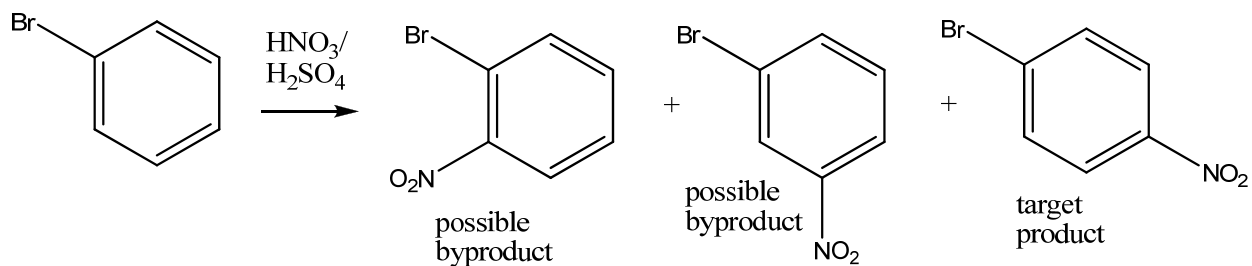


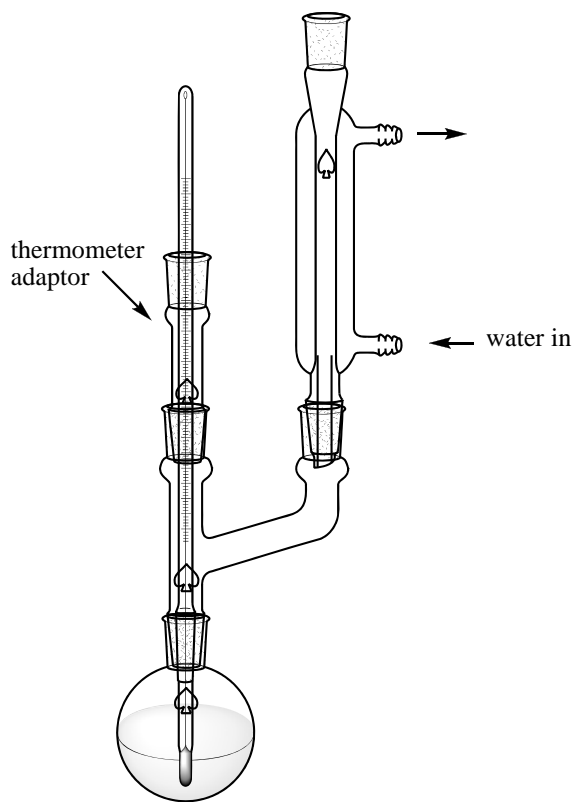
Synthesis of 4-bromonitrobenzene

Purpose: To synthesize 4-bromonitrobenzene and purify it from other isomers of bromonitrobenzene

Main Reaction:



Reaction Glassware:



Procedure:

In the hood, place 4.0 mL of concentrated sulfuric acid into a 25 mL roundbottom flask and slowly add 4.0 mL of nitric acid (caution: release of heat.) Cool the roundbottom to room temperature then fit it with a Claisen adaptor to make the apparatus pictured above. Use a magnetic stirplate to begin stirring the mixture.

In portions of about 0.5 mL, add a total of 4.5 mL of bromobenzene through the condenser. The addition should take about 10 minutes. Do not allow the temperature of the reaction to go above 50-55 °C. If it starts to get too hot, cool the reaction with an icebath and/or add the bromobenzene slower.

After the addition is complete, heat the reaction for 15 minutes, keeping the temperature below 60 °C. Cool the reaction to room temperature and pour it slowly and carefully, with stirring, into 40 mL of ice-cold water in a beaker.

Use vacuum filtration to isolate the precipitate. Wash the solid in the filter thoroughly with ice-cold water until the washes are neutral. (Test with pH paper.) Pull water through the filter until the solid is almost dry.

Transfer the crude solid to an Erlenmeyer flask and recrystallize the crude product from 95% ethanol. (Remember good recrystallization procedure to maximize your yield.) Isolate the purified product with vacuum filtration. Wash the purified product with two small portions of ice-cold 95% ethanol. Continue to pull air through the purified product until it is dry. Transfer the solid to a watch glass to obtain its mass. Obtain a melting point of the purified product.

Do a TLC analysis. Obtain authentic samples of the isomers of bromonitrobenzene that are provided and make solutions of them by dissolving about 1 mg of each of them into separate vials with 0.5 mL of dichloromethane. Using 9:1 hexanes:ethyl acetate as the eluting solvent, develop a TLC with the Authentic 2-bromonitrobenzene column(s), purified product, and the filtrate from your recrystallization. Develop the TLC plate and visualize it under a UV lamp.

Dispose of chemicals as directed by your AI's.

Discussion:

Did you make your intended product? Was it made in low, modest, or high yield? What data support this conclusion?

How pure is your product? What data support this conclusion? Was the recrystallization effective? Was the recrystallization necessary? Did you form any byproducts during the reaction? What data support these conclusions?

Be sure to reference sources of any data that you are citing but did not obtain yourself.

How would you improve this reaction in the future?