REUSE AND RECOVERY OF ORGANIC COMPOUNDS FROM LABORATORY WASTE
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Abstract: Preparation of meta-dinitrobenzene from Nitrobenzene is an elementary synthesis carried out in any organic preparation experiments in laboratories. The substrate nitrobenzene is a pale yellow coloured liquid with a characteristic almond like odour. Upon continuous vigorous agitation with nitration mixture at cooler temperatures, nitrobenzene solidifies as a yellow coloured solid called meta-dinitrobenzene that gets precipitated when added with a suitable quantity of ice cold water. As a result, a lot of filtrate gets accumulated during the separation of pure product hence obtained. It is observed that large quantities of filtrate containing harmful organic residual effluents are discarded directly into the drains. This makes the treatment of waste water extremely dangerous. If left untreated it can prove to be deleterious to all the aquatic living forms, when it enters the water bodies. The objective of this study is to use the filtrate and recover as many organic compounds as possible before draining it out as waste. The recovered compounds can be reused within the laboratories or supplied as raw materials to other small scale manufacturers.

Keywords: Distillation, Filtrate, Meta-Dinitrobenzene, Nitrobenzene, Sulphuric Acid, Qualitative Analysis.
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INTRODUCTION
Benzene is the smallest organic aromatic compound. Nitrobenzene is a significant derivative of benzene with a molecular formula C₆H₅NO₂. New Jersey Department of Health Right To Know [NJDOH RTH] Hazardous Substance List classifies Nitrobenzene as 1st degree Reactive, 2nd degree Flammable, Carcinogen and a Teratogen. However, it is used in manufacture of perfumes, cosmetics, agro-chemicals, fertilizers, pesticides, dyes alongside its derivatives like meta-dinitrobenzene and aniline. Conventionally nitrobenzene is prepared by treating a charge of benzene with a nitrating mixture which is ideally a mix of concentrated sulphuric acid and concentrated nitric acid in the ratio 2:1 by volume. This is thereafter digested in the same vessel (Edward, 1958). Nitrobenzene is subjected to another round of regulated nitration to form meta-dinitro benzene [1, 3 dinitrobenzene]. An aliquot of nitrobenzene on treatment with the nitrating mixture followed by vigorous agitation forms a solid meta-dinitrobenzene which is precipitated by adding ice cold water.

Figure 1. Preparation of Nitrobenzene from benzene

Figure 2. Preparation of m-dinitrobenzene from Nitrobenzene
The products obtained namely nitrobenzene and m-dinitrobenzene have substantial uses in the field of pharmaceuticals and chemical industries. The filtrate however is likely composed of unreacted nitrobenzene, remnants of concentrated acids like sulphuric acid and traces
of nitric acid. These organic reactions produce large volumes of filtrates which are discarded untreated. Such a deed has grave impact on the natural environment and life in it. Several strategies are being researched upon to combat the effect of these effluents. More recently, manufacture process of nitrobenzene and m-dinitrobenzene in industries are focussed on a continuous preparation that involves reuse of spent acid which remains at the end of nitration with fresh reactants to use up major portion of any unreacted nitric acid in the spent acid mixture (Edward, 1958). This study aims to separate and carry out subsequent qualitative analysis of the three major components: water, sulphuric acid and nitrobenzene of filtrate or the spent obtained during nitration of benzene and nitrobenzene. The separated components can be reused as raw materials to carry out other chemical reactions or organic preparations. The study intends to reduce the wastage of expensive chemicals by using basic principles of chemistry. It reduces the possibility of those detrimental chemicals reaching the environment.

EXPERIMENTAL

Preparation of m-dinitrobenzene form Nitrobenzene: 1mL of nitrobenzene was taken in a clean dry 100mL beaker. 3mL of concentrated nitric acid and 7mL of concentrated sulphuric acid were added slowly into the beaker. The reaction mixture was vigorously stirred for 20 minutes using a glass rod by placing the beaker in a water bath till a solid was observed to be formed. The reaction mixture was tested for completion of reaction by dipping the glass rod in a test tube filled with ice cold water. If pale yellow solid is not precipitated, stirring was continued. Once yellow solid of m-dinitrobenzene precipitated, about 50mL of ice cold water was added to cease the reaction and separate the product completely. The precipitate of m-dinitrobenzene was filtered and dried. The filtrate was separately collected for further analysis.

Distillation of Filtrate: Distillation is the process of separating the components from a liquid mixture by exploiting their relative volatile characteristics. Distillation results in complete separation forming nearly pure components or partial separation that increases the concentration of individual components in the mixture (Schaschke, 2014). Components having low boiling point distils out first leaving behind components with higher boiling point. The different components of the spent vary in their boiling points. Water has a boiling point of 100ºC, Nitrobenzene has a boiling point of 210.9 ºC and sulphuric acid has a boiling point of 337 ºC. This was used to separate them to a large extent. Upon distillation, water evaporates first followed by yellow coloured nitrobenzene leaving behind sulphuric acid.

The experimental setup was arranged as shown in Figure 3. Bunsen burner was turned on and kept below the round bottomed flask. On gradual heating the temperature remained constant at 95 ºC - 100 ºC until water boiled off which was collected in a beaker. Heating was further continued till temperature remained constant at 211 ºC. At this temperature nitrobenzene boiled off and was collected in a separate beaker. Care was taken to avoid spilling of the spent from the round bottomed flask during heating.

Figure 3. Distillation setup for separating the components of spent

RESULTS AND DISCUSSION

Qualitative analysis of distillates has been done after recovery of the organic compounds.

Qualitative Test for Sulphuric acid

a) Litmus Test: Blue litmus paper was dipped in sulphuric acid. Blue litmus turns red proving that it is an acid.

b) Barium chloride Test: Barium chloride when added to sulphuric acid, a white
precipitate of Barium Sulphate was formed insoluble in excess hydrochloric acid.

\[ \text{BaCl}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{BaSO}_4 + 4\text{HCl} \]

**Qualitative Test for Nitrobenzene**

a) Janovsky Reaction (Janovsky, 1891): 10 mL of dry distilled acetone was added to the sample of nitrobenzene in a beaker. 2g of dry sodium hydroxide was added and the reaction mixture was heated for 5 minutes gently in a hot water bath. The mixture did not turn pink confirming the presence of mono nitro compound (Maiti and Patel, 1982).

b) Preparation of dinitrobenzene derivative: Approximately 1mL of nitrobenzene separated from the spent was taken in a clean dry 100mL beaker. 3mL of concentrated nitric acid and 7mL of concentrated sulphuric acid were added slowly into the beaker. The reaction mixture was vigorously stirred for 20 minutes using a glass rod. 50mL of ice cold water was added. A pale yellow precipitate of a di-nitro derivative was formed confirming the presence of nitrobenzene.

Waste management is a crucial area of research in recent times. It focuses on reducing the harmful effects of waste on human and environmental health (Tonini et al., 2018). An effective way to achieve this is by reducing, reusing and recycling the waste generated to a large extent even before disposing it at domiciliary and industrial levels. One such simple yet efficient strategy is detailed in this study which can be employed in all institutions associated with organic preparations. Basic, fundamental and simple scientific techniques can be used to treat the waste generated and recover as many organic compounds from it which can otherwise prove to be deleterious to aquatic life and environmental equilibrium.

**CONCLUSION**

Organic compounds and their by-products constitute a major proportion of the waste disposed by either large scale industries or small scale units like chemical laboratories. Such chemical waste causes several short term and long term catastrophic effects on exposure to environment. Direct disposition of organic compounds induces subsequent reactions with an otherwise neutral natural environment making it toxic for survival of life forms ranging from beneficial micro-flora to plants and aquatic animals. Certain compounds cannot be degraded by the surrounding habitat making them arduous deleterious components of macrocosm. These possess the ability to be accumulated and persist within biological systems correlating to their pernicious repercussions for eternity. The rampantly growing need for effective waste management can be attributed to the harmful effects of chemicals being generated as waste. This situation obligates new age researchers and environmentalists around the globe to profess strategies for chemical waste disposal. One of the creditable strategies is to reduce the disposal of such chemicals to a large extent possible. This can be achieved by recovery of as many organic compounds as possible from waste generated prior to its disposal to environment. The reuse of these recovered organic compounds contributes to the preservation of environment quality and reduction of water pollution in specific. This study emphasizes the importance of minimization strategies to waste production to achieve substantial environmental improvements.

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**REFERENCES**


