Chemistry 4631

Instrumental Analysis
Lecture 8

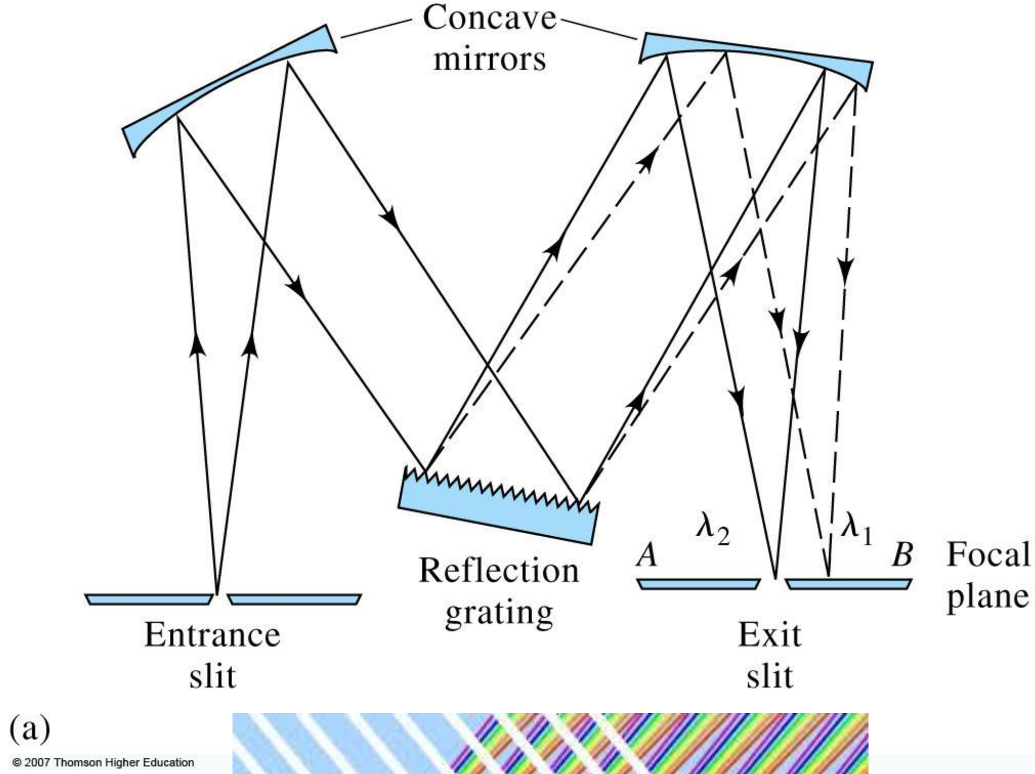
Components of Optical Instruments

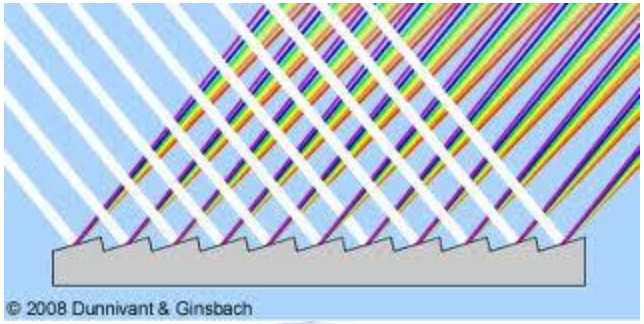
UV to IR

Basic components of spectroscopic instruments:

- stable source of radiant energy
- transparent container to hold sample
- device to isolate selected region of the spectrum for measurement
- detector to convert radiant energy to a signal
- signal processor and readout

Components of Optical Instruments





Components of Optical Instruments

Holographic Gratings

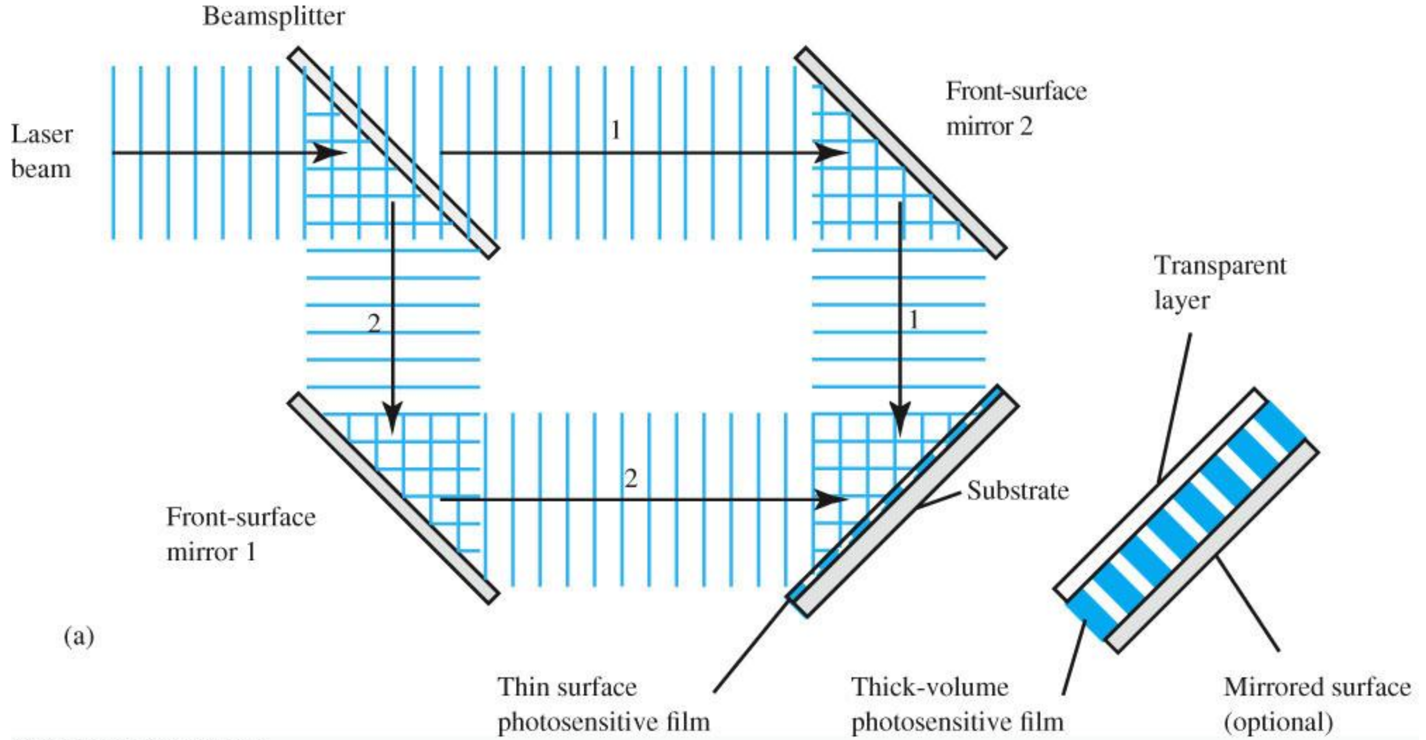
Gratings formed from an optical technique (lasers) on a plane or concave glass. (semiconductor industry)

Formed by an interference fringe field of two laser beams whose standing wave pattern is exposed to a polished substrate coated with photoresist. Processing of the exposed medium results in a pattern of straight lines with a sinusoidal cross section.

Components of Optical Instruments

Filters

Holographic Filters



Components of Optical Instruments

Holographic Gratings

Advantages

- Greater perfection
- Less stray radiation and ghost
- Low cost

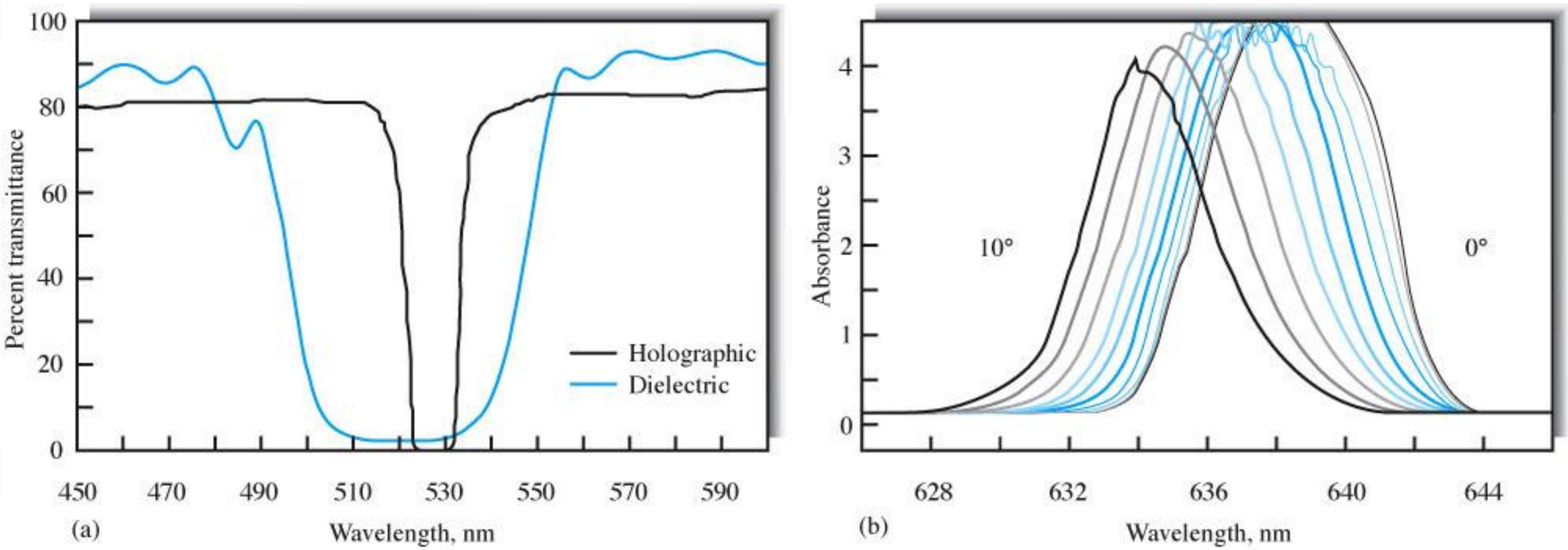
Disadvantages

- Sinusoidal cross section decreases efficiency (exception when groove spacing to λ ratio is near 1)

Components of Optical Instruments

Filters

Holographic Filters



Components of Optical Instruments

Quality of Monochromator depends on

- Purity of radiation output
- Resolution of adjacent wavelengths
- Light gathering power
- Spectral bandwidth

Components of Optical Instruments

Spectral Purity

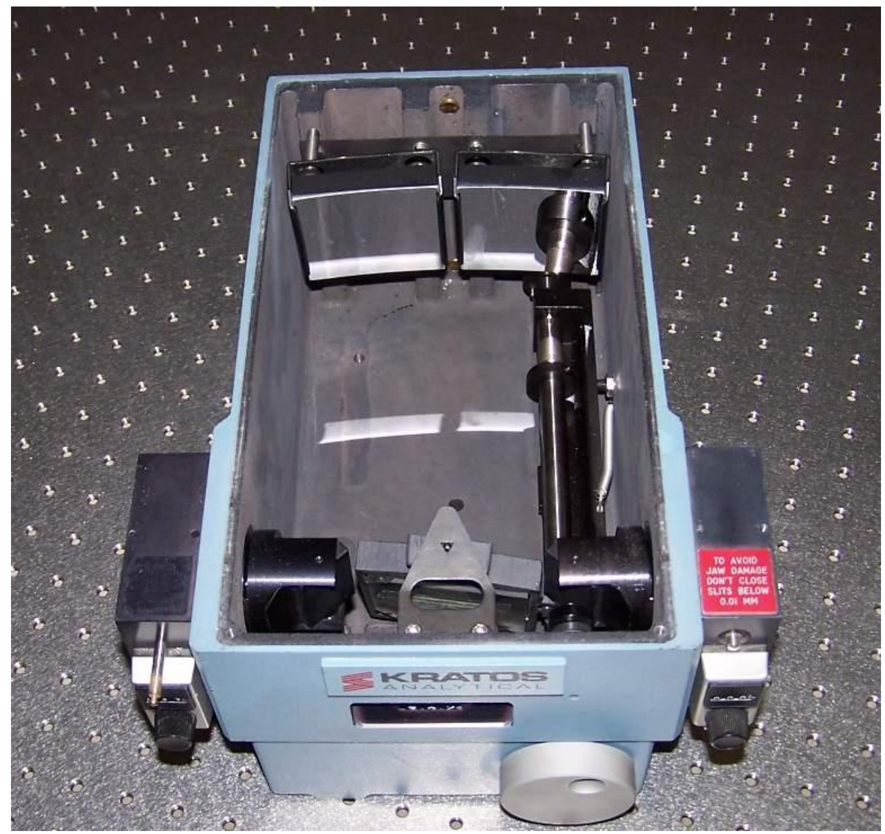
Scattered and stray radiation at other wavelengths interfere with measurements.

Source of unwanted radiation

- Reflection from monochromator housing
- Surface imperfections
- Dust particles

Components of Optical Instruments

Source of unwanted radiation



Components of Optical Instruments

Dispersion

Ability of monochromator to separate different wavelengths.

Components of Optical Instruments

Dispersion

Angular dispersion is given by $dr/d\lambda$

dr – change in angle of reflection

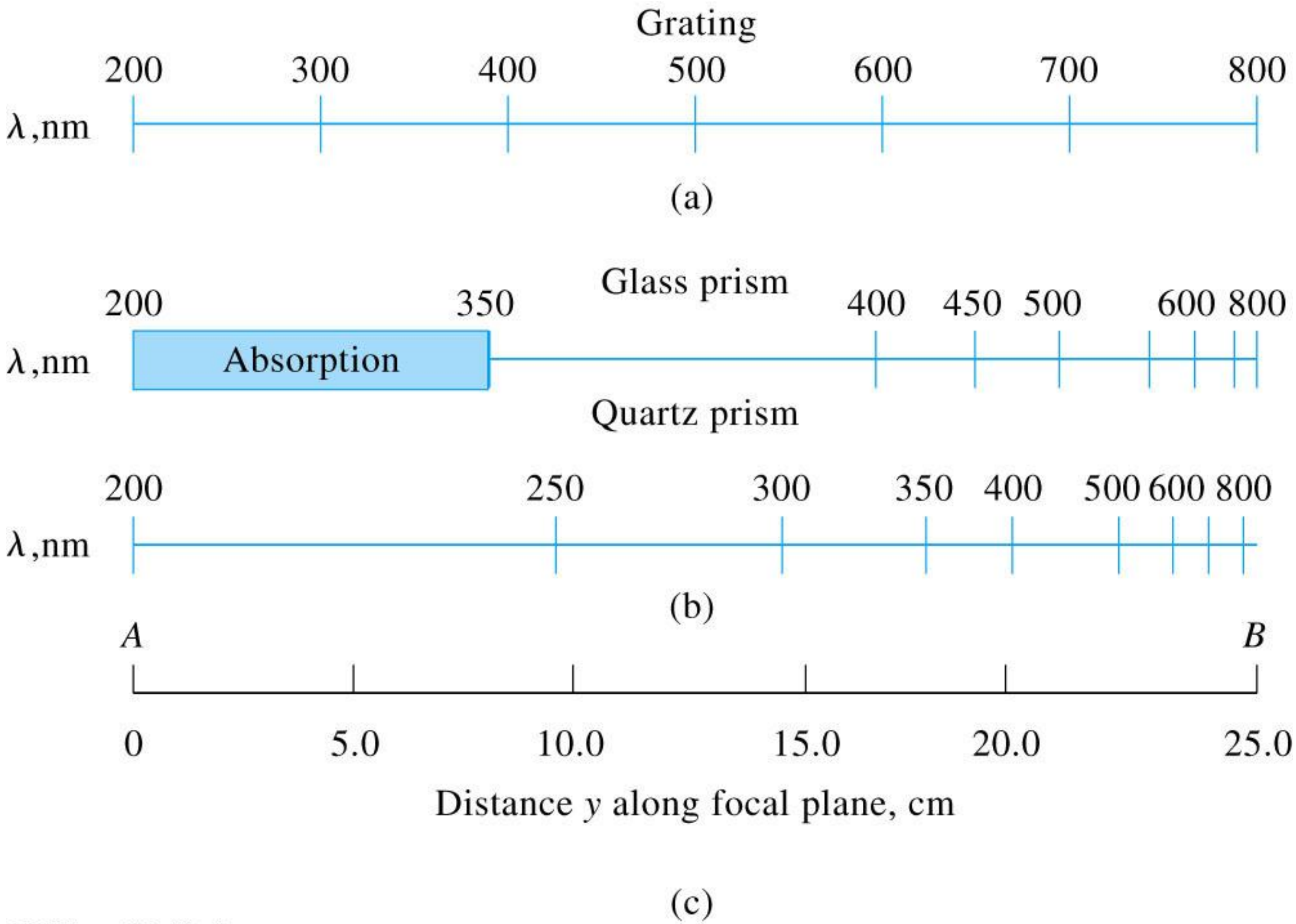
$d\lambda$ – change in wavelength

$D = f\, dr/d\lambda$

D – linear dispersion – is the variation in λ along the focal plane

f - focal length of the monochromater

Components of Optical Instruments



Components of Optical Instruments

Light–Gathering Power

Amount of light reaching detector.

Needs to be high to keep signal-to-noise ratio high.

Ability of monochromator to collect radiation emerging from entrance slit called f/number or speed.

Components of Optical Instruments

Light–Gathering Power

F = f/d

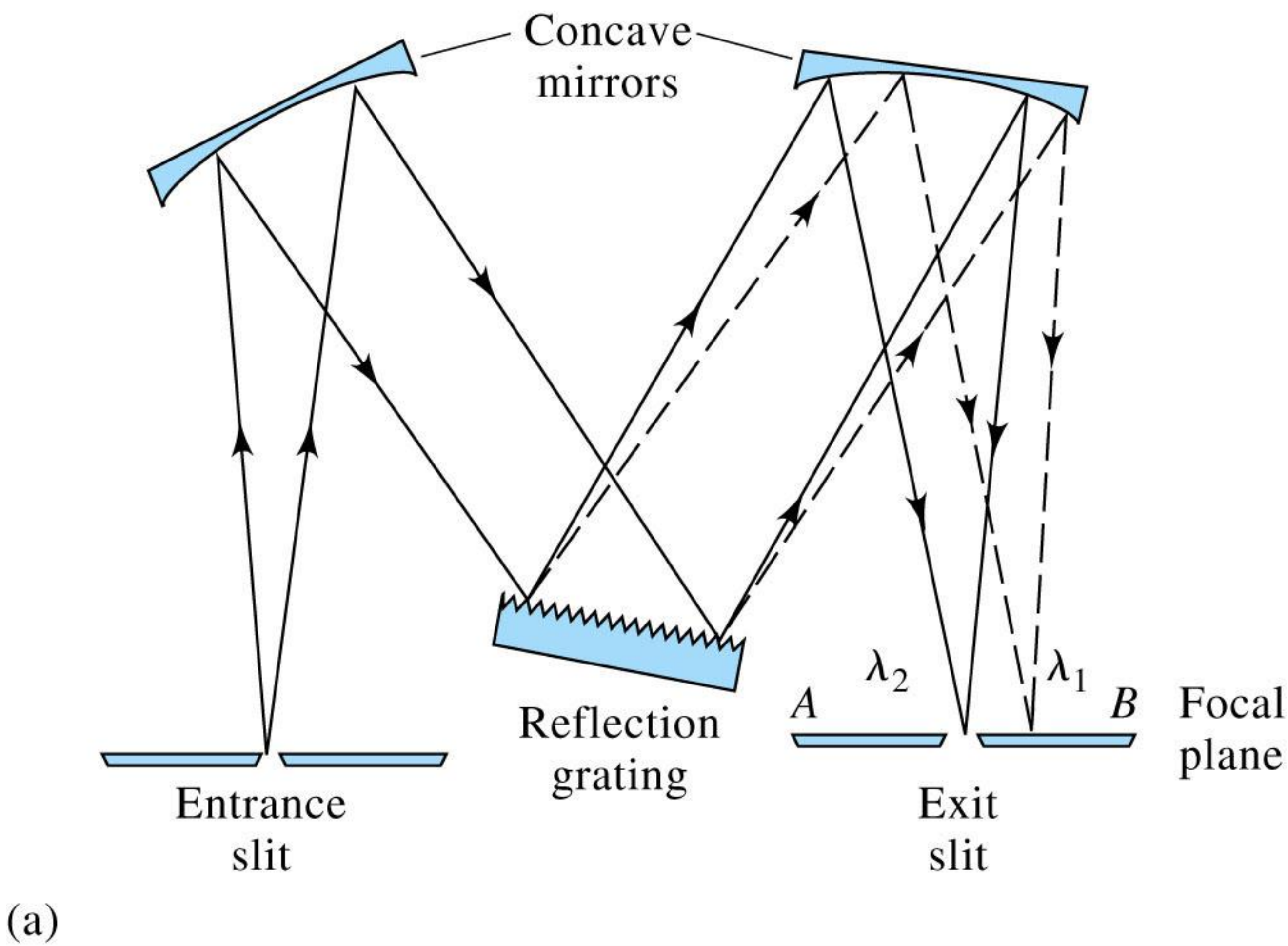
f- focal length of collimating mirror

d – diameter

F – f-number or speed – measure of ability of monochromater to collect radiation

f-number is between 1-10

Components of Optical Instruments



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Components of Optical Instruments

Radiation Transducers

Transducer – converts radiant energy into an electrical signal.

Properties of Ideal Transducers:

- high sensitivity
- high signal-to-noise ratio
- constant response
- fast response time
- no background

Components of Optical Instruments

Radiation Transducers

Two types of radiation transducers:

- Response to photons
- Response to heat

Components of Optical Instruments

Radiation Transducers

Photon transducers

Contain an active surface capable of absorbing radiation.

Absorbed radiation causes:

- emission of electrons giving a photocurrent
- promotion of electrons into conduction bands (photoconduction)

Components of Optical Instruments

Photon Transducers

which respond to radiation:

- Phototubes (emission of electrons from a photosensitive solid)
- Photomultiplier tubes
- Photovoltaic cells (current generated at the interface of a semiconductor layer)
- Photoconductivity (production of electrons and holes in a semiconductor)
- Silicon photodiodes (conductance across a reverse bias pn junction)
- Charge transfer (charge develops in silicon crystal)

Components of Optical Instruments

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Components of Optical Instruments

Photomultiplier Tubes (PMTs)

- The photomultiplier tube is made up of a series of photocathodes (dynodes).
- The photocathodes are a photosensitive material made up of cesium-antimony intermetallic compound.
- Light strikes the 1st photocathode and electrons are ejected.

Components of Optical Instruments

Photomultiplier Tubes

- These electrons are accelerated toward the next dynode by a potential difference (ΔV).
- Each dynode is 90 V more positive than the proceeding one.
- As electrons hit the next dynode, more electrons are produced (multiplication).
- Last dynode is connected to a circuit.

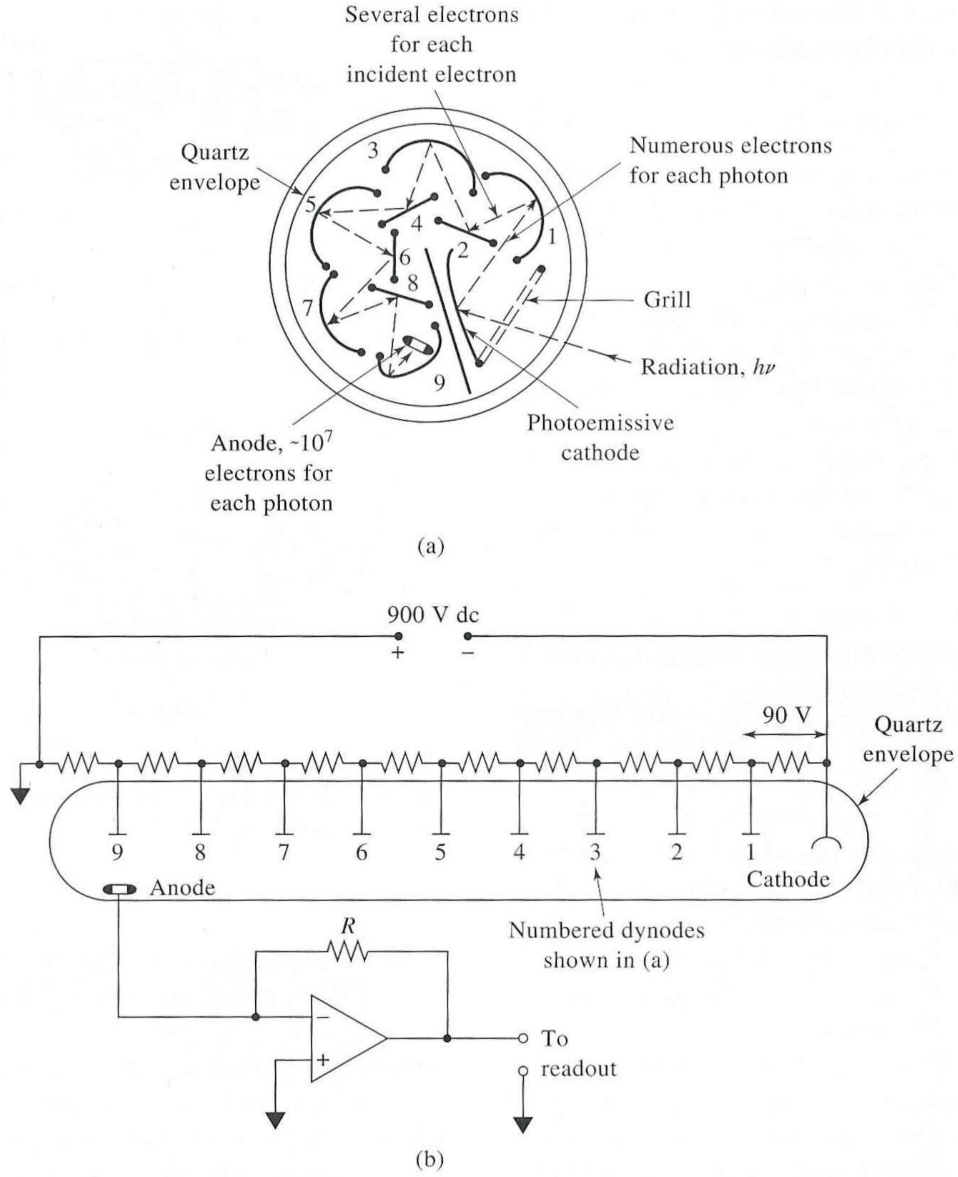


Figure 7-29 Photomultiplier tube: (a) cross-section of the tube and (b) electrical circuit.

Components of Optical Instruments

Photomultiplier Tubes

Total Gain of the photomultiplier tube is:

$$G = (f)^n$$

where f - secondary emission factor (range 3 - 50)

n - # of stages

If the Gain per dynode is ~5

(1 electron knocks out 4 to 5 electrons):

With 10 dynodes, there is a multiplication factor of 5^{10} or 10^7.

Components of Optical Instruments

Photomultiplier Tubes

This whole process takes less than a μsec.

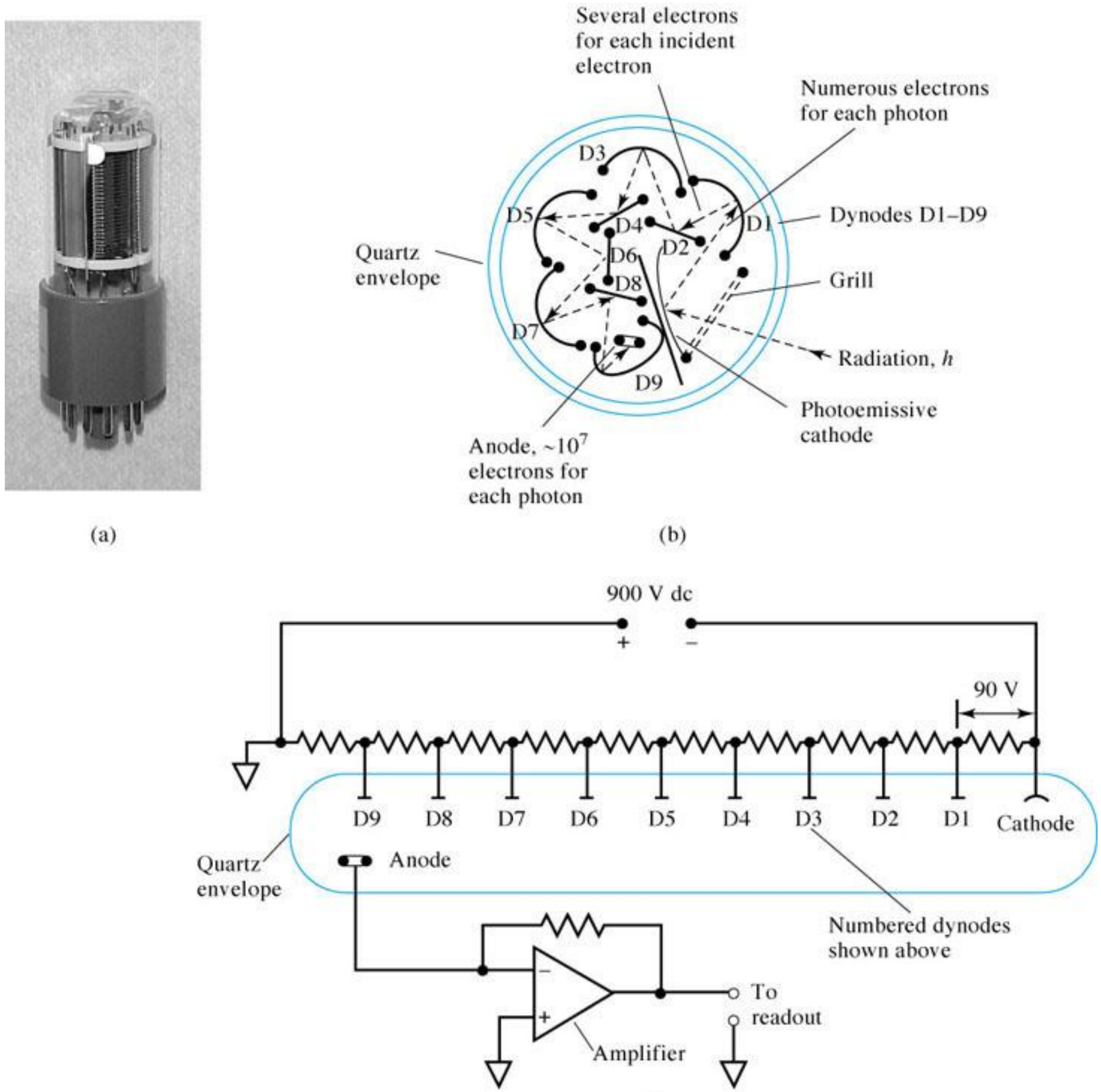
So detector can handle rates of 10^5 counts/sec without loss.

Advantages:

- Very sensitive in UV and vis region
- Fast response time

However dark current limits sensitivity.

Thermal dark currents can be reduced by cooling the detector to –30 °C.


Several electrons for each incident electron
Numerous electrons for each photon
D3
D5
D4
D2
D1
Dynodes D1–D9
Quartz envelope
D6
D8
Grill
D7
Radiation, h
D9
Photoemissive cathode
Anode, ~10^7 electrons for each photon
900 V dc
+
–
90 V
D9
D8
D7
D6
D5
D4
D3
D2
D1
Cathode
Quartz envelope
Anode
Numbered dynodes shown above
To readout
Amplifier

(a) (b) (c)

Assignment

- Read Chapter 7
- Read Chapter 13
- HW4 Chapter 13: 1, 2, 5-8, 12, 13, 16-19
- HW4 Chapter 13 Due 2-6

- Exam 1- Lectures 1 to 8 - Feb 9th

Instrument Lab

II. Instrumentation

The Instrument used was a Spectrophotometer Beckman Double Beam # 1401100834. And the light source was a Beckman Hydrogen Lamp Power Supply # 337997.

Schematic drawing: Figure 1: Double Beam Spectrometer

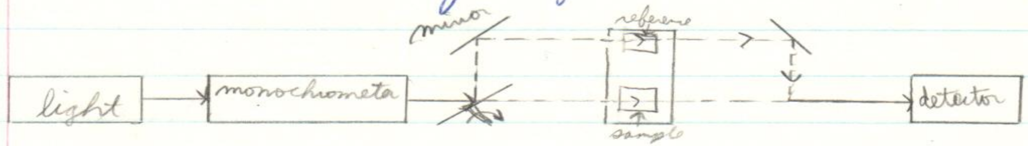
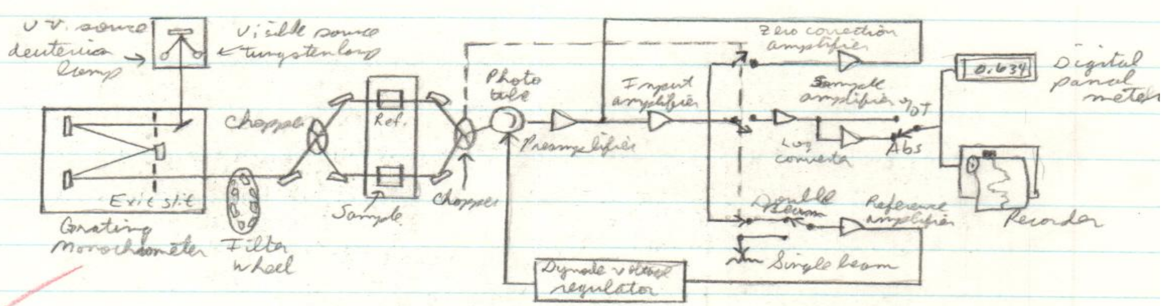


Figure 2: A scanning double-beam spectrometer with dual source, single grating. pg. 57, Instrumental Analysis, Dean & Settle



Instrument Lab

Block diagram of EG+G PARC Model 174A Polarographic Analyzer (Instrumental Method of Analysis, 6th Ed, Dean & Settle)