Overview of Experiments II-V

CU- Boulder

CHEM-4181

Instrumental Analysis Laboratory

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Lecture will be posted on course web page

Lab Report Due Dates

- There seems to be some confusion about when the lab reports are due
- If you read the first sentence on p.6 of the lab manual:
  - "The first 8 experimental laboratory reports are to be submitted to your TA within the first 15 minutes of lab one week after completion of the experiment."
- The penalties for being late are also discussed there:
  - "Each report is worth 60 points. Late reports will be assessed the following penalties: one day late = -5 points, two days late = -10 points, three days late = -20 points and four or more days late = no credit. A report submitted more than 15 minutes after the beginning of lab will be considered a day late."

Experiments II-V

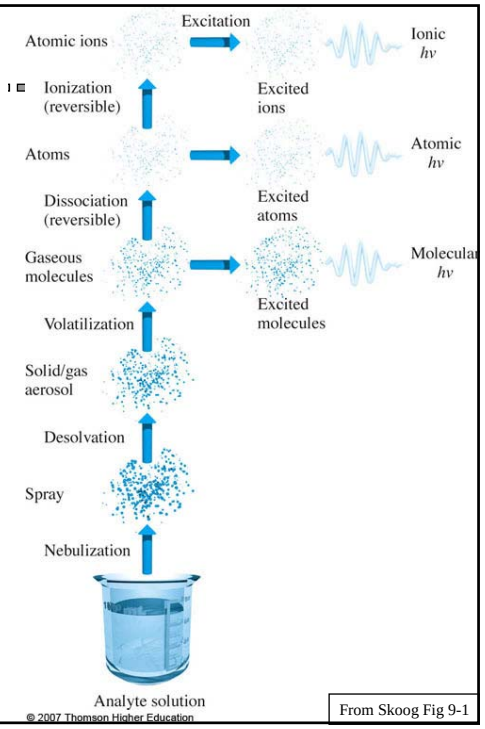
- Weeks of Feb 5 to 26
- Four types of optical instruments
- E2: Pb in soil
  - Atomic absorption
- E3: Phosporus in water
  - UV/Vis absorpption
- E4: Oil in water
  - Fluorescence
- E5: CO in car exhaust
  - Infrared absorption (FTIR)

E2: Lead in Soil

- Summary:
  - Soil sample with Pb
  - Digest with acid to extract Pb into aqueous phase
- Safety:
  - Concentrated acid (gloves, goggles)
  - Flame (explosion danger)
  - Also pay attention to waste
- Quality control
  - Wash all glassware with HNO_3 (poss. big errors otherwise)
  - Pb standards by dilution of provided solution
    - Determine linear dynamic range
  - Blank (Ottawa sand)
  - Blank spike
    - Look for matrix effects (extraction eff.)

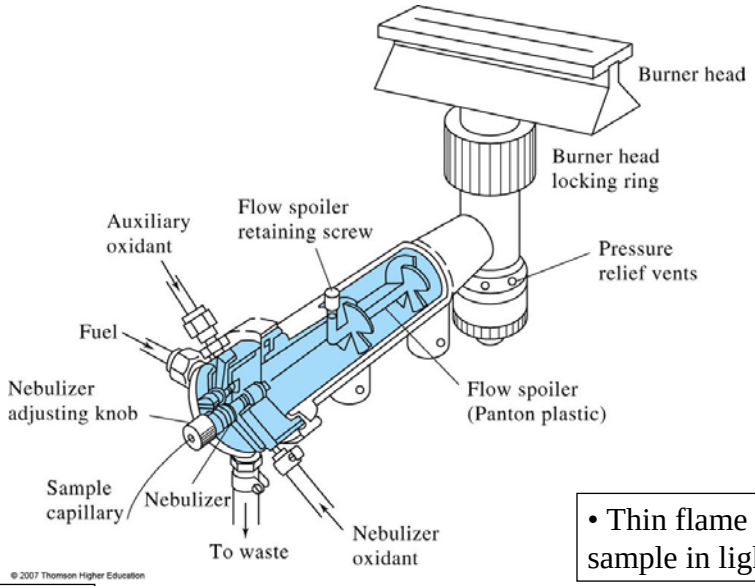
Flame Atomic Absorption I

- Solid or liquid sample
- Need to turn into free atoms in the gas-phase
  - Use a flame



From Skoog Fig 9-1

Flame Atomic Absorption II

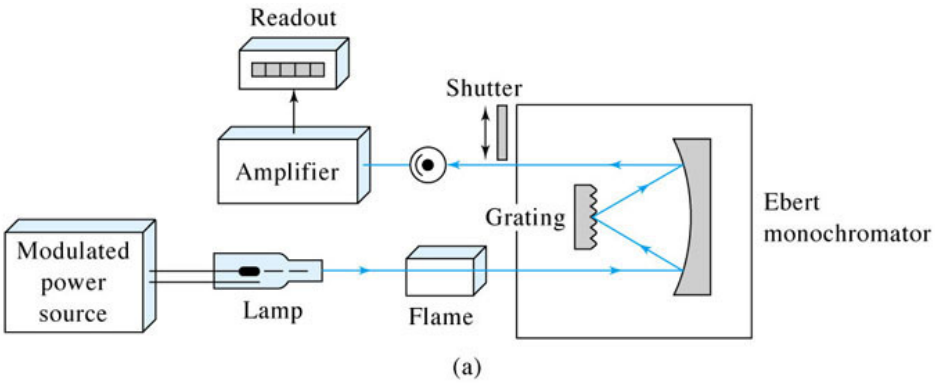


From Skoog Fig 9-5

• Thin flame to get all sample in light path

Flame Atomic Absorption III

- Lamp generates light that element absorbs
  - Different lamps for different elements
- Q: why do we need monochromator?



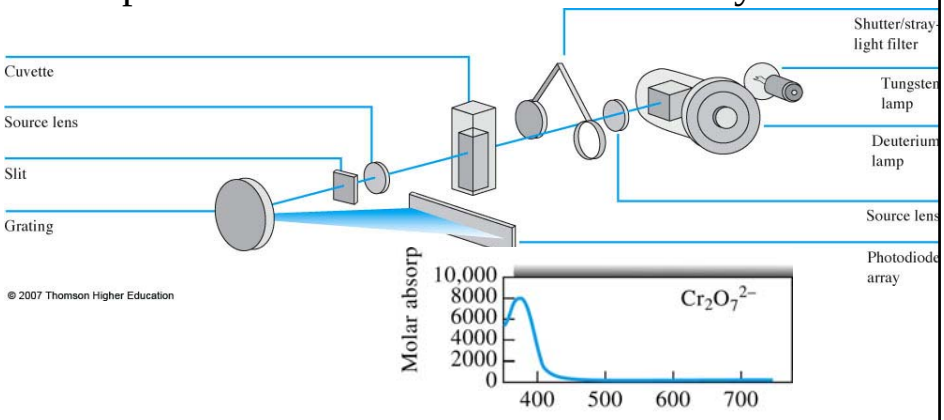
From Skoog Fig 9-13a

E3: Phosporus in Water

- Env. Relevance:
  - Phosphates in detergents and fertilizers
  - Stimulate algal growth, algae die $\Rightarrow$ BOD $\uparrow$
- Summary:
  - Treat w/ acid to convert to PO_4^{3-}
    - Usual acid precautions
  - Add ammonium molybdate and stannous chloride to form molybdenum blue (MB)
    - Absorbance of MB measured at 696 nm
    - Measure 10-12 min. after adding reactants (timing!)
- Quality control
  - Standards as before
  - Blank
  - Replicates
  - Matrix spike

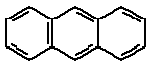
UV/Vis Spectrometer

- Tungsten lamp
- Light passes through your sample in cuvette
- Spectrum determined with diode array

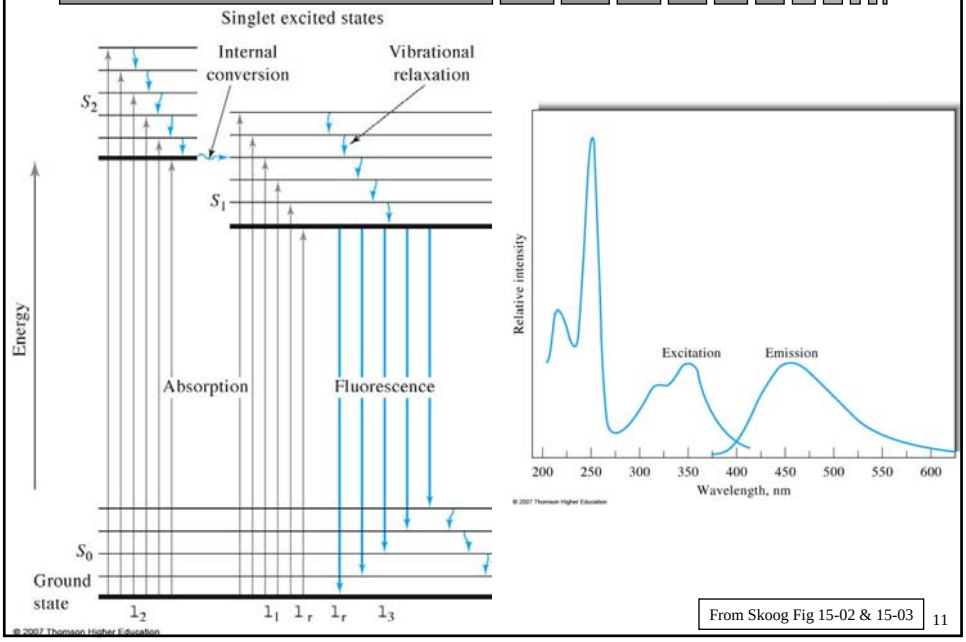


From Skoog Fig 13-25 & 14-3a

E4: Oil in water

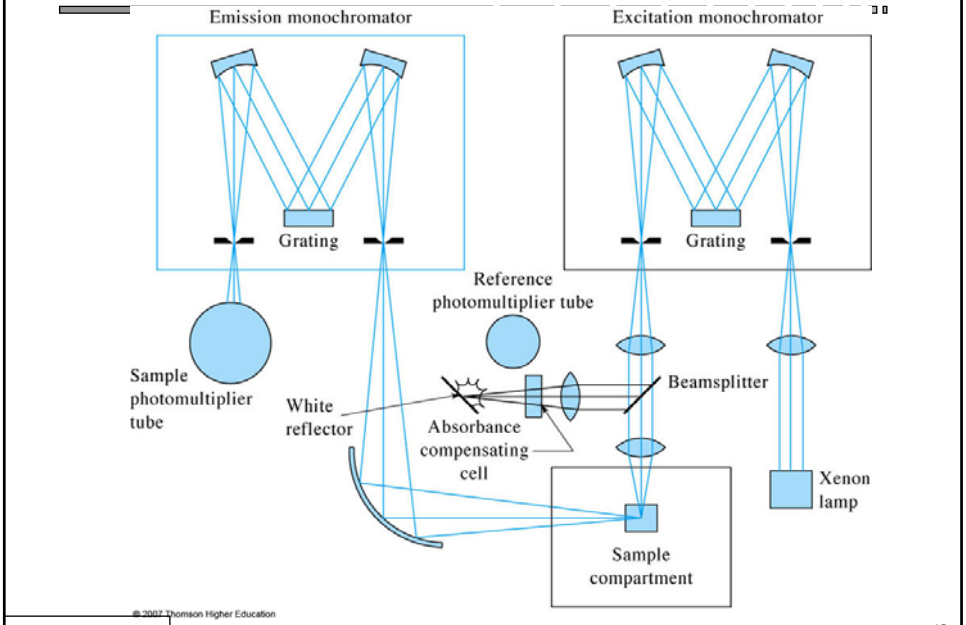
- Summary 
  - Analyze for anthracene, a component of oil
- Fluorescence spectrometer
  - Quartz cuvettes
    - Careful with fingers
  - Quick measurements, sample will photobleach
- Quality Control
  - Standard addition method for quantification
  - Blank (milli-Q water)
  - Evaluate effect of time on fluorescence signal
  - Evaluate the effect of positioning cuvette

Fluorescence I



From Skoog Fig 15-02 & 15-03

Fluorescence II: A spectrofluorometer

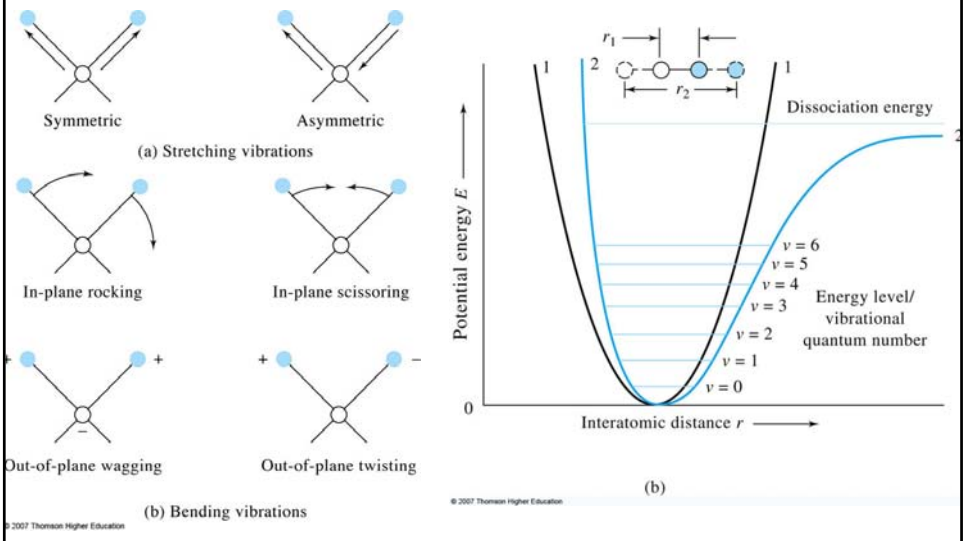


From Skoog Fig 15-11

E5: CO in Auto Exhaust

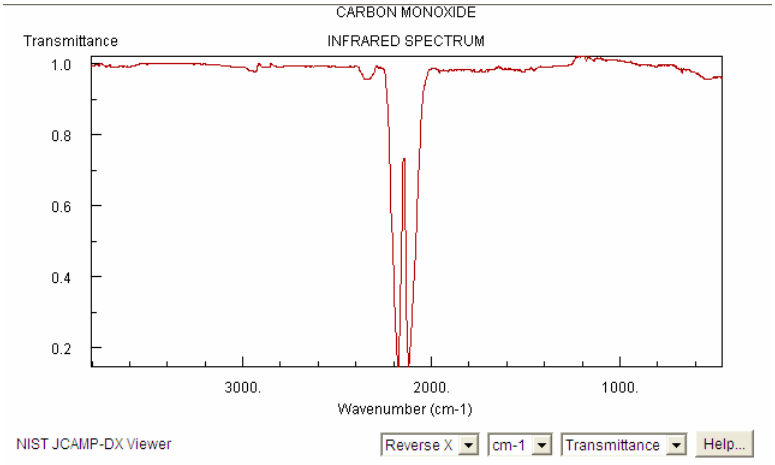
- Summary
  - Capture auto exhaust samples
  - Introduce into vacuum manifold
  - Analyze with FTIR
- Precautions
  - Easy to break glass parts in manifold
  - Can fill the manifold with pump oil if not careful
  - Explosion of glass manifold possible
    - Use goggles
  - Pay attention!
- Quality control
  - Standards @ *the same pressure*
  - Determine detection limit

Infrared Absorption I



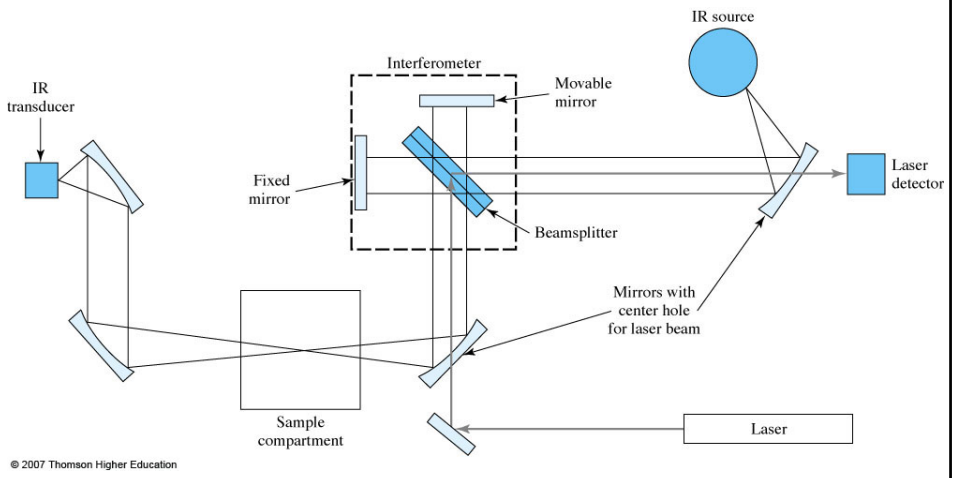
From Skoog Fig 16-02 & 16-03b

IR Spectrum of CO



- http://webbook.nist.gov/cgi/cbook.cgi?ID=C630080&Units=SI&Type=IR-SPEC&Index=1#IR-SPEC

FTIR (Fourier-Transform Infrared Spectrometer)



- http://www.infrared-analysis.com/info1.htm

From Skoog Fig 16-08

Practical Notes

- Professional & courteous behavior
- Part of grade on how you work together
- Patient w/ TA if tied up w/ someone else
- Real world: have to work with various people that you haven't chosen
- Rotate jobs in group, so everyone gets skills
  - Meet w/ groups before lab to plan