MANIPAL ACADEMY of HIGHER EDUCATION
(Institution of Eminence Deemed to be University)

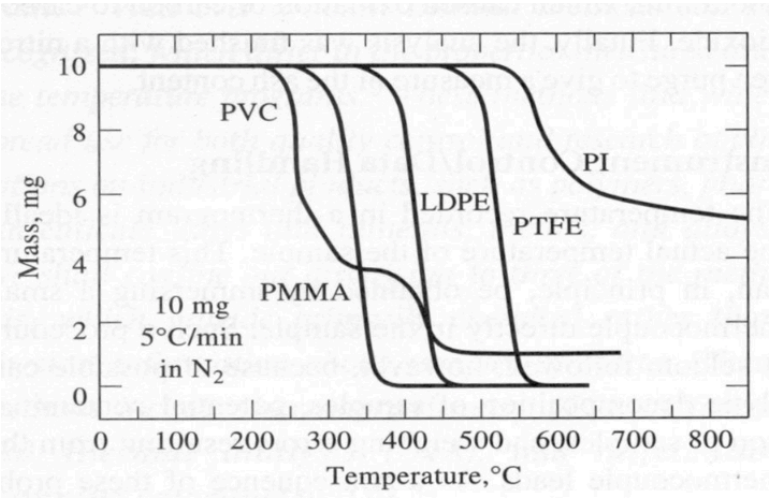
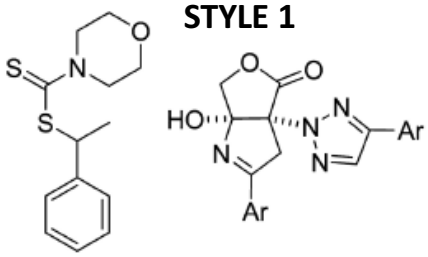
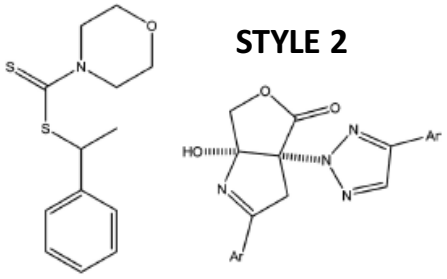
Reg.No.									

DEPARTMENT OF SCIENCES, II SEMESTER M.Sc. (Chemistry)
END SEMESTER EXAMINATIONS, MAY/JUNE 2023
Research Methodology and Technical Communication [CHM 5207]
(CHOICE BASED CREDIT SYSTEM - 2021)

Time: 3 Hours Date: 29/05/2023 MAX. MARKS: 50

Note (i) Answer ALL questions and draw diagrams, and write equations wherever necessary.

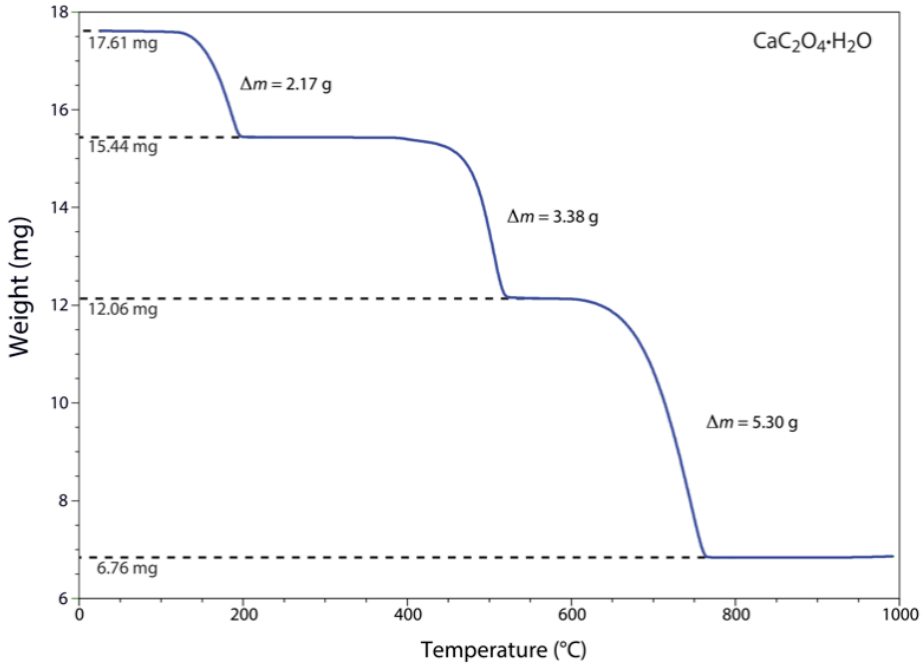
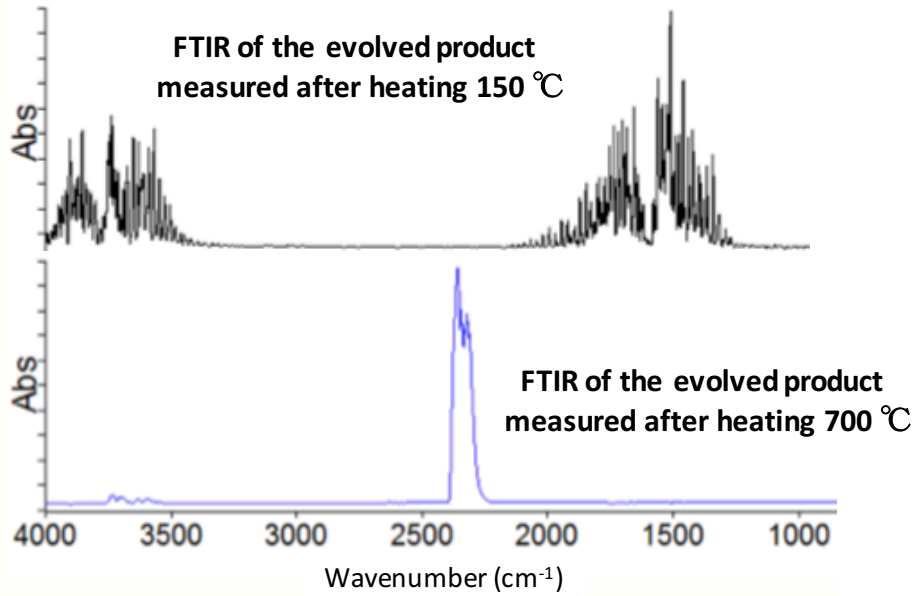
	Questions **(5+3+2) marks**	Marks	CO	BL
1	(a) Write a detailed note on the following. (i) the importance of literature review and how to perform it (ii) ethics in conducting research and on how to find plagiarism	5	2	2
	(b) Explain the difference between copyright, patent and trademark.	3		
	(c) What are primary and secondary data in research. Explain with an example for each**.**	2		
2	(**a**) Explain preferred type of thermal analysis technique you would like to use for studying and getting information on the following phenomena or chemical reaction. Justify your choice. (i) To determine different allotropic forms of elemental sulfur (ii) To find the crystallization temperature of polymers (iii) To differentiate whether the chemical/material is degrading via decomposition or sublimation (iv) polymerization of alkenes (v) To find dehydration behavior of copper sulfate pentahydrate	5	1	3
	(**b**) Write representative qualitative DSC graphs for the reactions mentioned below. Justify your answer. (i) $2Pb(NO_3)_2 \rightarrow 2PbO + 4NO_2 + O_2$ (ii) $C + O_2 \rightarrow CO_2$ (iii) $N_2 + O_2 + Heat \rightarrow 2NO$	3		

	(**c**) Using scientific English, explain the data presented in below graph.  	2		
3	(**a**) What is differential scanning calorimetry (DSC) analysis and explain on how we can get information on the enthalpy (heat flow) from this analysis. Explain the differences between DTA and DSC techniques. What are the advantages and limitations of thermogravimetric analysis (TGA) as an experimental tool? (**b**) What are the best practices for delivering oral presentation in a scientific meeting or in a conference? To write the structure of organic molecules in a manuscript, which of the following styles do you choose, and why? **STYLE 1**  **STYLE 2**  (**c**) Explain the principle involved in using TGA as a tool for the following. (i) to find metallic impurities (contamination) in carbon nanotubes (ii) to find the ratio and compositional analysis of mixed materials	5 3 2	1	2

4	(**a**) Read the below text and write the Research Gaps using scientific English. **Text** The nitrogen cycle is a vital biogeochemical process for the utilization of atmospheric nitrogen. Nitrogen plays an essential role in life on earth, in which nitrogen is a building block of proteins and nucleic acids. Naturally abundant dinitrogen (N_2) in the atmosphere is chemically inert. Therefore, N_2 cannot be utilized for humankind unless it has not converted into a more reactive form through either oxidation or reductive pathway in the nitrogen fixation process. In the nitrogen cycle, atmospheric N_2 is converted into ammonia (NH_3) through biological nitrogen fixation by microbes and industrially by Haber-Bosch process. Formation of NH_3 through electrocatalytic nitrogen reduction reaction (NRR) is a sustainable alternative to the industrial Haber-Bosch process. Electrocatalytic NRR occurring at ambient conditions could be an energy storage pathway for storing excess energy produced at times by the intermittent renewable energy sources such as wind and solar. Electrocatalysts that selectively and efficiently reduce N_2 to NH_3 still elusive due to low NH_3 production and current efficiency. Necessary parameters required to develop a catalyst for the electrochemical conversion of the N_2 to NH_3 are (a) the selectivity of NRR over the hydrogen evolution reaction (HER), (b) the high efficiency of the overall NRR process, and (c) the high yield of NH_3 synthesis. It is difficult to dissociate the N_2 molecule owing to its strong π and σ bonds with high bond dissociation energy of first of three bonds. A valuable solution to this problem is achieved by noble and non-noble metal-based electrocatalysts with nitrogen binding ability. Transition metal exhibits acceptable behavior as a catalyst for the NRR process due to its tendency to induce π backdonation from its filled d orbital to the antibonding molecular orbital of the N_2 molecule. This back-bonding is the driving force for activating the N≡N triple bond and facilitates the NRR process. The orientation of the metal ion in the molecular complex can control the effectiveness of the back-bonding. Substantial efforts have been made for the development of heterogeneous electrocatalysts to enhance the NRR activity. This was achieved by tailoring the morphology of the nanostructure, creating vacancies, alloying, etc. In addition, well-defined metal complexes have been reported to break the strong N≡N bond in N_2. Bimetallic and trimetallic complexes are advantageous to	5	3	4

activate the N_2 molecule. Holland and co-workers have shown that metal complexes with thiolate auxiliary ligands are preferred in N_2 activation over O/N donors, due to increment in the electron density on the metal, and during protonation, thiolate may assist with H^+ transfer required for the conversion. These thiolate complexes mimic the activity of metalloenzymes like Ni superoxide dismutase and Ni−Fe hydrogenase.

Recently, thiol-protected metal nanoclusters (NCs) have gained much interest in the field of catalysis and photo-physical properties due to their unique structure, core geometry, and metal-ligand staple motif. The increase in accessible dimension owing to their ultra-small size, purity in atomic level, and unique structures of NCs has prompted the utilization of these for different types of catalytic reactions. Precision at the atomic level is a crucial factor in the metal cluster. As the catalytic properties of the metal NCs firmly depend on the number of metal atoms, even a single atom can differ the activity in catalysis by metal NCs. A well-defined structural information of the nanocluster can be used as a model to provide in-depth understanding of their catalytic property. Furthermore, the uniformity of the catalyst in the electrolyte solution provide more activity and selectivity towards desired products. This NCs activated N_2 molecule and transformed to NH_3 (without any other nitrogenous products) with Faradic efficiency of ≈25 %. Both experimental results and theoretical calculations help to elucidate the mechanistic aspect of the NRR by this NCs.

	(**b**) Using scientific English, explain the thermal decomposition profile of $CaC_2O_4\ H_2O$ by comparing TGA and FT-IR analysis of the evolved products.   	3		
	(**c**) In TGA graph, the initial mass of the sample containing adsorbed CO_2 gas is 5.258 mg. After heating to 500° C, the sample weight reduced to 4.829 mg owing to in CO_2 removal/desorption from the sample. Find out how many mol of CO_2 was absorbed per gram of the sample taken.	2		

5	(**a**) A new automated procedure for determining glucose in serum (Method A) is to be compared to the established method (Method B). Both methods are performed on serum from the **same six patients** to eliminate patient-to-patient variability. Do the following results confirm a difference in the two methods at the 5% significance level? Substantiate with relevant hypothesis. (t-table is provided)	5	2	4

Patient	**Method A** glucose (mg/L)	**Method B** glucose (mg/L)
1	1044	1028
2	720	711
3	845	820
4	800	795
5	957	935
6	650	639

(**b**) Comment on the spread and consistency of the two data sets (A and B) provided below using standard deviation and coefficient of variation. Justify your answer. — 3

Data set A=1, 2, 3

Data set B=101, 102, 103

(**c**) On heating a sample of 25 mg of hydrated compound with a molecular weight of 250 g/mol in TGA analysis, 16 mg of dehydrated compound remains. The number of water molecules lost per molecule of hydrated compound is….? — 2