

SAULT COLLEGE
of Applied Arts and Technology
Sault Ste. Marie

COURSE OUTLINE

CHM-209-3
PHYSICAL CHEMISTRY-LAB

revised June, 1981 by D. Heggart



PHYSICAL CHEMISTRY LABORATORY

The laboratory section of physical chemistry consists of a three hour lab per week for the full semester. It is intended that the student gain experience in some of the classical physical chemistry experiments as well as some physical testing, which will aid him in chemical processes (CHM 203-4) which is taken concurrent with physical chemistry.

TEXT:

Laboratory Manual of Physical Chemistry; Crockford and Nowell;
-Wiley (1967)

REFERENCE TEXTS:

Experimental Physical Chemistry - 7th Ed., Daniels et al;
-McGraw-Hill (1970)

Experiments in Nuclear Science - Chase et al; -Burgess (1968)

PHYSICAL CHEMISTRY - LABORATORY OUTLINE

You are required to complete ten (10) experiments, and to submit formal reports one week after completion of each experiment. A pre-lab report is due at the first theory class of the week, on the lab for the following week.

A data book is to be kept with you in the lab and it will be inspected and given a mark equivalent to one experiment.

EXPERIMENTS

Lab - 3 hrs/wk

<u>No.</u>	<u>Experiments</u>	<u>(Hrs.)</u> <u>Time</u>
1.	Viscosity of Liquids - D-157 - to use different viscosimeters -Saybott, Oswald, MacMichael - and investigate affect of temperature on viscosity	3
2.	Molecular Weight Determination - D183 - C&N-72-to determine the MW of non-volatile solutes	3
3.	Fractional Distillation - D-70-to investigate column efficiency and separate a binary mixture	6
4.	X-ray Diffraction - D-321-to determine lattice type for cubic crystals and identify unknown	6
5.	Heat of combustion - Bomb Calorimeter - D-18 to determine heat of combustion of an organic substance C&N-79	6
6.	Bromination of Acetone - D-152 to investigate the kinetics and determine order of reaction	3
7.	Inversion of Sucrose - D-149 to study inversion of encrose using polarimeter	3
8.	Effect of Temperature on Reaction Rate - C&N-124 to determine effect of temperature on reaction rate.	
9.	Nuclear Chemistry - to become familiar with use & operation of Geiger-Muller Counter. C.R.S.	6
10.	Ion-Exchange - D-341	3

CODE

B-Butler

C&N- Crockford & Nowell

D - Daniels et al

K- Kittsley

M&S - Masterton & Slowinski

B&T - Babor & Thiessen

C.R.S. - Chase, Rituper, Sulcoski

PHYSICAL CHEMISTRY - LABORATORY

A Student Guide for CHM - 209

Table I - Physical Chemistry

Laboratory experiments for fourth semester physical chemistry course. You will be required to complete 6 lab experiments. Selected experiments will require formal written reports. A lab book must be maintained at all times and this will be your record of completing the program. Failure to complete the above will result in your obtaining incomplete standing.

<u>Experiment No.</u>	<u>Title</u>
1	M. W. by Boiling Point Elevation
2	M. W. by Freezing Point Method
3	Fractional Distillation
4	Viscosity
5	X-Ray Diffraction
6	Heat of Combustion
7	Bromination of Acetone
8	Temperature Effect on Reaction Rate
9	Polarimeter (inversion of sucrose)
10	Nuclear Chemistry
11	Heat of Solution, Heat of Neutralization, Calorimetric Procedure
12	Refractive Index

All experiments will be allowed 2 lab days. Grades will be assessed in the following:

6 experiments	C maximum
7 experiments	B maximum
8 experiments	A maximum

Lab books must be initialled by the student and instructor at the end of each day. Lab Reports are due one week after completion of experiment. Late reports will be considered as failure to meet deadline and will be graded accordingly.

Molecular Weight Determination by the Boiling Point Method

The student will:

1. Prepare a pellet of known sample of .2 to .4 gms, using the pellet press.
2. Set the Beckmann thermometer so as to read in the lower half of the scale.
3. Determine the boiling point elevation upon addition of the non-volatile solute.
4. Calculate the molecular weight of known sample.
5. Repeat experiment with second sample of known sample.
6. Calculate % error, precision, accuracy.
7. Prepare a formal report of work including all calculations.

Ref. Crockford & Nowell - p. 11

Barrow - p. 83

Reference: Crockford & Nowell
Barrow - pg. 79

Fractional Distillation

The student will:

1. Calibrate the Fisher Refractometer, using distilled water.
2. Prepare a set of solutions of CCl_4 in C_6H_6 and determine the RI of each.
3. Prepare a calibration curve by plotting RI vs. Mole % of solution.
4. Determine the number of theoretical plates for three distillation columns, using a $\text{CCl}_4 - \text{C}_6\text{H}_6$ mixture.
5. Analyze the distillate from the most efficient column by collecting every 4 mls. and recording temperature and RI of each sample.
6. Prepare graphs of temperature vs. Mole % and RI vs. Mole %.
7. Submit a formal report outlining his work, with discussion of all data and graphs.

Reference: Crockford & Nowell
Daniels - pg. 70

Viscosity

The student will:

1. Determine the viscosity of H_2O at 30° , 25° , and $20^\circ C$, using an Ostwald viscosimeter.
2. Determine (as in 1) the viscosity of methanol.
3. Determine the viscosity of castor oil at 30° and $20^\circ C$ using the Falling Ball method.
4. Determine (as in 3) the viscosity of a series of selected motor oils using the Mac Michael Viscosimeter.
5. Submit a formal report on findings.

X-Ray Diffraction

The student will:

1. Learn the operation of the Phillips diffractometer, including loading of sample.
2. Run a sample of Na_2CO_3 and do all calculations on known sample.
3. Run an unknown inorganic and unknown organic and determine unknown.
4. Submit a formal report on his findings.

Daniels - pg. 321

Heat of Combustion - Parr Bomb Calorimeter

The student will:

1. Learn the operation of the Parr Bomb Calorimeter.
2. Standardize the instrument with benzoic acid.
3. Determine the heat of combustion in duplicate, of naphthalene.
4. Submit a formal report including all calculations, and analyze his data completely.

C & N - pg. 79

Bromination of Acetone

The student will:

1. Prepare all solutions using analytical technique -- 4M acetone, 1M HCl, 0.02M Br₂.
2. Determine time for reaction in seconds at T°C.
3. Calculate the reaction order with respect to acetone, H⁺ and Br₂.
4. Predict reaction rate.
5. Determine energy of activation.
6. Submit a formal report of his findings.

The Effect of Temperature on Reaction Rate

The student will:

1. Determine the effect of temperature on the specific rate constant for the H^+ catalyzed hydrolysis of methyl acetate.
2. Calculate the energy of activation from the specific rate constant.
3. Submit a formal report on his work.

Polarimeter

The student will:

1. Learn the operation of the Polarimeter and constant temperature bath.
2. Follow the inversion of a sucrose solution at 30°C.
3. Study the role of H⁺ in the catalysis of the reaction.
4. Submit a formal report on the findings.

Ion Exchange

The student will:

1. Prepare ion-exchange column.
2. Separate a Ni^{++} - Co^{++} solution by ion exchange at a rate of 2.5 - 3.0 mls/min.
3. Calibrate Spectronic 20 using citrate buffer.
4. Prepare calibration curves for Ni^{++} and Co^{++} solutions, using Spectronic 20 spectrophotometer.
5. Analyze eluted solutions - at 510 m μ for Co^{++} and 650 m μ for Ni^{++} .
6. Convert absorbancy to concentrations for each eluted sample.
7. Plot concentration vs. volume of effluent.
8. Determine quantity of Ni^{++} and Co^{++} recovered and calculate % recovery.
9. Submit a formal report on the work.

Daniels - pg. 341

Nuclear Chemistry

The student will:

1. Determine the plateau and operating voltage of a Geiger-Muller counter, using Tl^{204} or some other β emitter (5)
2. Investigate background radiation in shielded and unshielded areas (6)
3. Determine the efficiency of a G-M counter for calibrated sources of Tl^{204} , Ba^{133} , C^{14} (8)
4. Investigate the effect of distance upon the intensity of radiation and to measure the shelf ratio of a sample holder (10)
5. Study methods for increasing the accuracy of measurement (19)
6. Determine the half-life of a radioisotope (20)
7. Determine the efficiency of commercial detergents
8. Submit a formal report on his work.

Experiments in Nuclear Science - Chase, Retuper, Sulcoski