Kingdom of Saudi Arabia
lmam Muhammad ibn Saud lslamic University
College of Science
Department of Chemistry

Synthesis and Characterisation of

1-(Aryldiazenyl)naphthalen-2-ol

A graduation Research Project

Submitted to the Department of Chemistry in partial fulfillment of the requirements for the completion of the degree of Bachelor of Science in Chemistry

BY

Ruba Faisal AL-harbi 442013676

Alanoud Mogeb AL-qahtani 442013240

Razan Nawaf AL-harbi 442013274

Aisha ghazi al-shammari 441023021

Under Supervision

Dr.Amal Ali AL-muatiri

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Abstract

1-(Aryldiazenyl) naphthalene-2-ol was synthesised via conventional diazo-coupling of aniline derivatives and 2-napthol, yielding approximately 2.5% and 39% respectively. The 1-(aryldiazenyl) naphthalene-2-ol structures were confirmed by FT-IR spectroscopy

الملخص:

في هذا المشروع، تم تحضير ١-(أريل ديازينيل) نفثالين-٢-أول من خلال عملية اقتران ثناي الأزو التقليدية بين مشتقات الأنيلين و٢-نافثول، مما أدى إلى إنتاج ما يقارب من ٣٩% و من المنتج. تم تأكيد بنية ١-(أريل ديازينيل) نفثالين-٢-أول بواسطة **%** ٢,٥ مطيافية الأشعة تحت الحمراء.

List of Abbreviations

Celsius	**°C**
Centimetre -1	**cm^{-1}**
Dimethylformamide	**DMF**
Fourier transform infrared spectroscopy	**FTIR**
Milliliter	**Ml**
Melting point	**M.P.**
Tetramethylsilane	**TMS**
Potassium Bromide	**KBr**
Percent sign	**%**
Sodium hydroxide	**NaOH**
Tetrahydrofuran	**THF**

1. Introduction

Azo dyes are organic compounds that possess an N=N functional group and absorb in the UV-visible and infrared spectrum (Figure 1) [1, 2]. They are commonly used in the plastic industry as a colourant. [3]

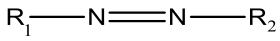
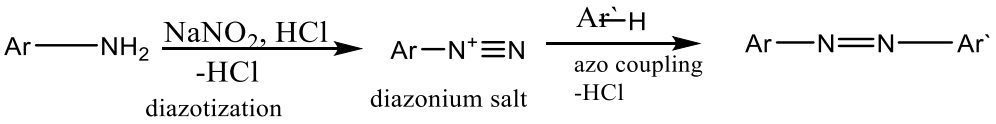


Figure 1: Structurer of Azo compounds

Azo dyes are typically produced through a conventional process that involves converting an aromatic amine or heterocyclic amine into a diazonium salt. This reaction typically occurs at low temperatures in the presence of salts and acids, followed by the reaction of the diazonium complex with nucleophilic coupling agents like phenol, naphthol, or amines, as shown in Scheme 1. [4-5]



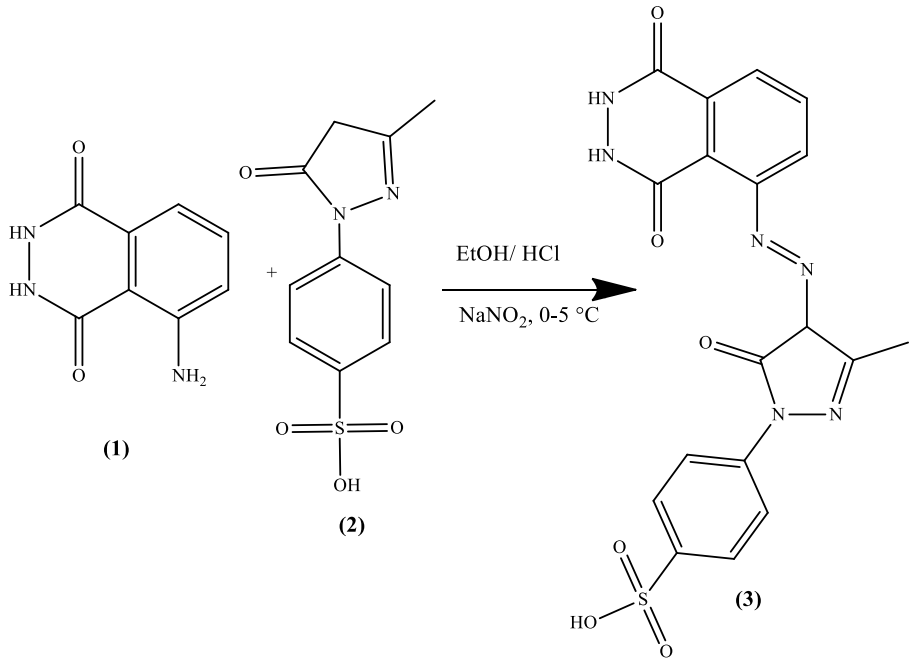
Scheme 1: Diazotization and azo coupling

These dyes exhibit excellent colour fastness to light, temperature, washing, and other factors, making them popular in the textile industry for dyeing fabrics such as silk [6],cotton [7], polyester [8], wool and nylon [9],Additionally, azo dyes are used as food colourants and preservatives to enhance the visual appeal and preserve the flavour of food .[10] They also have applications in electroplating, cosmetics, nonlinear optical devices, molecular switches, sensors, fluorescent devices, dye-sensitized solar cells (DSSCs), and anticorrosive materials. Furthermore, azo dyes have shown significant potential in biological fields demonstrating antibacterial, antifungal, anticancer, antituberculosis, antiproliferative, and antioxidant activities. [11]

2. Synthesis of azo dye derivatives

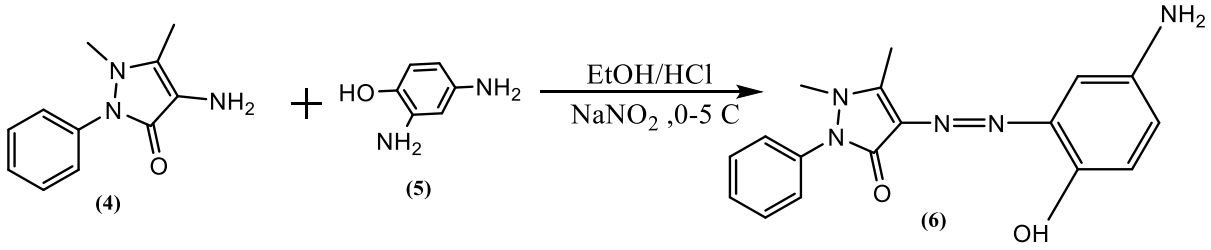
2.1 Azo dyes containing pyrazolone

The synthesis of a new azo dye **(3)** by Taha *et al.* involved the reaction of luminol **(1)** with 1-(4-sulfophenyl)-3-methyl-5-pyrazolone **(2)** [12]:



(1) (2) (3)

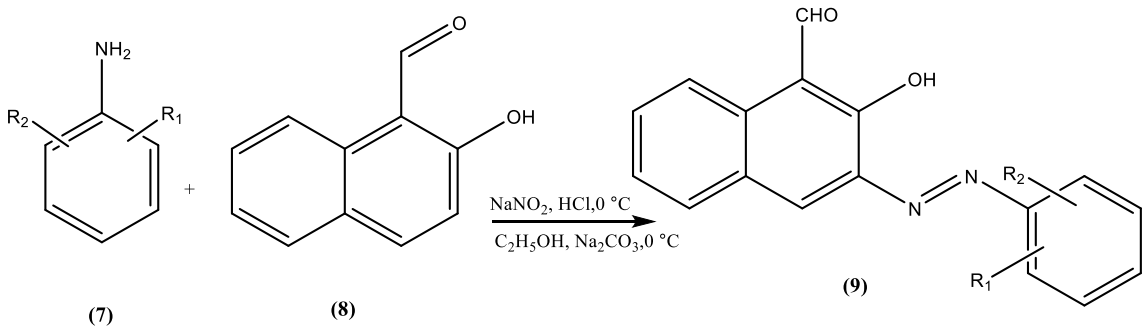
Al-Gaber and coworkers reported the synthesis of an azo dye **(6)** through diazotization of 4-amino-antipyrine **(4)** and coupling 2,4-diaminophenol **(5)** in an acidic ethanol condition [13]:



(4) (5) (6)

2.2 Azo dyes containing naphthol

Kaur *et al*. synthesised azo dyes **(9)** containing 2-hydroxy 1-napthaldehyde through diazotization by aniline derivatives **(7)** with 2-hydroxy 1-napthaldehyde **(8)** in an alkaline ethanolic solution [14]:

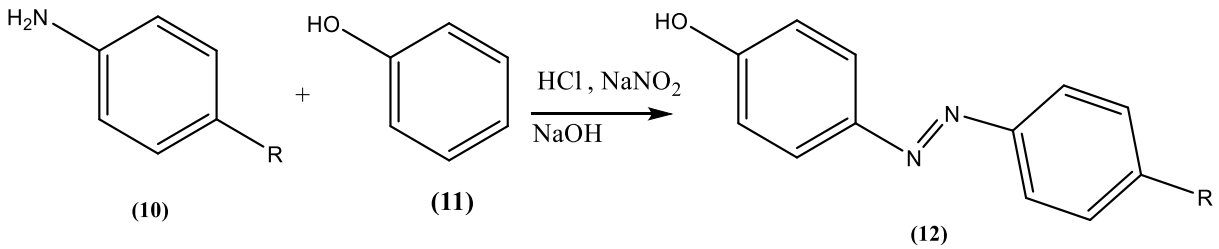


(7) **(8)** **(9)**

;R_1=4-COOH,R_2=H
;R_1=2-Cl,R_2=5-Cl
R_1=2-CH_3,R_2=4-CH_3
;R_1=2-NO_2,R_2=4-Cl

2.3 Azo dyes containing phenol

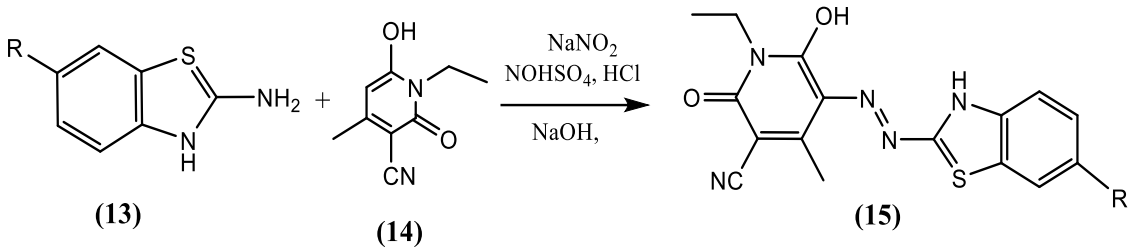
The synthesis of a new azo dye by Chen *et al.* involved the reaction of phenol with its corresponding aryl diazonium salt prepared by diazotizing 4′-substituted aniline **(10)** with phenol **(11)** to create 4-hydroxy-4′-substituted azobenzene **(12)** [15]:



(10) **(11)** **(12)**

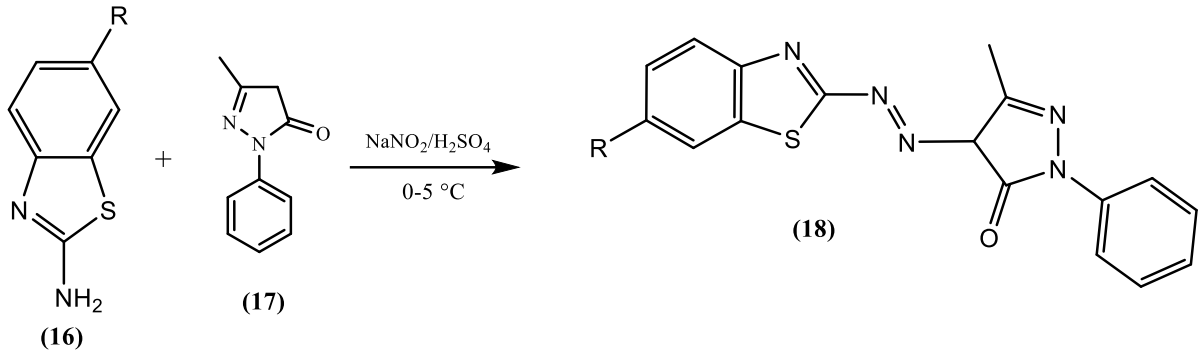
2.4 Azo dyes containing benzothiazole

Song *et al*. reported the synthesis of bi-heterocyclic dyes **(15)** from benzothiazole derivatives **(13)** through diazotization in the presence of $NaNO_2$ and nitrosylsulfuric acid followed by reaction with N-ethyl-3-cyano-4-methyl-6-hydroxy-2-pyridine **(14)** [16]:



(13) **(14)** **(15)**

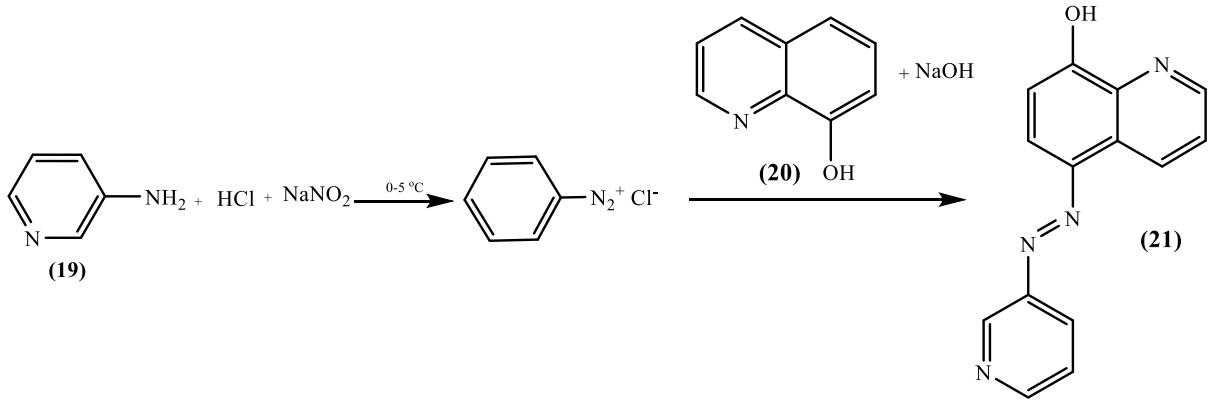
Benzothiazole-based dispersed azo dyes (**18**) were synthesised by Maliyappa *et al*. from the reaction of 2-phenyl-2,4-dihydro-pyrazole-3-one **(17)** with benzothiazole derivatives **(16)** [17]:



(16) **(17)** **(18)**

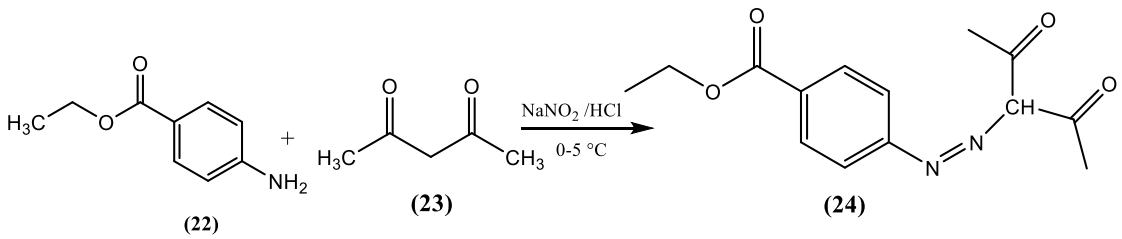
2.5 Azo dyes containing quinolin-8-ol

Alzamili *et al.* synthesised azo dyes **(21)** by gradually adding 3-aminopyridine **(19)** to the solution of 8-hydroxyquinoline **(20)** in sodium hydroxide at 0-5°C [18]:



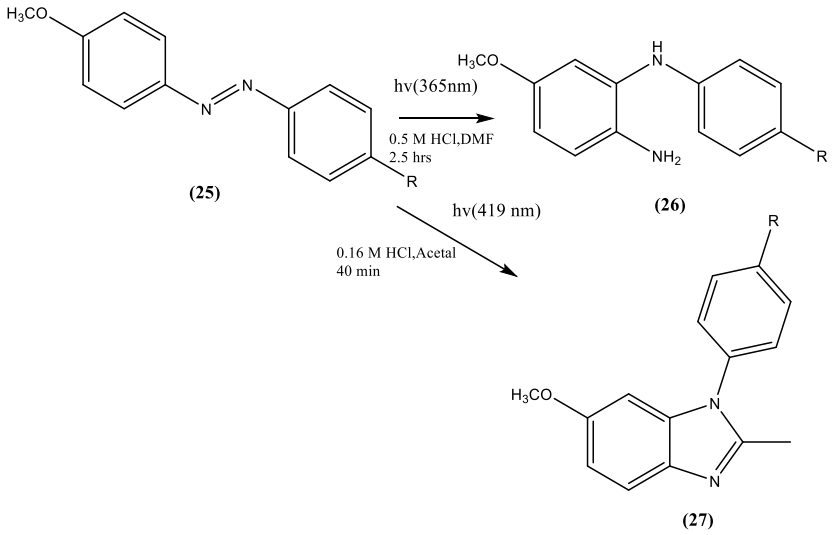
2.6 Azo dyes containing acetylacetone

Al-Battat *et al.* reported the synthesis of azo dye **(24)** by diazotizing ethyl p-aminobenzoate **(22)** in the presence of sodium nitrite/aqueous HCl at 0–5°C and coupling with acetylacetone **(23)** in alkaline solution [19]:

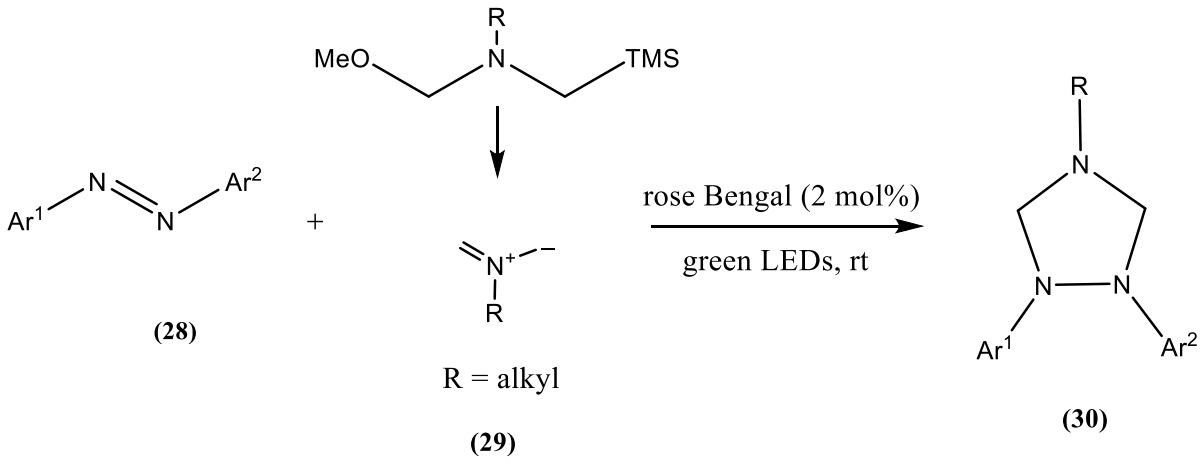


3. Azo dye derivative reactions

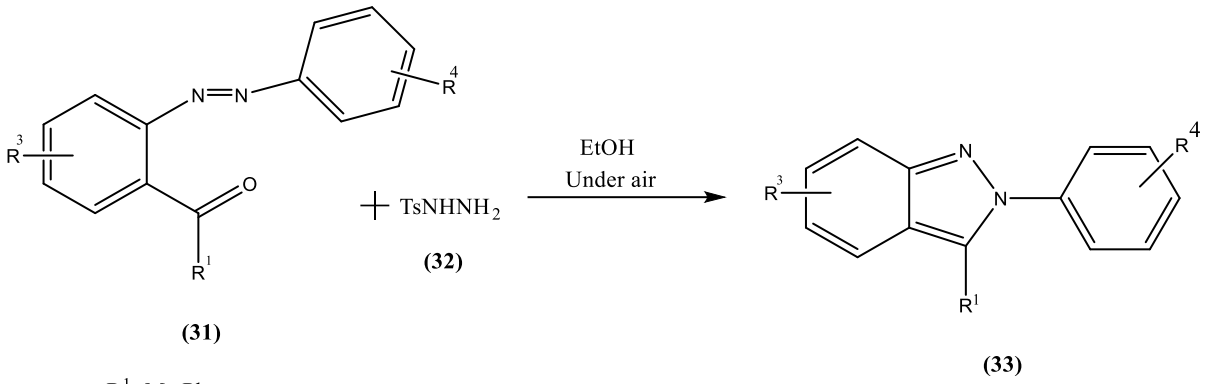
Chen *et al.* synthesised 5-methoxy-N-phenylbenzene-1,2-diamines **(26)** and 6-methoxy-2-methyl-1-phenyl-benzimidazoles **(27)**. The irradiation of 4-methoxyazobenzenes **(25)** in the presence of DMF containing 0.5 M hydrochloric acid produced compound **(26),** while irradiation in the presence of acetal with 0.16 M hydrochloric acid produced compound **(27**) [15]:



Yang *et al.* added azobenzene **(28)** and azomethine **(29)** under visible light irradiation at room temperature to synthesise 1,2,4-triazolidine **(30)** with yields of up to 96% [20]:



Son *et al.* synthesised 2-aryl-2H-indazoles **(33)** by condensation of 2-acyl azobenzenes **(31)** with Tosyl hydrazine **(32)** in a metal-free medium [21]:



(31) **(32)** **(33)**

R^1=Me,Ph

R^2(in the case of Ar =Ph)=Me,MeO,F,Cl,Br,CO_2Me,NO_2,thiophen-2-yl

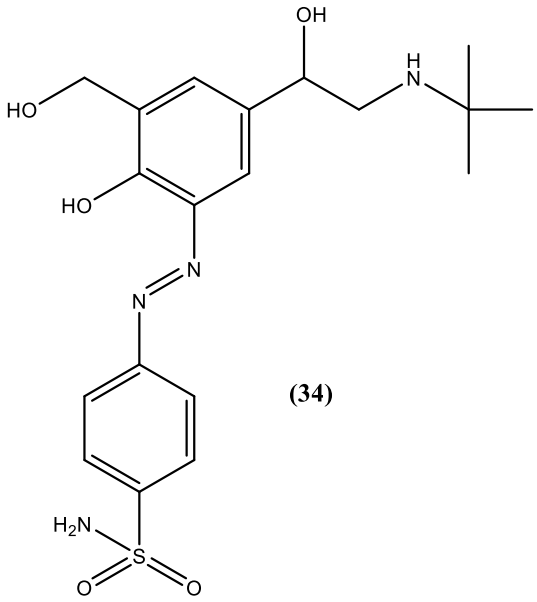
R^3,R^4=H,Me,MeO,Cl,Br,4-CO_2Et

4. Applications of Azo dyes

4.1 Medical and pharmaceutical applications

4.1.1 Antibacterial activity

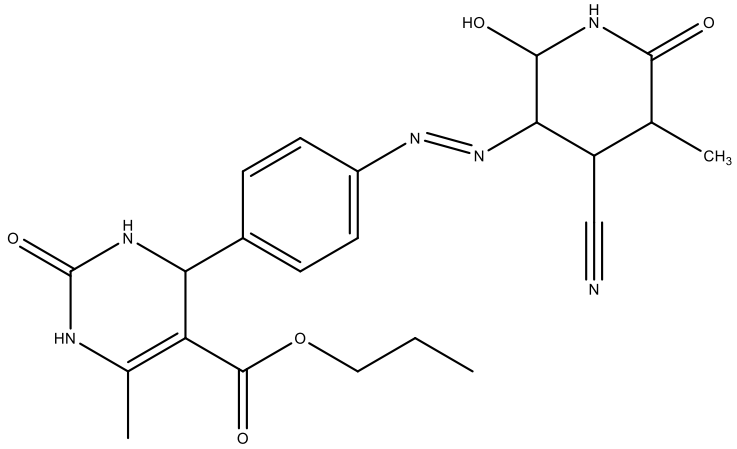
Asaad and Hazim reported the synthesis of azo dye **(34)** from sulfanilamide, sulfadiazine, and sulfathiazole with salbutamol sulfate *via* the diazo-coupling method. Azo dyes **(34)** showed the most potent antimicrobial activity. [22]



(34)

4.1.2 Anticancer activity

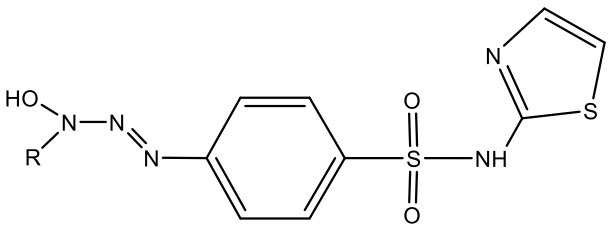
Tadić *et al.* reported the synthesis of azo dye **(35)** which possessed anti-cancer activity. [23]



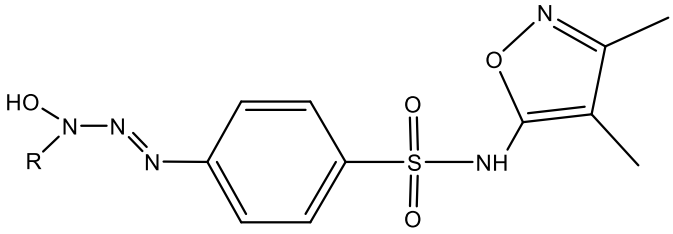
(35)

4.1.3 Antidiabetic activity

Dayma *et al.* synthesised compounds containing azo groups with hydroxytriazenes by coupling hydroxylamine with its diazonium salt from the diazotization of sulpha drugs at 0–5°C in an acidic condition. The azo dyes **(36, 37)** and **(38)** were potent antidiabetic agents. [24]



(36)



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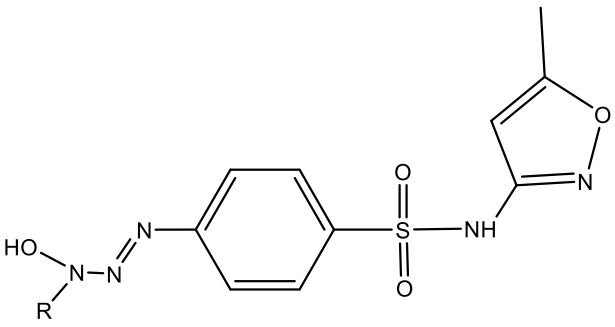
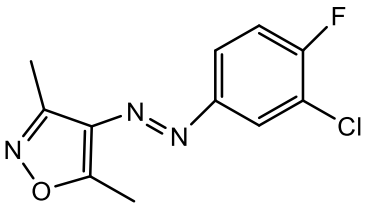


(38)

4.1.4 Antioxidant activity

Ketan *et al*. synthesised 3,5-dimethyl-4-(3'-chloro-4'-fluorophenylazo) isoxazole **(39)** and 3,5-dimethyl-4-(2'-fluoro-4'-methylphenylazo) isoxazole **(40)** which demonstrated antioxidant activities. [25]



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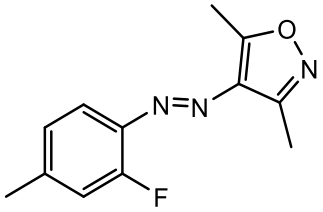
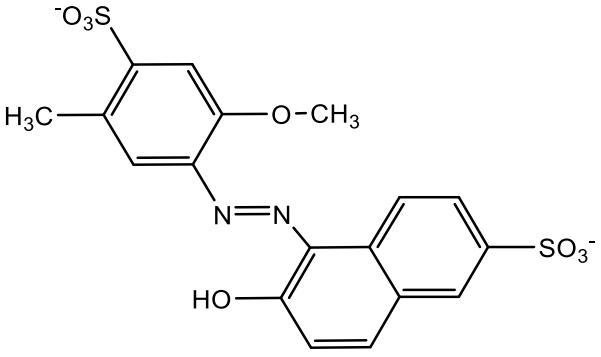


(40)

4.2 Industrial Applications

4.2.1 Food industry

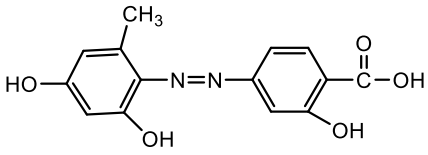
Mono-azo red dye **(41)** is commonly used in products such as powder mixes, candy, ice cream, spices, drink colouring, sauces, jellies, gelatins, and dairy items. [26]



(41)

4.2.2 Anti-corrosive steel

Abd Alrazzak reported the synthesis of azo compounds **(42)**, **(43)** and **(44)** based on 4-aminosalicylic acid to prevent the corrosion of steel. [27]



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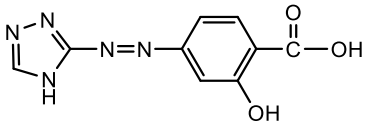

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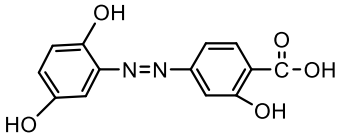
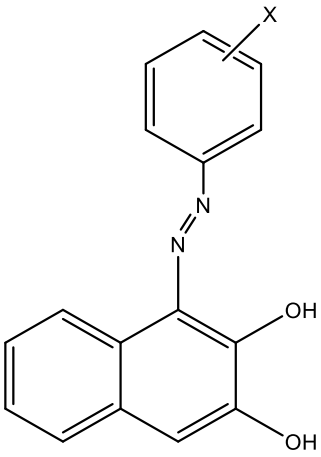


(44)

4.2.3 Dyeing polyester fabrics

Omer *et al.* reported the use of azo derivatives of 1-(aryldiazenyl) naphthalene-2,3-diol to dye polyester fabrics. [28]



(45)

5. Experimental

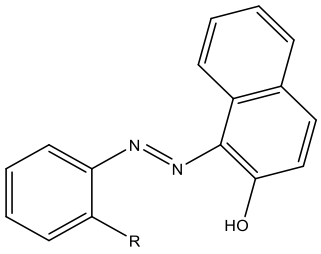
5.1. Materials:

Conc. HCl, Aniline, sodium Nitrate, 2-naphthol, 10% NaOH, Ethanol, 2-Nitroaniline

5.2. Physical measurements:

Melting ranges has been determined via Stuart apparatus SMP 30 melting point. The FT-IR . spectra were collected on the use of KBr pellet on IR Prestige-21 SHIMADZE

5.3 Synthesis of 1-(aryldiazenyl) naphthalene-2-ol (III a,b)



IIIa R= H
IIIb R= NO_2

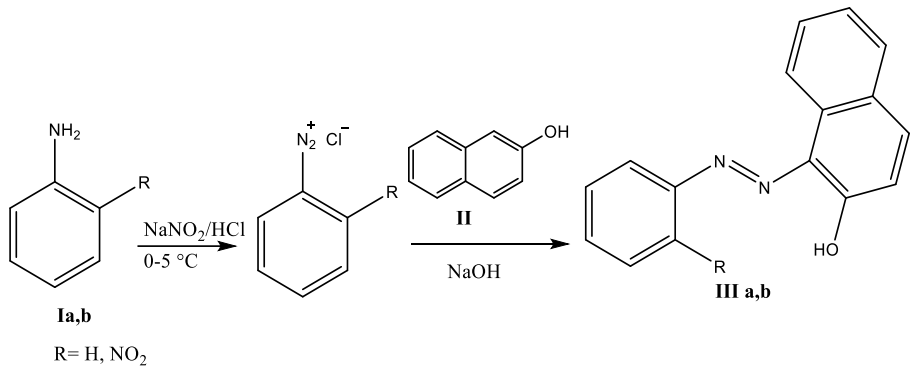
A mixture of aniline derivatives (0.0234 mol) was dissolved in 10 ml of H2O and 2.5 ml of conc. HCl, stirred and cooled at 0−5°C in an ice bath. Then, 2.5 ml of sodium nitrate solution was added dropwise into the aniline hydrochloride solution. A solution of 0.1 g of 2-naphthol was dissolved in 2 ml of 10% NaOH . The 2-naphthol solution was vigorously stirred followed by the slow addition of cold diazonium salt. The mixture was stirred for 30 min to 1.5 hrs. The reaction progress was monitored by TLC using a mobile phase of ethyl acetate: hexane (1:6). [29]

IIIa yield (2.5%) **(**M.P. 122-123°C). FT-IR (KBr, υ/cm^{-1}): 3427 (OH), 3049 (C-HAr.), 1618 (C=C Ar), 1501(N=N).

IIIb yield (39%) **(**M.P. 151°C). FT-IR (KBr, υ/cm^{-1}): 3434 (OH), 1611 (C=C Ar), 1522 (N=N).

5.4 Results and Discussion

1-(Aryldiazenyl) naphthalene-2-ol **IIIa** and **IIIb** were synthesised via the conventional diazo-coupling method in an acidic condition. Diazotization of aniline derivatives **Ia** and **Ib** with the coupling of 2-napthol **II** yielded 2.5 and 39% respectively, as shown in Scheme 2. The structure of products **IIIa** and **IIIb** was confirmed by FT-IR spectrometry.



Scheme 2: Synthesis of 1-(Aryldiazenyl) naphthalen-2-ol **IIIa,b**

The FT-IR spectrum of compound **IIIa** in **Figure 2** shows the absorption of a broad band at 3427 cm^{-1} attributed to the stretching of the OH group and 3049 cm^{-1} for the stretching of aromatic C-H. The medium intensity bands at 1618 and 1501cm^{-1} were due to stretching of the aromatic (C=C) and azo group (N=N), respectively.

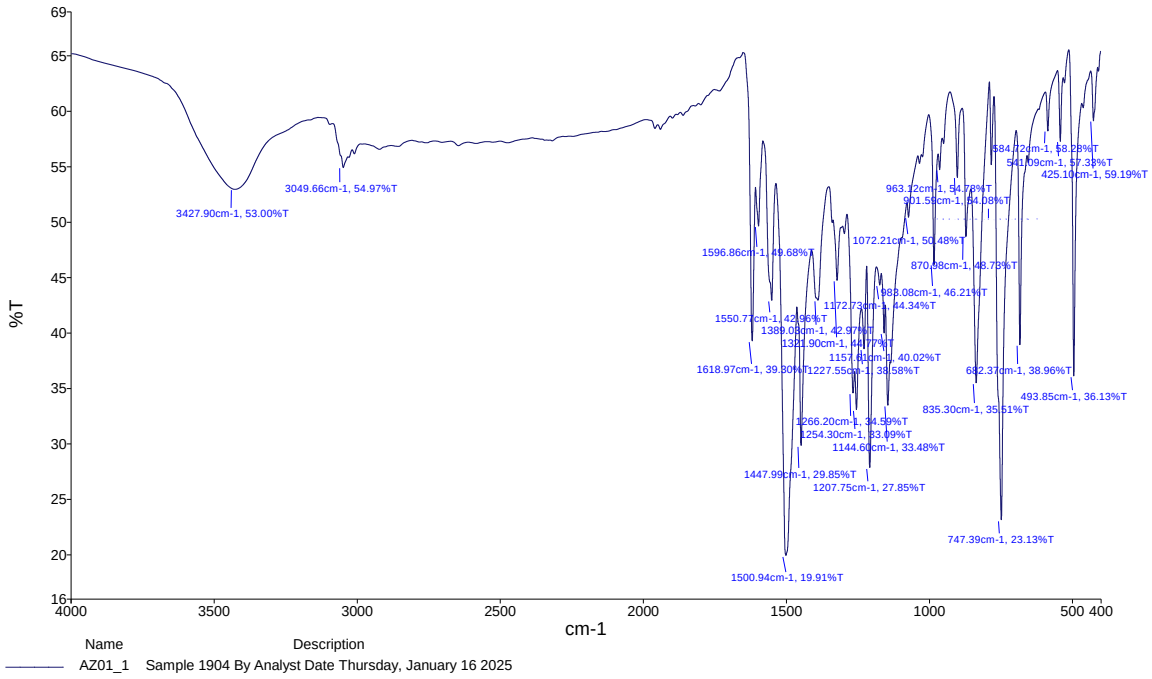


Figure 2: The Infrared spectrum of compound **IIIa**

The FT-IR spectrum of compound **IIIb** in **Figure 3** shows the absorption of a broad band at 3434 cm^{-1} attributed to the stretching of the OH group. The medium intensity bands at 1611 and 1522 cm^{-1} were due to the stretching of an aromatic (C=C) and azo group (N=N), respectively. The sharp band at 3311 cm^{-1} is an intermediate (NH).

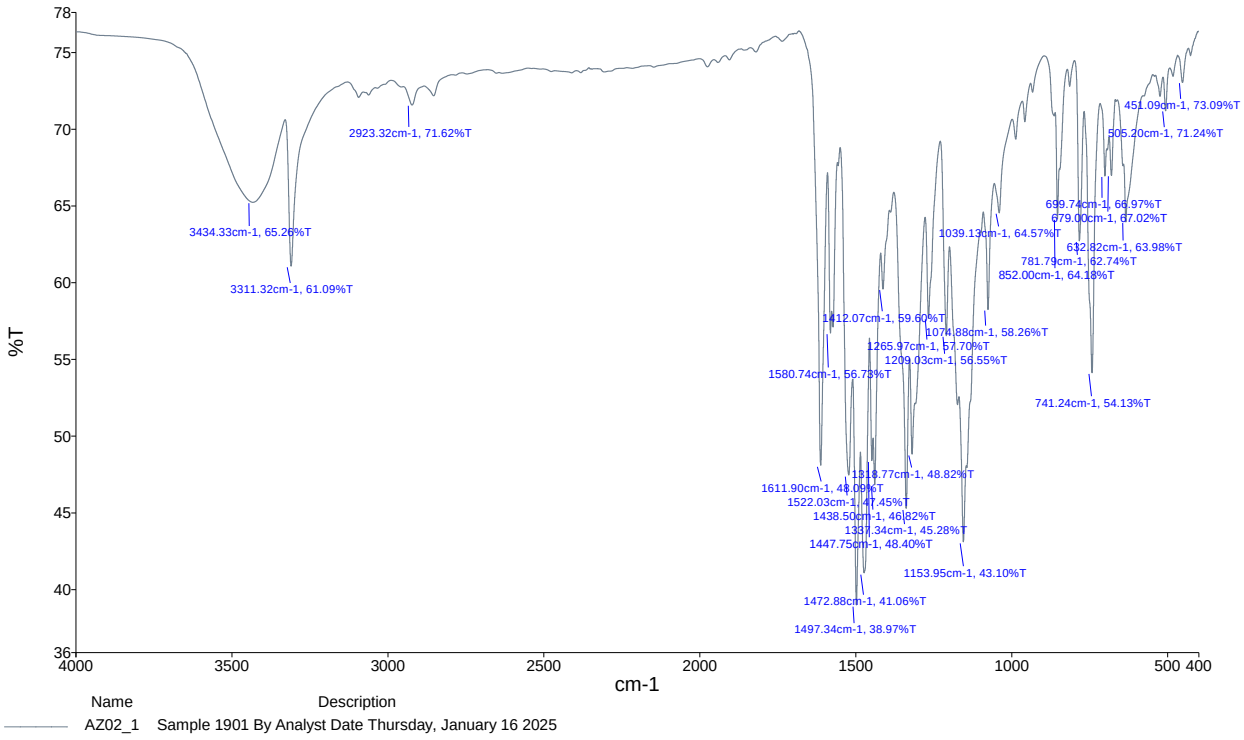
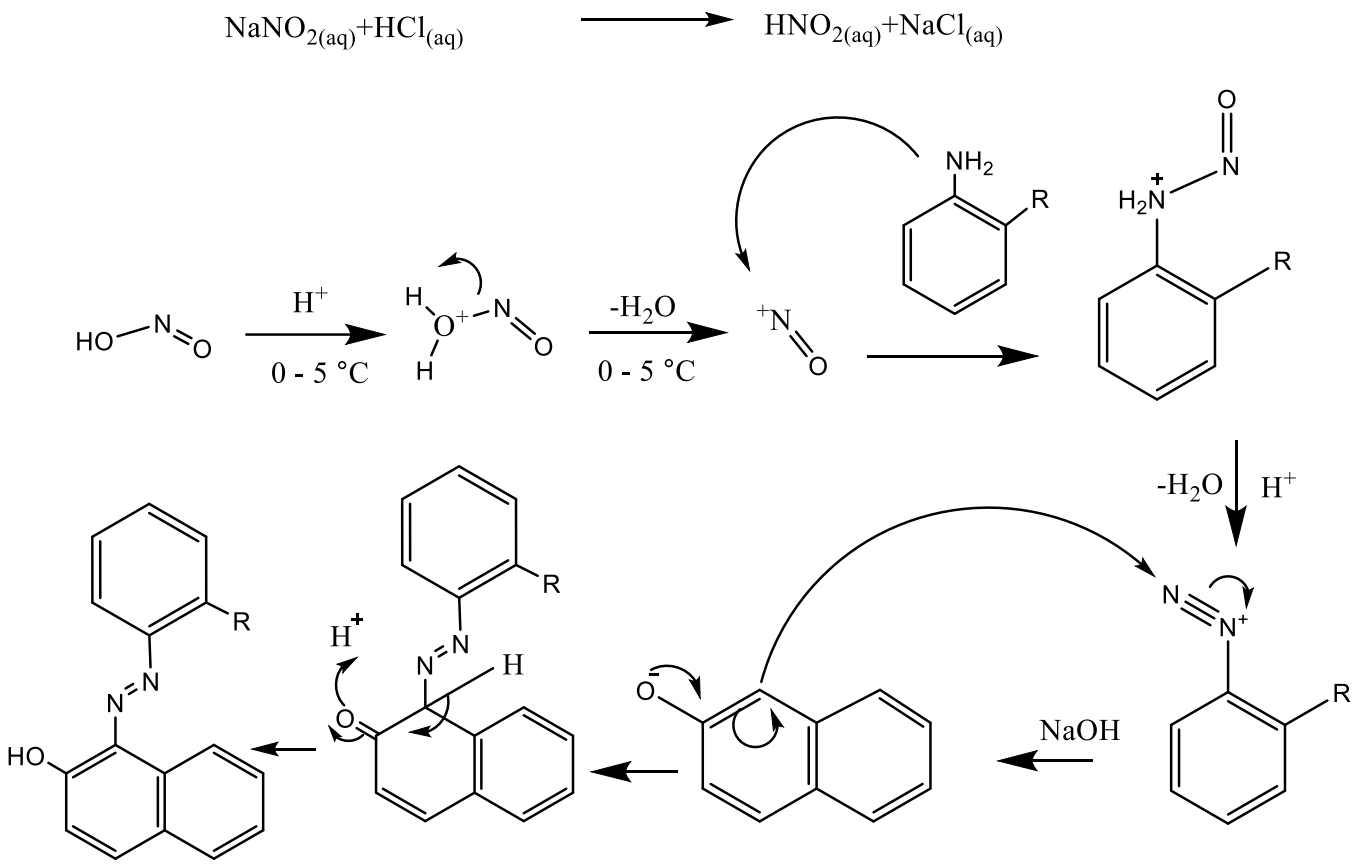


Figure 3: The Infrared spectrum of compound **IIIb**

Scheme 3 presents the proposed mechanism for synthesising 1-(aryldiazenyl) naphthalene-2-ol



Scheme 3:proposed mechanism to the 1-(Aryldiazenyl)naphthalen-2-ol

Conclusion:

In conclusion, azo dyes are an important group of organic compounds that have gained significant attention due to their diverse biological activities and chemical applications. 1-(Aryldiazenyl) naphthalene-2-ol was synthesised and characterised using the conventional diazo-coupling method in an acidic condition. Diazotization of aniline derivatives **Ia** and **Ib** with the coupling of 2-naphthol **II** yielded 2.5 and 39% respectively. The structure of the azo dyes (**IIIa** and **b**) was confirmed by FT-IR spectroscopy.

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A dedicated chemistry student at Imam University, with a strong foundation in organic chemistry. Skilled in laboratory techniques, data analysis. Passionate about applying chemistry knowledge to solve real-world problems. Eager to contribute to academic and research excellence.

-EDUCATION:

IMAM MOHAMMAD BIN SAUD ISLAMIC UNVERSTY
BACHELOR SDEGREE INCHEMISTRY

-SkILLS:
TeamWork
Chemical Safety and Handling
Problem Solving
Team Leadrship
Organization and Time Management

-COURSES:
Basics of scientific research
Biological regulation of wildlife
Manufacture of nanomaterials for energy production

- CONTACT
ihir48@icloud.com
0530307253
Riaydh city

Ruba Faisal Alharbi.

A committed chemistry student at Imam University, with a solid background in organic chemistry. Proficient in laboratory procedures and data interpretation. Enthusiastic about leveraging chemistry expertise to address practical challenges. Driven to contribute to academic achievement and research innovation.

Education: IMAM MOHAMMAD BIN SAUD ISLAMIC UNIVERSITY Bachelor's degree in chemistry

Skills:
TeamLeadership
Hard working
Strong analytical and problem-solving
Committed to learning and development

Courses:
Quality in medical laboratories
Strategic planning
Professional and academic certificates
Basics of scientific research

Contact:
Rubaalharbi11@gmail.com
0563558344
Riyadh city

I'm seeking to work a job with an in environment which provides career advancement to Gain new experiences and skills, develop my good scientific knowledge, and be an effective member in the work environment

Education: IMAM MOHAMMAD BIN SAUD ISLAMIC UNIVERSITY Bachelor's degree in chemistry

King Abdulaziz City for Science and Technology Cooperative Training.
King Abdullah City for Science and Technology Specialized programs in artificial intelligence.
Skills:
TEAM WORK
SCIENTIFIC WRITING
MICROSOFT OFFICE (WORD,EXCEL,POWERPOINT).

COURSES:

Occupational safety and health according to US(OSHA)Standars-30hours
INTRODUCTION TO INDUSTRIAL OCCUPATIONAL SAFETY AND HEALTH
ISO 17025 – 60 HOURS .
FUNDAMENTALS OF SUSTAINABLE ENERGY TECHNOLOGIES .
PHARMACEUTICAL SYNTHESIS .
CRISIS AND RISK MANAGEMENT.
ANALYTICAL DEVICES (UV-VIS SPECTTROPHOTOMETER, FTIR, TGA,DR6000 , HPLC,Contact angle).

Contact:
aishaalshammari182@gmail.com
0542083973
Riyadh city.

Alanoud Mogeb AL-qahtani

I am a student at Imam University, pursuing a Bachelor's degree in Chemistry. I have a strong interest in the fields of science and industry, and I am keen to explore the practical applications of chemistry in various industrial settings, and I have the ability to deal with chemicals, and precision devices.

Education: IMAM MOHAMMAD BIN SAUD ISLAMIC UNIVERSITY Bachelor’s degree in chemistry

Skills:
Analysis and problem solving
TeamLeadership
Preparing samples and solutions and testing the accuracy of tests
Effective communication skills, critical thinking, computer and administrative skills
Committed to learning and development
Accuracy and observation

Courses:
Fundamentals of Scientific Research
Supply Chain Management

Contact:
alalanoud425@gmail.com
0530258246
Riyadh city