KARPAGAM ACADEMY OF HIGHER EDUCATION
(Deemed to be University)
(Established Under Section 3 of UGC Act 1956)
Coimbatore – 641 021.

SYLLABUS

DEPARTMENT OF CHEMISTRY

STAFF NAME: Dr. A. THANGAMANI

SUBJECT NAME: ORGANIC SPECTROSCOPY PRACTICAL

SUB.CODE:17CHU413

SEMESTER: IV CLASS: II B.Sc (CHEMISTRY)

17CHU413 ORGANIC SPECTROSCOPY PRACTICAL 4H 2C

Instruction Hours/week:L:0 T:0 P:4 Marks: Internal: 40 External: 60 Total:100

Scope

This lab course deals with the spectroscopy practical's involving the Identification of simple organic compounds by IR spectroscopy and NMR spectroscopy, qualitative analysis of organic compounds preparation of formaldehyde resin, extraction of caffeine and preparation of methyl orange.

Objectives

The lab course enables the student to

1. Extraction of caffeine from tea leaves.
2. Preparation of urea formaldehyde resin.
3. Understand the qualitative analysis of unknown organic compounds.
4. Identify simple organic compounds by IR spectroscopy and NMR spectroscopy.
5. Prepare of methyl orange.

Methodology

Spectroscopic methods UV, IR and NMR.

1. Extraction of caffeine from tea leaves.
2. Preparation of urea formaldehyde resin.
3. Qualitative analysis of unknown organic compounds containing monofunctional groups (carbohydrates, aryl halides, aromatic hydrocarbons, nitro compounds, amines and amides) and simple bifunctional groups, e.g. salicylic acid, cinnamic acid, nitrophenols etc.
4. Identification of simple organic compounds by IR spectroscopy and NMR spectroscopy (Spectra to be provided).
5. Preparation of methyl orange.

Suggested Readings:

Text Books:

1. Vogel, A.I. (2012). *Quantitative Organic Analysis*. Part 3. Pearson.
2. Mann, F.G. & Saunders, B.C. (2009). *Practical Organic Chemistry*. Pearson Education
3. Furniss, B.S., Hannaford, A.J., Smith, P.W.G. & Tatchell, A.R. (2012). *Practical Organic Chemistry*. 5th Ed. Pearson.

Reference Books:

1. Ahluwalia, V.K. & Aggarwal, R. (2000*). Comprehensive Practical Organic Chemistry: Preparation and Quantitative Analysis.* University Press.
2. Ahluwalia, V.K. & Dhingra, S. (2000). *Comprehensive Practical Organic Chemistry: Qualitative Analysis*. University Press.

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LECTURE PLAN

DEPARTMENT OF CHEMISTRY

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S.No.	Lecture Duration Period	Topics to be Covered	Support Material/Page Nos.
1	4	Extraction of caffeine from tea leaves and identification by spectroscopic methods.	T1:162
2	4	Preparation of urea formaldehyde resin and identification by spectroscopic methods.	T1: 316
3	4	Qualitative analysis (spectroscopic methods) of unknown organic compounds containing monofunctional groups (carbohydrates and aryl halides).	T1:392
4	4	Qualitative analysis (spectroscopic methods) of unknown organic compounds containing monofunctional groups (aromatic hydrocarbons and nitro compounds).	T1:281 & 313
5	4	Qualitative analysis (spectroscopic methods) of unknown organic compounds containing monofunctional groups (amines and amides).	T1:295 & 309
6	4	Viva-voce questions discussion.	
7	4	Qualitative analysis (spectroscopic methods) of unknown organic compounds containing simple bifunctional groups (salicylic acid,	T1:303

		cinnamic acid and nitrophenols).	
8	4	Identification of simple organic compounds by IR spectroscopy and NMR spectroscopy (Spectra to be provided).	T1:272-273 & 330-332
9	4	Preparation of methyl orange and identification by spectroscopic methods.	T1:340
10	4	Model Practical Examination.	
	Total No. of Hours Planned For Practical's = 40		

Suggested Readings:

Text Book:

1. Vogel, A.I. (2012). *Quantitative Organic Analysis*. Part 3. Pearson.

Experiment. No: 1

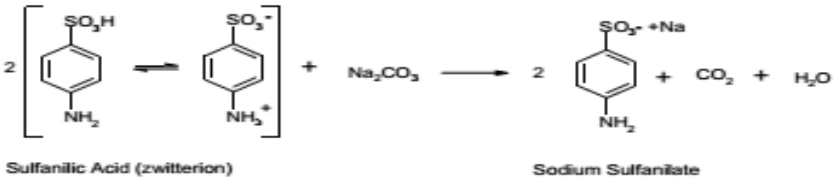
PREPARATION OF METHYL ORANGE

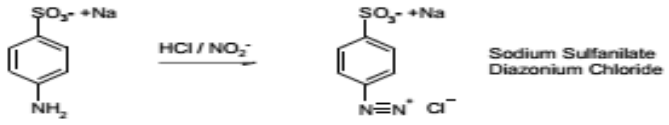
Aim:

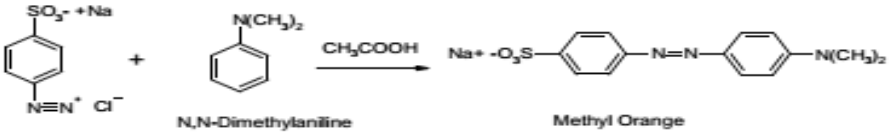
To prepare a pure sample of Methyl Orange by treating sulphanilic acid and dimethylaniline.

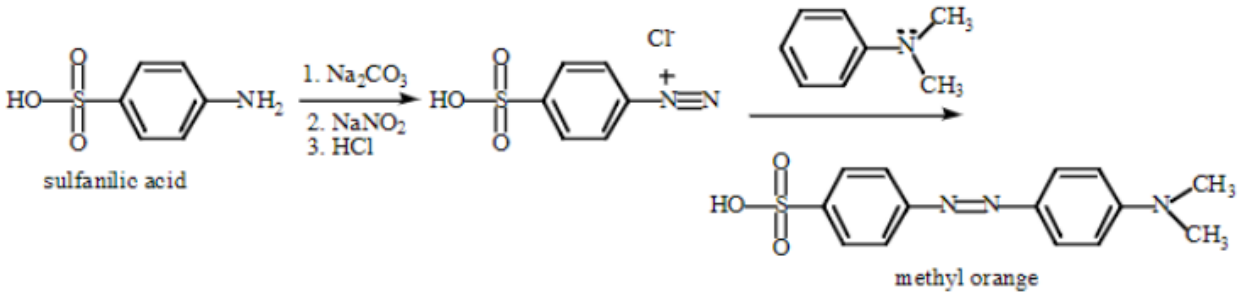
Principle:

Sulphanilic acid is diazotized by allowing sodium salt to react with a mixture of sodium nitrate and HCl at ice cool condition. This diazotized salt readily complexes with nitrogen, N-Dimethyl Aniline to yield methyl orange. It is precipitated as it sodium salt by adding sufficient quantity of sodium hydroxide solution.









Chemicals Required:

Sulphanilic acid, Anhydrous sodium carbonate, Sodium nitrite, Con.HCl, Dimethyl aniline, Acetic Acid, 20% NaOH , Sodium chloride

Procedure

Diazotized Sulfanilic Acid

Dissolve 0.29 g of anhydrous sodium carbonate in 12.5 mL of water. Use a 50 mL Erlenmeyer flask. Add 1.0 g of sulfanilic acid monohydrate to the solution and heat it until it dissolves. A small amount of suspended material may render the solution cloudy. Gravity-filter the still hot solution and rinse the used filter paper with a little (0.5 -1.0 mL) hot water. Cool the filtrate to room temperature, add 0.375 g of sodium nitrite, and stir until solution is complete. Pour this mixture, while stirring, into a 100-mL beaker containing 8 mL of ice water to which 1.25 mL of concentrated hydrochloric acid have been added; add the HCl dropwise to maintain a temperature of 0-5 ^{o}C. The diazonium salt of sulfanilic acid should soon separate as a finely divided white precipitate. Keep this suspension cooled in an ice bath until it is to be used.

Preparation of Methyl Orange

In a test tube, mix together 0.7 mL of dimethylaniline and 0.5 mL of glacial acetic acid. Add this solution dropwise to the cooled suspension of diazotized sulfanilic acid in the 100-mL beaker. Stir the mixture vigorously. In a few minutes, a red precipitate of helianthin should form. Keep the mixture cooled in an ice bath for about 15 minutes to ensure completion of the coupling reaction. Add 7.5 mL of a 10% aqueous sodium hydroxide (NaOH) solution. Do this slowly and with stirring, as you continue to cool the beaker in an ice bath. Check with litmus or pH paper to make sure the solution is basic. If it is not, add more base. Heat the mixture to boiling with a Bunsen burner for 10 to 15 minutes to dissolve most of the newly formed methyl orange. When all (or most of it) the dye is dissolved, add 2.5 g of sodium chloride, and cool the mixture in an ice bath. The methyl orange should recrystallize. Filter using a Buchner funnel.

To purify the product, transfer the filter cake and paper to a large beaker containing about 40 mL of boiling water. Maintain the solution at a gentle boil for a few minutes, stirring it constantly. (Note: Not all the dye will dissolve, but the salts with which it is contaminated will

dissolve.) Remove the filter paper and allow the solution to cool to room temperature. Cool the mixture in an ice bath, and when it is cold, collect the product by vacuum filtration, using a Buchner funnel. Allow the product to dry, weight it, and calculate the percentage yield. Note: The product is a salt. Since salts do not generally have well-defined melting points, the melting-point determination should not be attempted. The sample gives the NMR analysis.

Recrystallization

About 1g of the sample is recrystallized from hot water. Red orange crystals of methyl orange are separated and dried.

Report:

The yield of the methyl orange = ----------

Experiment. No: 2

PREPARATION OF UREA FORMALDEHYDE RESIN

AIM: -

To prepare urea formaldehyde and phenol formaldehyde resins.

Apparatus required: -

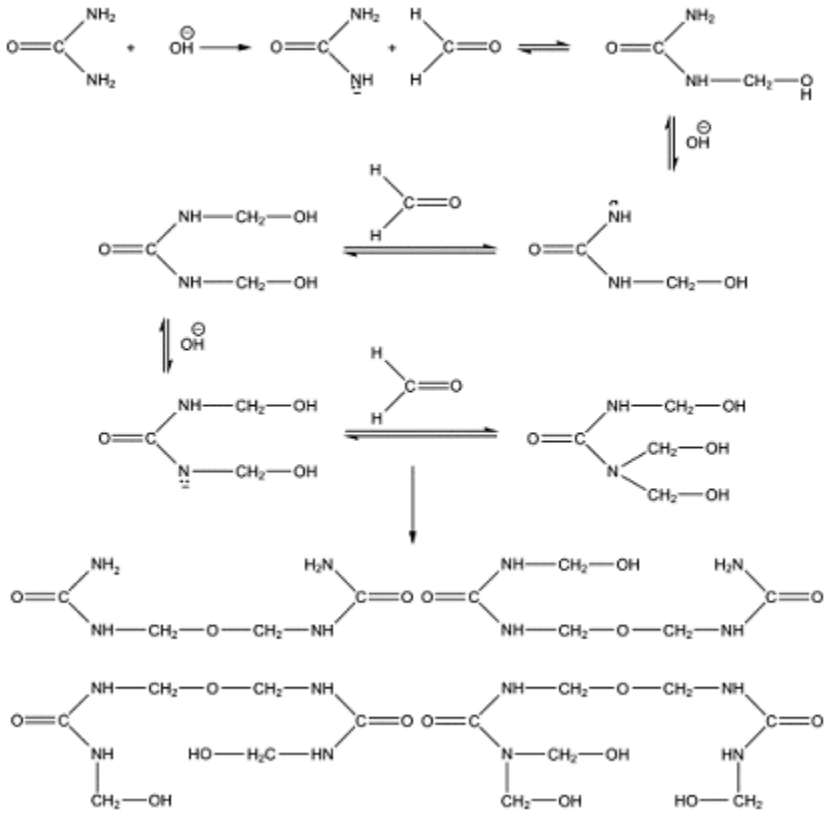
Beaker, glass rod, funnel, filter paper and chemical balance.

Chemicals: -

Urea, formaldehyde sol., conc. H2SO4, distilled water.

Principle:

Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium. The first product formed during the formation of resin is monomethylol and dimethylol ureas.



Procedure:

1. Place about 5 ml of 40% formaldehyde solution in 100 ml beaker.
2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
3. Add a few drops of conc. H_2SO_4, with constant stirring.
4. A voluminous white solid mass appears in the beaker.
5. Wash the white solid with water and dry it in the folds of filter paper.
6. Weight the yield of product

Precautions:-

1. While adding concentrated H_2SO_4, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

Observations:-

Mass of the beaker (W1) = ---------g.

Mass of the beaker with urea formaldehyde (W2) = --------g.

Therefore mass of urea formaldehyde (W2 – W1) = --------g.

Result:-

The yield of urea formaldehyde = -------g

Experiment. No: 3

EXTRACTION OF CAFFEINE FROM TEA LEAVES

Aim:

To extract Caffeine from Tea leaves– Caffeine is an alkaloid.

Materials Required:

Dichloromethane, sodium carbonate, Anhydrous sodium sulfite, Distilled water, Whatmann filter paper no.40, funnel, glass rod, Tea leaves, Melting point apparatus.

Procedure:

Tea bags or tea leaves are used as the source of caffeine for this experiment.

1. Take 5 tea bags and record the weight of these tea bags.
2. Take 500 ml beaker adds 200 ml of distilled water to it. Now place the 5 tea bags in this beaker.
3. Boil the contents in the beaker vigorously using a hot plate.
4. Allow the mixture to cool for 5 minutes and then decant the mixture into another beaker.
5. Gently squeeze the tea bags to liberate the rest of the water.
6. Cool the aqueous solution to near room temperature.
7. Continue cooling in an ice box, the tea must be cool (20° C) before coming in contact with dichloromethane (boiling point = 40° C).
8. Extract the solution three times with 30-mL portions of dichloromethane (CH_2Cl_2). Do not get dichloromethane on your hands.

Extraction step:

The tea solution is poured into a separating funnel and 20ml of dichloromethane is added to it. The mixture will separate into two layers - the top layer is the tea layer and bottom layer is the dichloromethane since it is denser than tea. Remove the funnel from the stand and keep your fingers on the stopper and carefully shake the separating funnel. Vent the separating funnel periodically (every 30 sec) to relieve vapour pressure created inside the funnel. When the contents have been sufficiently shaken place the separating funnel back on the ring stand and let the two layers separate. Drain the bottom layer into a conical flask because now the caffeine is

extracted into the dichloromethane layer. Cover the mouth of the conical flask to avoid evaporation of solution. Repeat steps a) through e) twice.

Dry the combined dichloromethane solutions with anhydrous Sodium sulfite. Add about 1 teaspoon of the drying agent until it no longer clumps together at the bottom of the flask. Mix well and leave it for 10 minutes. Decant the dichloromethane into a conical flask (100ml). Evaporate the dichloromethane solvent in a hot water bath. When all the solvent is removed you observe a residue of yellowish green - white crystalline caffeine.

Sublimation step:

a. Take the conical flask containing crystalline caffeine.

b. Sublime the crude caffeine at atmospheric pressure by placing the flask directly on a pre-heated hot plate. Caffeine melts at 238°C and sublimes at 178°C.

c. Collect your sublimed caffeine by keeping a test tube on the mouth of the conical flask.

d. White vapour of caffeine sticking onto the test tube and the walls of the conical flask is observed.

e. Now cool the conical flask.

Take a clean watch glass and record its weight in a weigh balance.

Now strip off the caffeine from the conical flask and the walls of the test tube into the watch glass using a spatula.

Record the weight of the watch glass + caffeine in a weigh balance and then find out the weight of extracted pure caffeine.

The melting point of the extracted caffeine is determined using the melting point apparatus.

Report

Yield of the Product = ----------

Experiment. No: 4

IDENTIFICATION SIMPLE ORGANIC COMPOUNDS USING IR SPECTROSCOPY

Aim:

To identify the simple organic compounds using IR spectroscopy technique.

Materials Required:

KBr, FT-IR instrument.

Principle:

FTIR relies on the fact that the most molecules absorb light in the infra-red region of the electromagnetic spectrum. This absorption corresponds specifically to the bonds present in the molecule. The frequency range are measured as wave numbers typically over the range 4000 – 600 cm^{-1}.

Procedure:

KBr Pellet procedure for solid samples

Take about 1/8" of the solid sample on a micro spatula and about 0.25-0.50 teaspoons of KBr. Mix thoroughly in a mortar while grinding with the pestle. If the sample is in large crystals, grind the sample separately before adding KBr. Place just enough spl. to cover bottom in pellet die. Place in press and press at 5000-10000 psi. Check pellet press brochure for details. Carefully remove the pressed sample from die and place in the FTIR sample holder. The pressed disc should be nearly clear if properly made. If it is translucent, regrind and repress.

Turn on the IR spectrometer and allow it to warm up.Obtain an unknown sample from the instructor and record the letter and appearance of the sample.Collect a background spectrum.Using a metal spatula, place a small amount of sample under the probe.Twist the probe until it locks into place.Record the IR spectrum of the unknown sample.Repeat if necessary to obtain a good quality spectrum.Record the absorption frequencies indicative of the functional groups present.Clean the probe with acetoneTurn off the spectrometer

Characteristic Stretching Frequencies: The Five Zones (Table)

Bond	Stretching Frequency (cm^{-1})	Intensity and Shape
	ZONE 1: 3700-3200 cm^{-1}	
Alcohol O-H	3650-3200 cm^{-1}	Usually strong and broad
Alkyne ≡C-H	3340-3250	Usually strong and sharp
Amine or Amide N-H	3500-3200	Medium; often broad
	ZONE 2: 3200-2700 cm^{-1}	
Aryl or Vinyl sp^2 C-H	3100-3000	Variable
Alkyl sp^3 C-H	2960-2850	Variable
Aldehyde	~2900 and ~2700	Medium; two peaks
Carboxylic Acid O-H	3000-25000	Usually strong; very broad
	ZONE 3: 2300-2000 cm^{-1}	
Alkyne C≡C	2260-2000	Variable and sharp
Nitrile C≡N	2260-2220	Variable and sharp
	ZONE 4: 1850-1650 cm^{-1}	
Ketone C=O	1750-1705	Strong
Ester C=O	1750-1735	Strong
Aldehyde C=O	1740-1720	Strong
Carboxylic Acid C=O	1725-1700	Strong
Amide C=O	1690-1650	Strong
	ZONE 5: 1680-1450 cm^{-1}	
Alkene C=C	1680-1620	Variable
Benzene Ring C=C	~1600 and ~1500	~1600 often has 2 peaks