Bromination of acetanilide

BROMINATION OF ACETANILIDE OBJECTIVE

Aim of the experiment was too brominate acetaldehyde to form p-bromoacetanilide.

INTRODUCTION

Acetanilide is a chemical compound with the chemical formula of C6H5NH(COCH3). It is a crystalline solid that is prepared by acetylation of analine and is widely used in the dye industry. This crystalline solid is an odourless solid chemical that has a flake or leaf like appearance. It is also known as N phenylacetamide, acetanil or acetanilide. Its former trade name was antifebrin. As mentioned earlier acetanilide can be produced by reacting acetic anhydride with aniline.

C6H5NH2 + (CH3CO)2O > C6H5NHCOCH3 + CH3COOH (http://www. mendelset. com/articles/680/preparation-recrystallization-acetanilide) (regensburg. de/Fakultaeten/nat_Fak_IV/Organische_Chemie/Didaktik/Keusch/Grafik/ box_brom_ar1. gif) The second figure illustrates the process used in the laboratory to form bromoacetanilide. Recrystallization is of key importance in the experiment because this method is used to obtain the crystals. Recrystallization is a purification technique that removes impurities from a sample. An impure solid is placed in a liquid for example water or ethanol. It is then heated making the solid dissolve into the solvent.

When the solvent cools back down again, the solid will precipitate out of the solution and leave the impurities behind, still dissolved in the solvent. This method purifies the solid. Acetanilide has many uses in both pharmaceutical

and manufacturing industry. It is used as a precursor in the synthesis of penicillin and other pharmaceuticals. Originally it was the first aniline discovered to posses' analgesic and antipyretic properties. A. Cahn and P. Hepp introduced it in the medical field in 1886. It was later to be discontinued in the medical field because of its toxic nature that was believed to cause cyanosis in the patient.

Through further research it was also discovered that acetanilide is metabolized in the body to form paracetamol which was responsible for the analgesic and antipyretic property. Acetanilide is also used as an inhibitor in hydrogen peroxide and is used to stabilize cellulose ester varnishes. It is also used in intermediation in rubber accelerator synthesis, dyes and dye intermediate synthesis and camphor synthesis. It is also used in the manufacture of sulfa drugs as it used for the production of 4-acetamidobenzenesulfonyl chloride, which is a key intermediate during this manufacture. METHOD 4. g of acetanilide was placed in a 100cm3 conical flask. It was then dissolved by adding 20 cm3 of glacial acetic acid, which was measured out in a measuring cylinder. 7. 0 cm3 was dispensed directly into the conical flask by use of a burette in one portion. The flask was then swirled to mix the contents. It was then covered with a clock glass and left in the fume hood for precisely 30 minutes long and swirled occasionally during the duration. Aqueous sodium hydrogen sulphite solution was added into the beaker until the red colour of the reactant disappeared this was to ensure that excess bromine formed during the reaction was destroyed.

Feature Article -Free-Radical Bromination

The lumps of solid found in the bottom of the flask were broken up to ensure no trapped bromine remained. As a precaution the first part of the experiment, which is outlined above, was carried out in a fumed cupboard. The flask was then taken out of the cupboard and the solid was then filtered by suction filtration using a Buchner funnel and washed with distilled water (2x 25cm3 portions). As much water as possible was removed from the solid by suction filtration then transferred to a 100cm3 conical flask.

The crude solid was recrystallized by use of a minimum volume of a mixture of ethanol and water in the ratio 2: 1 at its boiling point. This mixture was added in portions using a steam bath as a heat source. The clear solution was then set aside to cool to near room temperature. The flask was cooled thoroughly in an ice/water bath afterwards the crystals were collected by suction filtration using a Hirsch funnel. The crystals were then washed with approximately 1cm3 of ice-cold ethanol/water mixture, dried by suction filtration and the mass and melting point range recorded.

RESULTS Mass of empty weighing boat | 2. 25g| Mass of empty weighing boat+crystals| 5. 09g| Mass of crystals| 2. 8g| Melting range| 162 – 170C | DISCUSSION Bromine is generally a toxic, corrosive and a severe irritant to lung and eye tissues. Necessary precautions were taken during the experiment. One of the precautions taken was handling of the bromine solution in the reaction was done in a fumed cupboard. The excess bromine that was in the reaction was completely destroyed by adding aqueous sodium hydrogen sulphite solution.

Protective gloves were worn to prevent the bromine liquid, ethanoic acid and sodium hydrogen sulphite from irritating the skin, as they are all skin

irritants. The reaction that was carried out in the first part of the experiment is an electrophilic aromatic substitution. In the electrophilic aromatic substitution, an electrophile reacts with the benzene ring this result in an electrophile replacing hydrogen in one of the phenyl carbons such that the product of the reaction has the electrophile bonded to one of the phenyl carbon.

We did not use aniline in this practical as it has carcinogenic properties instead we used acetic anyhidride which is a milder version. The other products formed in this reaction hydrogen bromide and can be collected by hydrolizing(removing the hydrogen) to obtain the bromine. The percentage yield of the rection was calculated as follows Maas of products was 2. 84g Molar mass of bromo acetanilide= 214 0. 284/ 214 = 0. 00133 4. 5g of acetanilide used Molar mass = 135 0. 45/134 = 0. 003 Yield 0. 00133/0. 003x 100 = 44. 3% yield.

As clearly illustrated the percentage yield is less than a 100% this could be due to a number of reasons like some of the solid might have been lost during the suction or while washing the crystals with the water and ethanol mixture the weighing scales could also have been inaccurate. CONCLUSION Better practical methods are required during the experiment to ensure a 100% yield. REFERENCES http://www. mendelset. com/articles/680/preparation-recrystallization-acetanilide regensburg. de/Fakultaeten/nat_Fak_IV/Organische_Chemie/Didaktik/Keusch/Grafik/ box_brom_ar1. gif)