Expt. No.: **Date:**

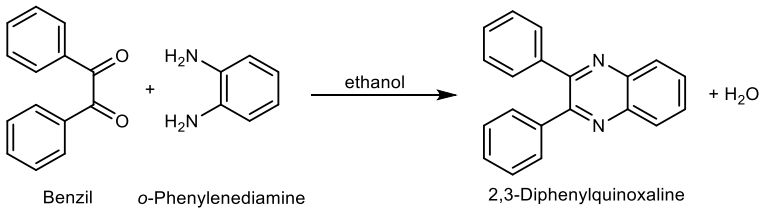
Experiment	**Lab scale preparation of important drug intermediate- Synthesis of 2,3-Diphenylquinoxaline**
Aim	• Preparation of 2,3-Diphenylquinoxaline from benzil • Calculating the yield of the reaction and confirming the product obtained by analytical data
Problem definition	Preparation of a medicinally important organic molecule by condensation reaction
Solution	Synthesis of 2,3-Diphenylquinoxalin will be carried out by condensation reaction. After completion of the reaction, monitored by thin-layer chromatography, the product will be filtered and recrystallized. The identification of the product will be carried out by infrared spectroscopy.
Student learning outcomes	Students will be able to: • monitor the reaction by using thin-layer chromatography and calculate the yield of the reaction • learn about basic organic chemistry transformations and the usage of spectral data for the identification of the product

Significance of the reaction.

Quinoxaline derivatives are an important class of heterocyclic compounds in pharmaceutical industries due to their wide range of therapeutic uses and potential activities, such as antimicrobial agents, cytotoxic agents, anti-tubercular, anxiolytic, anti-HIV, antioxidant, anti-inflammatory, antimalarial, anticancer, antidepressant, antibacterial, antifungal. Besides, they are also used in the agricultural field as fungicides, herbicides and insecticides, pesticides. Also, they are used as dyes, efficient electroluminescent materials, organic semiconductors, corrosion inhibitor etc. Owing to these plethora of applications, the synthesis of quinoxalines is very significant in the field of organic chemistry.

Principle.

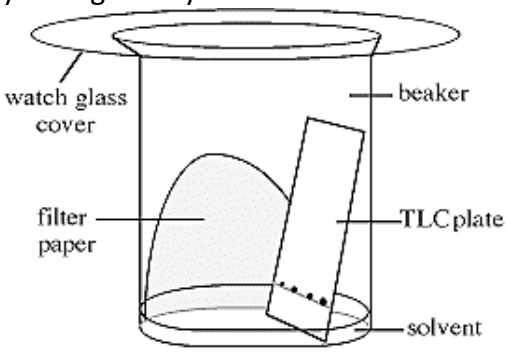
When benzil or diphenylethanedione (1,2-dicarbonyl compound) is treated with *o*-phenylenediamine (1,2-diaminobenzene) in ethanol (solvent), the amine groups and carbonyl groups get condensed via removal of two water molecules, resulting the formation of 2,3-diphenylquinoxaline.



Benzil *o*-Phenylenediamine 2,3-Diphenylquinoxaline

Working of thin-layer chromatography (TLC).

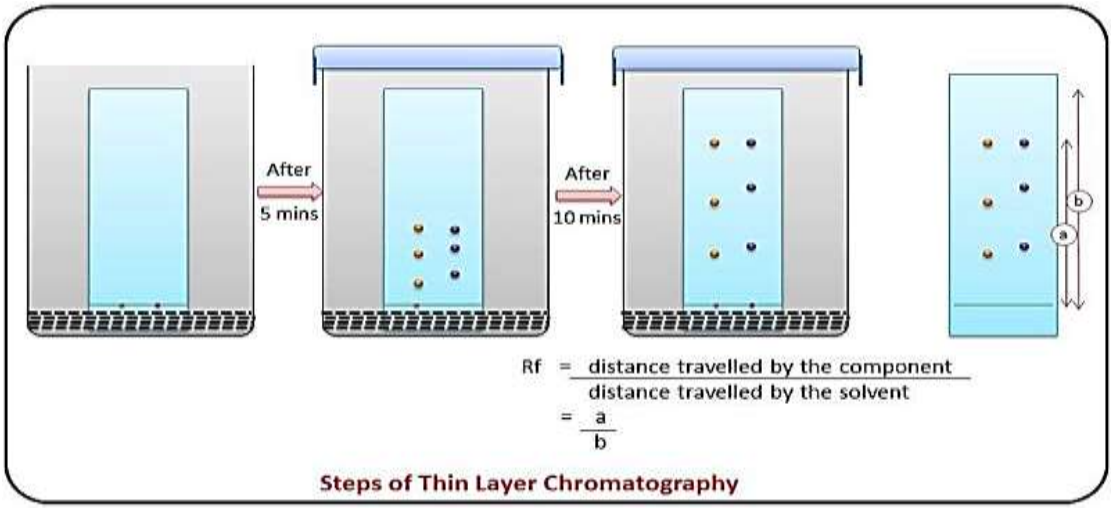
Thin Layer Chromatography can be defined as a method of separation or identification of a mixture of components into individual components by using finely divided adsorbent solid/(liquid) spread over a plate and liquid as a **mobile phase**.



Thin-layer chromatography is performed on a sheet of glass or plastic, or aluminium foil, which is coated with a thin layer of adsorbent material, usually silica gel, aluminium oxide (alumina), or cellulose. This layer of adsorbent is known as the **stationary phase**.

After the sample has been applied on the plate, a solvent or solvent mixture (known as the mobile phase) is drawn up the plate via capillary action. Because different analytes ascend the TLC plate at different rates, separation is achieved.

It is based on the principle of adsorption chromatography or partition chromatography or a combination of both, depending on the adsorbent, its treatment, and the nature of solvents employed. The components with more affinity towards the stationary phase travel slower. Components with less affinity towards the stationary phase travel faster. Once separation occurs, the individual components are visualized as spots at a respective level of travel on the plate. Their nature or character is identified through suitable detection techniques.



Steps of Thin Layer Chromatography

Chromatography is an important technique that enables the separation, identification, and purification of the components of a mixture for qualitative and quantitative analysis.

In this physical method of separation, the components to be separated are distributed between two phases, one of which is stationary (stationary phase) while the other (the mobile phase) moves in a definite direction. Depending upon the stationary phase and mobile phase chosen, they can be of different types.

TLC system consists of:

TLC plates - preferably ready-made with a stationary phase: These are stable and chemically inert plates, where a thin layer of stationary phase is applied on its whole surface layer. The stationary phase on the plates is of uniform thickness and is in fine particle size.

TLC chamber - The chamber maintains a uniform environment inside for the proper development of spots. It also prevents the evaporation of solvents and keeps the process dust-free.

Mobile phase- This comprises a solvent or solvent mixture. The mobile phase used should be particulate-free and of the highest purity for the proper development of TLC spots. The solvents recommended are chemically inert with the sample and the stationary phase.

Applications of Thin Layer Chromatography (TLC):

1. In monitoring the progress of reactions
2. Identify compounds present in a given mixture

Required reagents, apparatus, and conditions:

Reagents and solvents:

Benzil, *o*-Phenylenediamine, Ethanol

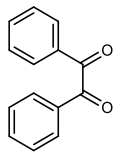
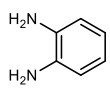
Apparatus:

Beaker, Buchner funnel, Measuring cylinder, Filter paper

Procedure

- To a solution of 2.1 g of benzil in 8 mL of ethanol, add 1.1 g of o-phenylenediamine in 8 mL ethanol in a beaker.
- Warm the reaction mixture in a water bath for 30 min, then add water until a slight cloudiness persists and allow the reaction mixture to cool.
- Filter the product and dry.
- Recrystallize the product from aqueous ethanol. Filter the recrystallized product and weigh it.

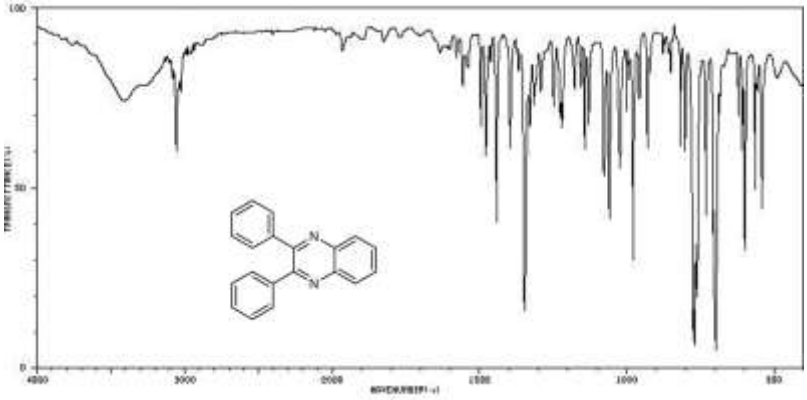
Table: Calculated amounts of reagents in the stoichiometric reaction

S. No.	Compound	Weight	Molecular weight	Moles = $\frac{weight\ taken}{Molecular\ weight}$
1		2.1 g		
2		1.1 g		

Melting Point: 125-126 °C

IR spectral data: Infrared (IR) spectroscopy deals with the infrared region of the electromagnetic spectrum, that is light with a longer wavelength and lower frequency than visible light. It works mainly on the absorption of infrared radiation by the molecules/ materials due to the change in the dipole moment. This vibrational spectroscopy is used to study and identify chemical substances or functional groups in solid, liquid, or gaseous forms.

IR spectrum of 2,3-diphenylquinoxaline:



Results:

Molecular formula of 2,3-diphenylquinoxaline = $C_{20}H_{14}N_2$

Molecular weight of 2,3-diphenylquinoxaline = ……

Theoretical yield of 2,3-diphenylquinoxaline = ……. g

Weight of the product obtained =……. g

% Yield = (Weight of the product obtained by the experiment) x 100/(Theoretical Yield)

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