Ministry of Higher Education and Scientific Research
AL Mustaqbal university college
Chemical Engineering Department

Petroleum Chemistry Laboratory

Experimental No.3

Reaction Coupling of dysonium salts

Prepared by

Asst. lect. Ban Ali Hassan
Eng. Difaf Ayman

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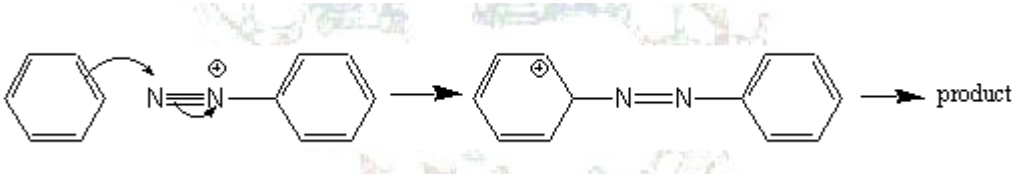
The purpose of the experiment: <u>Dyes Azo of Preparation</u>

Introduction And Theory

Amino groups on aromatic rings especially are convenient because they can be used to attach many other types of groups to the ring. For example, we can attach halogens, we can remove the amino group completely, and we can attach the hydroxy group to produce a phenol. Often starting with a nitro group, we can reduce it to an amino group, and then carry out other modifications.

Diazonium salts are very useful synthetic intermediates; they can be converted to a large number of functional groups, and, in many cases, are used as the key reaction step in the only practical synthetic route to yield substituted benzene compounds. Another set of important synthetic uses for diazonium salts is the diazonium coupling reaction. The terminal nitrogen of the diazonium cation is used as an electrophile in an EAS reaction:

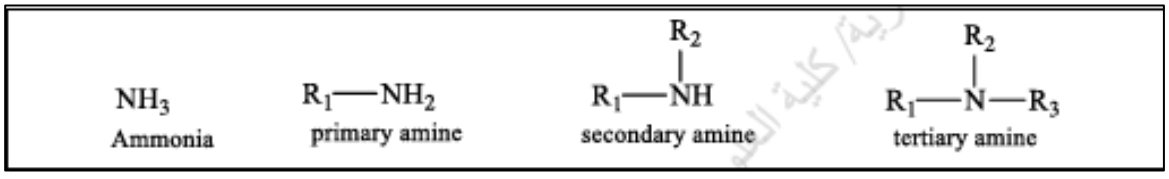


Most of the products of this type of diazonium coupling reaction are brightly colored, and many of the dyes used in textiles in the past century are made in this way. These dyes belong to the class of chemicals called "azo-dyes." As a dye, these chemicals tend to stick to objects, including clothing and your skin. To avoid "dying" items you don't wanted dyed, wear gloves when handling any of these colored compounds.

<u>Theoretical part:</u>

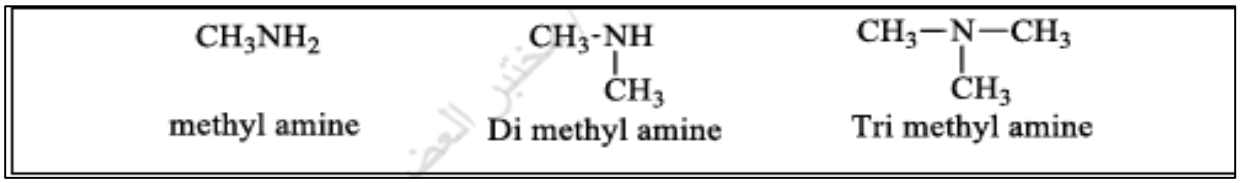
Amines: Are organic compounds that show appreciable basicity ,the general formula RNH_2, R_2NH, or R_3N, where R is any alkyl or aryl group.

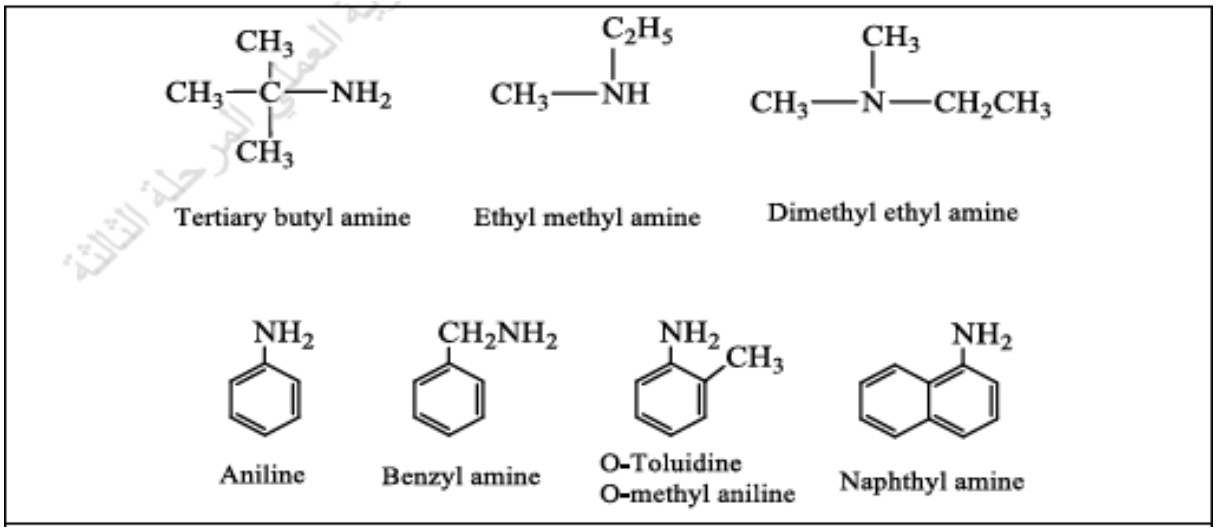
Amines are classified as primary, secondary, or tertiary according to the number of groups are attached to the nitrogen atom.



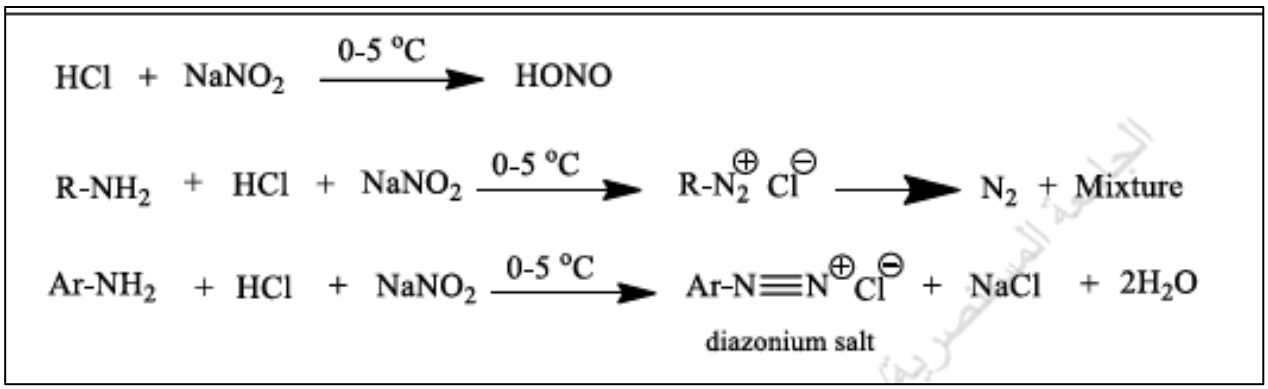
Nomenclature:

amines are named by naming the alkyl group or groups attached to nitrogen, and following these by the word-amine.

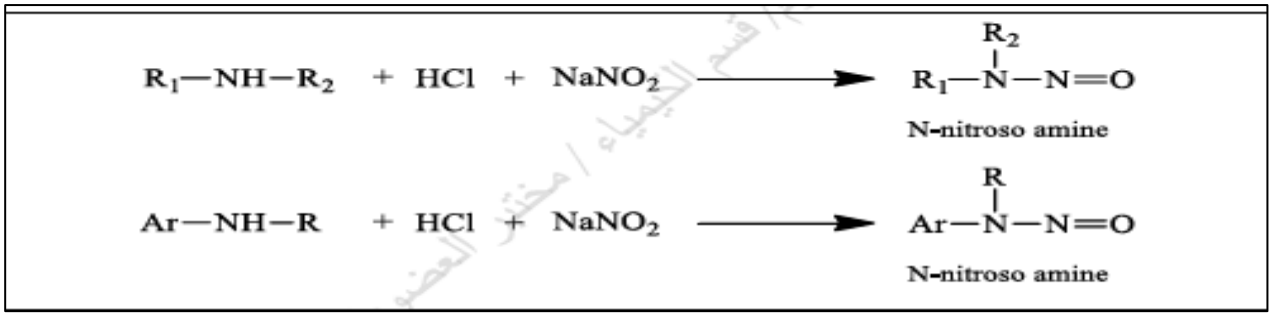




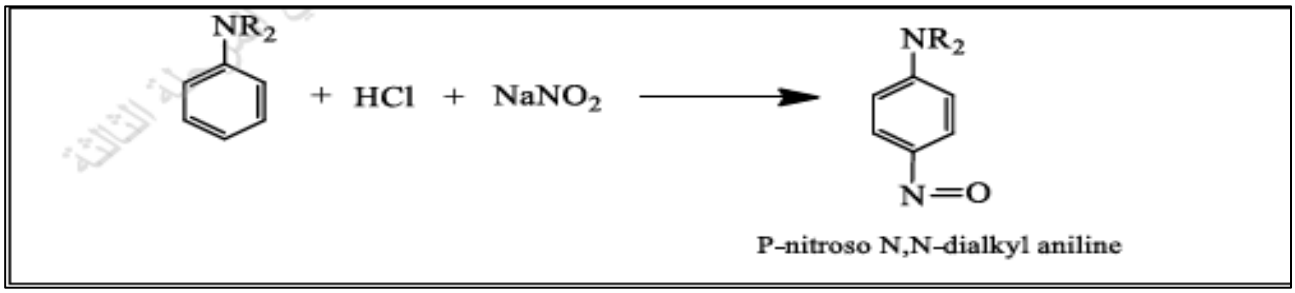
Amines are react with nitrous acid (HONO) to yields a different kind of product, HONO prepare by the action of mineral acid on sodium nitrite Primary amines react with nitrous acid to yield diazonium salt



Secondary amines, both aliphatic and aromatic reacted with nitrous acid to yield N nitrosoamines

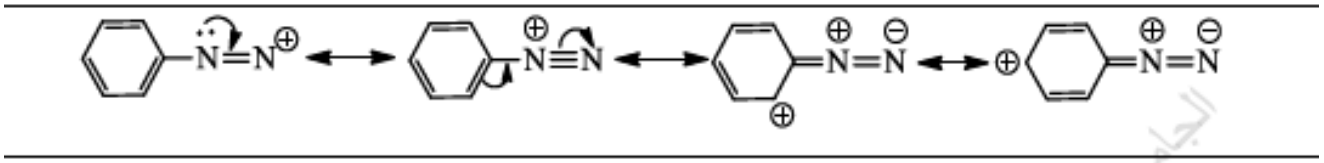


Tertiary aromatic amines undergo ring substitution, to yield compounds in which a nitroso group, -N-O is joined to carbon in p- position



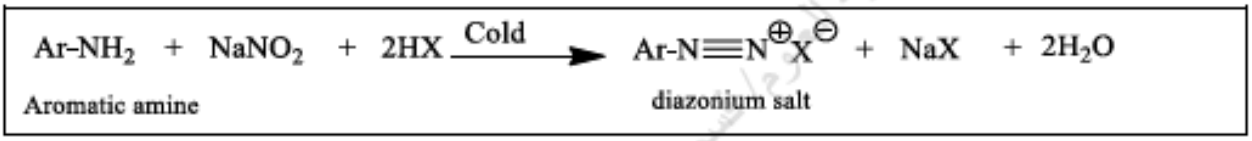
Primary aromatic amines react with nitrous acid to yield diazonium salt , this is one of the most important reactions in organic chemistry .

aromatic diazonium salt is more stable than aliphatic diazonium salt .



diazonium salt reaction :

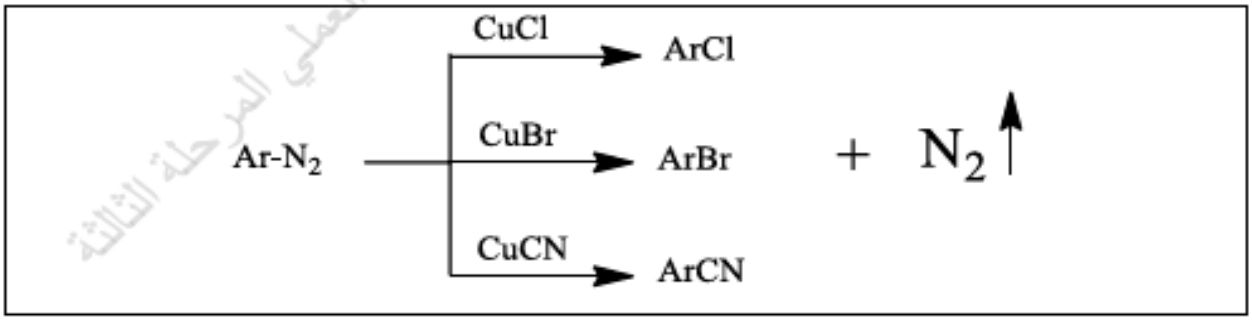
when primary aromatic amine is dissolved or suspend in cold aqueous mineral acid & treated with sodium nitrite , there is formed a diazonium salt



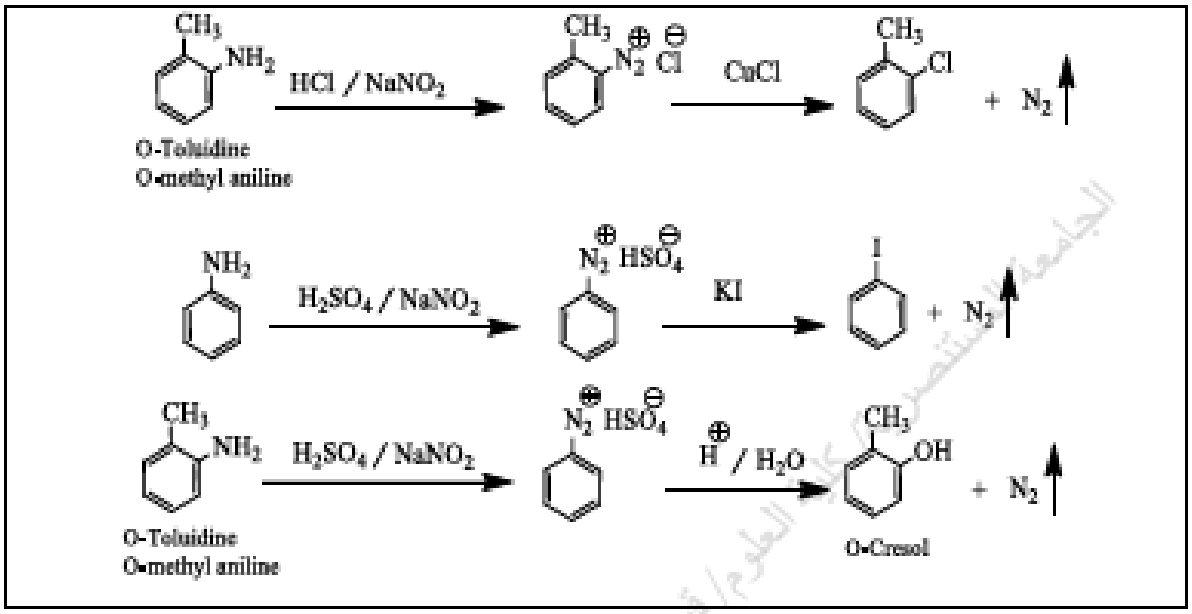
diazonium salt slowly decomposes even at ice-bath temperatures , the solution is used immediately after preparation .

There are large numbers of reaction undergo by diazonium salts may be divided in two types:

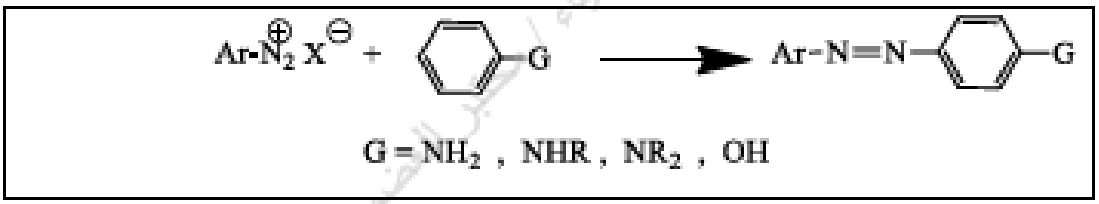
1- **Replacement reaction:** in which nitrogen is lost as N_2 , and some other atom or group becomes attached to the ring in its place .



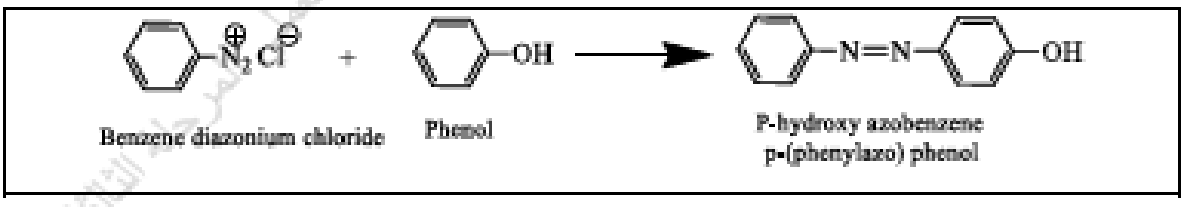
Examples:



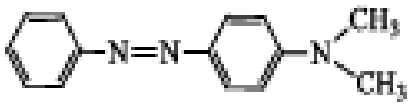
2- **Coupling reaction** : in which the nitrogen in diazonium salt is retained in the product .



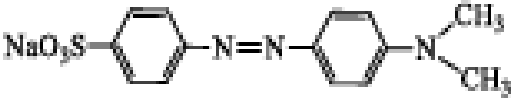
Example:



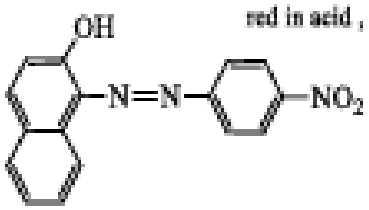
Examples for AZO dye :



p-di methylamino azobenzene

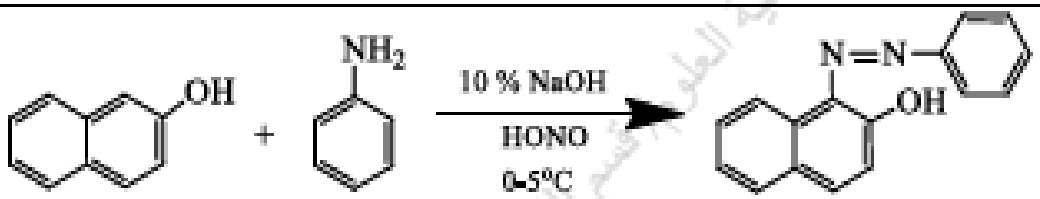


methyl Orange and acid base indicator
red in acid , yellow in base



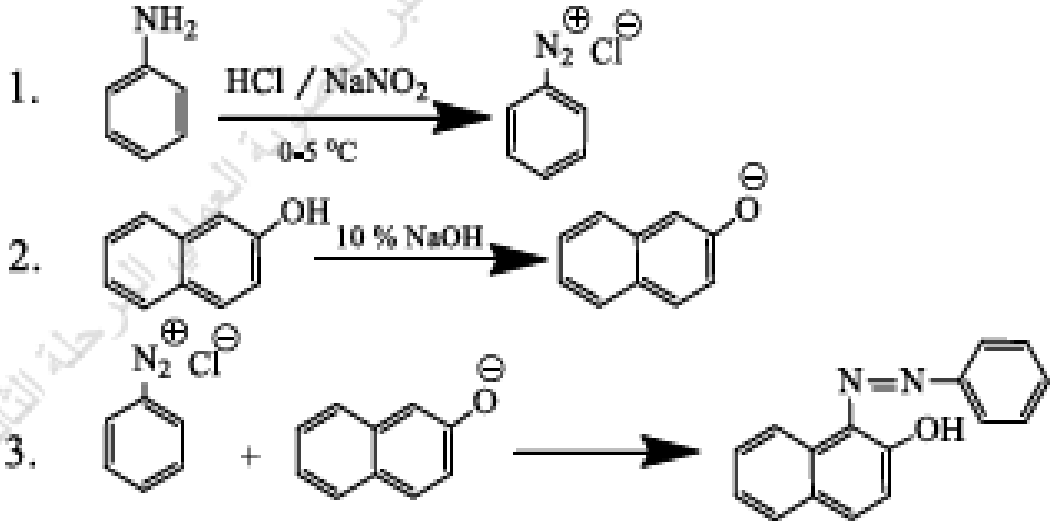
P-nitro azo-2-naphthol para red

E.G. : preparation of 1-phenylazo-2-naphthol :



1-phenyl azo -2-naphthol

mechanism preparation of 1-phenylazo-2-naphthol:



Instruments and Chemicals

- Beaker
- conical flask,
- graduated cylinder,
- thermometer,
- buechner funnel
- Naphthol,
- sodium hydroxide,
- prepared dysonium salt solution.

Procedure :

1) Prepare a solution of 0.1g of 2 naphthol dissolved in 2ml of 10% sodium hydroxide dilute with 5ml of water. Cool the solution to 5°C. Add a few grams of ice grits to the reaction mixture at necessity.
2) Vigorously stir the naphthol solution, then add 2ml of the diazonium salt solution prepared from the previous experiment
3) After that, the red crystals (1 phenyl azo 2 naphthol) separate.
4) Place the finished solution in an ice bath for 15 minutes for the solution to settle.
5) Filter with a Buechner funnel and wash the crystals with water

Discussion :

1- What is the purpose of the experiment,

2- What is the purpose of using sodium hydroxide in preparing azo dye?

3- Why do we put the final solution in a cold bath?

4- Is it possible to get azo dyes in different colors?