3590/3550 - Laboratory

- Time: Monday to Thursday 2:30 – 5:30
- Location: 318 Parker Bldg.

- Five two week labs must be completed. Two students per experiment. Individual lab reports, sharing same data.

- There are prefab questions which are to be answered (in paragraph form) and are due at start of each new laboratory experiment.

- Lab reports are due the beginning of the following lab period the week after the lab is completed.

- The lab report and marks can be viewed, but labs are not returned until the end of the course.

Lab Report Overview

- Each student is responsible for 50% of lab work, and for individual, original reports.

- The lab report constitutes 50% of the final grade. The organization, accuracy, completeness, interest in topic, results and explanations determine report grade.

- The format of the report is that of a journal submission. Check out a Chemistry or Science journal to see how the report should be written.

Grading

- Graded on:
  - Readability- good graphs, well made, self explanatory
  - Completeness
  - Flow of report.
  - Interest in researching subject.
  - Accuracy of results. Explanations of results. Good standard curves. Accurate and/or precise analysis of unknown
  - Discussion of results. Why results are as seen. If exp. didn't work, explain problems and solutions.
  - Comprehensive conclusion. Questions answered.
  - References on similar material

Introduction

- Description of problem.
- Importance of solution of problem. Why are you working on the problem?
- Why use this instrument to solve the problem? Some theory and advantage of using instrument.
- Do not give basic description of instrument operation. This is usually part of prelab questions.
- Include reference to other work done.

Materials and Methods

List in paragraph format all methodology, including instruments used with model and manufacturer, columns, solvent systems, reagents (supplier). Methods should be detailed and complete, but not a copy of lab manual. Should not include particulars of operating the equipment from the laboratory manual. Do not explain which buttons you pushed on the instrument.

Results

- All the pertinent data needed for understanding of experiment. Include tables and/or graphs, sample calculations.

- Each table or graph should have a paragraph of explanation in the results section. No graph or table should stand alone without detailed description of what the reader is seeing.

- If you are comparing results, for example, you can show % differences. You can include standard deviation if you have multiple results.

Discussion: (Separate from results)

- Discuss the results and the explanations
- Interpret the meaning of the results.
- If problem in results explain and give solution.
- Refer to other work on the subject

Conclusion

- What was demonstrated by experiment.
- A short summary of what you found or accomplished. Was experiment a success? Why? Why not?
- Advantage of this method and instrument.
- What additional experiment(s) could tell you more about result you are looking for?

Questions and References

Questions: Answer the questions at the end of experiment. The answers to the questions are in addition to the conclusions even if the answer is covered in the conclusions.

References: References you used to interpret results. Do not reference lab manual.

Marks

- Prelab ……2 marks
- Introduction……2.5
- Materials and Methods…….2.5
- Results ……5
- Discussion……..5
- Conclusion……...1
- Questions……2

Total 20

Course Comment

- The information you learn in lectures and lab applies to real job situations.
- Know the material (theory) from the lectures and understand the labs before you come to lab.
- Much of theory of instruments were presented in second year analytical course (look up notes)

Laboratory Procedure

- There is only six hours for each experiment.
  - Read and understand experiment before lab.
  - Summarize exp. (in hard covered lab book) before lab.
  - Dilution tables (In your lab book) can be made-up ahead of lab.

- Be familiar with theory of operation of instrument before lab.
  - Understand which aspect of the instrument used is being demonstrated by the experiment you are running.

Pipetting and Dilution

- Students should be familiar with automatic pipettes for making up specified concentrations or diluting samples.

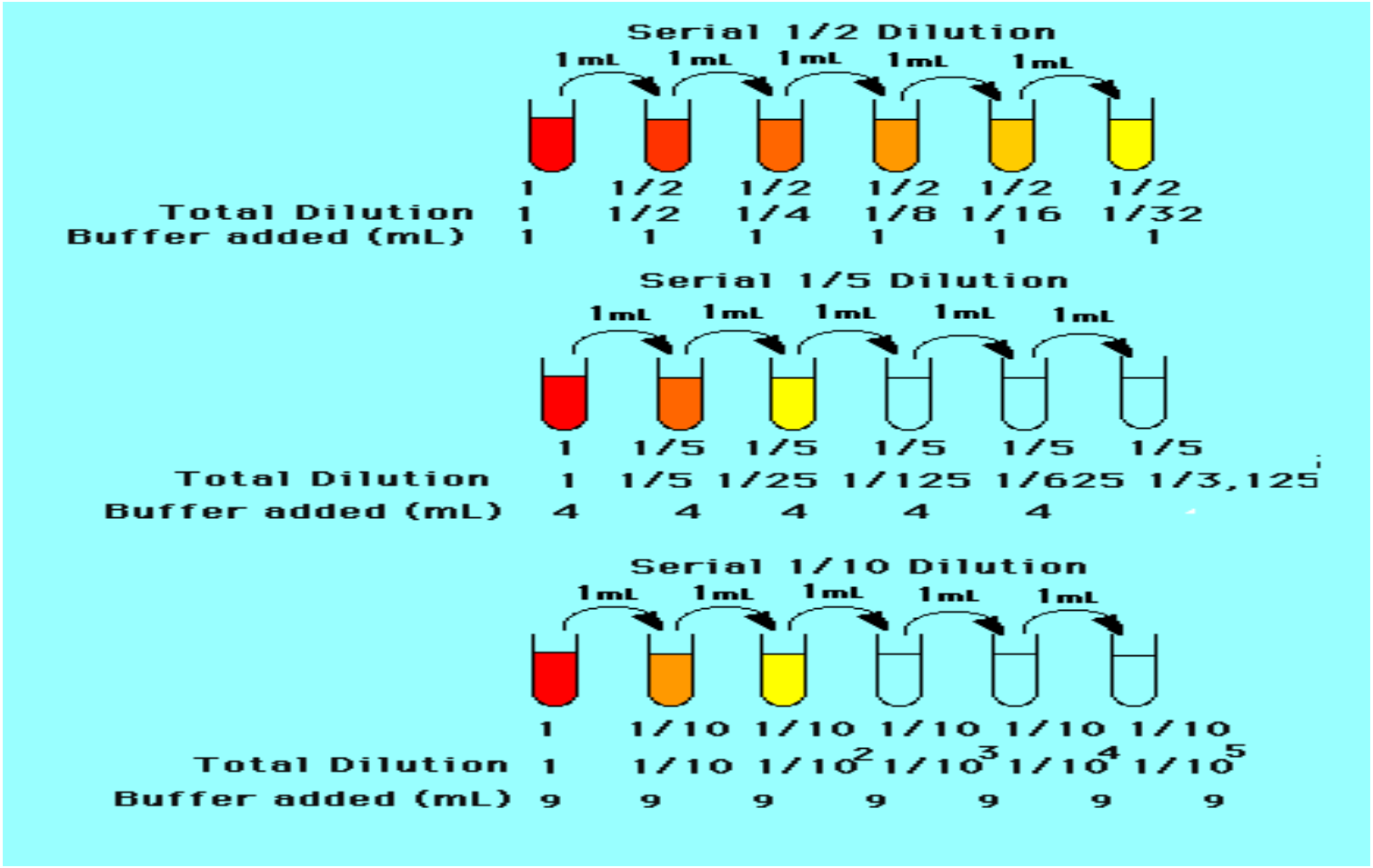
- Students should be familiar with standard and serial dilution techniques.

Normal Dilution

Dilution*	Mls sample	Mls solvent	Total volume
1 to 10	1 0.1 0.5	9 1.9 4.5	10 1 5
1 to 20	0.5 0.05 0.25	9.5 1.95 4.75	10 1 5

*Dilutions are normally done with auto pipettes

Serial Dilution



Housekeeping Comments:

- All glassware and equipment used must be cleaned at the end of each lab period and vials emptied and cleaned or disposed of. If you are short of time you may come in following day to finish cleaning.
- Chemicals should be put away.
- Lab coats, goggles, closed toed shoes, eye wash station, use fume hood of volatiles, care in handling chemicals

Do's and Don'ts

- Most of the instruments available in the MCAL are **very expensive**; please treat them carefully. If you do not understand something, please feel free to **ask for help.**

- The experiments must be recorded in a bound **lab book** as the work is done (**not on a loose piece of paper**).

- Please **clean-up** your work area before you leave (*this is strictly enforced*). Remove any waste papers, spills etc. and clean-up all the glassware used. All the chemicals and equipments must be returned to their proper location. *You have to get the signature of one of the demonstrators on your lab book before leaving the lab.*

- Do not eat or drink in the lab.

IN THE CASE OF A FIRE ALARM:

- REMAIN CALM
- IF IT IS SAFE, EVACUATE THE CLASSROOM OR LAB
- GO TO THE CLOSEST FIRE EXIT
- DO NOT USE THE ELEVATORS

  IF YOU NEED ASSISTANCE TO EVACUATE THE BUILDING, INFORM THE INSTRUCTOR NOW

- IF YOU NEED TO REPORT AN INCIDENT OR A PERSON LEFT BEHIND DURING A BUILDING EVACUATION, CALL SECURITY SERVICES **204-474-9341**
- DO NOT REENTER THE BUILDING

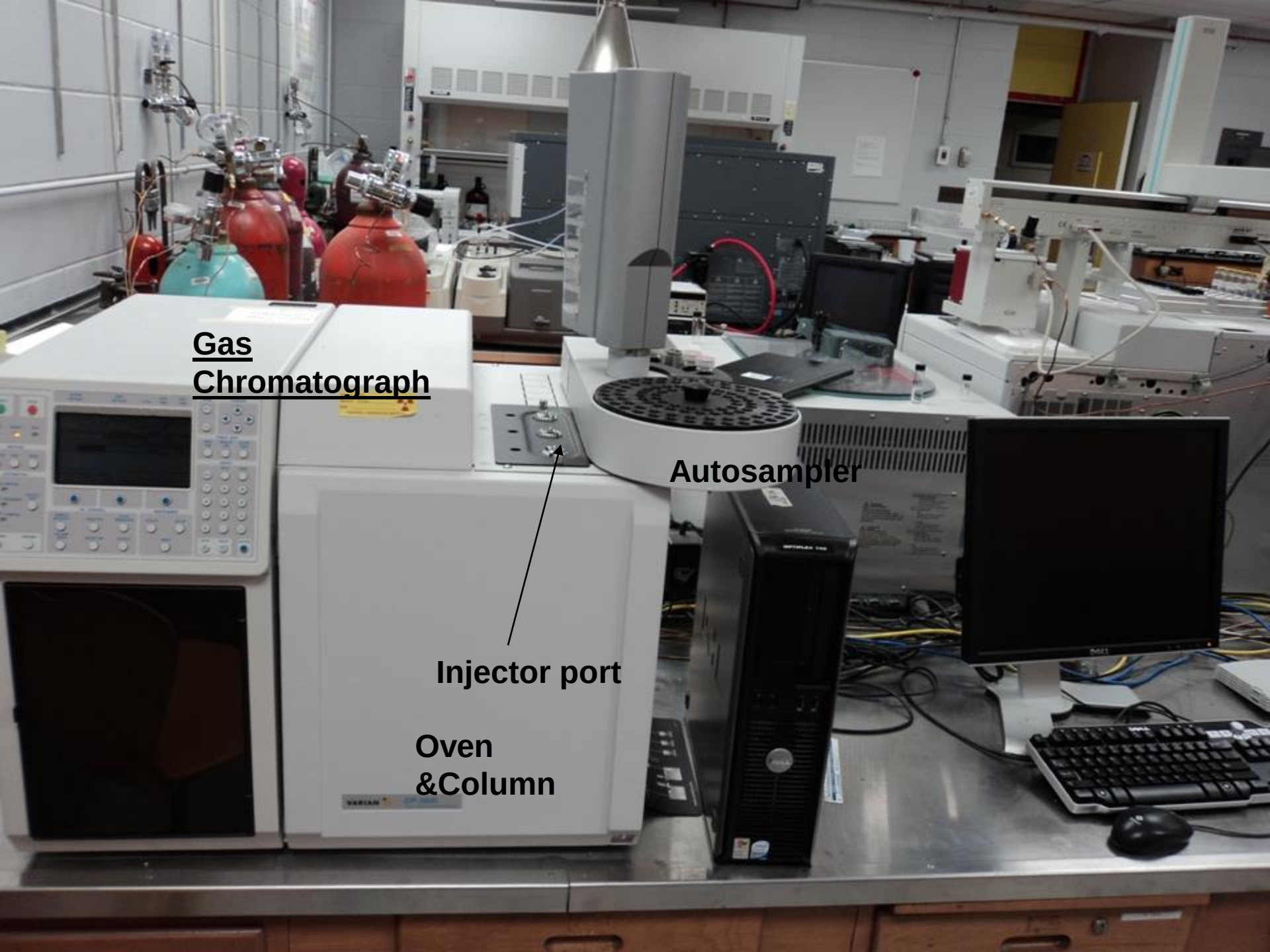
  UNTIL THE **"ALL CLEAR"** IS DECLARED BY A FIRE WARDEN, SECURITY SERVICES OR THE FIRE DEPARTMENT

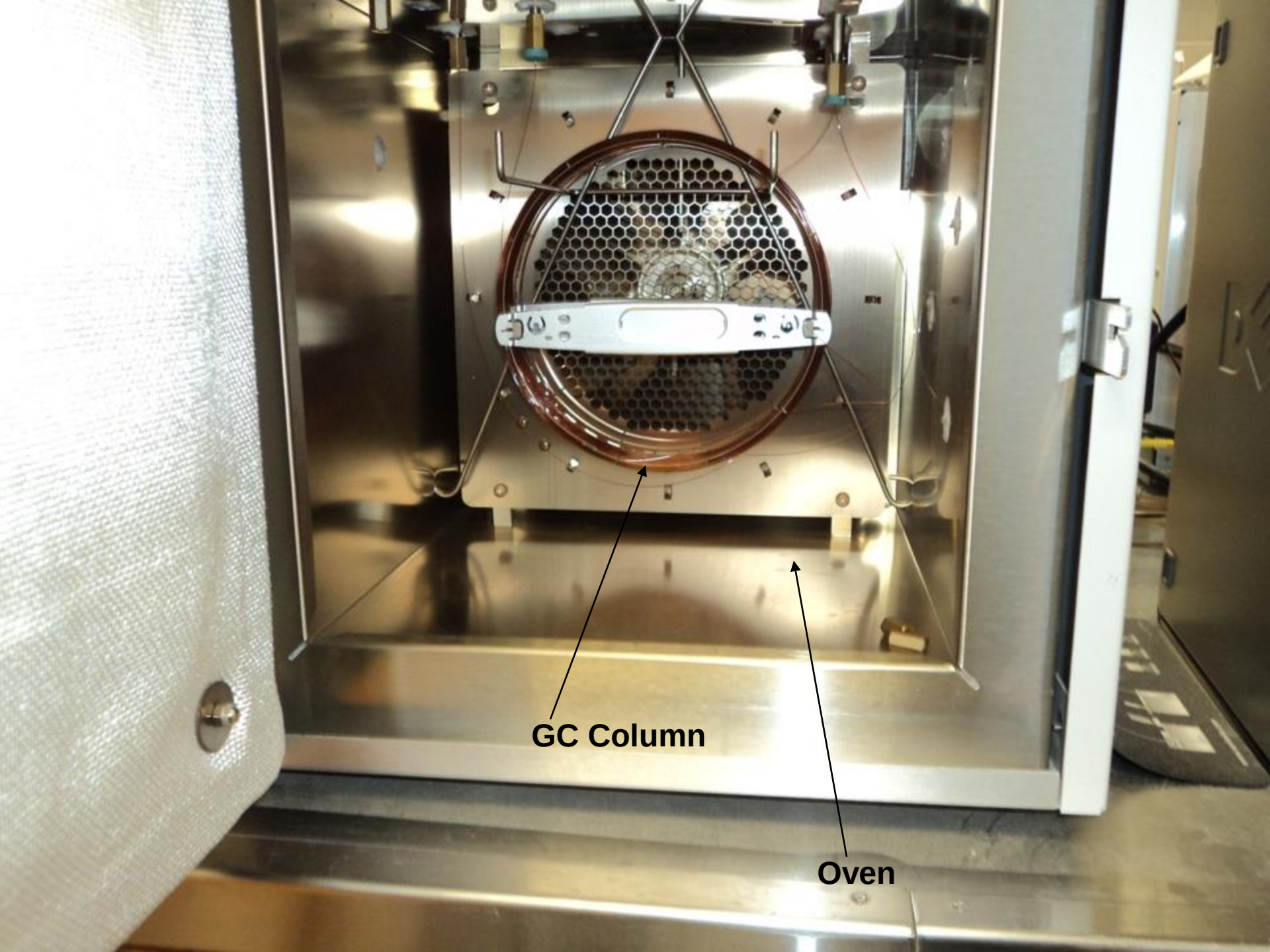
Laboratory Experiments

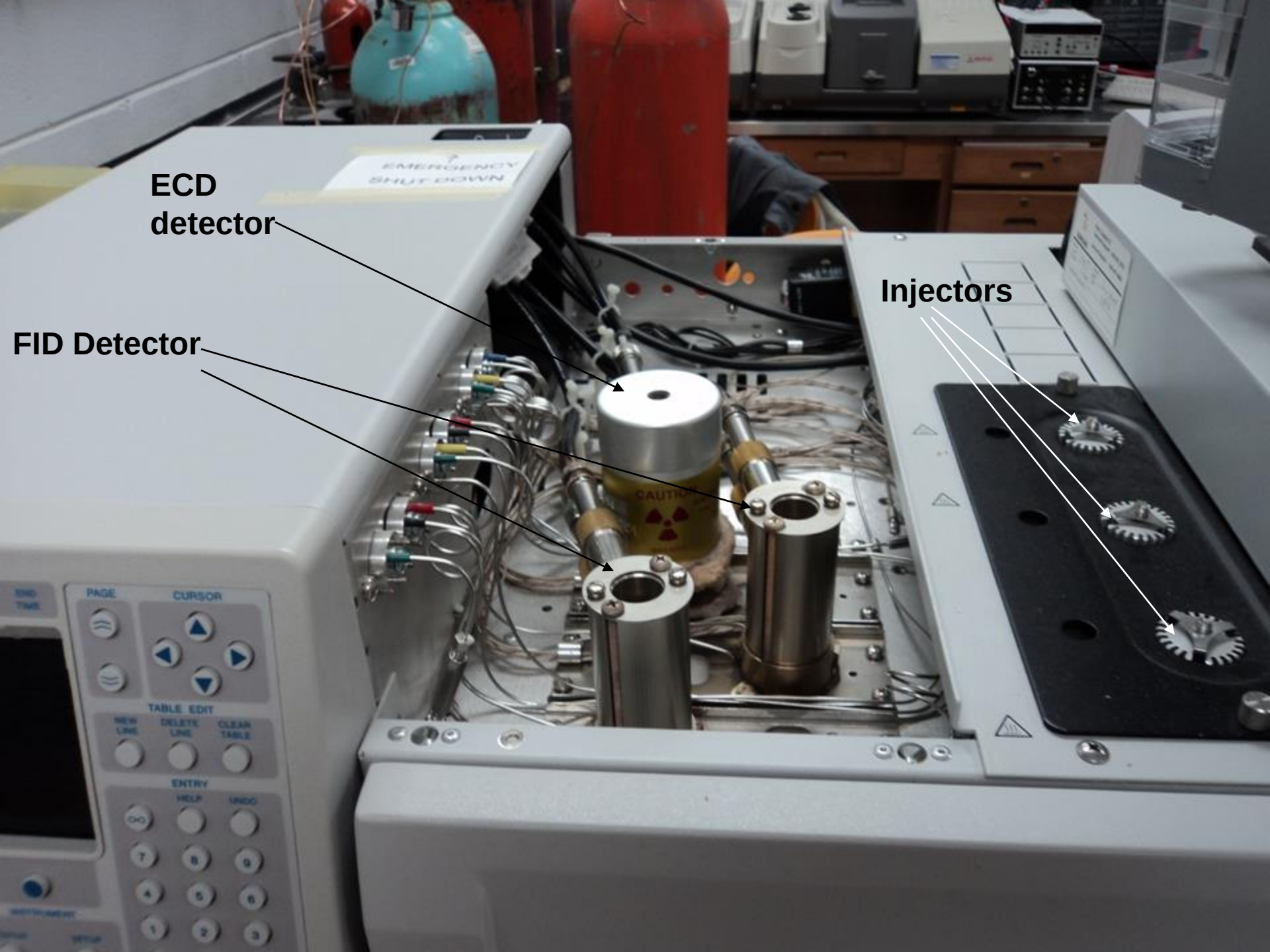
The five experiments include:

- **Spectrophotometry Experiment**
- **Gas Chromatograhy Experiment**
- **HPLC Experiment**
- **Mass spctrophotometry (LC) Experiment**
- **ICP Experiment**

Gas Chromatography (GC) Experiment


Gas Chromatograph
Autosampler
Injector port
Oven &Column


GC Column
Oven


ECD
detector
FID Detector
Injectors
EMERGENCY
SHUT DOWN
CAUTION
PAGE
CURSOR
TABLE EDIT
NEW LINE
DELETE LINE
CLEAR TABLE
ENTRY
HELP
UNDO

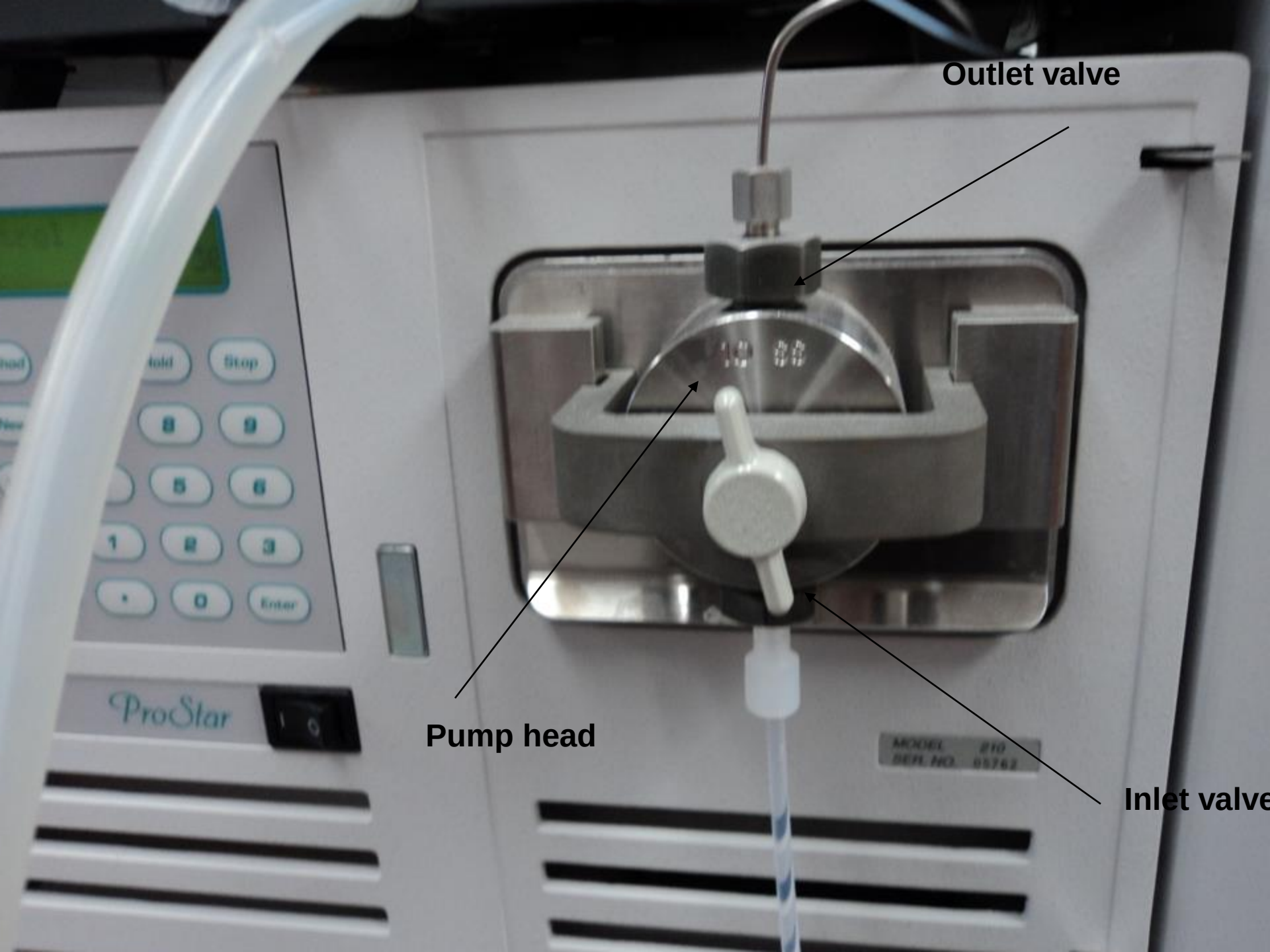

GC 3900
EMERGENCY
SHUT DOWN
CAUTION

GC Experiment

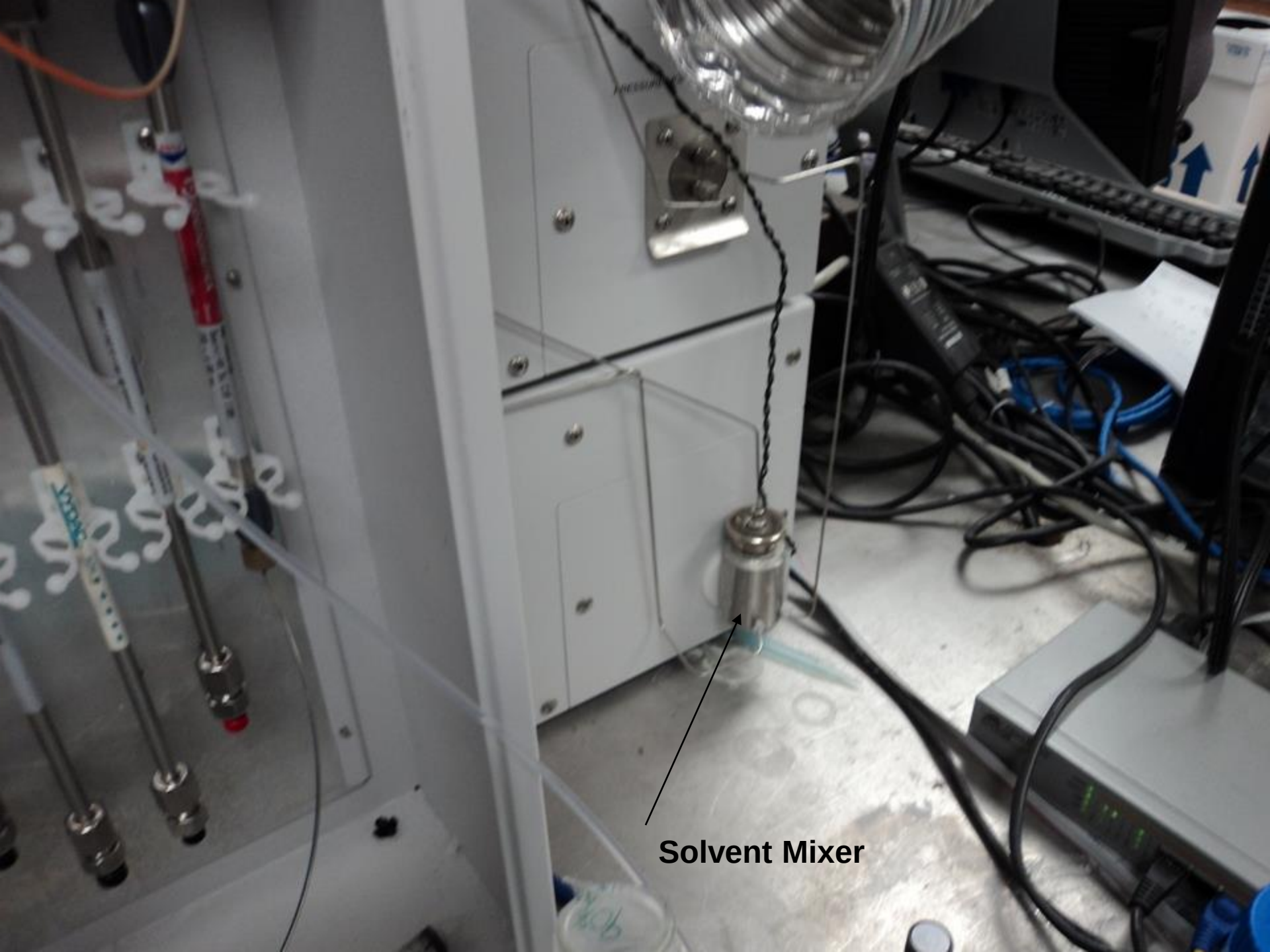
- Seperation of alcohols to determine effect of chain length and boiling point on retention time.
- Determine separation of polar and non-polar compounds on different columns
- Optimizing the 3800 GC for analysis
- Determining the sensitivity (LOD) of a GC with microbore column.

High Precision Liquid Chromatography Experiment (HPLC)


HPLC
Experiment
Solvents
Fluorescence
detector
pumps
Uv-Vis
detector


Outlet valve
Pump head
Inlet valve
ProStar
Stop
MODEL 210


ual
ctor
HPLC
columns


Solvent Mixer

HPLC Experiment

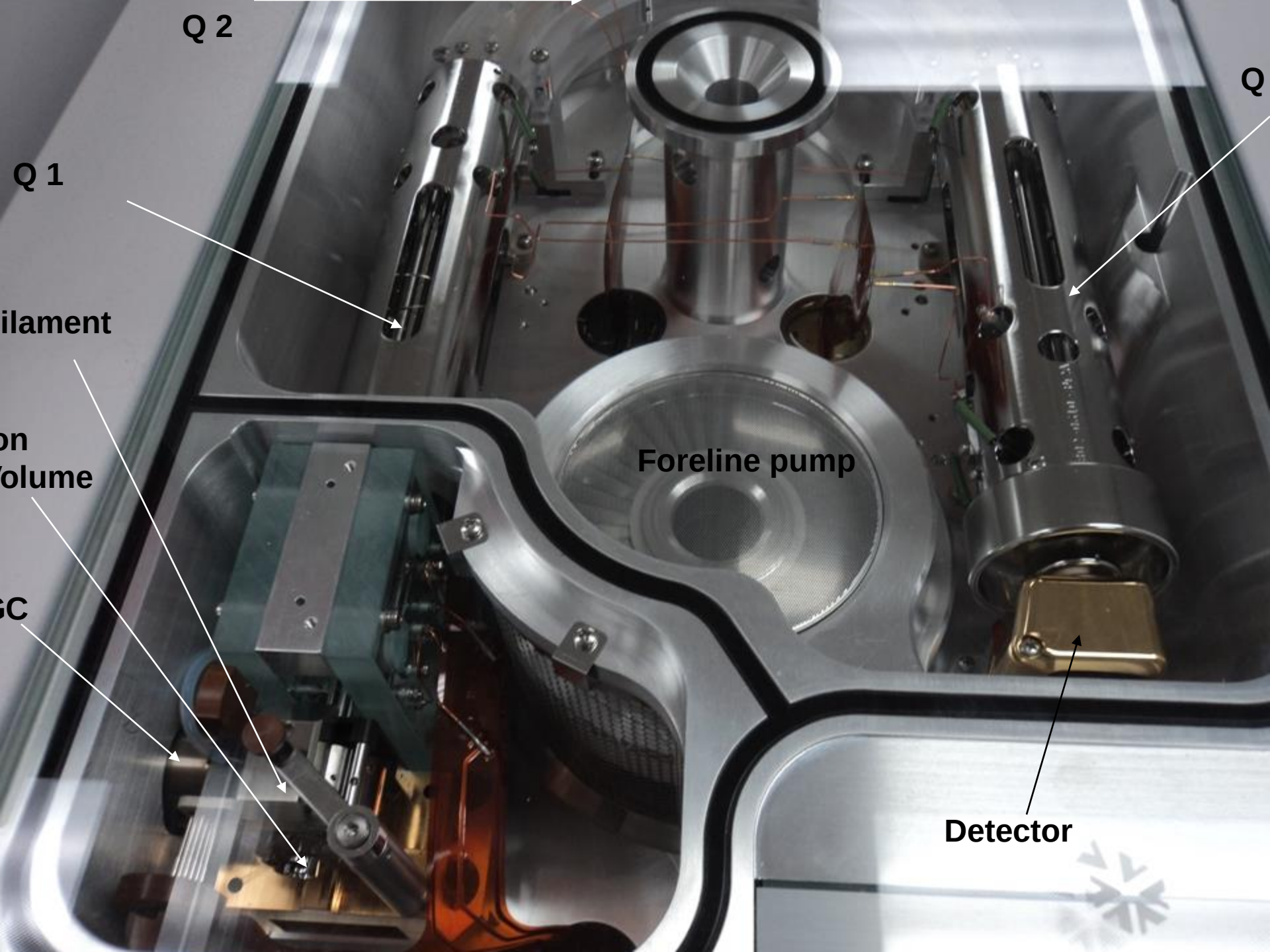
- The MCAL HPLC is a automated binary pump system, using a reverse phase C_{18} analytical column, with manual and automated sampling.

- The LC experiment consists of:
  - The identification and seperation of UV absorbing and fluorescing compounds found in common beverages.
  - The determination of the concentration of beverage constituents by preparation of external standards.
  - The optimizing of the LC seperation method

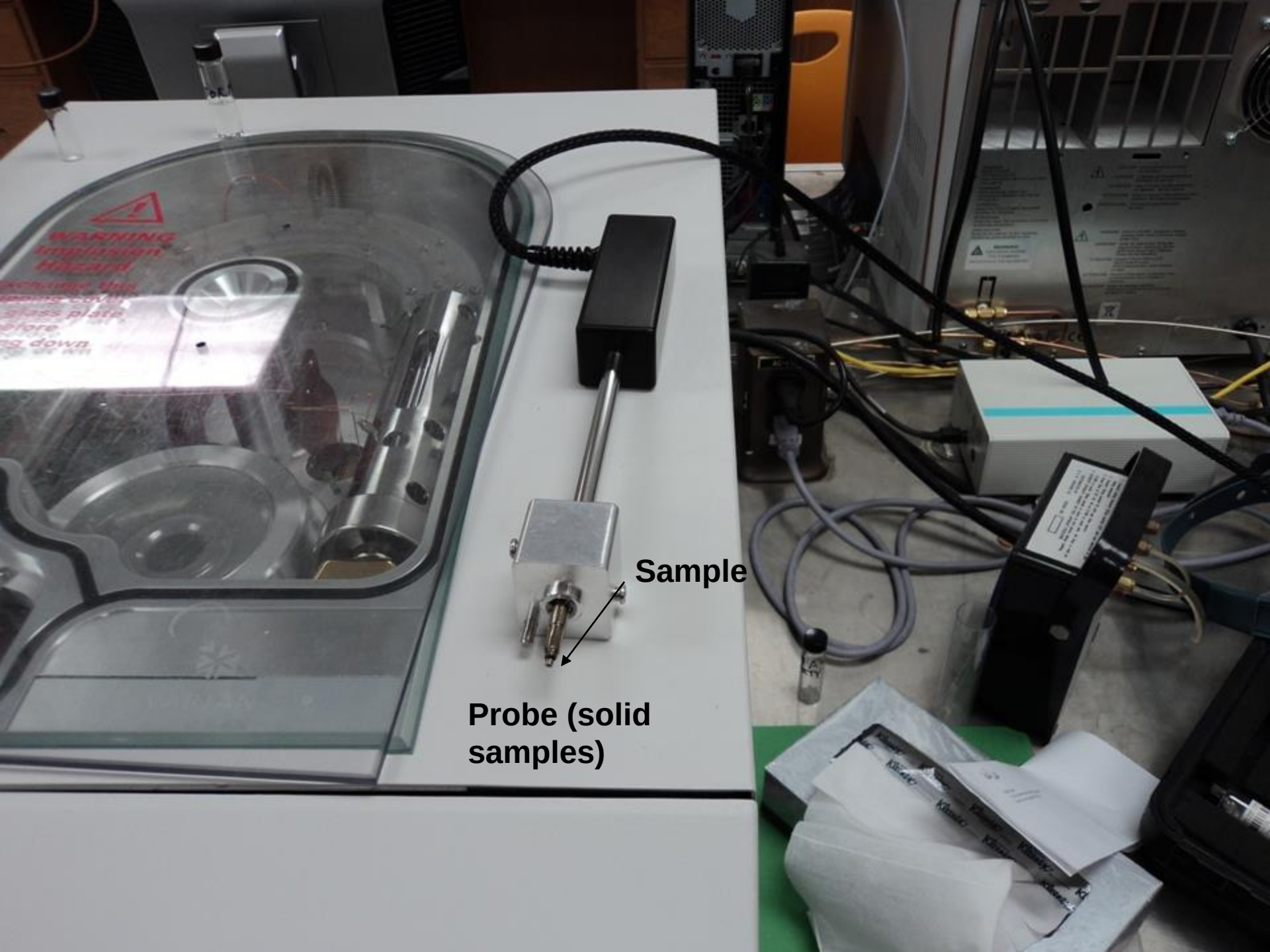
GC-MS Experiment

There is no GC-MS Experiment


Combipal
Syringe-tenex-heater
Mixer-heater
GC Mass Spec.


Q 2
Q 1
Q
ilament
on
olume
GC
Foreline pump
Detector

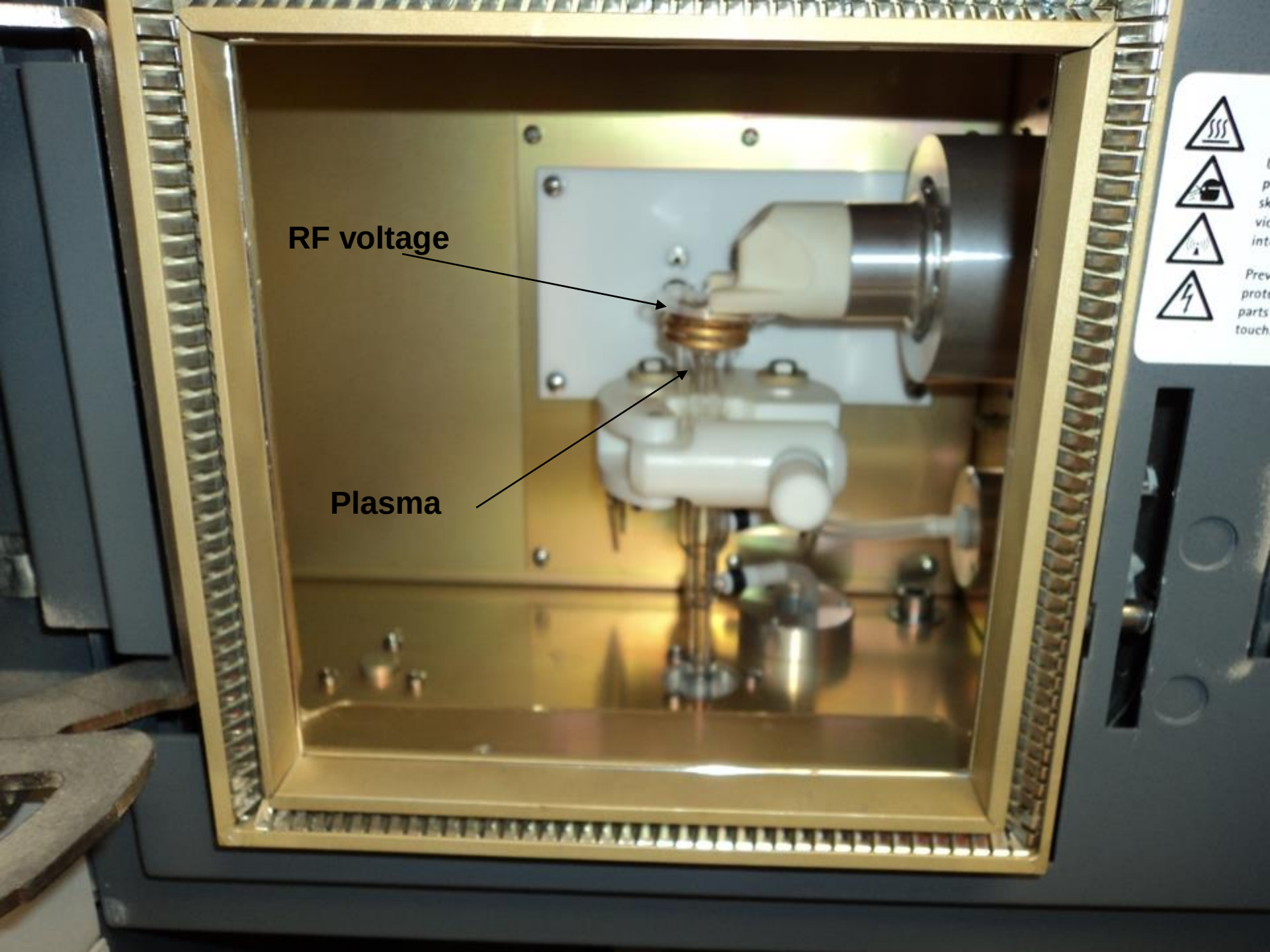

GC column
VF-5ms
30m, 0.25mm, 0.25um
VARIAN
VARIAN

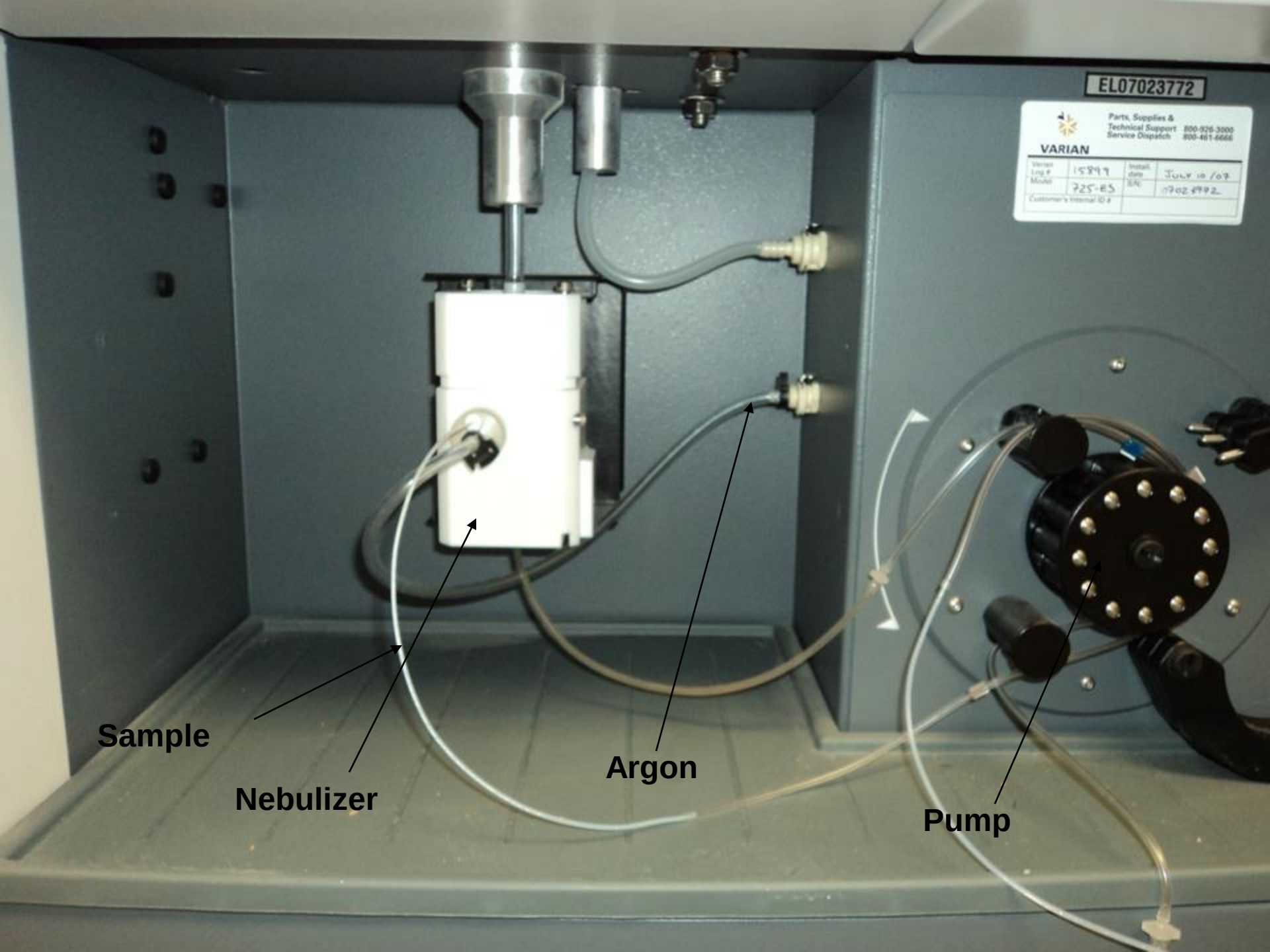

Sample
Probe (solid samples)


Probe
Source On
Scanning
Power

ICP Experiment


VARIAN
ICP-OES
Varian 725-ES

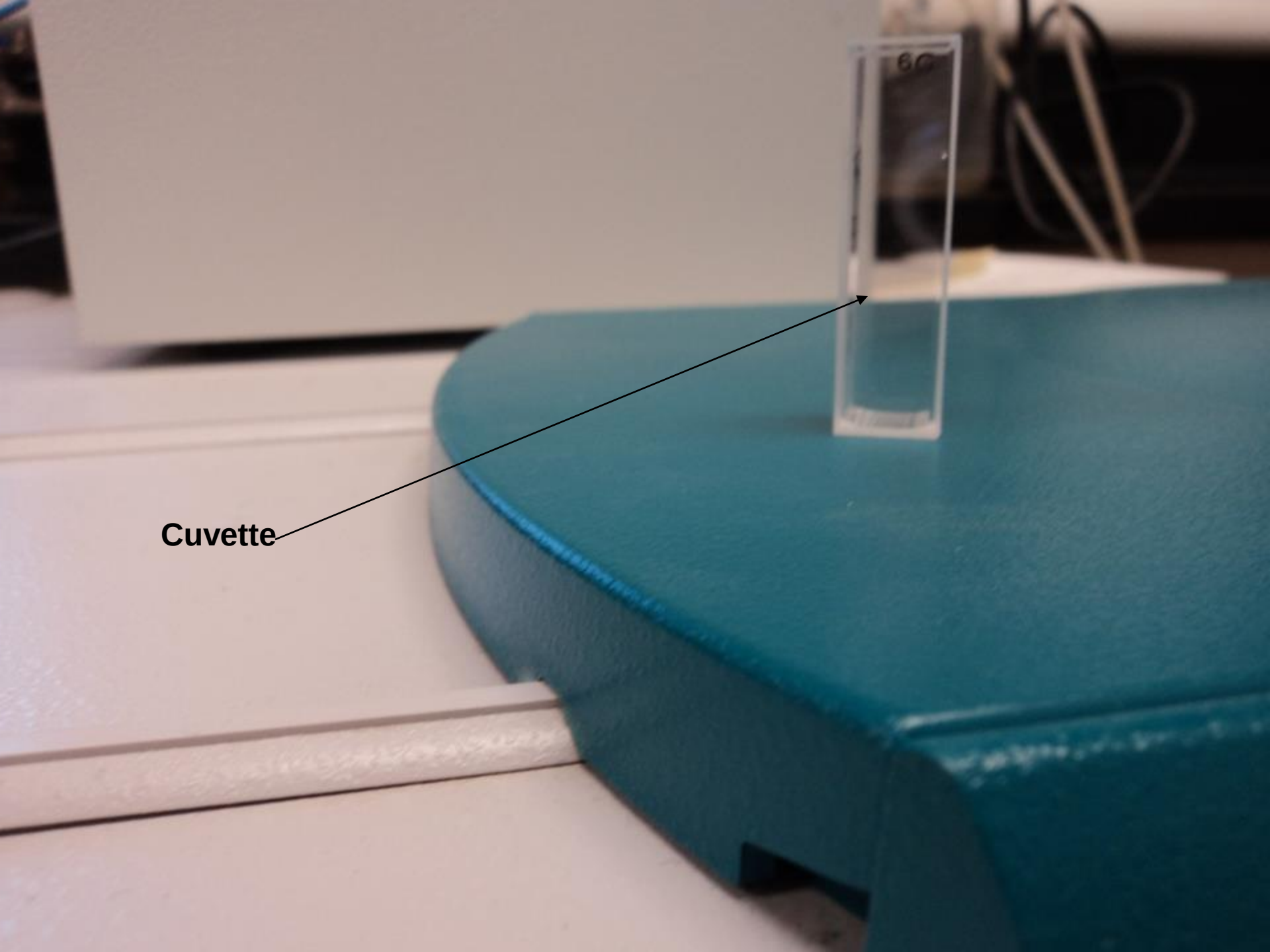

RF voltage
Plasma


EL07023772
VARIAN
Parts, Supplies &
Technical Support 800-926-3000
Service Dispatch 800-461-6666
Sample
Nebulizer
Argon
Pump

ICP-OES

- Elemental analysis of a liquid sample
  - Must be solubilized, with conc. acid.
- Experiment measures the presence and concentration of elements (in ppb).
  - Look at conc. of minerals in water, in food, in rock, in sediment, in metals.
- Must use standards (purchased or made up)
  - weigh and dilute accurately.

Spectrophotometry Experiment


Cuvette


Cuvette holder

UV/Vis – Fluorescence Experiment

Spectroscopy experiments

- Measure UV absortion of vitamin B1 (thiamine) and B6 (pyridoxine) at different wavelengths.

- UV-Vis measuring absorption and relates to concentration by Beer-Lambert Law.

- Fluorometer measures emmission of light from energized fluorescing B6 (pyridoxine) solution.

LC-MS Experiment


LC-MS
Column
LC
Pumps
Autosampler
Electrospray
chamber
Infusion pump
VARIAN

LC-MS

- LC for seperation and ESI mass spec with an ion trap as detector.
- Measures polar, water soluble compounds.
- Compared to GC-MS system which works well with volatile, non-polar samples.

LC-MS Experiment

- Optimization of the LC for the components of interest in the analgesic
- Seperation of the components of the medication.
- Quantitation of the medication components