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# Azo Compounds Synthesis and Antimicrobial Analysis

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**Abstract:** ***The purpose of the experiments conducted in this paper is to synthesize a new series of azo compounds from P-hydroxy acetophenone and aromatic amines by azo coupling reaction conducted near ice temperature. This study was designed to show antimicrobial activity and products were analyzed by $^1$H NMR, IR spectroscopic technique giving positive results.***

**Keywords:** Azo coupling, antimicrobial activity, p-hydroxy acetophenone

## 1. Introduction

Chemistry of azo compounds is one of the leading lines of investigations in the organic chemistry. These products have a huge contribution not only to the chemical industry but also to the biological. These are widely distributed in nature and are essential for life as they are fantastic precursors to make complex molecules such as medicines, agrochemicals and dyes. Azo dyes which are characterized by one or more azo bonds are predominant class of colorants and are used in tattooing and consumer products. In theory, azo dyes can supply a complete rainbow of colors, however commercially they tend to supply more yellows, oranges and reds than any other colors. In this experiment compounds containing p-hydroxy acetophenone moiety are reported. These newly synthesize dyes range in color from yellow and orange to red and brown.

Aryl azo compounds are usually stable, crystalline species. Their biggest advantage is their cost effectiveness, which is due to the processes involved in manufacture and most of the chemistry completed at or below room temperature and also their environmental impact is low due to the use of water as solvent in all of the reaction. A small proportion of azo dyes contains or can break down to form a class of chemical substances referred to as aromatic amines. It is only certain azo dyes that are problematic but the majority do not result in exposure to hazardous aromatic amines. They are very important type of organic compounds due to their implication in drugs and industrial studies and they form 60-70% of all synthetic dyes used as commercial colorants.

## 2. Materials and Experimental Method

All the chemicals used in these experiments were of analytical grade. All the melting points were determined and are uncorrected. The products were confirmed by $^1$H NMR and IR technique. The biological activity was evaluated against two kinds of bacteria gram positive and gram negative. The products were recrystallized by ethanol as solvent. The complete reaction was conducted in two steps.

**Stage 1-Diazotisation**
This stage involves a primary aromatic amine(0.01M) called the diazo component. It is treated in low temperature acid conditions (concentrated HCl) with sodium nitrite to form unstable diazonium salt.

**Stage 2-Azo coupling**
The unstable diazonium salt was reacted with a coupling component p-hydroxy acetophenone (0.01M). This forms the stable azo dye in low temperature i.e. 0-5°c.

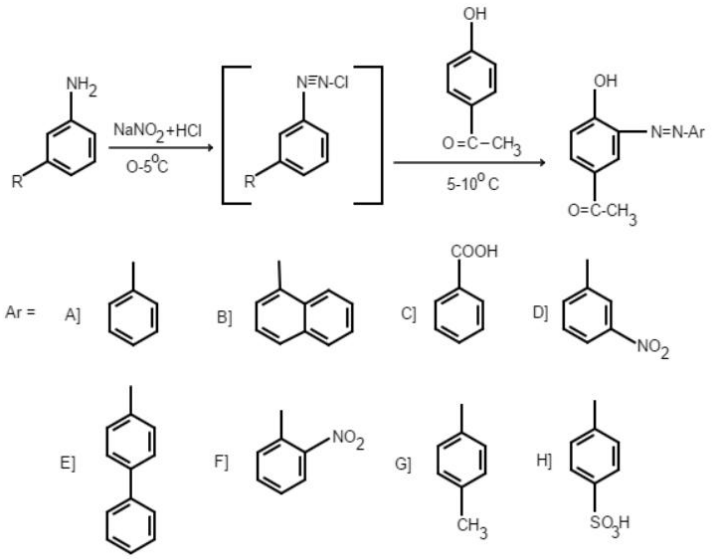


**Figure 1:** Chemical reaction of azo compounds

## 3. Safety and Regulations

This experiment should be performed in a well ventilated laboratory or fume hood. Many azo pigments are non-toxic, although some are mutagenic; likewiseseveral case studies have linked azo pigments with basal cell carcinoma. Azo dyes derived from benzidine are carcinogens and exposure to them has classically been associated with bladder cancer. Expert authorities such as the World Health Organization International Agency on cancer have classified some of these amines known or suspected human carcinogens.

**Table 1:** Anti-Microbial activity for the resultant products

**Anti-Microbial Activity of Product IVA-IVH**

| Symbol | Zone inhibition diameter (mm) | | | |
|---|---|---|---|---|
| | E. Coli | S. Typhi | P. Aeruginosa | S. Aureous |
| IV A | Resistant | 12.5 | 21 | 16 |
| IV B | 11 | 13 | 17 | 22 |
| IV C | 15 | Resistant | 22 | 18 |
| IV D | 12.5 | 16 | 15 | Resistant |
| IV E | 22 | 12 | 17.5 | 16 |
| IV F | 18 | 11 | 19.5 | 22.5 |
| IV G | 13 | 18 | 22 | 19 |
| IV H | 17 | 13 | 16.5 | 22 |

**Table 2:** IR and NMR absorption frequencies of product IVA to IVH

**IR and NMR absorption frequencies of product IVA to IVH**

| *Symbol* | *Name Of Compound* | *IR Spectra* | *NMR Spectra* |
|---|---|---|---|
| IV A | 1-{4-hydroxy-3-[(E)-2-phenyldiazen-1-yl]phenyl} ethan-1-one | 3309(OH), 1660(C=O), 1444(N=N),3182(C-H,Ar),1600(C=C,Ar) | 10.3(s,1H,OH), 2.4(s,3H,$COCH_3$), 7.7(s,2H,Ar-H), 7.8(s,1H,Ar-H), 6.8(d,2H,Ar-H) |
| IV B | 1-{4-hydroxy-3-[(E)-2-(naphthalene-1-yl]phenyl}ethan-1-one | 3311(OH), 2997(C-H,Ar), 1442(N=N),1568(C=C,Ar), 1666(C=O), 2997(C-H,Ar) | 10.3(s,1H,OH), 2.4(s,3H,$COCH_3$), 7.9(s,1H,Ar-H), 7.8(s,1H,Ar-H), 7.83(s,1H,Ar-H),7.6(1H,Ar-H),6.9(2H,Ar-H) |
| IV C | 4-[(E)-2-(5-acetyl-2-hydroxyphenyl)diazen-1-yl]benzoic acid | 3118(OH), 3018(C-H,Ar), 1730(C=O of COOH), 1444(N=N), 1556(C=C,Ar) | 10.2(s,1H,OH), 2.4(s,3H,$COCH_3$), 7.8(2H,Ar-H), 6.9(1H,Ar-H), 6.8(1H,Ar-H) |
| IV D | 1-{4-hydroxy-3-[(E)-2-(3-nitrophenyl)diazen-1-yl]phenyl}ethan-1-one | 3311(OH), 3190(C-H,Ar),1600(C=C,Ar), 1693(C=O),1444(N=N),1379($NO_2$) | 10.3(s,1H,OH), 2.4(s,3H,$COCH_3$), 7.8(t,2H,Ar-H),6.9(m,2H,Ar-H) |
| IV E | 1-{3-[(E)-2-{[1,1'-biphenyl]-4-yl}diazen-1-yl]-4-hydroxyphenyl}ethan-1-one | 3309(OH), 3186(C-H,Ar), 1602(C=C,Ar)1660(C=O of COOH),1444(N=N) | 10.3(s,1H,OH), 7.8(d,2H,Ar-H), 6.8(s,2H,Ar-H), 2.4(s,3H,$COCH_3$) |
| IV F | 1-{4-hydroxy-3-[(E)-2-(2-nitrophenyl)diazen-1-yl]phenyl}ethan-1-one | 3130(OH), 3020(C-H,Ar),1568(C=C,Ar), 1440(N=N), 1666(C=O of COOH), 1379($NO_2$) | 10.3(s,1H,OH), 2.4(m,3H,$COCH_3$), 7.8(s,2H,Ar-H), 6.9(d,2H,Ar-H) |
| IV G | 1-{4-hydroxy-3-[(E)-2-(4-methylphenyl)diazen-1-yl]phenyl}ethan-1-one | 3311(OH), 3074(C-H,Ar), 1444(N=N), 1562(C=C,Ar), 1681(C=O of COOH), 1300(C-H of $CH_3$) | 10.2(s,1H,OH), 7.8(d,2H,Ar-H), 6.9(s,1H,Ar-H), 6.8(s,1H,Ar-H), 2.4(s,3H,$COCH_3$) |
| IV H | 4-[(E)-2-(5-acetyl-2-hydroxyphenyl)diazen-1-yl]benzenesulfonic acid | 3307(OH), 3074(C-H,Ar), 1435(N=N), 1573(C=C,Ar), 1693(C=O of COOH), 1166(S=O), 638(S-O of $SO_3H$) | 9.9(s,1H,OH), 7.7(d,2H,Ar-H), 6.8(m,2H,Ar-H), 2.4(s,3H,$COCH_3$) |

## 4. Results and Discussion

From the data presented here, it can be concluded that a series of azo compounds IV A-IVH were synthesized from p-hydroxy acetophenone and are useful for many applications such as colors of azo dyes include different shades of yellow, red, orange, brown which can be used for dying in different industries including pharmaceutical industry. The remarkable activity against Gram-negative and Gram-positive bacteria of the newly synthesized dyes will be subjected to further study for the action mechanism of each compound.

## 5. Acknowledgement

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