

April 11, 2005

➤ **Review Session for Exam #3:**

Monday, April 18th, 5 pm, B203

➤ **Stay tuned for:**

- Exam #3 Info Page
- Problem Set #4 Solutions
- Winning Lottery Numbers

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Multichannel Detection

➤ Another way to get *multiplex advantage*:
use multichannel detector (CCD)

- **Example:** with low-power UV excitation:
 - ✓ *ppb detection limits* for PAHs
 - ✓ surface studies: *sub-monolayer* detection
 - ✓ *speed* (30 secs to acquire spectrum that would have taken 5 hours with scanning instrument)
 - ✓ *ns time-resolution*

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Near-IR Excitation: *Revisited*

Goal: Raman for the masses!

➤ **Near-IR excitation**

- ✓ fluorescence gone
- ✓ inexpensive diode laser source

➤ **Multichannel detection**

- ✓ gives S/N boost
- ✓ fast

➤ **Fiber Optic probe "sample cell"**

- ✓ eliminates laser/sample alignment issues

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Simple, Low-Cost Raman?

• **Source:**

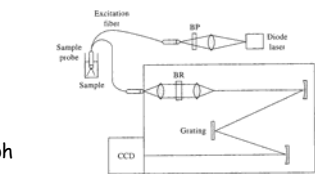
30-mW, 783-nm diode laser

• **Dispersion:**

$\frac{1}{4}$ -m, 600-g/mm grating spectrograph ($D^{-1} = 50 \text{ \AA}/\text{mm}$)

• **Detector:**

1152 pixel CCD (22.5 $\mu\text{m}/\text{pixel}$)



~ 1.6 $\text{cm}^{-1}/\text{pixel} \sim \sim 2000 \text{ cm}^{-1}$ ∇ -range

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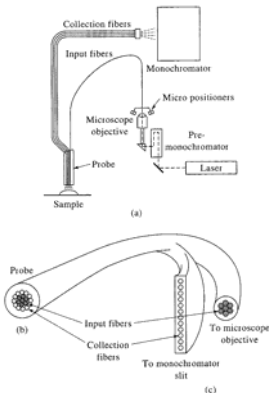
Fiber Optic Sampling

"User-Friendly Sampling"

• eliminates need for precise alignment of laser and sample

• place *directly in solution* containing the sample

• place *directly above solid* sample

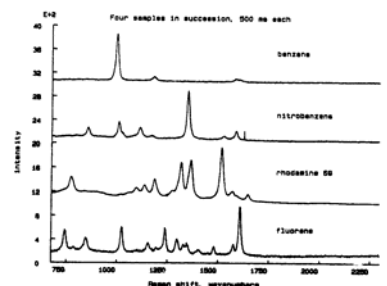


Spectral Results

• **rapid spectral acquisition** (500 ms)

• **easy sampling** of solutions and solids

• **no fluorescence background**



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Raman: What is it good for?

➤ Vibrational Spectral Analysis

- ✓ complementary with IR
- ✓ molecular structural info
- ✓ easy and fast!

➤ Quantitative Analysis

- ✓ can be as sensitive as fluorescence
- ✓ more selective than fluorescence

Raman as a detection method for separations? See:
<http://www.umich.edu/~morgroup/virtual/labeled/virtual.html> 7

Mass Spectrometry

Chem 221
 Instrumental Analysis
 Spring 2005

Background

- Method for *separating and quantifying gas phase ions* according to their **mass-to-charge ratio**
- ***NOT A SPECTROSCOPIC METHOD!***
 -no EMR interactions measured
- **Mass Spec is a SEPARATIONS technique**
 -ions are *separated physically* based on mass

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General Instrumental Scheme

Ion Source:

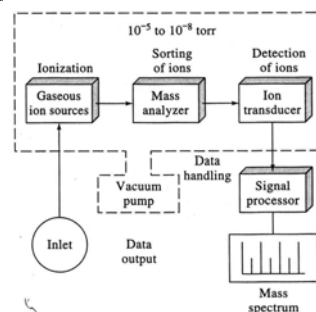
- converts sample to gas phase
- ionizes sample

Mass Analyzer:

- "sorts" ions according to mass-to-charge ratio

Detector:

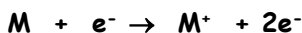
- converts ions to electrical signal proportional to # of ions



How are ions generated?

- Simplest, most common method:

Electron Impact (EI) Ionization



Analyte molecule High energy electron Molecular ion

- if M^+ has sufficient *internal energy* it can **fragment**:

