# Organic synthesis of aspirin chemistry formal lab essay sample

Science, Chemistry

Abstract

The purpose of this experiment is to synthesize a common organic product called acetylsalicylic acid (aspirin), and to become familiar with the optimum conditions needed for successful yields. Aspirin is produced from an acid catalyzed reaction between salicylic acid with acetic anhydride. The crystalline aspirin is synthesized and purified by recrystallization, although there is not a hundred percent yield due to sources of error.

Introduction

Aspirin is a medicine commonly found in households around the world. It also is one of the least expensive and most useful drugs in the market. A Chemist named Felix Hoffmann first synthesized aspirin, otherwise known as acetylsalicylic acid, in 1897 from salicylic acid. 1

Salicylic acid is a naturally formed substance. For example, willow trees have been recognized since ancient times as a natural source. Salicylic acid is found mainly in the willow’s leaves and bark. The pure acid possesses several useful medicinal properties. It is an antipyretic (a fever reducer), an analgesic (a pain reducer), and an anti-inflammatory (a swelling reducer).

Unfortunately, pure salicylic acid makes for a particularly unpleasant medicine. The salicylic acid molecule contains two acidic functional groups, the phenol group and the carboxylic acid group. These groups make salicylic acid irritating because it burns the sensitive linings of the mouth, throat, esophagus, and stomach.

These harsh qualities are alleviated by replacing the acidic hydrogens with less reactive groups of atoms: the acetyl group (COCH3). This results in acetylsalicylic acid or “ aspirin”.

Acetylsalicylic acid is synthesized in labs and does not occur naturally. It is produced from adding acetic anhydride to salicylic acid in the presence of sulfuric acid (H2SO4), an acid catalyst: (Diagram from 1)

The impurities left over (unreacted salicylic acid, acetic acid and sulfuric acid) are removed by the process of recrystallization. This process separates pure aspirin by precipitating it out of the impure solution.

Pure acetylsalicylic acid contains only one acidic functional group which bypasses most of the digestive system without causing burns. 2 Ultimately, acetylsalicylic acid hydrolyses with water, mainly in the bloodstream, to regenerate salicylic acid: (Diagram from 1)

There are four mathematical calculations given by the instructor. They are:

1. Density = Mass / Volume

2.# of moles of the limiting reagent = Weight of the limiting reagent (grams) / Molecular weight limiting reagent (grams/mole)

3. Theoretical Yield = # of moles of limiting reagent X

Of aspirinMolecular weight of aspirin (Acetylsalicylic Acid)

4. Percent actual Yield = (Actual yield / Theoretical Yield) X 100%

Experimental

Chemicals Used:

*Salicylic Acid: C7H6O3(S)(Provided)

*Acetic Anhydride: CH3COOCOCH3(l)(Provided)

*Sulfuric Acid: Conc. H2SO4(l)(Provided)

*Ethanol: C2H5OH(l)(Provided)

*Acetic Acid: C2H4O2(l)(Prepared)

*Acetylsalicylic Acid: C9H8O4(S)(Prepared)

Procedures:

Followed the procedures listed in the University of Winnipeg, Department of Chemistry Laboratory Manual, page 43.

Results

Data:

PropertyMeasurement Method of Determination

Mass of salicylic acid1. 498 gAnalytical Balance

Volume of acetic anhydride5. 0 mLGlass Graduated Cylinder

Density of acetic anhydride1. 08 g/mLGiven by Lab Manual (page 45)

Weight of acetic anhydride5. 4 gSee #1 in Calculations below

Molecular weight of salicylic acid138. 12 g/molGiven by Lab Manual (page 45)

Molecular weight of acetic anhydride102. 09 g/molGiven by Lab Manual (page 45)

Molecular weight of acetylsalicylic acid180. 16 g/molSee #2 in Calculations below

Mass of filter paper + aspirin2. 103 gAnalytical Balance

Mass of filter paper0. 962 gAnalytical Balance

Mass of aspirin1. 141 gSee #3 in Calculations below

Percent yield of aspirin58. 42%See #4 in Calculations below

Calculations:

#1)Density = Mass/Volume

Mass (of acetic anhydride) = Density X Volume

= 5. 0mL X 1. 08 g/mL

Mass (of acetic anhydride) = 5. 4 g

#2)Molecular weight of

acetylsalicylic acid (C9H8O4(S)) = molecular weight of its components.

= molecular weight of 9(C)+8(H)+4(O)

= 9(12. 01g/mol) + 8(1. 00g/mol) +

4(16. 00g/mol)

Molecular weight of

acetylsalicylic acid (C9H8O4(S)) = 180. 16 g/mol

#3)Mass of aspirin = (Mass of filter paper + aspirin) – Mass of filter paper

= (2. 103g) – 0. 962g

Mass of aspirin = 1. 141 g

Calculations continued:

#4)# of moles of the limiting reagent = Weight of the limiting reagent (grams) / Molecular weight limiting reagent (grams/mole)

= weight of salicylic acid / molecular weight of salicylic acid

= 1. 498g / 138. 12g/mol

= 0. 01085 moles of salicylic acid

Theoretical Yield = # of moles of limiting reagent X

Of aspirin Molecular weight of aspirin (Acetylsalicylic Acid)

= 0. 01085 mol X 180. 16 g/mol

= 1. 953 g

Percent actual yield of aspirin = (Actual yield of aspirin/ Theoretical Yield of aspirin) X 100%

= (1. 141g/ 1. 953g) X 100%

Percent actual yield of aspirin = 58. 41%

#5)# of moles of salicylic acid = 0. 01085 mol (see Calculation #4 above)

# of moles of acetic anhydride = weight of acetic anhydride / molecular weight of acetic anhydride

= 5. 4 g/ 102. 09 g/mol

= 0. 052 mol

Since: 0. 052 mol > 0. 01085 mol

Therefore:# of moles of acetic anhydride > # of moles of salicylic acid

Discussion

Lab Manual Questions (page 44)

1)The limiting reagent is salicylic acid. From the reaction equation (located in the Introduction of this report) one mole of Salicylic acid reacts with one mole of acetic anhydride to form product. Since there are fewer moles of salicylic acid relative to acetic anhydride (see Calculation #5 above), the

amount of product that can be formed depends on the amount of Salicylic acid, making it the limiting reagent.

The theoretical yield is 1. 935g and the percent yield of the aspirin is 58. 41%. The calculations for the theoretical yield and the percent yield can be seen in the results section above.

2)The acetylsalicylic acid is washed with cold water in order to keep the acetylsalicylic in a solid (precipitate) form. Cold water would lower the kinetic energy of the molecules, slow down their vibrational movement, and allow them to crystallize into a solid. This makes it easier for us to filter and collect the aspirin solid. Warm water would increase the kinetic energy of the aspirin molecules, increase their vibrational speed, and break their crystal structures. This would dissolve the aspirin into solution and make it much more difficult for us to collect and measure the aspirin.

Although aspirin was obtained using recrystallization, the percent yield (58. 41%) was low. This due to the many sources of error present in the lab. For example, residual amounts of aspirin were left over on many of the equipment used, especially on stir rods and the inside of flasks. Even with distilled water, the aspirin was difficult to remove from the filter paper and glass surfaces in steps three to five in the laboratory manual3 (Page 44). Some of it was left over on the paper and glass surfaces. A solution for this is taking more care when transferring the product from the different surfaces. Another solution is that in step 5 of the lab3 (page 44 one could try to wash

down the aspirin with four small 5mL portions of water rather than two large 10 mL portions as stated in the lab manual.

Another source of error is not allowing enough time for the aspirin to recrystallize and precipitate in the ice bath. This would mean that some of the aspirin would be dissolved in solution and not be caught by the filter. A simple solution to this is allowing the aspirin to recrystallize for a longer period without disturbance. One could also add more ice to the ice bath which would cool the solution further and allow precipitate to form faster.

An additional source of error can occur during the drying of the filter paper in step 5 of the lab manual3 (page 44). If one does not dry the paper enough, then the residual moisture on the filter paper could make the weight measurements inaccurate. This can be solved by heating the filter paper for a longer period of time.

Human errors, such as inaccurate measurements due to negligence could also affect the results. The obvious remedy to this problem is to pay attention and try to do the lab work precisely.

Conclusion

Aspirin, also known as acetylsalicylic acid, is derived from an acid catalyzed reaction between salicylic acid and acetic anhydride. This crude product has impurities such as unreacted salicylic acid, acetic acid, and sulfuric acid that need to be separated from the pure aspirin. Recrystallization is a process which allows pure aspirin to precipitate out of this crude product. In ideal

circumstances, the theoretical yield should be 1. 935 grams of aspirin. Yet the actual yield was only 1. 141 grams of aspirin. This gives a low percentage yield of only 58. 41%. Although the method used in the lab works, it is in inefficient process of producing pure aspirin. This is due most likely to the many errors, most of which are solvable, that can occur during the procedures.

Reference

1Shusterman, Alan, Reed College Laboratory Reference, November 20, 2003,.

2Shlafer, Marshal, Ototoxic Drugs, November 20, 2003,

3The University of Winnipeg Department of Chemistry, Laboratory Manual of Introduction to the Chemical Properties of Matter 1111, 2003.