Appendix C: Tutorial Information

https://web.uvic.ca/~chem213/tutorials/213%20tutorial.htm
(updated after publication of 2018 paper manual)

Fall 2019 Schedule

Date (Mon-Fri)	Tutorial	Location
Sept 9-13	T1: Practical Spectroscopy (Laboratory session)	Starts in regular tutorial location. This tutorial will move between a lab and a classroom through the period. 2 h lab + 2 h classroom.
Sept 16-22	T2: Practical IR (Laboratory session)	Starts in regular tutorial location. This tutorial will move between a lab and a classroom through the period. 2 h lab + 2 h classroom.
Sept 23-27	T3: Theoretical IR	Regular tutorial location. 4 h classroom tutorial.
Sept 30 - Oct 4	T4a: ^{1}H NMR	Regular tutorial location. 4 h classroom tutorial.
Oct 7-11	T4b: Practical NMR (Computer lab session)	T4b is a computer-based tutorial **in Cle A105** (4 h).
Oct 14-18	Thanksgiving (Oct 14) Review for midterm #1	Starts in regular tutorial location. This tutorial will include a short lab session and a review session for midterm #1.
Oct 21-25	T4c: ^{1}H NMR	T4c in regular tutorial location (4 h).
Oct 28 - Nov 1	T5a: ^{13}C{^{1}H}, DEPT, 2D NMR	Regular tutorial location. 4 h classroom tutorial.
Nov 4-8	T5b: Multi-nuclear Organic	Regular tutorial location. 4 h classroom tutorial.
Nov 11-15	Remembrance Day (Nov. 11) Reading Break (Nov 12-13)	No tutorial sessions this week.
Nov 18-22	T7: Multi-nuclear Inorganic	Regular tutorial location. 4 h classroom tutorial.
Nov 25-29	T8: Mass Spectrometry & UV-Vis	Regular tutorial location. 4 h classroom tutorial.

Table of Contents

Senior Lab Instructor

Kelli Fawkes
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Office: Ell 334a Phone: 250-472-5212
Please contact me if you have any questions.

Introduction

Welcome to the tutorial for Chemistry 213 ☺. This tutorial is designed to provide you with the opportunity to solve problems and practice interpreting spectra similar to those in the lecture. This is an intensive, four hour per week, mandatory part of the course. The tutorial will build upon the information in the lectures and will also introduce a few additional topics better suited to the smaller classroom. The tutorial sections are limited to 24 students per instructor to allow for lots of interaction between the instructor and the students as well as to encourage peer-learning in small groups.

There are three main formats for the tutorial sessions.

All of the tutorials are self-contained in the 4hr session and there is no homework. There are no pre-lab questions for marks, but instead practice problems are included in the manual to help you prepare for your tutorial if you choose. These are not collected or graded by your instructor. Answers to the practice problems will be posted on the website for you to check your own work prior to your tutorial.

1. Laboratory based - T1, T2, T4b.

These labs, in a designated chemistry laboratory, will focus on instrumentation, sample preparation and techniques of analysis. Your instructor will give a brief (~20 min.) introduction at the start of the session and then you will work in groups of 2-4 on the main tasks for the remainder of the period, at which time thetutorial question booklet will be due. These are typically very busy sessions and it is strongly advised that you come well prepared by reading the introductory materials in the manual prior to your lab. Check the website for the scheduled location, as some of these labs may not begin in your regular classroom. ***Please wear appropriate shoes and bring a labcoat and safety glasses***. Lab notebooks are not required.

2. Classroom based - T3, T4a, T4c, T5a, T5b, T7, T8.

These sessions, in your regularly scheduled location, use problem-based exercises to focus on interpretation of spectra. Your instructor will give an introduction (~30-50 min.) to the material before you start working on the assigned problems. The introduction will be based on the notes given in this manual. The problems are designed to be completed by the average student working at a steady pace with a small group in about 3 hours, but you will have four hours if needed. You can work individually or in groups, as you prefer, to complete the problems but it is generally acknowledged that a group experience is most beneficial to your learning. The opportunity to both learn from and teach to your fellow students can significantly enhance your understanding of the material.

Any remaining time in the tutorial is best used to work on the lecture assignments while your instructor is available to answer questions.

3. Homework based - T6.

In an effort to reduce student workload this multi-week assignment has been removed from the course. Many students have expressed a desire to continue to access the material so the instructions remain. If you would like to challenge yourself please try the problem posted on the website. The answer key is also supplied. The T6 assignment is a chance for you to apply your skills to a more in-depth problem.

Absences

If you plan to be absent for any reason please contact me, preferably in advance, to arrange to attend another section or to be excused. Medical excuses may require a doctor's note. The easiest way to contact me is by email, but feel free to drop by my office any time if you prefer to meet in person.

Preparing for the Tutorials

It is strongly suggested that you come to the tutorials prepared to work intensively for four hours on the topic. The best ways to prepare for each tutorial include:

- attend the lectures and read the relevant portions of the lecture manual
- read the introductory information in these instructions for each topic
- complete the practice problems and check your answers on the website

Organization will also allow for you to work more efficiently during the tutorial. It is suggested that you keep all the tutorials and handouts together in a binder - many exercises build on earlier problems and you may wish to refer back to these. Other supplies needed each week include:

- calculator, ruler, pencil, eraser
- lab coat and glasses (for T1, T2 and T4b lab sessions only)
- 213 course manual (optional, but most students prefer to bring this along)

Italicized keywords for each tutorial will provide a guide for finding further information on the topic, in the glossary for the course manual. Most general (1st year) and introductory organic chemistry textbooks should have more than enough information to supplement these instructions. There is no need to print this appendix.

Webpage

Answers to the practice problems, links to instructors' contact information, tutorial marks, schedules for each tutorial section, additional problems, and much more can be found at http://web.uvic.ca/~chem213/tutorials/213%20tutorial.htm

Grades

Please check your marks on the website regularly. Errors should be brought to the attention of your instructor as soon as possible. Grade disputes should be brought to my attention as soon as possible.

Office Hours

The tutorials are completed in class and there is no associated homework, so the tutorial instructors do not have office hours.

Suggestions

This tutorial format is constantly changing based on feedback from students. The material presented in the manual and tutorials is prepared by me each year and I encourage you to share your feedback with me. Comments and suggestions are welcome at any time, by email or in person. Anonymous suggestions can be slipped under my office door.

Thank you for taking the time to read the introductory portion of the tutorial manual. If you have any questions or concerns, please feel free to contact me at your convenience. I look forward to a successful series of tutorials and I hope you enjoy the course!

Sincerely,

Kelli Fawkes
Chem 213 Senior Lab Instructor
fawkesk@uvic.ca
Ell 334a 250-472-5212

C4

Policy Regarding Absences, Exemptions, and Extensions
By registering in the course & section, you have implicitly agreed to attend at the designated meeting times and to submit the required work by the published deadlines. We recognize that circumstances beyond your control can impede your ability to meet these obligations. The following is a guide as to how you might reasonably expect alternative procedures to be applied. It is not meant to be an all-encompassing document - merely an indication as to how the Senior Laboratory Instructors will approach such issues.

In general, only medical or compassionate grounds will excuse you from meeting a deadline or completing laboratory work. These absences will require documentation where appropriate and must be shown to the Senior Lab Instructor immediately on return to campus. The amount of work excused or re-scheduled will depend on the severity of the circumstance.

When you know that you are going to be absent from a scheduled class, for whatever reason, then contact the relevant Senior Lab Instructor as soon as possible. Contact may be made by email, phone, written note or in person. Please give sufficient information so that you can be given a prompt reply. If you fall ill during a class, leave a note addressed to the SLI with the lab instructor.

Absences and extensions will be considered in the light of:
(a) how much the circumstance is within the choice or control of the student.
(b) whether the same benefit could be extended to other students in the course in a similar situation.
(c) whether space is available for rescheduling. Rescheduling is not automatic, and if granted, may be for a reduced grade.

Please bear in mind that in order to pass a laboratory or tutorial course you must complete and have graded at least 70% of the work assigned, regardless of circumstance. This ensures that a passing mark also reflects a competency on the bulk of the course material.

Without an exemption from the relevant Senior Lab Instructor, all work not completed, or not handed in, will be given a mark of zero.

If you are sick, expect to provide adequate documentation to support your request for a concession which may be in the form of an exemption, a re-scheduled lab or an extension of a deadline.

You are encouraged to consult with the Senior Lab Instructor if you are in need of special considerations through a temporary or permanent disability.

The Senior Lab Instructors for second year courses are:
Dave Berry, ELL334e, phone 250-721-7170, email berryde@uvic.ca
Inorganic Chemistry (Chem 222, Chem 260) & Lab Supervisor

Kelli Fawkes, ELL334a, phone 250-472-5212, email fawkesk@uvic.ca
Spectroscopy (Chem 213, Chem 260)

Peter Marrs, ELL334c, phone 250-721-7172, email pmarrs@uvic.ca
Organic Chemistry (Chem 232, Chem 260)

Mehraveh Seyedalikhani , ELL334d, phone 250-721-7175, email mehraveh@uvic.ca
Analytical Chemistry (Chem 212)

Policy Regarding Tutorial Grades in Chemistry
In accordance with University policy, a student may elect (by writing to the instructor) to not have her/his marks posted. In any event, marks may only be posted against student number or other private identifier. Names may not be used on a public notice.

For courses that have a combined laboratory/tutorial and lecture content (all Chem 1XX and 2XX):

1. In arriving at the overall grade for a course, normally the distribution shall be tutorial 25%, lectures 75%; the tutorial contribution may be increased at the discretion of the lecturer with the agreement of the Chair at the start of the course.
2. The pass mark in tutorial courses is 50%, and at least 70% of the work (based on total points) must be completed and graded.
3. Students are required to attain passing grades in both tutorial and lecture portions of a course. The pass mark in the lecture portion of a course is set at the discretion of the lecturer.
4. In the case of a student passing the tutorial but failing the lecture portion of the course, s/he has the option - *dependent on available space* - of repeating the tutorial during a second attempt at passing the course.
5. If the tutorial is not repeated, then the original tutorial grade will be counted in the normal formula the second time around.
6. A tutorial grade can only count for <u>one</u> subsequent attempt at passing the course during any one of the following 3 academic years.
7. The decision to repeat/not repeat the tutorial portion of the course will be final after the last day of the period for *adding* courses in that term.
8. A student who wishes to repeat a course without retaking the tutorial must contact the Senior Lab Instructor at the time of registration (in the A section) to request permission to use the previous tutorial mark. If permission is granted, registration in a T section is not required. If permission is denied for any reason, the tutorial must be retaken, and the student must register in a T section of the course in the same term. A late request that is denied will not be grounds for waiving the need to repeat the tutorial.
9. No credit will be given for tutorial work done for a failed course at any other institution.
10. Dropping the lecture component of a course at any time removes all present or future credit for the tutorial portion of that course.
11. In the case of a student repeating a passed course for a better mark, all of the above rules apply.

Safety
The chemistry laboratory-based portions of the tutorials will adhere to the safe practices guidelines of the department. Details can be found on the UVic Chemistry Department website under the Safe Practices tab at https://www.uvic.ca/science/chemistry/undergraduate/safety/index.php

Policy on Cheating and Plagiarism

There are many forms of beating the system that are considered unacceptable methods of gaining credit. Experience has shown that it is impossible to define every version, and therefore each case tends to be judged separately. The overall aim is to prevent unjustified credit being obtained for work that is not one's own.

The penalties for *attempting* to gain unjustified credit must necessarily appear harsh. All instructors must refer suspicious situations to the Senior Lab Instructor responsible for that course. **The penalties that may be applied include a mark of zero for the experiment in question or a grade of "F" for the course**.

The notes below give typical chemistry lab examples of situations that may help to clarify the broader definitions given in the University Calendar.

i) It is unacceptable to submit samples not prepared by the author; to record data from samples not prepared by the author without giving due credit to the donor; to present someone else's data without acknowledging credit (with or without their knowledge); to falsify data.

ii) It is unacceptable to copy written material without using quotation marks; to copy ideas or facts from any source without acknowledging credit; to use another report (be it marked or not) as a source of information.

iii) It is unacceptable to copy, or minimally paraphrase, large sections of text from any source, even if it is referenced and within quotation marks. You are expected to reprocess any reference material that you consult, so that the result is a combination of the information that you have discovered, expressed in your own words to demonstrate your own understanding and the facts are appropriately referenced.

iv) There is a fine distinction between (a) discussing an assignment before an answer is attempted and (b) producing a collaborative effort. Even if collaborative discussion has taken place, the material submitted for assessment must be the result of the author's individual effort.

v) A person who supplies, knowingly or not, material that is used by someone else to cheat is considered to be equally accountable, and will be subjected to similar penalties.

Please be aware that you are responsible for keeping your work secure. It is unwise to leave written or electronic material on the department computers. Do not save files on computers with no protected access. The University rules regrading academic integrity are available in the calendar at https://web.uvic.ca/calendar2019-05/undergrad/info/regulations/academic-integrity.html .

The Chem 213 tutorial is a special case as we strongly encourage you to work in a group to solve the problems and complete the assigned work. This does not mean that any one person can supply the answers to anyone else. Everyone in the group must be contributing to the solutions. If that is not the case, your tutorial instructor reserves the right to re-organize the groups or require students to work independently. If the problem persists you may be penalized.

Tutorial 1: General Concepts of Spectroscopy

This session will start in Ell 228 before moving to the lab in Ell 349.
Please bring a labcoat and safety glasses (lab notebooks are not needed).
Please wear proper shoes (no sandals) and long pants (no shorts, skirts or capris).

Goals

- Examine the fundamental components of a *spectrometer*. All spectrometers are based on the same principle of source, sample compartment, wavelength selector and detector. We will look at different examples of sources, cells and wavelength selectors to see how these vary.
- Become familiar with the *electromagnetic spectrum* and the various applications in the major regions.
- Explore the components of white light and the difference between observed, absorbed and transmitted colors.
- Learn to use a UV-Vis spectrometer and choose an appropriate cell.
- Understand the interactions between matter and light and how spectroscopy utilizes these properties.
- Meet your tutorial instructor and your classmates. Find out where and when your next tutorial is scheduled. Have some fun!

Lecture Material: This tutorial is based on pp. 1-12, 241 in the lecture manual and assignment #1. Please read over these pages in addition to this introduction, in preparation for the tutorial.

General Information

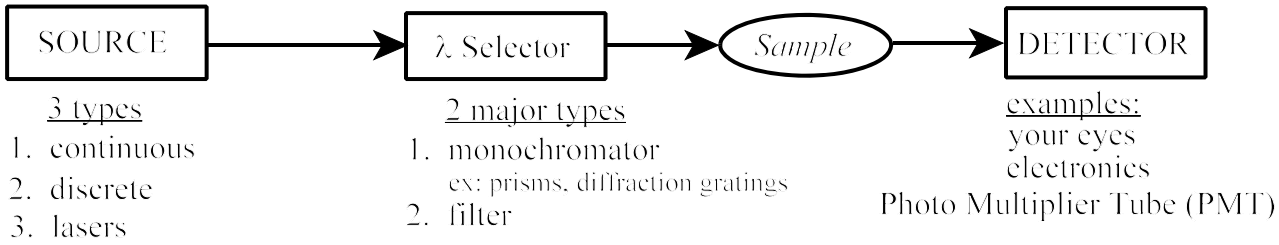
The instructor will begin the class with a brief introduction (~30 min) and then split the class into small groups to work at the various stations in turn. A handout will be given that contains the instructions and questions needed to complete the exercises. Average time = 3.5 hr.

Practice problems

1. Calculate the frequency of the ultraviolet line of wavelength 285 nm in MHz.
2. Calculate the energy of a photon of wavelength 232 nm. What region of the electromagnetic spectrum does this lie in?
3. An argon laser used to weld detached retinas to the human eye produces radiation with a frequency of 4.69×10^{14} Hz. What is the wavelength of this radiation in nm? What color would this laser appear?
4. Calculate the energy per mole (in kJ mol^{-1}) that an object can absorb from the 589 nm wavelength light emitted from a sodium lamp.
5. A unknown element with 1.00×10^{23} molecules in the ground state shows an emission at 656 nm. How many molecules are present in the excited state at 1741 K?

Answers to the practice problems are posted on the website.

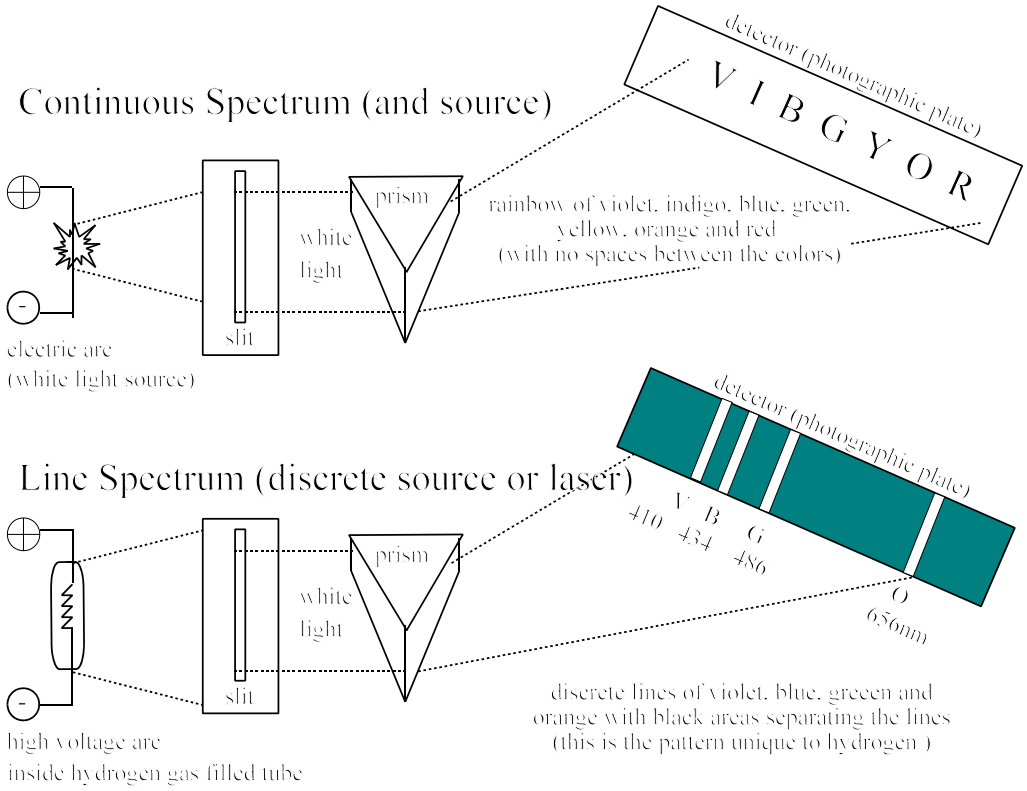
General spectrometer design



Sources

Most *spectrometers* require a radiation source of some type. In the examples used for this exercise, all of the sources visibly show *radiation*. The mercury lamp also has a high ultraviolet intensity, which is invisible (but potentially damaging) to the human eye, so avoid looking directly at the source without safety glasses.

Depending on the application, a radiation source can produce a *continuous* output of various wavelengths or it can produce selected *discrete* wavelengths. Most of the sources used for UV and visible absorption spectroscopy are continuous sources (such as tungsten lamps). Conversely, atomic absorption spectroscopy sources are discrete sources that produce emissions at particular wavelengths depending on the element in the lamp.

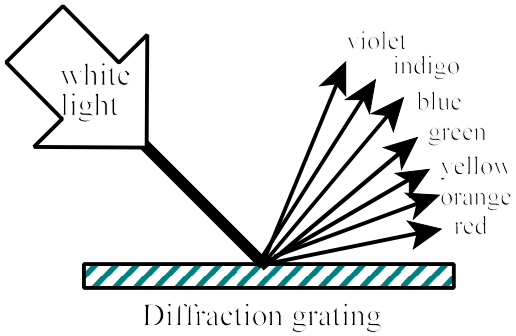


Lasers emit monochromatic light, so no wavelength selector is needed. Discrete and continuous energy sources need additional hardware to isolate the wavelengths of interest.

Wavelength Selectors

As discussed above, most spectroscopy sources produce more than one wavelength of radiation. To get a meaningful spectrum, it is usually necessary to measure a response at one wavelength, or at least one wavelength at a time. *Prisms* and *diffraction gratings* are the most common *monochromators* used to disperse radiation into its component wavelengths; entrance and exit slits are used to isolate a small portion of the dispersed radiation, which is then focused on the sample. Narrow slits let through very little radiation, with a very narrow range of wavelengths. Wider slits let through more radiation, but also a larger range of wavelengths.

A *prism* works by dispersing light through angled glass or quartz, or droplets of water in the case of rainbows. *Diffraction gratings* work by reflecting and dispersing light from a highly polished glass surface scored with thousands of etched grooves. This effect is visible when you look at the repeating 'rainbows' on the back of any CD.



Most spectroscopy instruments use diffraction gratings, instead of prisms, as less light is lost by absorption of the material and the angle of the etched grooves can be precisely controlled. Gratings require less space inside the instrument and can transmit UV radiation. Overall, this results in more speed and resolution per dollar compared to prisms and often a single grating can replace several prisms due to the greater dispersion of radiation.

If only a very simple, single-wavelength spectrometer is needed (to measure transmitted light intensity at one wavelength), a *filter* can be used instead of a monochromator. Filters are much cheaper and require no electronics; however, they have other disadvantages - low sensitivity, for example. A filter-based spectrometer would use one or more filters designed to remove (ie. absorb) all radiation except that within a very narrow range.

Filters are more commonly used to produce the lighting effects for theatrical productions using *visible* light sources. In these applications it is typically the removal of a large series of wavelengths from the electromagnetic spectrum that affects a particular color. For example, placing a blue transparency in front of a white light (which contains all colors) absorbs the red, orange and yellow wavelengths of light and transmits green, blue and violet to our eye which we observe as blue. Lighting technicians use filters designed to absorb a single color of radiation for much finer control of the color observed on the stage.

absorption region	absorption wavelength (nm)	color observed	absorption region	absorption wavelength (nm)	color observed
violet	375-435	yellow-green	yellow-green	560-580	violet
blue	435-480	yellow-orange	yellow	580-595	blue
green-blue	480-500	orange-red	orange	595-650	green-blue
green	500-560	red-violet	red	650-780	green

Detectors

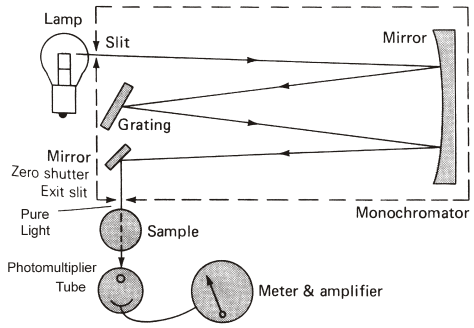
Just as there are several types of sources, there are also several types of detectors. The ideal detector is both very stable and very sensitive. Unfortunately, these two qualities are usually difficult to combine; a sensitive detector is often not very stable, and a stable detector is often not very sensitive. Your eye proves to be an excellent detector in the visible region.

A common detector for UV and visible light is the *photomultiplier tube* (PMT). It uses the photoelectric effect to convert photons into current. PMTs are used in UV/Vis spectrometers, fluorometers, atomic absorption spectrometers, etc. Infrared (IR) light isn't detected by PMT's very well, so most IR instruments use solid-state detectors made of single crystals. The undergraduate IRs use a crystal of lithium tantalate. This detector converts the transmitted radiation (*photons*) into electrons and the resulting current is then measured.

Simple UV-Vis spectrometer design

One of the earliest and most common types of *spectrometer* in the sciences is the UV-Visible instrument. You have likely already used this in your high school and introductory chemistry courses. Like all spectrometers, it has the basic design principles of *source* (of energy of the appropriate wavelength),*wavelength selector* (such as a prism or diffraction grating), *sample compartment*, and *detector*.

When electromagnetic radiation from the source is directed onto a sample, the light may be *reflected*, *transmitted*, or *absorbed*. The measurement of the absorption of radiation has been found to be a useful tool for the quantitative and qualitative analysis of many inorganic and organic molecules. This is because the ultraviolet (about 180 - 350 nm) and visible (about 350 - 850 nm) wavelength ranges correspond to the energy differences in the outer electronic orbitals of molecules.



Modern UV-Vis instruments have a tungsten lamp as the white light *source* for the visible region and a deuterium lamp for the ultraviolet region.

Monochromatic (single wavelength) light is generated by reflecting the incident light from a *diffraction grating*, not through a prism. This light is then passed through the sample before it is detected by a solid state detector. The diffraction grating can be rotated to generate a continuous scan of the region instead of a single wavelength of light, as needed.

The detector is a *photomultiplier tube* which counts the number of photons hitting it. Samples are typically dilute solutions in water or an organic solvent and are held in a specially designed *cuvette* or *cell*.

Cell selection

The choice of *cell* material is very important as many cells absorb radiation in portions of the UV-Vis spectrum. The cells used are made of various materials. They are made to exact dimensions (to give a known path length) and are highly polished on the two sides through which the light beam will pass (to avoid distortion of the light). There are three types of cells used routinely in this department:

(1) *Glass* is the most typical cell due to its chemical resistance and lower cost.

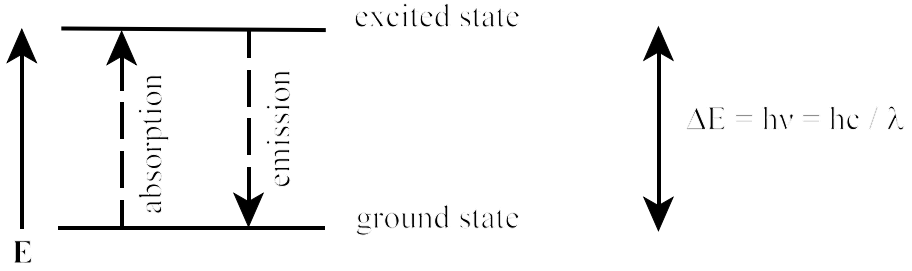
(2) Disposable *polystyrene* can only be used for aqueous solutions (polystyrene dissolves in organic solvents).

(3) *Quartz* cells are reserved exclusively for the UV region (below 350 nm) due to their high cost. These are manufactured with different grades of silica for different regions of the UV spectrum.

code	material	guaranteed transmittance	cost
P	Polystyrene	80% at 400 nm	6¢
G	Optical glass	80% at 365 nm	$38
Q	Quartz, standard silica, UV grade	80% at 200 nm	$87
I	Quartz, IR grade silica	80% at 220 nm	$96

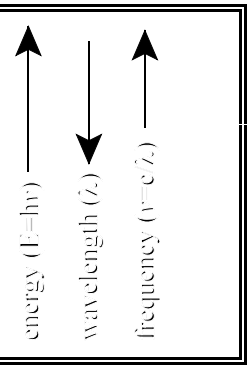
Why do we see a signal?

Atoms and molecules present in the sample are excited by *absorbing* radiation from the source. From these excited atoms only certain wavelengths are *emitted* as the excited electrons return to the ground state. The size of the energy gap between the excited and ground states determines the region of the spectrum in which the transitions can be observed.



Electrons in molecules occupy molecular orbitals with precise energy levels and their transitions involve absorption in the *ultraviolet* and *visible* regions of the spectrum. Much smaller quantities of energy are associated with changes in the vibrational energy of a molecule and these are observed in the *infrared* region of the spectrum. Energy changes associated with certain nuclei in magnetic fields are smaller still; these occur in the radio-frequency region and form the basis for *nuclear magnetic resonance* spectroscopy. Differences in the magnitudes of the energy changes involved dictate the necessity for different instrumental arrangements for UV, Visible, IR and NMR spectroscopy. Understanding why a particular wavelength of radiation is absorbed (or emitted) provides a key to the structure of the molecule under investigation.

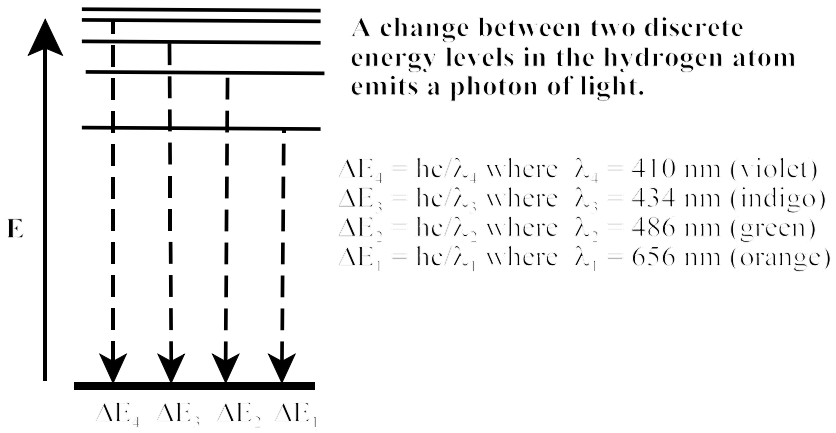
Spectral region	Energy Transitions
x-ray	nuclear changes (changes in energy of core electrons)
ultraviolet and visible	electronic (changes in energy of valence electrons)
infrared	vibrational (internal vibration of a molecule)
microwave	rotational (rotation of entire molecule)
radiowave	nuclear (nmr) and electronic (esr) changes in spin states



Atomic Emission Spectroscopy and the Flame Test

Atomic absorption (AA) spectroscopy is usually used for *quantitative* analysis. Atomic emission spectroscopy can be used for *qualitative* identification as many elements emit characteristic emission spectra when heated.

When a solution enters the burner flame in an AA, the molecules decompose into atoms due to the high temperature (about 2500 Kelvin). The flame temperature also determines the population of the energy levels in the atoms by the Boltzmann Distribution. Depending on the particular arrangement of energy levels, the thermal energy provided by the flame may be sufficient to populate some of the *excited states* of the atom. When these excited atoms return back to the *ground state*, they emit a photon of light with a wavelength (λ) determined by the energy difference (ΔE) of the levels. The arrangement of energy levels is *quantized* and unique for each element so the wavelength of the emitted photons is also unique.



Determining the atomic emission spectrum for each element allows for qualitative identification as each element will have a unique *line spectrum* consisting of a series of separate lines.

In its simplest form, this also forms the basis of the *flame test* that you may have seen in previous chemistry courses. The flame test is used to identify elements such as strontium, copper, sodium etc. that emit radiation in the visible range when heated by a bunsen burner flame. The characteristic wavelength(s) of light emitted by each metal is used to confirm the identity of the species present in the sample, simply by observing the color of the flame - no electronics needed.