N2020 A Level Planning Question – Suggested Solutions

4(a) $n(\text{benzene}) = n(C_6H_5NO_2) = \frac{10.0}{123} = 0.08180$ mol

Mass of benzene = 0.08180 x 78.0 = 6.34 g

As the yield is 70%, mass of benzene = $\frac{6.34}{0.70}$ = 9.06 g

4(b) Volume of benzene required = 9.06 / 0.8765 = 10.34 cm^3

We use 10.4 cm^3 of benzene for this reaction.

Modifying the quantities of reagents based on given procedure which used 8.0 cm^3 of benzene.

- Volume of concentrated nitric acid = volume of concentrated sulfuric acid = (10.4/8.0) x 8.0 = 10.4 cm^3
- Volume of DI water and Na_2CO_3 for washing organic layer = (10.4/8.0) x 75 = 97.5 ≈ 100 cm^3

(I) Preparation of nitrobenzene

1. Using separate 25.0 cm^3 measuring cylinders, 10.3 cm^3 of conc HNO_3 and 10.3 cm^3 of conc H_2SO_4 are added to a clean and dry 100 cm^3 conical flask placed in a water bath.
2. Using a 10.0 cm^3 measuring cylinder, 2.1 cm^3 of benzene is added to the mixture in the conical flask. The mixture is stirred with a thermometer. Care is taken to ensure that the temperature of the reaction mixture does not rise above 50 °C, for example by adding ice to the water bath to cool down the mixture if necessary.
3. Step 2 is repeated till a total of 10.4 cm^3 of benzene is added to the reaction mixture.
4. The temperature of the water bath is then adjusted to 60 °C and the reaction mixture is kept at 60 °C for 30 minutes.

(II) Purification of nitrobenzene

1. Using a 100 cm^3 measuring cylinder, 100 cm^3 of deionised water is added to a 250 cm^3 separating funnel. The impure nitrobenzene from part (I) is added and the resultant mixture is shaken thoroughly after stoppering the separating funnel. The bottom oily layer is drained into a clean and dry 100 cm^3 beaker from the tap of the separating funnel while the aqueous layer is poured out from the top of the separating funnel.
2. The oily layer in the beaker is poured back into the separating funnel. Using a 100 cm^3 measuring cylinder, 100 cm^3 of aqueous Na_2CO_3 is added and the separating funnel shaken thoroughly. The bottom oily layer is drained from the tap while the aqueous layer is poured out from the top of the separating funnel.
3. Step 1 is repeated with another portion of 100 cm^3 of deionised water.
4. A spatula of calcium chloride is added to the oily layer in the beaker. The mixture is stirred and left aside for 20 min.
5. The mixture is filtered using clean and dry filter funnel and paper into a 100 cm^3 round bottomed flask.
6. A distillation set up is assembled with the round bottomed flask being warmed using a water bath. At 80.1 °C, any remaining benzene will be distilled off.
7. When no more distillate is formed, benzene present as an impurity has been removed. The residue in the round bottomed flask contains the purified nitrobenzene.

4(c) Aqueous sodium carbonate is added to react with excess HNO_3 and H_2SO_4 present. The ionic salts formed from the reaction will dissolve in the aqueous layer and so will be separated from the oily layer.

4(d) Safety issue 1: benzene and nitrobenzene are toxic and volatile.
Precaution: Perform the experiment in a fume cupboard.
Safety issue 2: benzene and nitrobenzene are flammable.
Precaution: There should be no naked flame. Any heating is done with a hot plate or water bath.
(Another safety issue: Benzene and nitrobenzene are toxic and nitrobenzene is an eye irritant.
Precaution: Wear gloves and goggles when performing the experiment.)