KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University)

(Established Under Section 3 of UGC Act 1956)

Coimbatore – 641 021.

DEPARTMENT OF CHEMISTRY

SYLLABUS

CLASS: II B.SC CHEMISTRY

Batch: 2017

Semester-III

17CHU314A PHARMACEUTICAL CHEMISTRY PRACTICAL 3H 1C

Instruction Hours/week:L:0 T:0 P:03 Marks: Internal: 40 External: 60 Total:100

Scope

The course deals with the experimental skill enhancement techniques in the pharmaceutical chemistry.

Objective

The course enables the student to

1. Understand the synthesis and characterisation of pharmaceutical drugs like aspirin and magnesium bisilicate.

Methodology

Practicals

1. Preparation of Aspirin and its analysis.
2. Preparation of magnesium bisilicate (Antacid).

Suggested Readings

Text Books:

1. Patrick G.L. (1995): Introduction to *Medicinal Chemistry*. UK: Oxford University Press.
2. Hakishan, V.K. Kapoor, (1996) *Medicinal and Pharmaceutical Chemistry*. New Delhi: Vallabh Prakashan. Pitampura.

Reference Books

1. William O. Foye, Thomas L., Lemke ,& David A. William.(2008). *Principles of Medicinal Chemistry*. New Delhi: B.I. Waverly Pvt. Ltd.

KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University)

(Established Under Section 3 of UGC Act 1956)

Coimbatore – 641 021.

DEPARTMENT OF CHEMISTRY

Title of the Paper : **PHARMACEUTICAL CHEMITRY PRACTICAL**

Paper Code : **17CHU314**

Class : II- B. Sc-Chemistry

Year and Semester : 2018–2019 and I-Semester

LIST OF PRACTICALS

S.No.	Duration Hours	Name of the Experiments	Support Materials
1	3	Writing the experimental Procedure	
2	3	Preparation of Aspirin	R1:191
3	3	Melting point analysis of aspirin	R1:191
4	3	Estimation of Aspirin	W1
5	3	Spectroscopy analysis of aspirin	
6	3	Viva-voce questions discussion	
7	3	Preparation of magnesium bisilicate(antacid)	W2
8	3	Melting point analysis of antacid	W2
9	3	Estimation of antacid	W3
10	3	Revision	
11	3	Viva –voce questions discussion	
12	3	Model practical examinations	

Reference Books:

1. O.P.Pandey, D.N.Bajpai,S.Giri. Practical chemistry for B.Sc Students.2001.S.Chand and sons, New Delhi

Website:

W1.http//www. Estimation of Aspirin

W2:http//www.Preparation of antacid

W3: http//www. Estimation of Antacid

DEPARTMENT OF CHEMISTRY

PRACTICAL MANUAL

PHARMACEUTICAL CHEMISTRY

Karpagam Academy of Higher Education (KAHE)

(Deemed to be University Established Under Section 3 of UGC Act, 1956)

Pollachi Main Road,Eachanari Post,

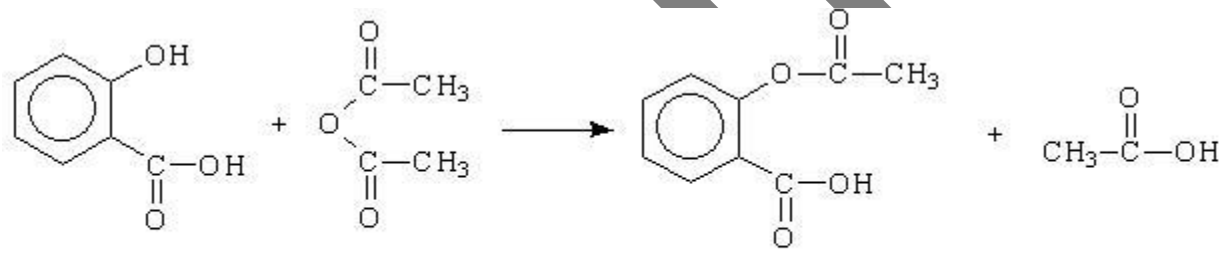
Coimbatore – 641 021,Tamil Nadu,India.

CONTENT

1. PREPARATION OF ASPIRIN

Principle:

To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride.A small amount of a strong acid is used as a catalyst which speeds up the reaction. The synthesis reaction of aspirin is shown below:



Salicylic acid	Acetic anhydride	Acetylsalicylic acid	Acetic Acid
($C_7H_6O_3$)	($C_4H_6O_3$)	($C_9H_8O_4$)	($C_2H_4O_2$)

Requirements

(i) Salicylic acid : 3 g

(ii) Acetic anhydride : 6 ml

(ii) Phosphoric acid 85% : 5-8 drops

Procedure:

Accurately weigh 3.00 grams of salicylic acid and transfer to a dry Erlenmeyer flask. Add 6 mL of acetic anhydride and 5-8 drops of 85% phosphoric acid to the flask. Gently swirl the flask to mix the solution. Place the flask in a beaker of warm water for 15 minutes. Add 20 drops of cold water drop wise to the warm solution to destroy the excess acetic anhydride. Add 20 mL of water to the flask. Set the flask in an ice bath to cool the mixture and speed crystallization. When the crystallization process

appears complete, pour the mixture through a Buckner funnel. Apply suction filtration through the funnel and wash the crystals with a few milliliters of ice cold water. Be sure the water is near freezing to minimize loss of product. Perform a recrystallization to purify the product. Transfer the crystals to a beaker. Add 10 mL of ethanol. Stir and warm the beaker to dissolve the crystals. After the crystals have dissolved, add 25 mL of warm water to the alcohol solution. Cover the beaker. Crystals will reform as the solution cools. Once crystallization has started, set the beaker in an ice bath to complete the recrystallization. Pour the contents of the beaker into a Buckner funnel and apply suction filtration. Remove the crystals to dry paper to remove excess water.

Result

Yield of aspirin…….g

2. MELTING POINT ANALYSIS OF ASPIRIN

Principle

Melting point (mp) is the temperature at which a solid becomes a liquid at standard atmospheric pressure; at this point, solid and its liquid are in equilibrium at a certain pressure. Melting point is one of physical properties of a compound by which it is identified.

Measuring the melting point of a substance is a good way to test for purity. A pure substance usually has a sharp melting point – *ie*a narrow temperature range during which it changes from a solid to a liquid. A substance which contains impurities often melts over a range of several degrees. Any impurities in the substance cause a lowering and broadening of this characteristic temperature.The melting point range of pure aspirin and the salicylic acid are

Substance	Melting point
2-Hydroxybenzoic acid	158–160 °C
Aspirin	138–140 ° C

Requirements

(i) Melting point tubes

(ii) Watch glass

(iii) Melting point apparatus

(iv) 0-360 °C thermometer

Procedure

Weigh the dry product to obtain the yield of the reaction. Pack a few crystals of aspirin product in a melting point capillary tube. The melting point tube is attached to the thermometer and place tube in the melting point apparatus.Allow the temperature of the melting point apparatus to increase 1 °C per minute starting from 120 °C. If impurities are present in crude sample, the melting point range will be lower than the range of pure aspirin. Record the melting point ranges of the pureaspirin.

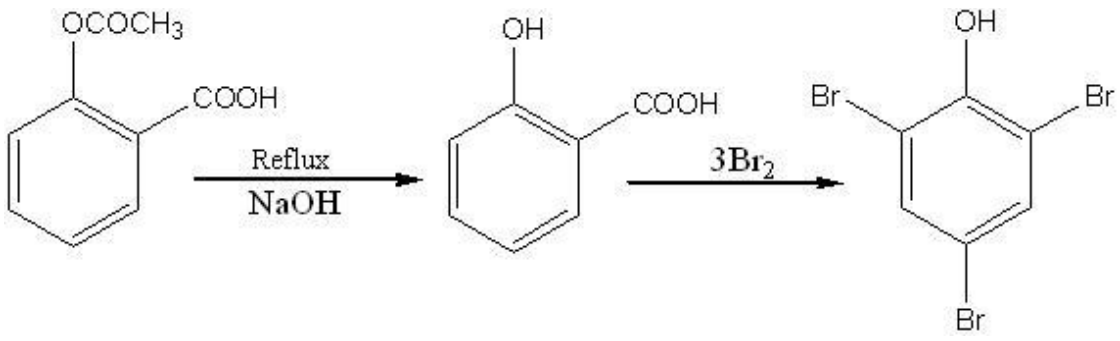
Result

Melting point of aspirin experimentally found to be……..

3. ESTIMATION OF ASPIRIN

Principle and Procedure:

The amount of aspirin can be determined by brominating using $KBrO_3$-KBr mixture. A definite amount of aspirin is refluxed with NaOH. Then salicylic acid is formed. The excess brominating mixture formed is titrated with standard thio.



Preparation of KBr-KBrO3 solution:- Dissolve 75 g KBr & 5.36 g of $KBrO_3$ in H_2O and make upto 1litre.

a) Standardisation of $Na_2S_2O_3$:

0.5 g $K_2Cr_2O_7$ is weighed accurately and made upto 100 mL. 20 mL is pipetted out into a conical flask. Then add 3 mL con.HCl followed by 5mL 10% KI and titrated against $Na_2S_2O_3$ using starch as indicator.

b) Estimation of aspirin:

1.5g aspirin is weighed out into an R.B flask. Then 40 mL 10% NaOH is added and refluxed for 15 min. Transfer the solution quantitatively into a 250 mL standard flask, made upto the mark. From that 20 mL is pipette, acidified with 2 mL con. HCl. Then add 50 mL of brominating mixture, shake well for 15min. Then 10 mL 10% KI is added & diluted with H_2O and titrated against standard $Na_2S_2O_3$ using starch as indicator.

Calculation:

Normality of thio = N_1
Weight of aspirin = W g
50 mL brominating mixture = X mL thio
20 mL aspirin + 50 mL brominating mixture = Y mL
Amount of thio = X - Y = Z mL
Normality of aspirin = Z x N_1 / 20 = N_2

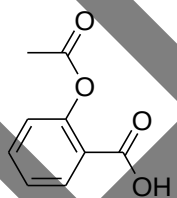
Amount of aspirin in the whole of the given solution = (N_2 x Equivalent weight of aspirin) / 4 = A g

% of aspirin = (A x 100) / W = B %

4. SPECTROSCOPY ANALYSIS OF ASPIRIN

Principle

Infrared spectroscopy (IR) is a characterization tool chemists use to help determine the molecular structure. IR capitalizes on the concept that functional groups absorb specific frequencies of energy based on their structure. Aspirin, or acetylsalicylic acid, has three functional groups. A benzene ring (aromatic group), A carboxylic acid (COOH) group An ester (R-C=O--O-R) group.



aspirin

Requirements

(i) IR instrument

(ii) KBr

(iii) Mortar and pestle

Procedure

In this technique a small amount of finely ground solid sample of aspirin is intimately mixed with about 100 times its weight of powdered potassium bromide, in a vibrating ball mill. This finely ground mixture is then under very high pressure in evacuable die or minipress to form a small pellet (about 1-2 mm thick and 1 cm diameter). A good KBr pellet is thin and transparent. Opaque pellets give poor spectra, because little infrared beam passes through them. White spots in a pellet indicate that the powder is not ground well enough, or is not dispersed properly in the pellets. The resulting pellet is transferred in to IR radiation and run. Predict the important functional groups of aspirin.

Result

The O-H stretch for a carboxylic acid is found in the range …… cm^{-1}

The Carbonyl (C=O) group stretch found in the range…… cm^{-1}

5. PREPARATION OF MAGNESIUM BISSILICATE

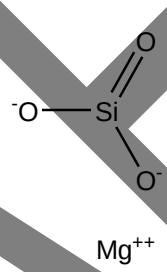
Principle

The most common route for the synthesis of magnesium silicate is via aprecipitation reaction between a soluble metal silicate (e.g., sodium orthosilicate, sodium metasilicate, or potassium silicate) and a soluble magnesium salt (e.g., magnesium sulfate, nitrate, or chloride).

$$Na_2SO_3 + MgCl_2 \xrightarrow{H_2O} MgSO_3 + 2NaCl$$

Sodium sillicate — Magnesium chloride — Magnesium sillicate

Magnesium silicate occurs as a fine, white, odorless, tasteless powder, free from grittiness.



A schematic of the structure of magnesium silicate

Requirements

(i) Sodium silicate

(ii) Magnesium chloride

(iii) Conical flask 250 ml

(iv) Measuring cylinder

Procedure

Accurately weigh 1.2 grams of sodium silicate and transfer to a dry Erlenmeyer flask containing 20 ml of distilled water. Add 0.96 g of magnesium chloride to the flask. Gently swirl the flask to mix the solution. Immediately, white odorless magnesium silicate is precipitated. The precipitate is further

stirred continuously for 5 minutes.The precipitated magnesium silicate is filtered, washed with excess amount of water and dried. The yield is noted.

Result

Amount of the magnesium silicate obtained……g

6. MELTING POINT ANALYSIS OF MAGNESIUM SILICATE

Principle

Melting point is the temperature at which a solid becomes a liquid at standard atmospheric pressure; at this point, solid and its liquid are in equilibrium at a certain pressure. Melting point is one of physical properties of a compound by which it is identified.

Measuring the melting point of a substance is a good way to test for purity. A pure substance usually has a sharp melting point – *ie*a narrow temperature range during which it changes from a solid to a liquid. A substance which contains impurities often melts over a range of several degrees. Any impurities in the substance cause a lowering and broadening of this characteristic temperature. The melting point range of sodium silicateand the magnesium chloride are

Substance	Melting point
Sodium silicate	1088 °C
Magnesium chloride	714 °C

Requirements

(i) Melting point tubes

(ii) Mortar and pestle

(iii) Melting point apparatus

(iv) 0-360 °C thermometer

Procedure

Weigh the dry product to obtain the yield of the reaction. Pack a grinded powder of magnesium silicate product in a melting point capillary tube. The melting point tube is attached to the thermometer

and place tube in the melting point apparatus. Allow the temperature of the melting point apparatus to increase 1 °C per minute starting from 120 °C. If impurities are present in the crude sample, the melting point range will be lower than the range of pure magnesium silicate. Record the melting point ranges of the pure magnesium silicate.

Result

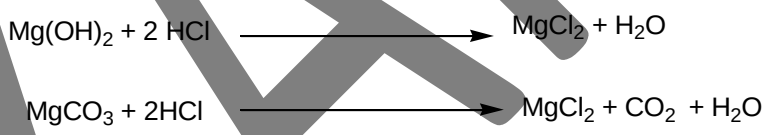
Melting point of magnesium silicate experimentally found to be……..

7. Titration of Antacids

Introduction

The purpose of this experiment is to determine the amount of solid neutralized by various commercial antacids. These products contain bases such as calcium carbonate, magnesium chloride and magnesium hydroxide. The later compounds are not very soluble in water, but direct titration can be carried out with HCl if sufficient time is allowed for the reaction between the solid and the titrant. A recurring and point may be obtained because this reaction is rather slow.

In the procedure below, excess acid is added to react with the antacid, the solution is heated to remove CO_2, and the excess acid is titrated with standard base. Phenolphthalein can be used as an indicator and reasonably sharp end point is obtained.



Procedure:

Take half a tablet of antacid and weigh on analytical balance. Tranfer the samples to 250 ml Erlenmeyer flask. Add 50 ml of standard 0.1 M HCl. Heat the solution to boiling then boil it gently for about 3 minutes. Cool the solution to room temperature. Add 4 drops o0f the indicator and titrate with standard base to the first permanent pink color. Calculate the grams of HCl neutralized by 1 g of antacid. Assume that 0.1M HCl has density of 1.00g/ml. Calculate the grams of 0.1m HCl solution neutralized by 1 g of the antacid

Report:

The grams of HCl neutralized by 1 g of antacid =----------g