Nitration of Chlorobenzene

NOTE

WEAR GLOVES DURING THE ENTIRE PROCEDURE
Chlorobenzene and the two nitrochlorobenzenes are irritating to sensitive skin areas. If you should have these materials on your hands and then accidentally touch your face, this can cause a severe burning sensation in the affected area. If this should happen, IMMEDIATELY:
1. Go to the restroom and wash the affected area with lots of soap and water.
   THE SOAP IN THE LAB IS NOT SUITABLE FOR THIS PURPOSE.
2. Return to the lab and apply mineral oil to the affected area.
3. The scenario to this warning is NOT TO TOUCH ANY PART OF YOUR BODY WHILE PERFORMING THIS EXPERIMENT.

If you must leave the lab for any reason:
1. First dispose of your gloves in the waste container
2. Immediately go to the restroom and wash your hands thoroughly with soap and water.
   AGAIN, LAB SOAP WILL NOT DO A SUFFICIENT CLEANING JOB
3. Upon returning to the lab, obtain another pair of gloves from the front of the room, and proceed with the experiment.

----------------------------- FOR SAFETY REASONS -----------------------------

1. Add 700mL of tap water to your 1 L Beaker.
2. Discard any acid washings, plus the contents of the filter flask (from step 9 below) into your 1 L Beaker, WITH STIRRING.
3. Wash the contents of your 1 L Beaker down the sink.

PROCEDURE

1. Prepare a mixture of 4 mL conc HNO₃ and 4 mL conc H₂SO₄ in a 25 x 150 mm test tube. cool it to room temperature by means of an ice-water bath.

2. Add 1.5 mL chlorobenzene to the test tube DROPWISE, using the plastic pipet provided. Cork the tube and shake after each drop added.

   Should the test tube become warm ( over 35°C ), immerse it momentarily in an ice-water bath until the contents are cooled to room temperature before adding more chlorobenzene.

3. After the addition is completed and the exothermic reaction has subsided, stopper and shake
the tube for 5 min, until most of the upper layer of chlorobenzene is gone.

4. Allow the tube to stand at room temperature for 10 mins., to complete the reaction.

5. Pour the reaction mixture into 25 gm. of ice in a 100 mL beaker.

6. Isolate the crude product by vacuum filtration.

7. Wash the filter cake thoroughly with cold (0-10°C) distilled water and dry the filter cake by allowing the vacuum apparatus to draw air through after you have finished washing.

9. Transfer the crystals to a TARED 50 mL Erlenmeyer flask and obtain the weight of your wet product. Place the washings into the 1L beaker.

10. Calculate the volume of 95%(v/v) ethanol needed to just dissolve the nitrobromobenzenes at 78.2°C (the boiling point of 95%(v/v) ethanol). You will need approx. 5 mL 95% ethanol per gram of crude product. Now, add 1 mL more of solvent. SHOW THIS CALCULATION IN THE PROCEDURE PORTION OF YOUR REPORT.

11. Bring this mixture to boiling to dissolve the crude product, then set the flask aside and allow the contents to cool slowly to room temp. If the product does not completely dissolve after 2 minutes of boiling, add 5 mL of 95%(v/v) ethanol. If solid still remains you will have to do a hot filtration.

12. Isolate the nearly pure crystals of 4-nitrochlorobenzene by vacuum filtration.

13. Wash the crystals with a little ICE COLD ethanol, allowing the washes to drain into the filter flask with the mother liquors.

**DISCARD THE LIQUID PORTION INTO THE ETHANOL RECOVERY BOTTLE PROVIDED IN HOOD #1.**

**WASH DOWN YOUR BENCHTOP WITH A SMALL PORTION OF 95% ETHANOL ON A PAPER TOWEL. THROW THE PAPER TOWEL INTO THE TRASH. DO NOT USE THE LAB SPONGE TO WIPE DOWN YOUR BENCHTOP !!!!!**

14. Allow the crystals to dry overnight in the top of your filter funnel

15. Determine their weight and melting point. WASH YOUR BENCHTOP AS ABOVE. *

16. Place your product in the recovery jar in Hood #1.