Teaching Instrumental Analysis without a Textbook

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There is strong motivation for instructors of Instrumental Analysis to use online supplements for their course or to possibly function without a textbook. But paring down the ASDL sources and links into one usable document takes quite a bit of time. Hopefully this document will allow those interested to be able to gather the information quickly.

The following contains learning objectives and corresponding web links for topics seen in most Instrumental Analysis courses. Instrumental Analysis is the second course of a two-semester sequence for analytical chemistry at the United States Military Academy. The textbook used in the course was different editions of *Principles of Instrumental Analysis*, by Skoog, Holler, and Crouch. Dr. Way Fountain, Dr. Dawn Riegner, and Dr. Tom Spudich have taught instrumental analysis at USMA from 1996 to the present. We have assembled this information for anyone to use in their course; all we request is that you acknowledge us and/or others that have provided the links for the work that has been done. Note that there is a plan to include problems (and access to solutions) for all the lessons, but we cannot guarantee a date as to when this will occur. If you have suggestions or comments, please do not hesitate to email us: tspudich@maryville.edu or dawn.riegner@usma.edu

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How to use this document:
These topics and learning objectives are currently grouped for a traditional 75 minute class and set up so they can be ported directly into a syllabus. There are general topics listed in the Table of Contents (below) that take you to a listing of possible learning objectives for this topic. There are references, which are mainly online links, for the general topic OR specific learning objective. All online links are underlined and in italics. Links that are highlighted in gray are references that are in the Analytical Sciences Digital Library (www.asdlib.org). Note that other than the Analytical Chemistry 2.0 textbook by David Harvey, and the Atomic Emission Spectroscopy learning module by Alexander Scheeline and Thomas Spudich, the links are not shortened in any way, thus giving the reference for the document. Additionally, there are no links or learning objectives for any electrochemistry, as it was not offered in the instrumental analysis portion of the course.

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Note: All online links are underlined and in italics. Links that are highlighted in gray are references that are in the Analytical Sciences Digital Library (www.asdlib.org).

Press the “Ctrl” button and Click on the title of interest to go to the learning objectives and links for that particular lesson.

1. What is Instrumental Analysis? / Data domains
2. Digital Electronics
3. Signals and Noise/DAQ Lab
4. Electromagnetic Radiation
5. Introduction to Optical Spectroscopy
6. More Optical Spectroscopy
7. Introduction to Atomic Spectroscopy
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11. Preparation of lab report/critical journal reading
12. Instrumentation for Molecular Absorption Spectroscopy
13. Applications of Molecular Absorption Spectroscopy
14. Molecular Luminescence Spectroscopy
15. Infrared Spectroscopy
16. Instrumentation for Infrared Spectroscopy
17. Nuclear Magnetic Resonance Spectroscopy (NMR)
18. Applications of NMR
19. Mass Spectrometry
20. Mass Spectrometry, continued
21. Re-introduction to Chromatography
22. Gas Chromatography
23. Liquid Chromatography/ Capillary Electrophoresis
What is Instrumental Analysis? / Data Domains

1. Be able to explain how there are physical and chemical characteristics of matter that can be probed by various instrumental techniques.
2. Define transducer.
3. Define data domain.
4. Understand the numerical criteria for selecting analytical methods.
5. Relate the usefulness and differences of calibration curves, standard additions, and internal standards (this means be able to create & use them to identify unknown concentrations as well).
   (Data Reduction Section of AES web page, Harvey – Chapter 5)

https:// facultystaff.richmond.edu/~rdominey/301/local/Intro_Instrum_Analysis.pdf

Digital Electronics
Note: You should be familiar with computer components and applications. If not, ask about them in class.

1. Convert between base-10 and binary numbers and vise versa.
   [Links to conversion resources]

2. Be able to explain how analog to digital conversion (ADC) occurs and be able to compare an analog plot to a digital plot of the same data.
   [Links to ADC resources]

3. Relate why more bits in an ADC leads to a reduced uncertainty as well as max % error. Relate cost to number of bits (research outside the text for approximate costs).
   [Links to ADC cost analysis]

4. Be able to explain the flexibility that is needed using computers and instrument specific hardware and software.

Signals and Noise/DAQ Lab
General Signal and Noise references:

1. Be able to explain what the S/N ratio represents; know how to calculate it, and how the relative ratio can be increased.
2. Know the five types of noise and characteristics in which to reduce noise.
3. Discuss ways to improve the S/N ratio both via analog and digital signal processing. Be able to use the ensemble averaging formula to determine the number of scans needed to improve the S/N ratio by a known numerical amount.
4. Be able to discuss potential problems associated with signal processing.
   [Links to signal processing resources]
Electromagnetic Radiation

1. Define, explain, apply, & use terms associated with wave and particle properties of electromagnetic radiation.
   Harvey – Chapter 10A1-10A.2
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/UV-Vis/spectrum.htm#uv1

2. Be able to calculate wavelength, frequency, and energy of electromagnetic radiation.
   Harvey – Chapter 10A.2

3. Understand the possible interactions of electromagnetic radiation with matter, i.e. scattering, transmission, diffraction, refraction, reflection, polarization. (AES web page)
   Harvey – Chapter 10A.2, 10H.1
   Scheelie & Spudich – links above
   Polarization -- http://en.wikipedia.org/wiki/Polarization_(waves)

4. Be able to explain the photoelectric effect, how spectra are generated, black-body radiation, absorption and emission of electromagnetic radiation. (AES web page)
   Scheelie & Spudich – links above
   Harvey 10A.1, 10A.3

5. Understand the quantitative aspects of emission and absorption methods. Be able to calculate Beer’s Law values.
   Harvey 10B.

Introduction to Optical Spectroscopy

1. Know the 5 components necessary for building optical instruments for absorption and emission studies.
   Harvey, Figure 10.27
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/UV-Vis/uvspec.htm#uv1

2. Know the differences between continuum and line sources. Be able to give examples of each.
   For blackbody radiation sources, be able to calculate the wavelength maximum at different temperatures.
   Harvey 10A.3

3. Be able to explain the differences between filters and monochromators, i.e. the benefits and inferiorities of each.
   Harvey 10A.3

4. Be able to explain the two types of monochromators. (AES web page goes into significant depth for single detector spectrometers and multiple detector spectrometers)
   Harvey 10C.1

5. Know what factors contribute to choosing the slit width of a monochromator.
   AES web page goes into great depth
More Optical Spectroscopy
1. Have a general understanding of the characteristics of an ideal transducer. Understand the differences between single channel and multichannel transducers.

2. Know the two general types of radiation transducers: photoelectric and heat. Specifically, be able to describe how the phototube, photomultiplier, silicon photodiode, photodiode array, CCD, thermocouple, and pyroelectric transducers operate. (AES web page)


Introduction to Atomic Spectroscopy
1. Be able to draw and explain atomic energy level diagrams. Be able to indicate atomic absorption and atomic emission (fluorescence) on the diagram, AES web page, [History & Theory, energy level diagrams].


3. Be able to explain the effects of temperature on atomic spectra, i.e. Boltzmann equation. (AES web page, Sources)

4. Be able to explain the functionality of an atomizer and be able to describe how samples (solid and aqueous) are prepared for eventual atomization. (Introduction of analyte, electrothermal vaporization and nebulizers).

Atomic Absorption
AA Schematic info: [http://slc.umd.umich.edu/slconline/ADVAA/AdvAA.swf](http://slc.umd.umich.edu/slconline/ADVAA/AdvAA.swf)

1. Be able to relate the differences between atomic absorption and atomic emission with respect to the transition that is observed. This includes why limits of detection are better in an analysis for some metals with a particular method (such as FAA vs. FAE, for example) as opposed to others. [http://www.andor.com/learning/applications/atomic_spectroscopy/](http://www.andor.com/learning/applications/atomic_spectroscopy/)

2. Be able to relate specifically why graphite furnace (electrothermal vaporization) AA has a much higher sensitivity than flame AA. [http://www.water800.com/jspx/AConceptBook.pdf](http://www.water800.com/jspx/AConceptBook.pdf)

3. Understand source modulation; be able to relate why there tends to be improved LODs using source modulation as opposed to not using source modulation. [http://www.water800.com/jspx/AConceptBook.pdf](http://www.water800.com/jspx/AConceptBook.pdf)

4. Be able to acquire and process data from an analysis. This includes obtaining a signal for a blank, standards, and unknown; taking into account how the sample was processed (sample blank), which includes dilutions of both the sample & standards; plot the data (Absorbance vs. Concentration) & obtain a calibration curve; calculating a limit of detection for the analyte. [http://slc.umd.umich.edu/slconline/ADVAA/AdvAA.swf](http://slc.umd.umich.edu/slconline/ADVAA/AdvAA.swf) – sample problems in part 9. [http://terpconnect.umd.edu/~loh/models/CalibrationCurve.html](http://terpconnect.umd.edu/~loh/models/CalibrationCurve.html)

Atomic Emission
1. Know the advantages of using an ICP for atomic emission spectroscopy. ([AES web page](http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icp/icp.html))

2. Be able to explain the differences between a sequential and a simultaneous multi-channel instrument for ICP spectroscopy. ([AES web page](http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icp/icp.html) (spectrometers))

3. Be able to explain the different sample introduction methods/techniques used in atomic spectroscopy as well as relate the advantages and disadvantages of all. ([AES web page](http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icp/icp.html), Introduction of Analyte).

4. Be able to relate the functionality of an internal standard with atomic emission spectroscopy. ([AES web page](http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icp/icp.html), Data Reduction)

### X-ray Fluorescence

1. Be able to describe the advantages and disadvantages of atomic X-ray Fluorescence. ([serc.carleton.edu](http://serc.carleton.edu/research_education/geochemsheets/techniques/XRF.html))

2. Be able to explain the five components of the X-ray fluorimeter that you use in the lab. Specifically, it has an X-ray tube source, a sample chamber (for solid, powder, or liquid samples), a monochromator, a scintillation counter with photomultiplier tube for detection, it requires a vacuum pump for operation of X-rays, and computer processing. ([serc.carleton.edu](http://serc.carleton.edu/research_education/geochemsheets/techniques/XRF.html), [innovx.com](http://www.innovx.com/products/handheld), [rigaku.com](http://www.rigaku.com/edxrf/nex-cg.html))

3. Be able to explain how a spectrum is generated from X-ray Fluorescence using an energy level diagram. Also be able to describe the features of the spectrum (i.e. continuous and line features).


### Preparation of Lab Report/Critical Journal Reading

Note that this is a class discussion the components needed for a lab report/journal article. [George Whitesides interview](http://www2.fiu.edu/~collinsl/Article_reading_tips.htm) --- Publishing your research via the ACS web page, May 11, 2011.

[serc.carleton.edu](http://www2.fiu.edu/~collinsl/Article_reading_tips.htm), [analytictech.com](http://www.analytictech.com/mb870/Handouts/How_to_read.htm), [biochem.arizona.edu](http://www.biochem.arizona.edu/classes/bioc568/papers.htm), [examiner.com](http://www.examiner.com/x-6378-Baltimore-Science-News-Examiner~y2009m7d21-Science-Literacy-101-How-to-read-a-scientific-paper)
Instrumentation for Molecular Absorption Spectroscopy

1. Have a working understanding of Beer’s Law and know the instrumental deviations to it.
   http://terpconnect.umd.edu/~toh/models/BeersLaw.html
   http://www.youtube.com/watch?v=O39avevqndU&feature=channel_page

2. Be able to explain the three categories of instrumental noise or uncertainties in transmittance measurements. Know typical sources of each.

3. Be able to explain the differences between H₂, D₂, W, and Xe arc lamps as well as light emitting diodes (LEDs) as sources for UV-Vis spectroscopy.
   Harvey 10A.3
   http://en.wikipedia.org/wiki/Fluorescent_lamp
   http://en.wikipedia.org/wiki/Light-emitting_diode
   http://electronics.howstuffworks.com/led.htm

4. Understand (and be able to apply the knowledge of) the general types of UV-Vis instrument (single beam, double beam, multi-channel), and the advantages and disadvantages of each.
   Harvey 10C.1
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/UV-Vis/uvspec.htm#uv1

Applications of Molecular Absorption Spectroscopy

1. Be able to describe the molecular absorption process. Review molecular orbital theory from organic chemistry.
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/UV-Vis/spectrum.htm#uv3
   http://www.chemguide.co.uk/analysis/uvvisible/bonding.html
   http://www.chemguide.co.uk/analysis/uvvisible/theory.html#top

2. Be able to explain what is happening to molecular electrons using energy level diagrams in molecular absorption spectroscopy.
   Harvey 10B.1
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/UV-Vis/spec-trum.htm#uv3
   http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/intro3.htm#strc8a

3. Understand kinetic methods and what happens to analyte concentration during a spectrophotometric titration.
   Harvey 13B.1

Molecular Luminescence Spectroscopy


1. Know the possible deactivation processes available to a molecule which absorbs a photon and undergoes an electronic energy transition.

   - [http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin1.htm](http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin1.htm)
   - [http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin3.htm](http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin3.htm)

2. Be able to explain the impact of molecular structure, temperature, solvent, and concentration on fluorescence.

   - Harvey 10F.3
   - [http://www.springerlink.com/content/qp463n1w010033h7/fulltext.pdf](http://www.springerlink.com/content/qp463n1w010033h7/fulltext.pdf) -- solvent effects

3. Be able to explain the various instrumental components and diagrams for fluorimeters and spectrofluorimeters.

   - Harvey 10F.1
   - [http://www.fluorescence-foundation.org/lectures/genova2006/lecture3.ppt](http://www.fluorescence-foundation.org/lectures/genova2006/lecture3.ppt) (This site asks for user name and password, but opens without them when “cancel” is selected)

4. Be able to acquire and process data from an analysis. This includes, but is not limited to (quantitative analysis): obtaining a signal for a blank, standards, and unknown; taking into account how the sample was processed (sample blank), which includes dilutions of both the sample and standards; plotting the data (Signal vs. Concentration) and obtaining a calibration curve; calculating a limit of detection for the analyte.

   - Harvey 10, end of chapter problems, 40-43

Infrared Spectroscopy


1. Understand the quantum treatment of molecular vibrations; be able to apply equation listed in the first reference.

   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/irspec1.htm#ir1](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/irspec1.htm#ir1)
   - [http://www.youtube.com/watch?v=DDTJgln86E&feature=channel_page](http://www.youtube.com/watch?v=DDTJgln86E&feature=channel_page)
   - [http://orgchem.colorado.edu/hndbksupport/irtutor/IRtheory.pdf](http://orgchem.colorado.edu/hndbksupport/irtutor/IRtheory.pdf)
   - [http://infrared.als.lbl.gov/content/web-links](http://infrared.als.lbl.gov/content/web-links)

2. Be able to explain what kinds of motions molecules are undergoing when they absorb IR radiation.

   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/infrared.htm#ir2](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/infrared.htm#ir2)

3. Be able to explain the advantages of Fourier transform spectrometers, and how the Michelson interferometer works.

   - [http://www3.wooster.edu/Chemistry/analytical/flir/default.html](http://www3.wooster.edu/Chemistry/analytical/flir/default.html)
**Instrumentation for Infrared Spectroscopy**


1. Understand attenuated total reflectance and transmissive IR absorption techniques.
   - [http://las.perkinelmer.com/content/TechnicalInfo/TCH_FTIRATR.pdf](http://las.perkinelmer.com/content/TechnicalInfo/TCH_FTIRATR.pdf)
   - [http://www.niu.edu/ANALYTICALLAB/ftir/index.shtml](http://www.niu.edu/ANALYTICALLAB/ftir/index.shtml)

2. Be able to discuss the disadvantages of performing quantitative IR analyses.

3. Review interpretation of IR spectra = qualitative analysis (including limitations).
   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/infrared.htm#ir3](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/InfraRed/infrared.htm#ir3)
   - [http://www.chem.ucla.edu/~webspectra/](http://www.chem.ucla.edu/~webspectra/)
   - [http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng](http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng)
   - [http://www.colby.edu/chemistry/JCAMP/IRHelper.html](http://www.colby.edu/chemistry/JCAMP/IRHelper.html)

4. Be aware of the variety of ways to prepare samples for specific instruments.

**Nuclear Magnetic Resonance Spectroscopy (NMR)**

General NMR references:

[http://www.cem.msu.edu/~reusch/VirtualText/Spectrpy/nmr/nmr1.htm](http://www.cem.msu.edu/~reusch/VirtualText/Spectrpy/nmr/nmr1.htm)
[http://www.asdlib.org/onlineArticles/ecourseware/Larive/qnmr1.htm](http://www.asdlib.org/onlineArticles/ecourseware/Larive/qnmr1.htm)
[http://www-keeler.ch.cam.ac.uk/lectures/](http://www-keeler.ch.cam.ac.uk/lectures/)
[http://www.biophysics.org/Portals/1/PDFs/Education/donne.pdf](http://www.biophysics.org/Portals/1/PDFs/Education/donne.pdf)

1. Understand and be able to explain the quantum and classical descriptions of NMR.
   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm)
   - [http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/nmr1.htm](http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/nmr1.htm)

2. Be able to describe relaxation processes in NMR.
   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm)

3. Understand and be able to explain the differences between continuous wave (CW) NMR and FT-NMR (aka "pulsed") instruments.
   - [http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm](http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm)

4. Review interpretation of NMR spectra = qualitative analysis (chemical shift and coupling).
   - [http://www.nd.edu/~smithgrp/structure/workbook.html](http://www.nd.edu/~smithgrp/structure/workbook.html)
   - [http://www.chem.ucla.edu/~webspectra/](http://www.chem.ucla.edu/~webspectra/)
   - [http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng](http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng)
Applications of NMR

1. Know the components of an FT-NMR instrument.

2. Be able to describe why we need a frequency lock system, shims, and sample spinning.

3. Understand and be able to explain the RF source used for excitation.

4. Know why we do not need a Michelson interferometer to perform FT-NMR but we do need one for FT-IR.

Mass Spectrometry

General links:

- [http://www.youtube.com/watch?v=J-wao0Q0_qM&feature=channel_page](http://www.youtube.com/watch?v=J-wao0Q0_qM&feature=channel_page)
- [http://www.shsu.edu/~chm_tgc/primers/gcms.html](http://www.shsu.edu/~chm_tgc/primers/gcms.html)
- [http://svmsl.chem.cmu.edu/vmsl/default.htm](http://svmsl.chem.cmu.edu/vmsl/default.htm)

1. Know the similarities and differences between gas-phase ionization sources and desorption ionization sources for mass spectrometry.
   [http://www.public.iastate.edu/~kamel/ci.html](http://www.public.iastate.edu/~kamel/ci.html)

2. Understand and be able to explain electron impact ionization (EI); chemical ionization (CI); matrix assisted laser desorption (MALDI); and electrospray ionization sources for mass spectrometry.
Mass Spectrometry, continued

1. Understand how ions are detected in mass spectrometers. Especially know how electron multipliers work.
   http://www.vias.org/simulations/simusoft_emultiplier.html

2. Understand the role of mass analyzers in mass spectrometry. Be able to describe how quadrupole, time-of-flight, double-focusing, and FTICR analyzers work.
   http://www.asms.org/whatisms/p8.html
   http://www.instrumentalchemistry.com/gasphase/pages/iontrap.htm
   http://www.instrumentalchemistry.com/gasphase/pages/tof.htm
   http://www.instrumentalchemistry.com/gasphase/pages/magsecone.htm

Re-introduction to Chromatography

1. Understand important chromatographic quantities and relationships, specifically retention time, width at half height, retention factor, linear flow velocity, resolution, number of plates and plate height.
   http://www.separatedbyexperience.com/glossary/
   http://web.njit.edu/~kebbekus/analysis/4CHROMAT.htm
   http://www.edusolns.com/gc/gctutorial/
   Harvey 12AB

2. Be able to describe the general elution problem.
   http://web.njit.edu/~kebbekus/analysis/4CHROMAT.htm
   Harvey 12C

3. Understand how to utilize a Van Deemter plot (ie. kinetic variables affecting zone broadening).
   http://en.wikipedia.org/wiki/Van_Deemter_equation
   http://www.edusolns.com/hplc/hplctutorial/
   Harvey 12C

4. Understand the major components of a chromatogram and quantitative analysis in chromatography.
   http://www.gmu.edu/depts/SRIF/tutorial/gcd/gc-ms2.htm
   http://www.aerosol.us/ADIweb/ResearchTAG.html
Gas Chromatography

1. Be able to describe the major components of a gas chromatograph.
   - http://www.edusolns.com/gc/gctutorial/
   - http://eu.shimadzu.de/products/chromato/gc/default.aspx
   - http://orgchem.colorado.edu/hndbksupport/GC/GC.html
      Harvey 12D

2. Know the differences between the flame ionization, thermal conductivity, electron capture, and flame photometric detectors.
   - http://www.edusolns.com/gc/gctutorial/
      Harvey 12D.5

3. Understand different factors that affect column efficiency.
   - http://orgchem.colorado.edu/hndbksupport/GC/GC.html
      Harvey 12C

Liquid Chromatography/ Capillary Electrophoresis

Video of instrument in action: http://www.youtube.com/watch?v=kz_egMtdnL4&feature=channel_page

1. Understand what is going on during partition chromatography. Know how to perform gradient and isocratic elutions.
   - http://www.chemistry.nmsu.edu/Instrumentation/Lqd_Chroma.html
      Harvey 12E.2

2. Know the differences between normal and reversed phase partition chromatography.
   - http://www.chemistry.nmsu.edu/Instrumentation/Lqd_Chroma.html
      Harvey 12E.1

3. Know the basic components of an HPLC instrument. Specifically, know the difference between bulk property detectors (refractive index detector) and solute –property detectors (UV-Vis detectors). Also be familiar with the mass spectral detector.
      Harvey 12E.4