# PRACTICAL

# MEDICINAL CHEMISTRY

## -I-

JERASH UNIVERSITY
FACULTY OF PHARMACY
PRACTICAL MEDICINAL CHEMISTRY I
COURSE OUTLINE
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LAB I:- Assay of Ammonium Chloride.

LAB II:- Assay of Ferrous Sulphate.

LAB III:- Assay of Promethazine.HCl Syrup.

LAB IV:- Assay of Mefenamic acid Tablets.

LAB V:- Assay of Ibuprufen Tablets.

LAB VI:- Assay of Aspirin Tablets and Salicylic acid in Aspirin.

LAB VII:- Assay of Frusemide Tablets.

LAB VIII:- Assay of Nalidixic acid Tablets.

LAB IX:- Assay of Dimenhydrinate Tablets.

LAB X:- Assay of Chlorpropamide Tablets.

Reports:- 10 marks
Quizes:- 10 marks
Mid-Term exam:- 30 marks
Final-Term exam:- 50 marks

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MEDICINAL CHEMISTRY I
LAB.I

# ASSAY AND IDENTIFICATION OF AMMONIUM CHLORIDES

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## PART I-IDENTIFICATION TEST FOR CHLORIDE ION:-

1- Make solution of 0.1 gm Ammonium chloride in water
2- Acidify with 2M Nitric acid
3- Add 0.4 ml of silver nitrate solution
4- Shake and allow to stand, see the result and explain.

## PART II-ACIDITY OR ALKALINITY OF AMMONIUM CHLORIDE:-

1-Dissolve 1 gm of Ammonium chloride in 10 ml of distilled water
2-Add 0.05 ml of Methyl red
3- Add 0.5 ml of 0.01M HCl, note the result
4- Add 1 ml of 0.01M NaOH, note the result
5- Write your conclusion about acidity or alkalinity of your solution.

## PART III- ASSAY OF AMMONIUM CHLORIDE:-

1- Dissolve 1 gm of Ammonium Chloride in 20 ml of water
2- Add a mixture of 5 ml of Formaldehyde previously neutralized to Phenolphthalein solution and 20 ml of water
3- Allow to stand for 1-2 mins
4- Titrate slowly with 1M NaOH, using 0.2 ml of Phenolphthalein solution as an indicator.

The official standard of Ammonium chloride row material is 99.0-100.5% according to the BP.

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LAB.II

# ASSAY AND IDENTIFICATION OF $FeSO_4.7H_2O$

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## PART I- IDENTIFICATION OF IRON:-

1-Take about 10 mg of the salt. Dissolve in 1 ml of water
2- Add 1 ml of Potassium hexacyano ferrate(III) solution
3- Note the result. Add 2M HC1 and see if any changes occur.

## PART II- IDENTIFICATION OF SULPHATE:-1-

Take about 45 mg of the salt. Dissolve in 5 ml of water
2- Add 1 ml of 2M HC1 and 1 ml of Barium Chloride solution. Note the result.

## PART III- ASSAY OF $FeSO_4.7H_2O$:-

1- Dissolve 2.5 gm of sodium hydrogen carbonate in a mixture of 150 ml water and 10 ml Sulphuric acid
2- When effervescence ceases, add 0.5 gm of ferrous sulphate
3- Shake gently to dissolve and titrate with 0.1M Ammonium cerium (IV) nitrate using 0.1 ml of Ferroin solution as indicator.

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LAB.III

# ASSAY OF PROMETHAZINE.HCL SYRUP

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Procedure:-

1- Take 10 ml of the syrup to be assayed volumetrically.
2- Add 5 ml of water and shake for 5 min.
3- Add 1.5 ml of 1.2N NaOH and shake for 5 min.
4- Extract with 2 successive quantities of ether, 7.5 ml each
5- Filter the organic layer then evaporate to dryness
6- Add 2.5 ml of alcohol to the residue and shake well to dissolve
7- Add 5 ml of 0.1 HC1 volumetrically
8- Titrate the excess acid with 0.1N NaOH using methyl red indicator
9- Carry out the blank titration.
10- Calculate the amount of Promethazine.HCl in your sample.

Content of Promethazine.HCl should be 90-110% of the prescribed amount.

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LAB.IV

# ASSAY AND IDENTIFICATION OF MEFENAMIC ACID CAPSULES

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## PART I- IDENTEFICATION:-

I- Dissolve 25 mg of Mefenamic acid in 15 ml and Chloroform and examine under UV light (365 nm), the solution exhibits a strong greenish-yellow Fluorescence, carefully add 0.5 ml of a saturated solution of trichloroacetic acid drop wise and examine under UV (365 nm) light, no fluorescence is seen

II- Dissolve 5 mg in 20 ml sulfuric acid and add 0.05 ml of 0.0167M potassium dichromate, an intense blue color is produced immediately, and it fades rapidly to brownish-green.

## ASSAY OF MEFENAMIC ACID:-

1- Dissolve a quantity containing 250 mg of the drug in 50 ml of warm absolute ethanol previously neutralized to phenol red solution

2- Titrate with 0.1M sodium hydroxide using phenol red solution as indicator.

Each 1 ml of 0.1M sodium hydroxide is equivalent to 24.13 mg of $C_{15}H_{15}NO_2$

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LAB.V

# ASSAY OF IBUPROFEN TABLETS

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Procrdure:-

1- Weigh and powder 20 tablets
2- Extract a quantity of tablets equivalent to 0.4 gm Ibuprofen with 20 ml of Chloroform for 15 min.
3- Filter under reduced pressure
4- Wash the residue with two 5 ml quantities of Chloroform
5- Evaporate the combined filtrate just to dryness in a current of air
6- Dissolve the residue in 80 ml of ethanol (previously neutralized to phenolphthalein indicator)
7- Titrate with 0.1M NaOH using 0.2 ml of phenolphthalein as an indicator
8- Repeat the same procedure with out the substance
9- The difference between the two titrations represents the amount of NaOH reacted with the drug.

Each 1 ml of 0.1M NaOH is equivalent to 20.63 mg Ibuprofen.

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LAB.VI

## ASSAY OF ASPIRIN TABLETS

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Procedure:-

1- Weigh and powder 20 tablets
2- To a quantity of the powder containing 0.3 gm of Aspirin add 30 ml of 0.5M sodium hydroxide VS
3- Boil gently for 10 min.
4- Titrate the excess of alkali with 0.5M HC1 VS, using phenol red solution as indicator
5- Repeat the operation with out the substance being examined
6- The difference between the titration represents the amount of sodium hydroxide required.

Each ml of 0.5M sodium hydroxide VS is equivalent to 45.04 mg of $C_9H_8O_4$.

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## ASSAY OF SALICYLIC ACID IN ASPIRIN TABLETS

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1- Shake a quantity of powdered tablets containing 0.2 gm of Aspirin in 4 ml of ethanol and dilute to 100 ml at a temperature not exceeding 10 ° C
2- Filter immediately and transfer 50 ml of the filtrate to Nessler cylinder
3- Add 1 ml of freshly prepared Iron III sulphate R1. Mix and allow to stand for 1 min
4- Any violet color produced is not more than that obtained by adding 1 ml of freshly prepared Ammonium iron III sulphate solution R1 to a mixture of 3 ml of freshly prepared 0.01% W/V solution of salicylic acid, 2 ml ethanol 96 % and sufficient water to produce 50 ml in a second Nessler tube.

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LAB.VII

# ASSAY OF FRUSEMIDE TABLETS

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Procedure:-

1- Shake a quantity of the powder containing 0.04 ofFrusemide with 60 ml of 0.Lm NaOH for 10 min
2- Add sufficient 0.1M NaOH to produce 100 ml and filter.
3- Dilute 1 ml to 50 ml with 0.1M NaOH
4- Measure the absorbance at the maximum at 271 nm

Calculate the content of $C_{12}H_{11}ClN_2O_5S$ taking 580 as the value of A (1% ,1cm) at 271 nm.

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LAB.VIII

# ASSAY OF NALIDIXIC ACID TABLETS

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Procedure:-

1- Weigh and powder 20 tablets of Nalidixic acid
2- To a quantity containing 50 mg ofNalidixic acid add 75 ml of 1M NaOH
3- Shake for 3 min.
3- Dilute to 100 ml with 1M NaOH, mix, allow to stand for 15 min.
4- Take 1 ml and dilute to 100 ml with water
5- Measure the absorbance at 334 nm taking 0.01M NaOH as a reference

Calculate the content of $C_{12}H_{12}N_2O_3$ as the value of A (l%, 1cm) at 334 nm.

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LAB.IX

# ASSAY OF DIMENHYDRINATE TABLETS

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Procedure:-

1- Weigh and powder 20 tablets
2- Dissolve a quantity of the powder containing 0.1 gm of Dimenhydrinate as completely as possible in 20 ml of water
3- Add 10 ml of 5M Ammonia, mix, extract with successive quantities of 15,15,15,10 and 10 ml of ether and wash with the combined extracts with 10 ml water
4- Evaporate the ether, warm the residue with 10 ml of Ethanol 96%, until dissolved, cool,add 50 ml of 0.01M HC1 VS
5- Titrate the excess of acid with 0.01M NaOH VS using methyl red as indicator.

Each ml of 0.01M HC1 is equivalent to 4.7 mg of $C_{17}H_{12}NO$,$C_7H_7Cl\ N_4O_2$

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LAB.X

# ASSAY OF CHLORPROPAMIDE TABLETS

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Procedure:-

1- Weigh and powder 20 tablets
2- Shake aquantity of the powder containing 0.25 gm of Chlorpropamide with 40 ml Methanol for 20 min.
3- Add sufficient Methanol to produce 50 ml, mix, filter and dilute 5 ml of the filtrate to 100 ml with 0.1M HCl.
4- Dilute 4 ml of this solution to 100 ml with 0.1M HC1
5- Measure the absorbance of the resulting solution at the maximum at 232 nm

Calculate the content of $C_{10}H_{13}ClN_2O_3S$ , taking 598 as the value of $E_1^{\%}$.

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LAB.XI

# Assay of Propranolol Tablets

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1- Weight and powder 20 tablets.
2- Shake a quantity of powder containing 20 mg of Propranolol Hydrochloride with 20 ml of water for 10 minutes.
3- Add 50 ml of methanol, shake for a further 10 minutes, add sufficient methanol to produce 100 ml and filter.
4- Dilute 10 ml of the filtrate to 50 ml with methanol
5- Measure the absorbance of the resulting solution at the maximum at 290 nm.
6- Calculate the content of $C_{16}H_{21}NO_2$,HCl taking 206 as the value of A ( 1% , 1 cm ) at the maximum at 290 nm.