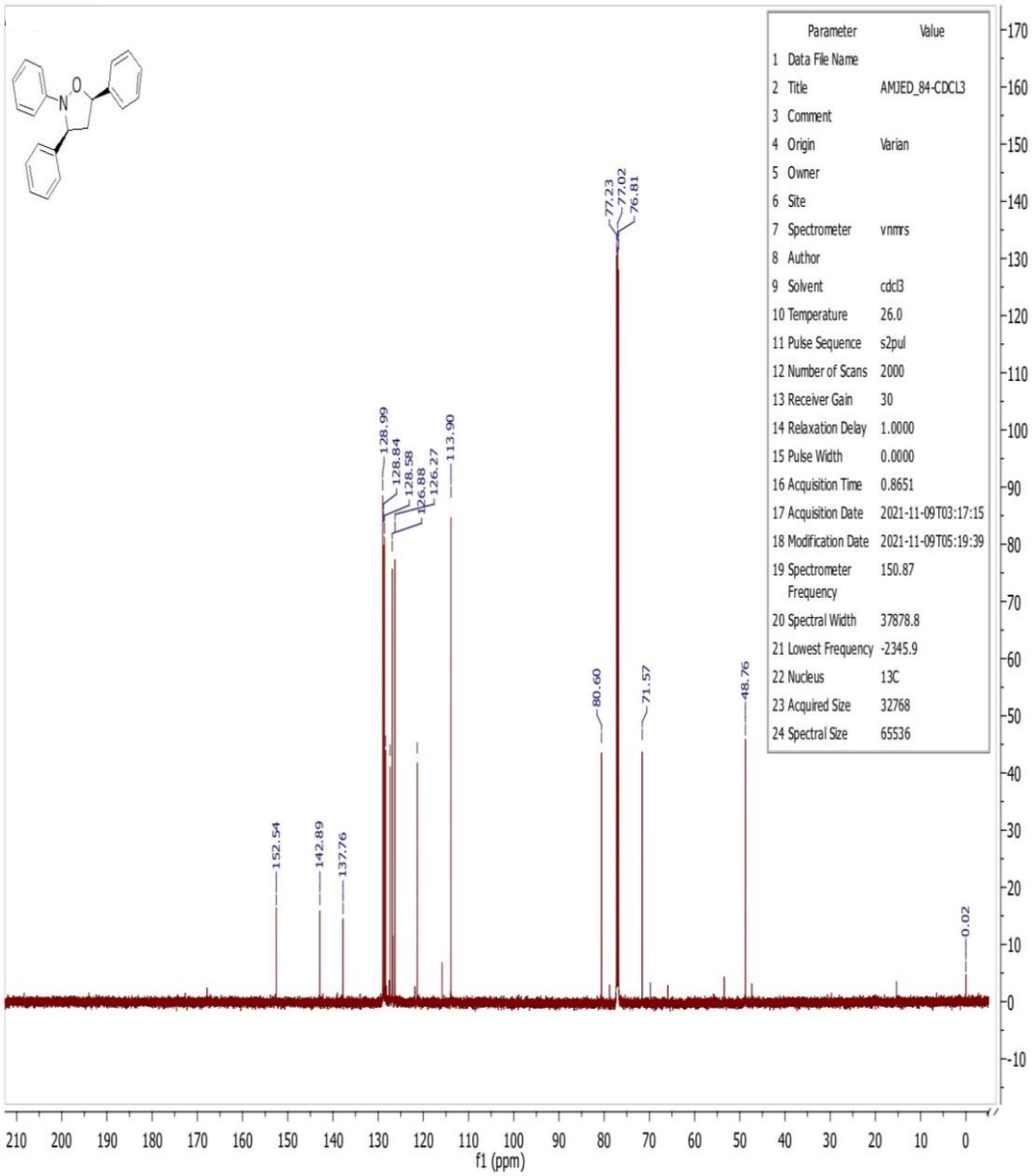
^{13}C NMR spectrum for the compound 55



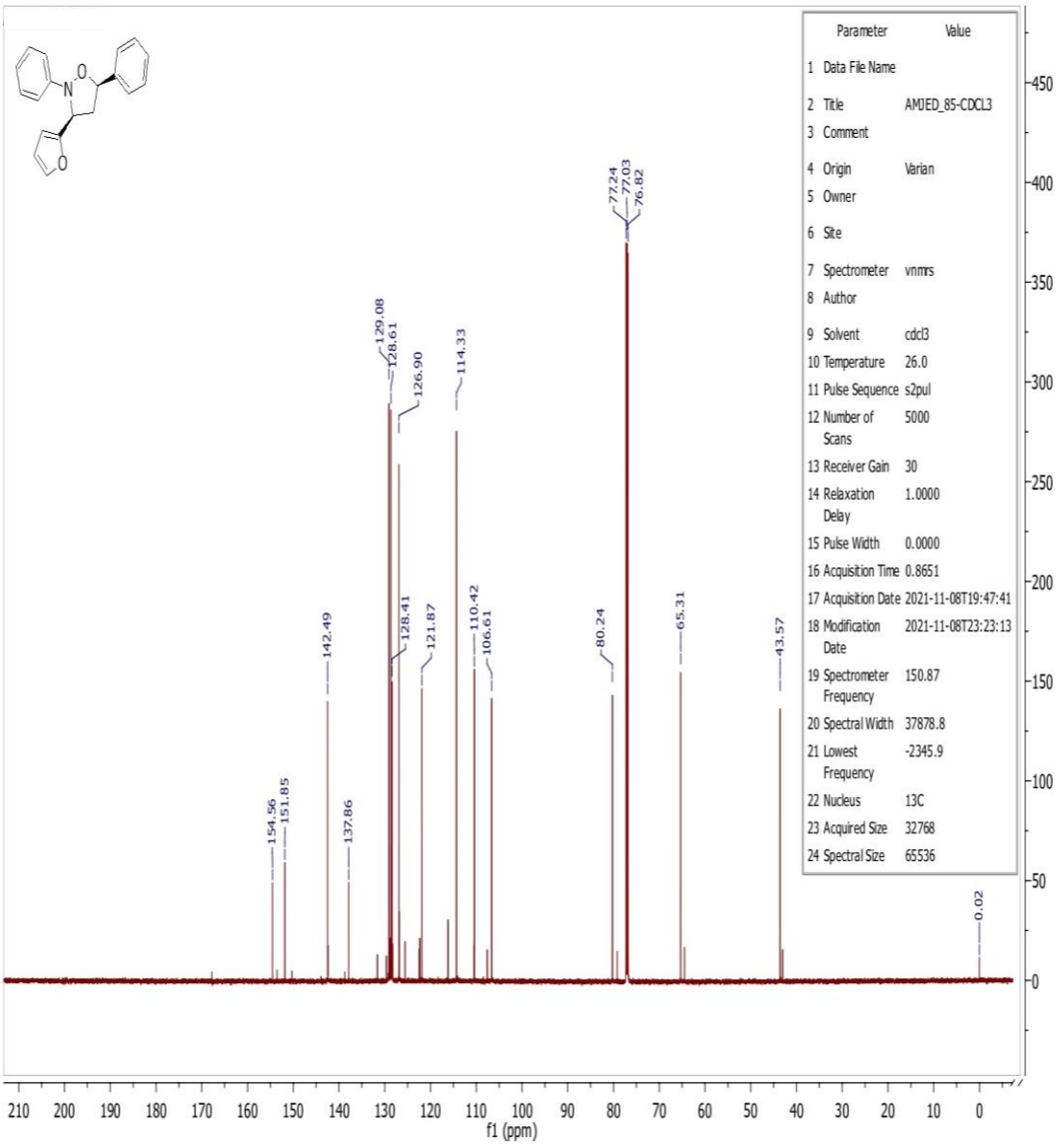
^{13}C NMR spectrum for the compound 56



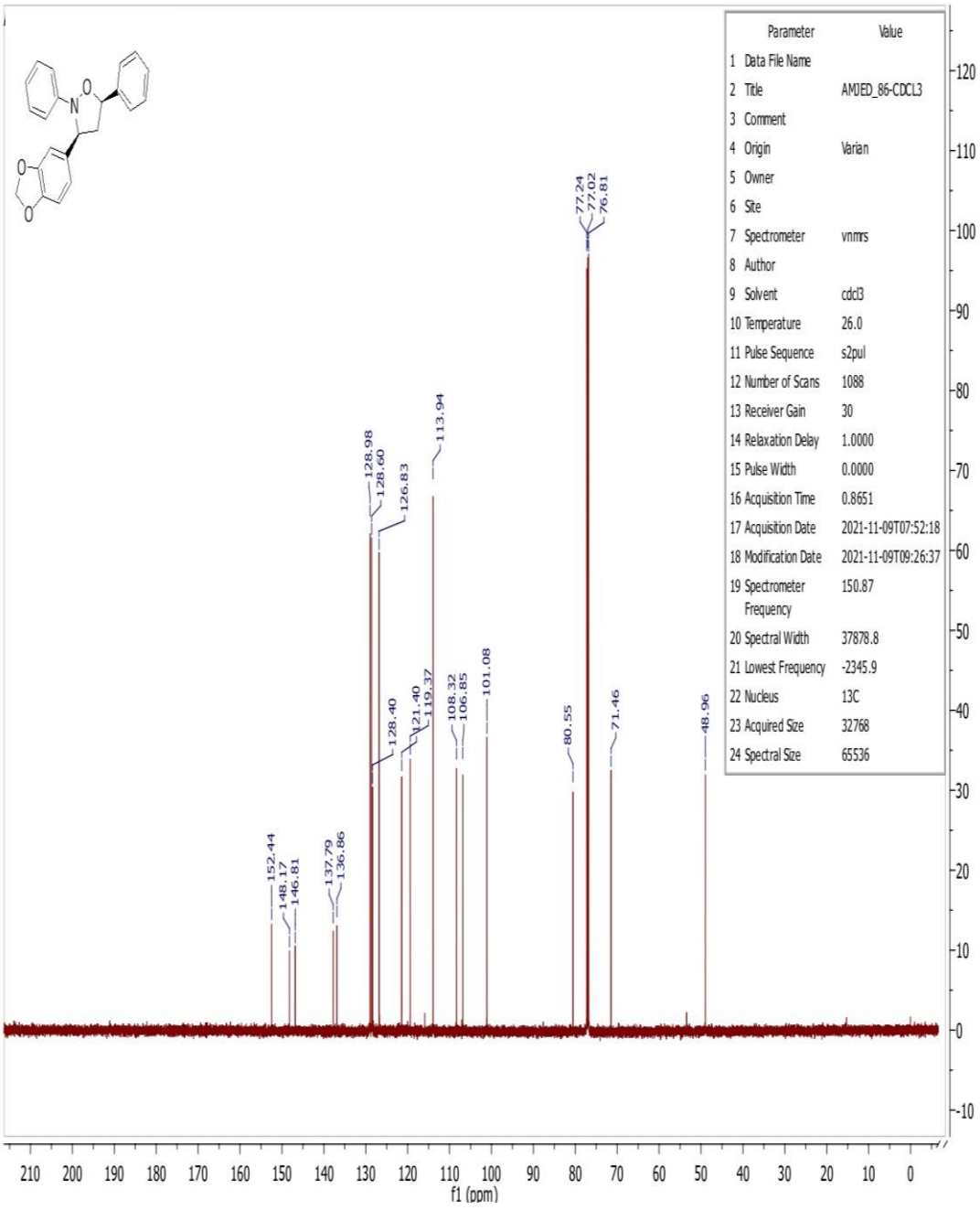
^{13}C NMR spectrum for the compound 57



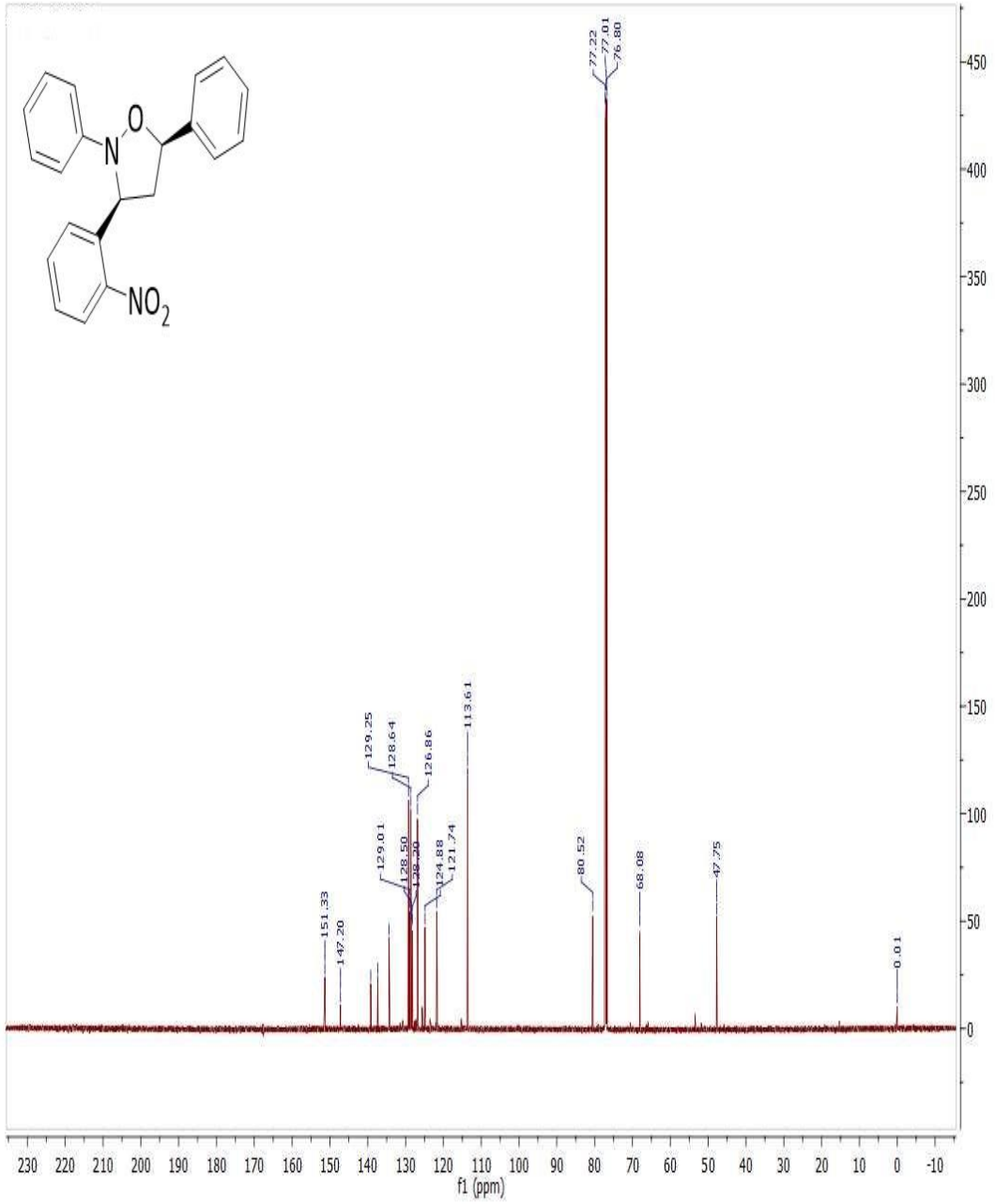
^{13}C NMR spectrum for the compound 58



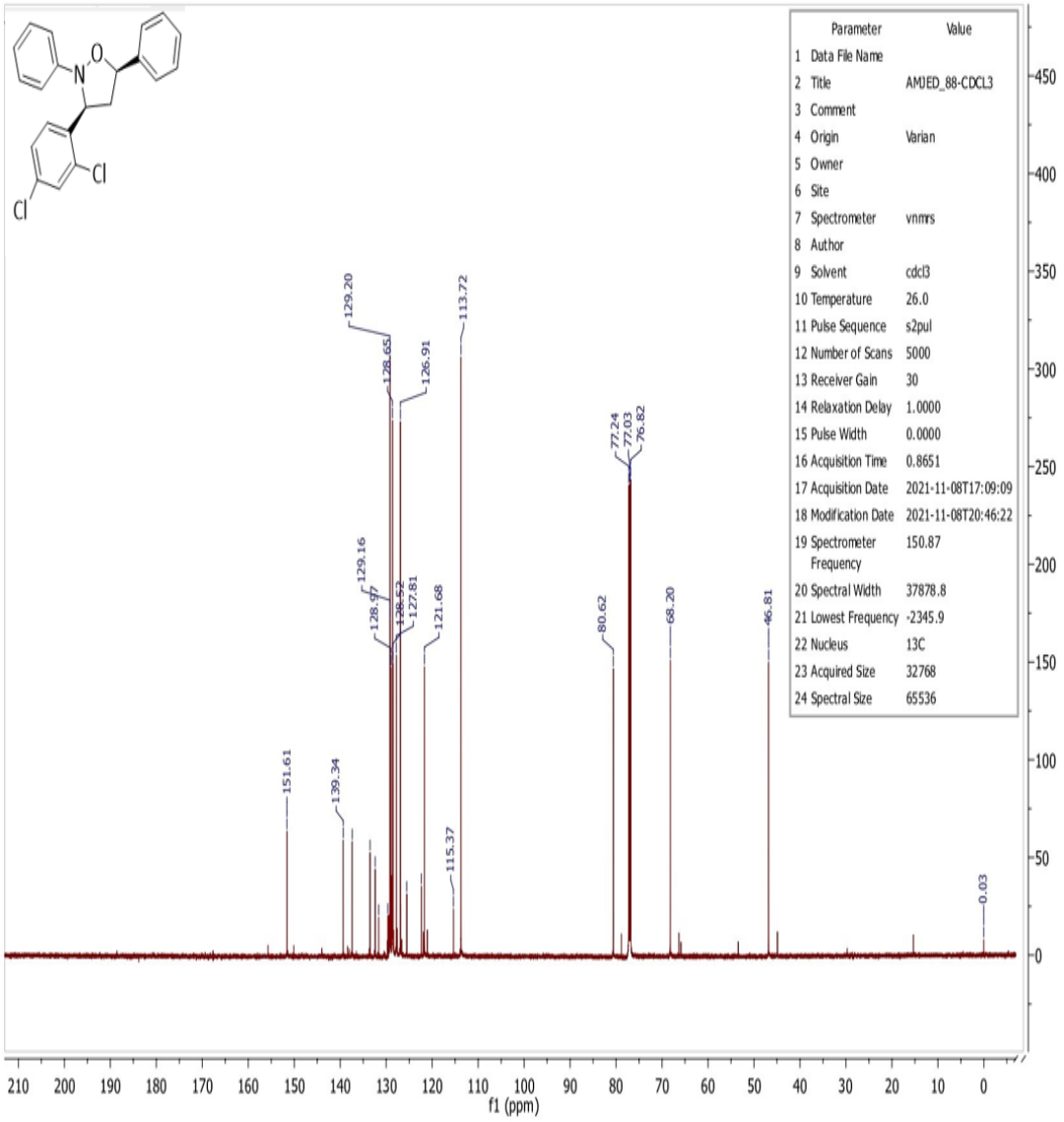
^{13}C NMR spectrum for the compound 59



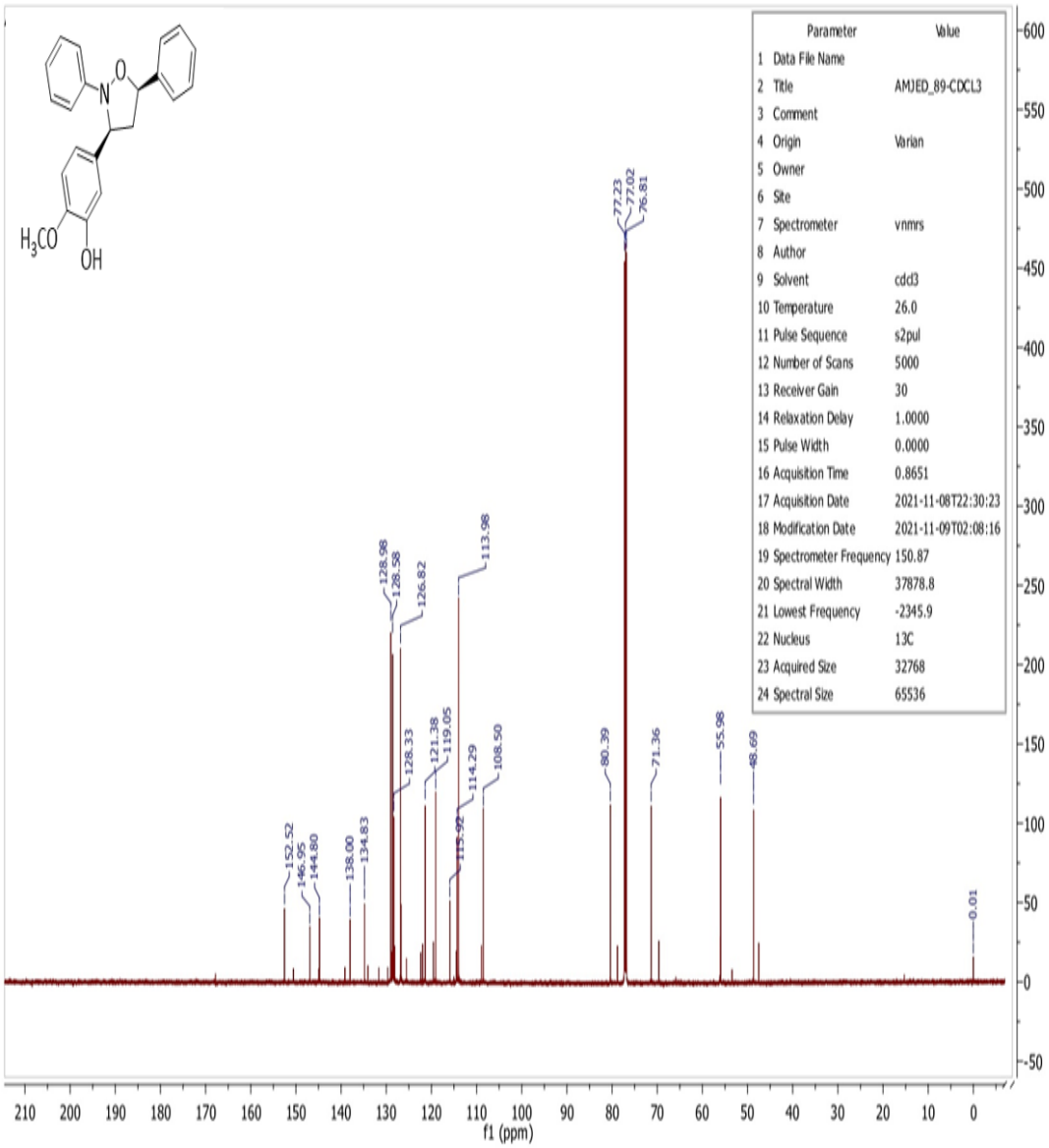
^{13}C NMR spectrum for the compound 60



^{13}C NMR spectrum for the compound 61



^{13}C NMR spectrum for the compound 62



^{13}C NMR spectrum for the compound 63

CURRICULUM VITAE

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Undergraduate	Tikrit University College of Science Department of Chemistry	1998-2003

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2007- Present	Ministry of Oil-North Refineries Company, Department of the Environment	Chemical

Academic Activities

Not currently available