

# **Bhakta Kavi Narsinh Mehta University**



**BSc Semester VI**

**CBCS**

**Chemistry Practicals**

**With effect from June - 2018**

**Instructions to Examiners**

## **CHEMISTRY PRACTICALS: SEMESTER VI**

**[Total: 150 Marks]**

- **Internal Evaluation [CCA]: 45 Marks**
- **External Evaluation [SEE]: 105 Marks**

### **CCA (Internal Evaluation): Total 45 Marks**

Equal weightage to all Sections i.e.

- Organic Chemistry : 15 Marks
- Inorganic Chemistry : 15 Marks
- Physical Chemistry : 15 Marks

### **SEE (External Evaluation): Total 105 Marks**

- Three Practical Exercises are to be given one from each section- Organic, Inorganic and Physicochemical Exercises.
  - Marks Distribution for Practicals Examination :
    - Organic Synthesis : 30 Marks
    - Inorganic Qualitative Analysis : 40 Marks
    - Physicochemical Exercise : 35 Marks
- Total : 105 Marks**

**Note:**

- **Internal Evaluation: [45 Marks] by the respective College**
- **External Evaluation: [105 Marks] by the Examiners appointed by Bhakta Kavi Narsinh Mehta University for the Practical Examination**

## **BSc Semester VI Chemistry Practical Examination**

### **Important Guidelines to the Examiners**

**Dear Colleagues**

**Welcome to the panel of Examiners!**

Most of you are experienced and knowledgeable, hence aware and familiar with the examination related responsibilities. However having being assigned the responsibility of preparing the guidelines for the forthcoming BSc Semester VI Chemistry Practical Examination June-2018, this a humble effort to formulate certain guidelines to ensure uniform evaluation at all centers and a smooth conduct of the examination.

The pages following the instructions contain:

- Specific Requirements for each Exercise
- Marking Scheme for different Exercises

**The Question Papers (Slip) for the Practical Examination and Formats A (Organic), B (Inorganic), C (Physicochemical & Chromatography), D (Viva) and E (Consolidated Mark sheet) are enclosed in a separate booklet.** The booklet also contains the format for detailed batch wise and group wise schedule of the examination to be displayed on the notice board at the examination centre.

In case the copies are not sufficient, the Examiners shall request the Lab Supervisor / principal to make arrangement for photocopying the same.

#### **General Instructions:**

- The Examiners appointed for the BSc Semester VI Chemistry Practicals must report to the Principal of the concerned College at the Examination centre at least one hour before the commencement of the Examination.
- The assessment of the practical answer sheets is to be confidential. The examination work shall not be carried out in presence of any other member.
- The Examiner named first is the Senior Examiner
- It shall be the responsibility of the Senior Examiner to ensure smooth conduct of the examination. The other Examiners are equally responsible and shall extend wholehearted cooperation to the Senior Examiner in the smooth conduct of the examination
- The detailed batch wise and group wise schedule of the examination at the allotted centre shall be sent to the centre by Senior Examiner at least one day prior to the

commencement of the examination. For the benefit of the candidates, the schedule shall be displayed on the notice board at the centre a day before the commencement of the examination,

- For the examination purpose the candidates shall be divided into two groups 'A Group' and 'B Group'.
- The Senior Examiner shall distribute the work to his/her colleague and guide him/her where necessary.
- The Senior Examiner shall inform the Lab Supervisor to keep the answer sheets ready for the examination. The Seat Numbers shall be written in blue ballpoint pen and the Table Number in red ballpoint pen.
- The Senior Examiner shall collect the certificate from the Lab Supervisor, indicating standardization of the laboratory equipments, before the commencement of the examination.
- On the day of the completion of the examination the senior examiner shall enter the mark in the mark sheet in Blue/ Black, seal the mark sheet and forward the same to the University immediately after the examination.
- The details in the mark sheet should be filled in English only.
- The attendance report should be filled in duplicate. One copy of the attendance report should be put in the cover along with the mark sheet.
- Another copy of the attendance report and the key forms (A-E) should be packed with the answer sheets and sent, so as to reach the University before the meeting for settling the marks.
- Candidates may use their own practical textbooks, typed or cyclostyled material as a reference. Under no circumstances should they be permitted to use their Journal during the Practical Examination.
- In case the candidate does not have his / her journal certified, he/she may be permitted to appear for the examination. However, the Examiner should submit a report of the same, to the Examination Controller, along with the Mark sheet.
- For authenticity, the examiners shall verify the fee receipt and ID cards of the candidates.

**Some Important Instructions:**

- The work distribution between the two Examiners is as follows:

Examiner 1: Inorganic Qualitative Analysis (Viva) + Project

Examiner 2: Physicochemical (Viva) + Organic Synthesis

- Marks Weightage

Inorganic Qualitative Analysis : 35 Marks

Organic Synthesis : 30 Marks

Physico-chemical / Chromatography : 30 Marks

Viva (Inorganic + Physicochemical) : 10 Marks

Total Marks : 105 Marks

- Practical Schedule

| DAY        | TIME                | 'A' GROUP                             | 'B' GROUP                             |
|------------|---------------------|---------------------------------------|---------------------------------------|
| FIRST DAY  | 10.00 am to 1.00 pm | Inorganic Qualitative Analysis (Viva) | Physicochemical (viva)                |
|            | 2.00 to 5.00 pm     | Physicochemical (viva)                | Inorganic Qualitative Analysis (Viva) |
| SECOND DAY | 10.00 am to 1.00 pm | Organic Synthesis                     | Project                               |
|            | 2.00 to 5.00 pm     | Project                               | Organic Synthesis                     |

**\* The arrangement of above practical session will be finalized by concerned examiner.**

- The number of exercises to be performed by each candidate is 3. The fourth session will be viva voce of Industrial project by external examiner. The Examination shall be conducted for two days with working time of 6 hours / day i.e. a total of 12 hours. The timings shall be from 10.00 am to 01.00 pm and 02:00 pm to 05:00 pm.)
- The Inorganic Mixture given for Inorganic Qualitative Analysis should not be repeated in a batch i.e. no two candidate of a batch should be given the same inorganic mixture.

- Not more than five candidates should be given the same organic Synthesis / Preparation. However, in each case the initial amount of the starting material should be different for all candidates.
- In case of Physicochemical Exercise no two candidates of a batch should be given the same Exercise.
- For Chromatography (Paper / TLC) not more than five candidates should be given the same exercise
- The allotment of the exercise shall be as per the lucky draw system wherein the candidate shall be asked to pick up a chit indicating the exercise number.
- The Examiners are requested to go through the Question Paper Slip before assigning the Exercise. In case of any typing mistake the Examiner shall make the necessary correction before assigning the Exercise.

# **BSc Semester VI**

## **Marking Scheme Chemistry Practicals**

**With effect from June-2018  
[CBCS]**

**Bhakta Kavi Narsinh Mehta University  
BSc Semester VI Chemistry Practical Examination**

**Inorganic Qualitative Analysis [35 Marks]**

**Note: The Inorganic mixture should not be repeated in a batch i.e. no two students of a batch should be given the same Inorganic mixture.**

**Instructions for preparing the Inorganic Mixture**

- At every centre at least 15-20 different mixtures should be prepared
- Not more than three radicals should be the same in the mixtures given to the candidates
- The inorganic salts used in the mixture should be of good grade
- If sulphates of Aluminum, Cadmium, Calcium or Barium is used in the mixture, check its solubility in HCl before giving the mixture to the candidates
- If Bismuth or Cadmium is given, check for its presence before giving the mixture to the candidates
- If the mixture contains nitrate and/or nitrite and iodide and/or bromide, prepare the mixture just a few minutes before giving it to the candidates
- The proportion of halide in the mixture should not be more than 10-15%
- If the mixture contains arsenate or arsenite or chromate, the proportion of the sodium or potassium salt should not be more than 10 %
- If the mixture contains cadmium, its proportion in the mixture should not be 15-20%
- If during the analysis, the radicals undergo oxidation or reduction or form their carbonates due to absorption of carbon dioxide, marks should not be deducted for detecting a wrong radical

**General Instructions:**

- First instruct the candidates to perform the dry tests and detect the radicals, present in the given mixture, within the first hour. The candidates should write the result of the dry tests and get it initialed by the Examiner.
- The Examiner should check and sign the preliminary and dry tests before candidates begin with the group separation.
- After the detection of the radicals present in the mixture, instruct the candidates to write the method of preparation of original solution to be used for the wet tests
- The candidates may then be instructed to proceed for detection of the radicals by wet tests



### Inorganic Qualitative Analysis – Exercise No. 1

#### Marks Distribution

**Total Marks: 35**

|  |                 |
|--|-----------------|
| 1. Preliminary Tests & Dry Tests (3+3)                                   | 6 Marks         |
| 2. Detection of Group<br>(1 marks for each radical)                      | 3 Marks         |
| 3. Group Separation<br>(1 marks for each radical)                        | 6 Marks         |
| 4. Confirmatory Tests for Positive Radical<br>(1 marks for each radical) | 6 Marks         |
| 5. Detection of Negative Radical<br>(1 marks for each radical)           | 6 Marks         |
| 6. Confirmatory Tests for Negative Radical<br>(1 marks for each radical) | 3 Marks         |
| 7. Systematic Working  | 3 Marks         |
| 8. Journal   | 2 Marks         |
| <b>Total Marks</b>   | <b>35 Marks</b> |

#### Note:

- For Inorganic Mixture refer to Appendix - I (List of Inorganic Mixtures)
- For entering the marks use Form A (Inorganic Qualitative Analysis)

### APPENDIX – I : LIST OF INORGANIC MIXTURES

| No | Mixture  | Radicals  |
|----|--|---|
| 1  | Pb(NO <sub>3</sub> ) <sub>2</sub> + KNO <sub>2</sub> + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>   | Pb <sup>+2</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , NO <sub>3</sub> <sup>-1</sup> , NO <sub>2</sub> <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup>    |
| 2  | CuSO <sub>4</sub> + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> + Na <sub>2</sub> SO <sub>3</sub>  | Cu <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , Na <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , CO <sub>3</sub> <sup>-2</sup> , SO <sub>3</sub> <sup>-2</sup>   |
| 3  | MnSO <sub>4</sub> + ZnS + KNO <sub>3</sub>   | Mn <sup>+2</sup> , Zn <sup>+2</sup> , K <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , S <sup>-2</sup> , NO <sub>3</sub> <sup>-1</sup>                              |
| 4  | BaCl <sub>2</sub> + CaCO <sub>3</sub> + (NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>   | Ba <sup>+2</sup> , Ca <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , Cl <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup> , PO <sub>4</sub> <sup>-3</sup>               |
| 5  | K <sub>2</sub> SO <sub>4</sub> + H <sub>3</sub> BO <sub>3</sub> + MgCO <sub>3</sub> + Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>                  | K <sup>+</sup> , Mg <sup>+2</sup> , Al <sup>+3</sup> , SO <sub>4</sub> <sup>-2</sup> , BO <sub>3</sub> <sup>-3</sup> , CO <sub>3</sub> <sup>-2</sup>                |
| 6  | AlPO <sub>4</sub> + FeCl <sub>3</sub> + KBr  | Al <sup>+3</sup> , Fe <sup>+2</sup> , K <sup>+</sup> , PO <sub>4</sub> <sup>-3</sup> , Cl <sup>-1</sup> , Br <sup>-1</sup>  |
| 7  | Mg <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> + ZnS + (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>  | Mg <sup>+2</sup> , Zn <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , PO <sub>4</sub> <sup>-3</sup> , S <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup>                |
| 8  | MgCO <sub>3</sub> + MnCl <sub>2</sub> + Na <sub>2</sub> SO <sub>3</sub>  | Mg <sup>+2</sup> , Mn <sup>+2</sup> , Na <sup>+</sup> , CO <sub>3</sub> <sup>-2</sup> , Cl <sup>-1</sup> , SO <sub>3</sub> <sup>-2</sup>                            |
| 9  | Sr(NO <sub>3</sub> ) <sub>2</sub> + CaCl <sub>2</sub> + KBr  | Sr <sup>+2</sup> , Ca <sup>+2</sup> , K <sup>+</sup> , NO <sub>3</sub> <sup>-1</sup> , Cl <sup>-1</sup> , Br <sup>-1</sup>  |
| 10 | SrCl <sub>2</sub> + NH <sub>4</sub> Br + KI  | Sr <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , K <sup>+</sup> , Cl <sup>-1</sup> , Br <sup>-1</sup> , I <sup>-1</sup>  |
| 11 | NaBr + KI + MgCl <sub>2</sub>  | Na <sup>+</sup> , K <sup>+</sup> , Mg <sup>+2</sup> , Br <sup>-1</sup> , I <sup>-1</sup> , Cl <sup>-1</sup>   |
| 12 | Na <sub>3</sub> PO <sub>4</sub> + NH <sub>4</sub> Cl + K <sub>2</sub> SO <sub>4</sub>  | Na <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , K <sup>+</sup> , PO <sub>4</sub> <sup>-3</sup> , Cl <sup>-1</sup> , SO <sub>4</sub> <sup>-2</sup>                  |
| 13 | Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> + FeCl <sub>3</sub> + KBr  | Al <sup>+3</sup> , Fe <sup>+3</sup> , K <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , Cl <sup>-1</sup> , Br <sup>-1</sup>  |
| 14 | Mn <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> + Co(NO <sub>3</sub> ) <sub>2</sub> + NiCl <sub>2</sub>  | Mn <sup>+2</sup> , Co <sup>+2</sup> , Ni <sup>+2</sup> , PO <sub>4</sub> <sup>-3</sup> , NO <sub>3</sub> <sup>-1</sup> , Cl <sup>-1</sup>                           |
| 15 | CuSO <sub>4</sub> + Bi <sub>2</sub> (CO <sub>3</sub> ) <sub>3</sub> + NH <sub>4</sub> Cl   | Cu <sup>+2</sup> , Bi <sup>+3</sup> , NH <sub>4</sub> <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , CO <sub>3</sub> <sup>-2</sup> , Cl <sup>-1</sup>               |
| 16 | FePO <sub>4</sub> + Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> + (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + H <sub>3</sub> BO <sub>3</sub> | Fe <sup>+3</sup> , Al <sup>+3</sup> , NH <sub>4</sub> <sup>+</sup> , PO <sub>4</sub> <sup>-3</sup> , SO <sub>4</sub> <sup>-2</sup> , BO <sub>3</sub> <sup>-3</sup>  |
| 17 | Bi(NO <sub>3</sub> ) <sub>3</sub> + KI + NH <sub>4</sub> Cl  | Bi <sup>+3</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , NO <sub>3</sub> <sup>-1</sup> , I <sup>-1</sup> , Cl <sup>-1</sup>                               |
| 18 | MnSO <sub>4</sub> + NH <sub>4</sub> NO <sub>3</sub> + KBr  | Mn <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , K <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , NO <sub>3</sub> <sup>-1</sup> , Br <sup>-1</sup>                 |
| 19 | MgSO <sub>4</sub> + Zn <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> + Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>                                    | Mg <sup>+2</sup> , Zn <sup>+2</sup> , Na <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , PO <sub>4</sub> <sup>-3</sup> , BO <sub>3</sub> <sup>-3</sup>               |
| 20 | Ba(NO <sub>3</sub> ) <sub>2</sub> + KI + NH <sub>4</sub> Cl  | Ba <sup>+2</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , NO <sub>3</sub> <sup>-1</sup> , I <sup>-1</sup> , Cl <sup>-1</sup>                               |
| 21 | PbCO <sub>3</sub> + KNO <sub>3</sub> + NaNO <sub>2</sub>   | Pb <sup>+2</sup> , K <sup>+</sup> , Na <sup>+</sup> , CO <sub>3</sub> <sup>-2</sup> , NO <sub>3</sub> <sup>-1</sup> , NO <sub>2</sub> <sup>-1</sup>                 |
| 22 | NiCl <sub>2</sub> + KBr + CuCO <sub>3</sub>  | Ni <sup>+2</sup> , K <sup>+</sup> , Cu <sup>+2</sup> , Cl <sup>-1</sup> , Br <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup>  |
| 23 | ZnS + MnSO <sub>4</sub> + Na <sub>2</sub> SO <sub>3</sub>  | Zn <sup>+2</sup> , Mn <sup>+2</sup> , Na <sup>+</sup> , S <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup> , SO <sub>3</sub> <sup>-2</sup>                             |
| 24 | CdCO <sub>3</sub> + (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + K <sub>2</sub> SO <sub>3</sub>   | Cd <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , K <sup>+</sup> , CO <sub>3</sub> <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup> , SO <sub>3</sub> <sup>-2</sup>    |
| 25 | Na <sub>2</sub> SO <sub>3</sub> + ZnS + NiSO <sub>4</sub>  | Na <sup>+</sup> , Zn <sup>+2</sup> , Ni <sup>+2</sup> , SO <sub>3</sub> <sup>-2</sup> , S <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup>                             |
| 26 | Na <sub>3</sub> AsO <sub>4</sub> + (NH <sub>4</sub> ) <sub>3</sub> AsO <sub>3</sub> + Zn <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>                  | Na <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Zn <sup>+2</sup> , AsO <sub>4</sub> <sup>-3</sup> , AsO <sub>3</sub> <sup>-3</sup> , PO <sub>4</sub> <sup>-3</sup> |
| 27 | Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> + MnCl <sub>2</sub> + NH <sub>4</sub> NO <sub>3</sub>  | Al <sup>+3</sup> , Mn <sup>+2</sup> , NH <sub>4</sub> <sup>+</sup> , SO <sub>4</sub> <sup>-2</sup> , Cl <sup>-1</sup> , NO <sub>3</sub> <sup>-1</sup>               |
| 28 | K <sub>2</sub> CrO <sub>4</sub> + (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + MgCO <sub>3</sub>  | K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Mg <sup>+2</sup> , CrO <sub>4</sub> <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup> , CO <sub>3</sub> <sup>-2</sup>   |
| 29 | CuCl <sub>2</sub> + As <sub>2</sub> O <sub>3</sub> + Na <sub>2</sub> CO <sub>3</sub> + Na <sub>2</sub> SO <sub>4</sub>                                 | Cu <sup>+2</sup> , As <sup>+3</sup> , Na <sup>+</sup> , Cl <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup>                            |
| 30 | FeSO <sub>4</sub> + CuCl <sub>2</sub> + ZnCO <sub>3</sub>  | Fe <sup>+2</sup> , Cu <sup>+2</sup> , Zn <sup>+2</sup> , SO <sub>4</sub> <sup>-2</sup> , Cl <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup>                           |
| 31 | Ba(NO <sub>3</sub> ) <sub>2</sub> + KCl + NaNO <sub>2</sub>  | Ba <sup>+2</sup> , K <sup>+</sup> , Na <sup>+</sup> , NO <sub>3</sub> <sup>-1</sup> , Cl <sup>-1</sup> , NO <sub>2</sub> <sup>-1</sup>                              |
| 32 | CrCl <sub>3</sub> + NiSO <sub>4</sub> + Na <sub>2</sub> SO <sub>3</sub>  | Cr <sup>+3</sup> , Ni <sup>+2</sup> , Na <sup>+</sup> , Cl <sup>-1</sup> , SO <sub>4</sub> <sup>-2</sup> , SO <sub>3</sub> <sup>-2</sup>                            |
| 33 | Co(NO <sub>3</sub> ) <sub>2</sub> + NiCl <sub>2</sub> + BaCO <sub>3</sub>  | Co <sup>+2</sup> , Ni <sup>+2</sup> , Ba <sup>+2</sup> , NO <sub>3</sub> <sup>-1</sup> , Cl <sup>-1</sup> , CO <sub>3</sub> <sup>-2</sup>                           |
| 34 | (NH <sub>4</sub> ) <sub>3</sub> PO <sub>4</sub> + NaNO <sub>3</sub> + KBr  | NH <sub>4</sub> <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , PO <sub>4</sub> <sup>-3</sup> , NO <sub>3</sub> <sup>-1</sup> , Br <sup>-1</sup>                  |
| 35 | Na <sub>2</sub> S + K <sub>2</sub> SO <sub>3</sub> + (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>   | Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , S <sup>-2</sup> , SO <sub>3</sub> <sup>-2</sup> , SO <sub>4</sub> <sup>-2</sup>                   |

**Bhakta Kavi Narsinh Mehta University**  
**BSc Semester VI Chemistry Practical Examination**  
**Organic Synthesis [30 Marks]**

**Slip No. 2: Exercise No. 2 Acetylation of Salicylic acid**

**Aim:** Synthesize Acetyl Salicylic acid (Aspirin) from Salicylic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements:**

- 10 gms Salicylic acid
- 50 gms (14ml) Acetic Anhydride
- 1 ml Conc H<sub>2</sub>SO<sub>4</sub>

Note: 9, 9.5, 10, 10.5, 11 gms Salicylic acid may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 10 gms Salicylic acid, the theoretical yield of Aspirin is 13 gms

**Slip No. 3: Exercise No. 3 Acetylation of Aniline**

**Aim:** Synthesize Acetanilide from Aniline and determine the percentage yield of the product. Re-crystallize the product and note its melting point

**Requirements:**

- 10 gms (9.9 ml) Aniline
- 13.8 gms (12.8 ml) acetic anhydride
- Aqueous solution of HCl (10ml HCl in 250 ml water)

Note: 9, 9.5, 10, 10.5, 11 gms Aniline may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 10 gms Aniline, the theoretical yield of Acetanilide is 14.5 gms

**Slip No. 4: Exercise No. 4 Acetylation of Phenol**

**Aim:** Synthesize Phenyl acetate from Phenol and determine the percentage yield of the product and note its boiling point.

**Requirements:**

- 12 gms Phenol
- 16.3 gms (15 ml) acetic anhydride
- 80 ml 10 % NaOH solution
- 10 ml CCl<sub>4</sub>
- Na<sub>2</sub>CO<sub>3</sub>

Note: 11, 11.5, 12, 12.5, 13 gms Phenol may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates.

From 12 gms Phenol, the theoretical yield of Phenyl acetate is 17.4 gms.

**Slip No. 5: Exercise No. 5 Benzoylation of Aniline**

**Aim:** Synthesize Benzanilide from aniline and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements:**

- 5.2 gms (5 ml) Aniline
- 8.5 gms (7 ml) Benzoylchloride
- 50 ml 10 % NaOH solution

**Note:** 4, 4.5, 5, 5.5, 6 ml Aniline may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 5 ml Aniline, the theoretical yield of Benzanilide is 11 gms.

**Slip No. 6: Exercise No. 6 Benzoylation of Phenol**

**Aim:** Synthesize Phenyl benzoate from Phenol and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements:**

- 2.5 gms Phenol
- 4.5 ml Benzoyl chloride
- 30 ml 10% Aqueous NaOH solution

**Note:** 1.5, 2.0, 2.5, 3.0, 3.5 gms Phenol may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 2.5 gms Phenol, the theoretical yield of aspirin is 5.26 gms.

**Slip No. 7: Exercise No. 7 Preparation of iodoform from ethanol**

**Aim:** Synthesize iodoform from ethanol and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements:**

- 2.5 ml (1.98 g) ethanol
- 25 ml 33 % aqueous  $K_2CO_3$  solution
- 5 gms solid Iodine

**Note:** 1.5, 2.5, 3.5, 4.5, 5.5 ml Ethanol may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 2.5 ml ethanol, the theoretical yield of iodoform is 16.96 gms.

**Slip No. 8: Exercise No. 8 Preparation of iodoform from Acetone**

**Aim:** Synthesize iodoform from acetone and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements:**

2.37 gm (3 ml Acetone)

15 ml 10% NaOH

100 ml Iodine solution (12.5 g I<sub>2</sub> in 25% KI solution)

Note: 2, 3, 4, 5, 6 ml Acetone may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 3 ml acetone, the theoretical yield of iodoform is 16 gms

**Slip No. 9: Exercise No. 9 Preparation of m-dinitrobenzene**

**Aim:** Synthesize m-dinitrobenzene from benzene and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

13 gm (15 ml) Benzene

33 ml Fuming HNO<sub>3</sub>

40 ml Conc H<sub>2</sub>SO<sub>4</sub>

Note: 11, 12, 13, 14, 15 gms Benzene may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 13 gms Benzene, the theoretical yield of m-dinitrobenzene is 28 gms.

**Slip No. 10: Exercise No. 10 Preparation of p-nitro acetanilide**

**Aim:** Synthesize p-nitro acetanilide from acetanilide and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

10 gms Acetanilide

10 ml Glacial Acetic Acid

20 ml conc H<sub>2</sub>SO<sub>4</sub>

Nitrating mixture: 6.2.gms (4.5 ml) conc HNO<sub>3</sub> + 5 g (3 ml) conc H<sub>2</sub>SO<sub>4</sub>

Note: 8, 9, 10, 11, 12 gms Acetanilide may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 10 gms acetanilide, the theoretical yield of p-nitro acetanilide is 13.33 gms.

**Slip No. 11 : Exercise No. 11 Preparation of p-bromoacetanilide**

**Aim:** Synthesize p-bromoacetanilide from acetanilide and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

9 gms Acetanilide

30 ml Glacial Acetic Acid

11.5 gms (3.6 ml) bromine in 17 ml glacial acetic acid

**Note:** 7, 8, 9, 10, 11 gms Acetanilide may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 9 gms acetanilide, the theoretical yield of p-bromoacetanilide is 14.27 gms.

**Slip No. 12: Exercise No. 12 Preparation 2:4:6 -tribromo phenol**

**Aim:** Synthesize 2, 4, 6 -tribromophenol from phenol and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

5 gms Phenol

9 ml liquid bromine

**Note:** 3, 4, 5, 6, 7 gms Phenol may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 5 gms Phenol, the theoretical yield of 2:4:6 -tribromo phenol is 17.61 gms.

**Slip No. 13 Exercise No. 13 Preparation of Methyl Orange**

**Aim:** Synthesize Methyl Orange from Sulphanilic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirement**

10.5 gm Sulphanilic acid

2.3 gm anhydrous  $\text{Na}_2\text{CO}_3$

3.7 gm  $\text{NaNO}_2$

6.3 ml N,N-dimethyl aniline

10.5 ml conc HCl

3 ml glacial acetic acid

35 ml 20% NaOH solution

**Note:** 7.5, 8.5, 9.5, 10.5, 11.5 gms Sulphanilic acid may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 10.5 gms Sulphanilic acid, the theoretical yield of Methyl orange is 19.85 gms.

**Slip No. 14: Exercise No. 14 Preparation of Methyl Red**

**Aim:** Synthesize Methyl Red from Anthranilic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

- 6.5 gm Anthranilic acid
- 12.5 ml conc HCl
- 3.6 gm NaNO<sub>2</sub>
- 8.9 ml N, N-dimethyl aniline
- 5 ml 20% NaOH solution
- 6.8 gm sodium acetate

**Note:** 3.5, 4.5, 5.5, 6.5, 7.5 gms Anthranilic acid may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 6.5 gms Anthranilic acid, the theoretical yield of Methyl Red is 12.76 gms.

**Slip No. 15: Exercise No. 15 Preparation of benzoic acid from benzaldehyde**

**Aim:** Synthesize Benzoic acid from benzaldehyde and determine the percentage yield of the product. Re-crystallize the product and note its melting point.

**Requirements**

- 11 gms Benzaldehyde
- 5 gms Na<sub>2</sub>CO<sub>3</sub>
- 1.5 gms KMnO<sub>4</sub>
- Conc HCl (to acidify)

**Note:** 9, 10, 11, 12, 13 gms Benzaldehyde may be given, however the proportion of the other requirements should be calculated accordingly and given to the candidates

From 11 gms Benzaldehyde, the theoretical yield of Benzoic acid is 12.66 gms.

## Marking Scheme - Organic Synthesis

Use Form B [Organic Synthesis] to enter the Marks

Marks Distribution Scheme for Exercise Nos. 2-15

**Total Marks: 30 Marks**

**Percentage yield of product : 18 marks**

Marks Distribution Percentage yield of product

> 90 to 100 % : 18 marks

> 80 to 90 % : 16 marks

> 70 to 80 % : 14 marks

> 60 to 70 % : 12 marks

> 50 to 60 % : 10 marks

Less than 50 % : 08 marks

**Calculations (Theoretical & % yield) : 03 marks**

**Re-crystallization of the product : 02 marks**

**Melting point / Boiling point of the product ( $\pm 3$ ) : 02 marks**

**Systematic working : 05 marks**

**Total Marks : 30 marks**



**Bhakta Kavi Narsinh Mehta University**  
**BSc Semester VI Chemistry Practical Examination**  
**Physico-Chemical Exercises**

**[30Marks]**

**Exercise No 16-28**

**Slip No. 16: EXERCISE No. 16 pH metry**

**Aim:** To determine Normality and gms/lit of given 'X'N HCl by pH meter.

**Requirements:**

0.1 N HCl solution (as unknown concentration)

Buffer solution

0.5N NaOH solution

Glass and Calomel Electrodes

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working                  | 4 Marks         |
| Neatness –Observation table (at least 8 readings) | 10 Marks        |
| Correct Graph (Two graphs)                        | 8 Marks         |
| Normality of HCl solution (From Graphs)           | 6 Marks         |
| Correct gms/litre of HCl solution                 | 2 Marks         |
| <b>Total Marks</b>                                | <b>30 Marks</b> |

**Slip No. 17: EXERCISE No. 17 - pH metry**

**Aim:** To determine the dissociation constant and Normality of given weak acid, CH<sub>3</sub>COOH, by pH meter.

**Requirements:**

0.1 N CH<sub>3</sub>COOH solution (as unknown concentration)

Buffer solution

0.5N NaOH solution

Glass and Calomel Electrodes

**Marks Distribution**

|  |                 |
|--|-----------------|
| Arrangement + Systematic Working                         | 4 Marks         |
| Neatness –Observation table (at least 8 readings)        | 8 Marks         |
| Correct Graph (Two graphs)                               | 8 Marks         |
| Correct Dissociation Constant                            | 4 Marks         |
| Normality of CH <sub>3</sub> COOH solution (From Graphs) | 6 Marks         |
| <b>Total Marks</b>                                       | <b>30 Marks</b> |

**Slip No. 18: EXERCISE No. 18 pH metry**

**Aim:** To determine the dissociation constant and Normality of dibasic acid Oxalic acid / Malonic acid using 0.1N NaOH, by pH meter.

**Requirements:**

- 0.1 N Oxalic acid / Malonic acid solution (as unknown concentration)
- 0.1N NaOH solution,
- Buffer solution,
- Glass and Calomel Electrodes

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working                  | 4 Marks         |
| Neatness –Observation table (at least 8 readings) | 8 Marks         |
| Correct Graph (Two graphs)                        | 8 Marks         |
| Correct Dissociation Constant                     | 4 Marks         |
| Normality of dibasic acid solution (From Graphs)  | 6 Marks         |
| <b>Total Marks</b>                                | <b>30 Marks</b> |

**Slip No. 19: EXERCISE No. 19: POTENTIOMETRY**

**Aim:** To determine normality and dissociation constant of benzoic acid using 0.5 N NaOH solution by potentiometry

**Requirements**

- |  |                       |
|--|-----------------------|
| 1. 'X' N Benzoic acid (0.08 to 0.12 N) | 4. Calomel Electrode  |
| 2. 0.5 N NaOH                          | 5. Platinum Electrode |
| 3. Buffer solution                     | 6. Quinhydrone Powder |

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working        | 6 Marks         |
| Presentation, Observations and Neatness | 12 Marks        |
| Graphs                                  | 6 Marks         |
| Correct Dissociation constant           | 4 Marks         |
| Correct Normality of Benzoic acid       | 2 Marks         |
| <b>Total Marks</b>                      | <b>30 Marks</b> |

**Slip No. 20: EXERCISE No. 20: POTENTIOMETRY**

**Aim** : To determine normality of given acid 'x' N HCl by potentiometric titration against 0.5 N NaOH solution.

**Requirements**

- |                               |                        |
|-------------------------------|------------------------|
| 1. 'X' N HCl (0.08 to 0.12 N) | 2. 0.5 N NaOH solution |
|-------------------------------|------------------------|

- |                           |                              |
|---------------------------|------------------------------|
| 3. Saturated KCl solution | 6. Calomel electrode         |
| 4. KCl salt bridge        | 7. Platinum/ Glass electrode |
| 5. Quinhydrone powder     |                              |

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working        | 6 Marks         |
| Presentation, Observations and Neatness | 12 Marks        |
| Graphs                                  | 6 Marks         |
| Correct Dissociation constant           | 4 Marks         |
| Correct Normality of HCl                | 2 Marks         |
| <b>Total Marks</b>                      | <b>30 Marks</b> |

**Slip No. 21: EXERCISE No. 21 POTENTIOMETRY**

**Aim:** To determine concentration of 'x' N  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  solution by potentiometric titration against 0.5 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution and determine the redox potential system.

**Requirements**

- |   |                              |
|---|------------------------------|
| 1. 'X' N $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ solution | 5. KCl salt bridge           |
| 2. 0.5 N $\text{K}_2\text{Cr}_2\text{O}_7$  | 6. Quinhydrone powder        |
| 3. 2N $\text{H}_2\text{SO}_4$ solution  | 7. Calomel electrode         |
| 4. Saturated KCl solution   | 8. Platinum/ Glass electrode |

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working        | 4 Marks         |
| Presentation, Observations and Neatness | 14 Marks        |
| Graphs                                  | 6 Marks         |
| Correct Normality                       | 6 Marks         |
| <b>Total Marks</b>                      | <b>20 Marks</b> |

**Slip 22: EXERCISE No. 22: POTENTIOMETRY**

**Aim:** To determine normality of each halide in the mixture of KCl, KBr and KI by potentiometric titration against 0.5N  $\text{AgNO}_3$  solution.

**Requirements**

1. Solution containing a mixture of 0.5 N KCl, KBr and KI (each)
2. 0.5 N  $\text{AgNO}_3$  solution
3. Saturated KCl solution
4. KCl salt bridge

5. Calomel electrode
6. Silver electrode

**Marks Distribution**

|   |                 |
|---|-----------------|
| Arrangement + Systematic Working        | 4 Marks         |
| Presentation, Observations and Neatness | 12 Marks        |
| Graphs                                  | 6 Marks         |
| Correct Normality of KCl, KBr & KI      | 8 Marks         |
| <b>Total Marks</b>                      | <b>30 Marks</b> |

**Slip No. 23: EXERCISE No. 23: SURFACE TENSION**

**Aim:** To determine the surface tension of the liquids A, B and C by using Drop number method and hence calculate the value of Parachor of liquids and CH<sub>2</sub> group.

**Requirements**

1. Liquids A, B & C  
(Benzene, Toluene, Xylene) or  
( n-propylalcohol, n-butylalcohol, n-amylalcohol)
2. Distilled water

**Marks Distribution**

|                                    |                 |
|------------------------------------|-----------------|
| Systematic Working                 | 4 Marks         |
| Determination of No. of drops      | 6 Marks         |
| Calculation of Density             | 6 Marks         |
| Calculation of Surface tension     | 5 Marks         |
| Calculation of Parachor            | 5 Marks         |
| Correct Surface tension & Parachor | 4 Marks         |
| <b>Total Marks</b>                 | <b>30 Marks</b> |

**Slip No. 24: EXERCISE No. 24: PAPER CHROMATOGRAPHY [ASCENDING]**

**Aim:** You are given three samples of amino acids and their mixture; separate them by ascending paper chromatography.

**Requirements:**

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H<sub>2</sub>O (4 :1: 5 ) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

**Rf value of amino acids**

|                |       |            |       |
|----------------|-------|------------|-------|
| Phenyl alanine | 0.62  | Prolamine  | 0.233 |
| Alanine        | 0.215 | Methionine | 0.503 |
| Histidine      | 0.11  | Lysine     | 0.184 |

|               |       |          |      |
|---------------|-------|----------|------|
| Glutaric acid | 0.288 | Arginine | 0.30 |
| Tyrosine      | 0.423 | Glycine  | 0.23 |

### Marks Distribution

|   |                 |
|---|-----------------|
| Chromatogram  | 6 Marks         |
| Rf value of individual components (4 marks each)        | 12 Marks        |
| Rf vale of each component in the mixture (4 marks each) | 12 Marks        |
| <b>Total Marks</b>                                      | <b>30 Marks</b> |

### Slip No. 25: EXERCISE No. 25: PAPER CHROMATOGRAPHY [CIRCULAR]

**Aim:** You are given three samples of amino acids and their mixture; separate them by circular paper chromatography.

#### Requirements:

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H<sub>2</sub>O (4 :1: 5 ) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

#### Rf value of amino acids

|                |       |               |       |
|----------------|-------|---------------|-------|
| Phenyl alanine | 0.62  | Lysine        | 0.184 |
| Alanine        | 0.215 | Glutaric acid | 0.288 |
| Histidine      | 0.11  | Tyrosine      | 0.423 |
| Prolamine      | 0.233 | Arginine      | 0.30  |
| Methionine     | 0.503 | Glycine       | 0.23  |

### Marks Distribution

|   |                 |
|---|-----------------|
| Chromatogram  | 6 Marks         |
| Rf value of individual components (4 marks each)        | 12 Marks        |
| Rf vale of each component in the mixture (4 marks each) | 12 Marks        |
| <b>Total Marks</b>                                      | <b>30 Marks</b> |

### Slip No. 26: EXERCISE No. 26: TLC CHROMATOGRAPHY

**Aim:** You are given three samples of amino acids and their mixture; separate them by TLC chromatography.

#### Requirements:

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H<sub>2</sub>O (4 :1: 5 ) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

#### Rf value of amino acids

|                |       |               |       |
|----------------|-------|---------------|-------|
| Phenyl alanine | 0.62  | Lysine        | 0.184 |
| Alanine        | 0.215 | Glutaric acid | 0.288 |
| Histidine      | 0.11  | Tyrosine      | 0.423 |
| Prolamine      | 0.233 | Arginine      | 0.30  |
| Methionine     | 0.503 | Glycine       | 0.23  |

**Marks Distribution**

|   |                 |
|---|-----------------|
| Chromatogram  | 6 Marks         |
| Rf value of individual components (4 marks each)        | 12 Marks        |
| Rf vale of each component in the mixture (4 marks each) | 12 Marks        |
| <b>Total Marks</b>                                      | <b>30 Marks</b> |

**Slip No. 27: EXERCISE No. 27: PAPER CHROMATOGRAPHY [ASCENDING]**

**Aim:** You are given three samples of metal ions and their mixture; separate them by ascending paper chromatography.

**Requirements:**

- ✓ Metal ions samples and their mixture
- ✓ **Developer:** Acetone + Ethylacetate + 6N HCl (9:9:2)
- ✓ **Spraying Agent:** 0.1 % Rubinic acid in Acetone

**Rf value of metal ions**

|                  |                     |
|------------------|---------------------|
| Ni <sup>+2</sup> | 0.1 (0.08 to 0.15)  |
| Co <sup>+2</sup> | 0.45 (0.35 to 0.55) |
| Cu <sup>+2</sup> | 0.65 (0.50 to 0.75) |

**Marks Distribution**

|   |                 |
|---|-----------------|
| Chromatogram  | 6 Marks         |
| Rf value of individual components (4 marks each)        | 12 Marks        |
| Rf vale of each component in the mixture (4 marks each) | 12 Marks        |
| <b>Total Marks</b>                                      | <b>30 Marks</b> |

**Slip No 28:EXERCISE No. 28 PAPER CHROMATOGRAPHY [CIRCULAR]**

**Aim:** You are given three samples of metal ions and their mixture; separate them by circular paper chromatography.

**Requirements:**

- ✓ Metal ions samples and their mixture [Ni<sup>+2</sup>, Cu<sup>+2</sup> and Co<sup>+2</sup>]
- ✓ **Developer:** Acetone + Ethylacetate + 6N HCl (9:9:2)
- ✓ **Spraying Agent:** 0.1 % Rubinic acid in Acetone

**Rf value of metal ions**

|                  |                    |
|------------------|--------------------|
| Ni <sup>+2</sup> | 0.1 (0.08 to 0.15) |
|------------------|--------------------|

Co <sup>+2</sup>                    0.45 (0.35 to 0.55)

Cu <sup>+2</sup>                    0.65 (0.50 to 0.75)

**Marks Distribution**

|   |                 |
|---|-----------------|
| Chromatogram  | 6 Marks         |
| Rf value of individual components (4 marks each)        | 12 Marks        |
| Rf vale of each component in the mixture (4 marks each) | 12 Marks        |
| <b>Total Marks</b>                                      | <b>30 Marks</b> |