Chemistry 4631

Instrumental Analysis

Lecture 7

Components of Optical Instruments

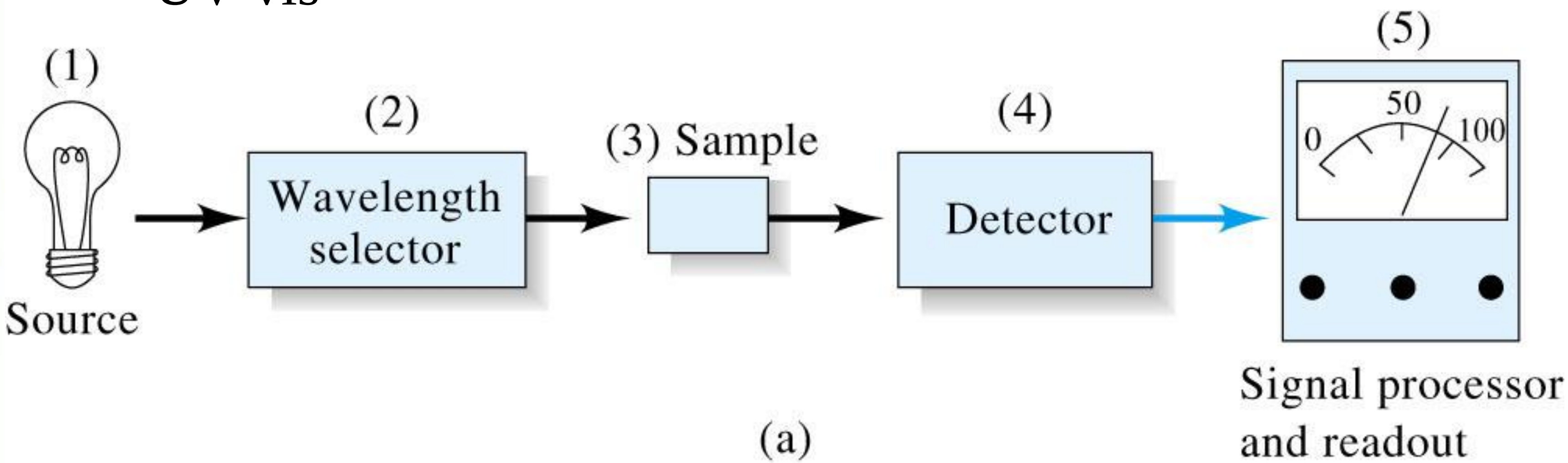
UV to IR

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

UV-vis



Components of Optical Instruments

Wavelength Selectors

- Since many sources are continuum sources, need a wavelength selector to narrow the bandwidth.
- This increases the sensitivity and selectivity of spectral methods.
- A wavelength selector gives a band with a measurable width.

Components of Optical Instruments

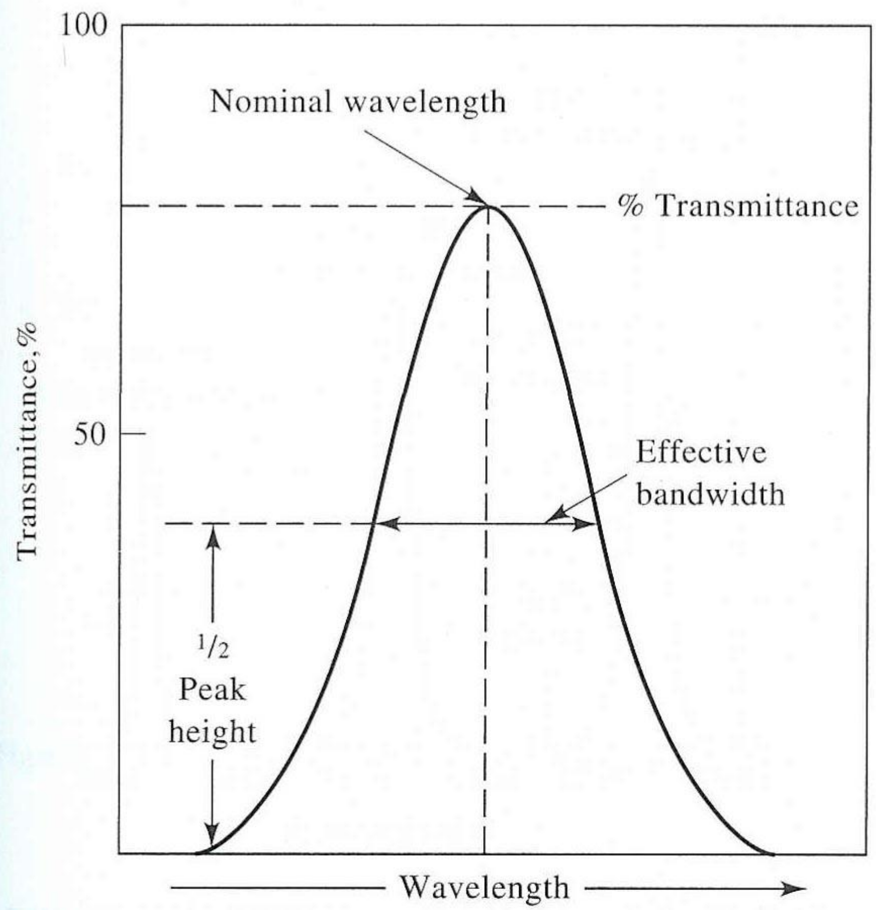


Figure 7-11 Output of a typical wavelength selector.

Components of Optical Instruments

Types of wavelength selectors:

- **Filters**
  - **Interference (Wedge)**
  - **Absorbance (Colored glass, plastic)**
- **Prisms**
- **Monochromators**
  - **Ruled**
  - **Holographic**

Components of Optical Instruments

Filters

Two types:

Interference Filters (Fabry-Perot) and Wedges - available in the UV, vis and IR.

Absorption Filters –

available for vis only.

Components of Optical Instruments

Filters

Interference Filters

Consist of transparent dielectric (i.e. CaF_2 or MgF_2) sandwiched between semitransparent metallic films, which are held between two plates of glass or other transparent material.

Components of Optical Instruments

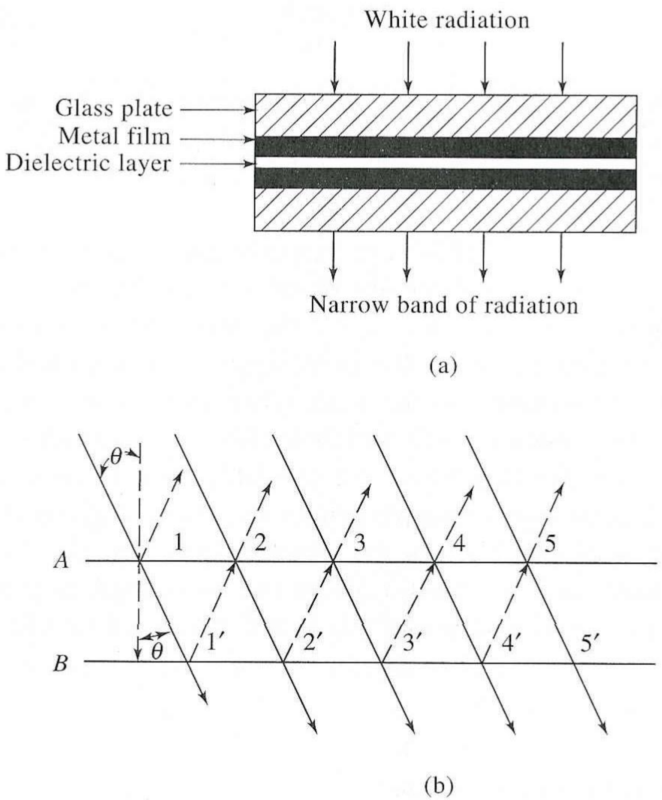


Figure 7-12 (a) Schematic cross section of an interference filter. Note that the drawing is not to scale and that the three central bands are much narrower than shown. (b) Schematic to show the conditions for constructive interference.

Components of Optical Instruments

Filters

Interference Filters

The wavelength of transmitted radiation is determined by the thickness of the dielectric layer.

As a beam strikes the array, a fraction passes through the 1^{st} metallic layer and the rest is reflected.

The fraction that passes through strikes the 2^{nd} metallic layer and part passes through while part is reflected.

Components of Optical Instruments

Filters

Interference Filters

It is the 2nd reflected part of the wave that interacts with radiation coming through the filter.

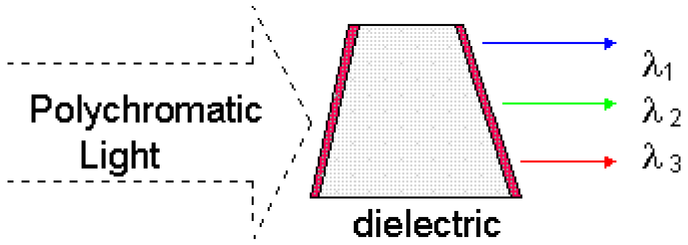
The 2nd reflected portion is in phase with some of the incoming waves, and combines <u>constructively</u> while other wavelengths undergo <u>destructive interference</u> with the reflected portion.

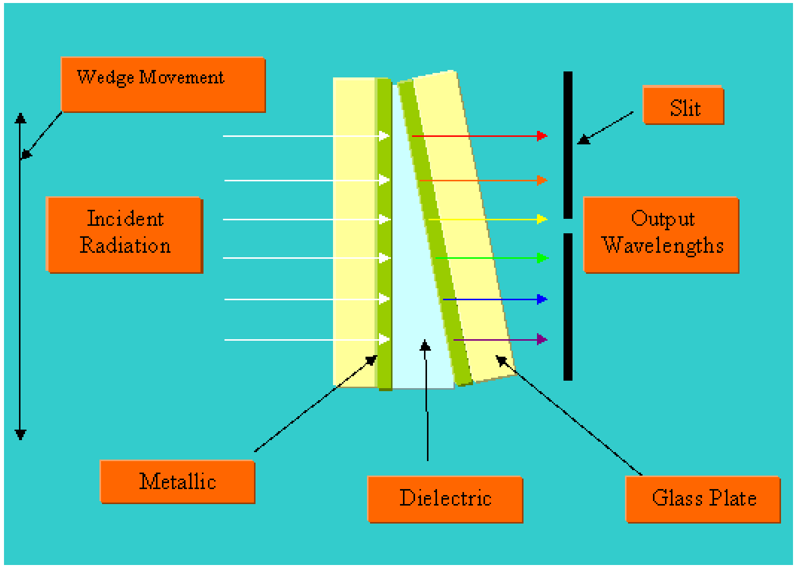
Components of Optical Instruments

Filters

Interference Wedges

Consist of a pair of mirrored partially transparent plates separated by a wedge shaped layer of dielectric material.





Components of Optical Instruments

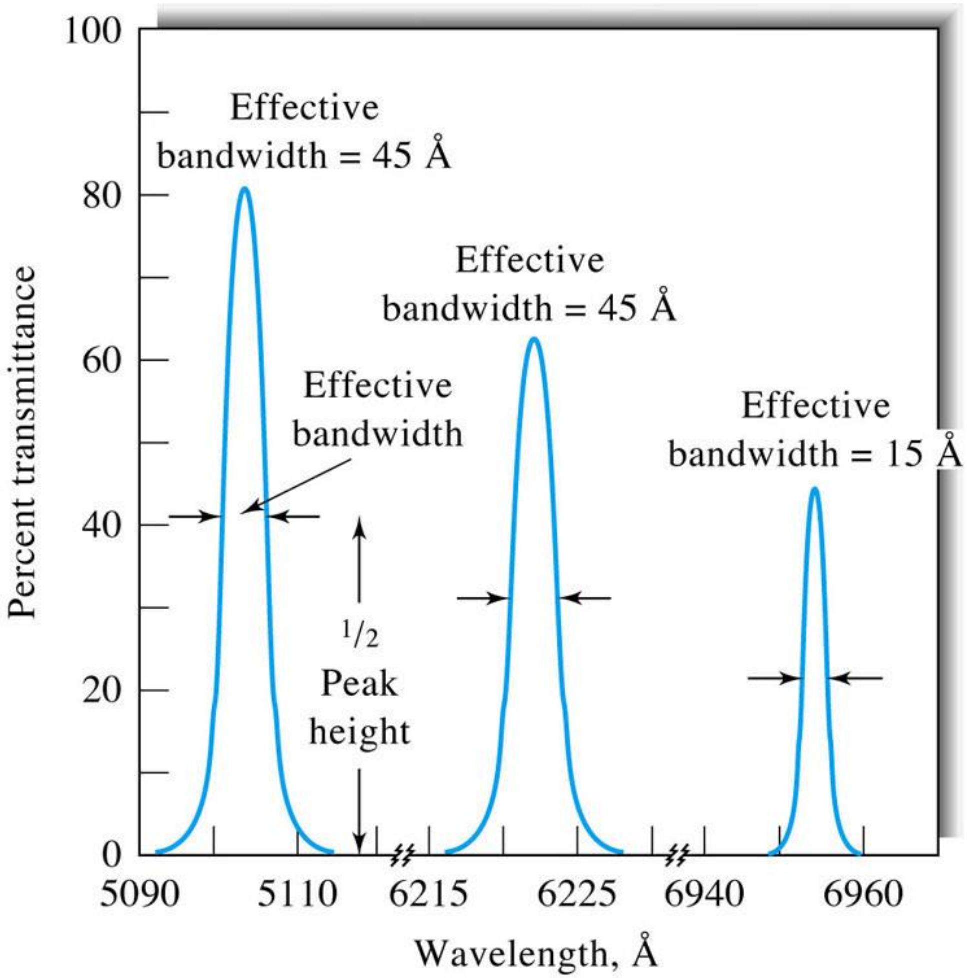
Filters

Interference Wedges

Transmitted wavelength varies with thickness of the wedge.

Wedges are available for the vis (400-700 nm), near IR (1000-2000 nm) and part of the IR region (2.5-14.5 μm).

Components of Optical Instruments



Transmission characteristics of an interference filter.

Components of Optical Instruments

Filters

Absorption Filters

- Less expensive
- Used for band selection in vis region.
- Filter absorbs part of the spectrum.
- Types:
- Colored glass
- Dye in gelatin between glass plates.
- Effective bandwidths ~ 30-250 nm.

Components of Optical Instruments

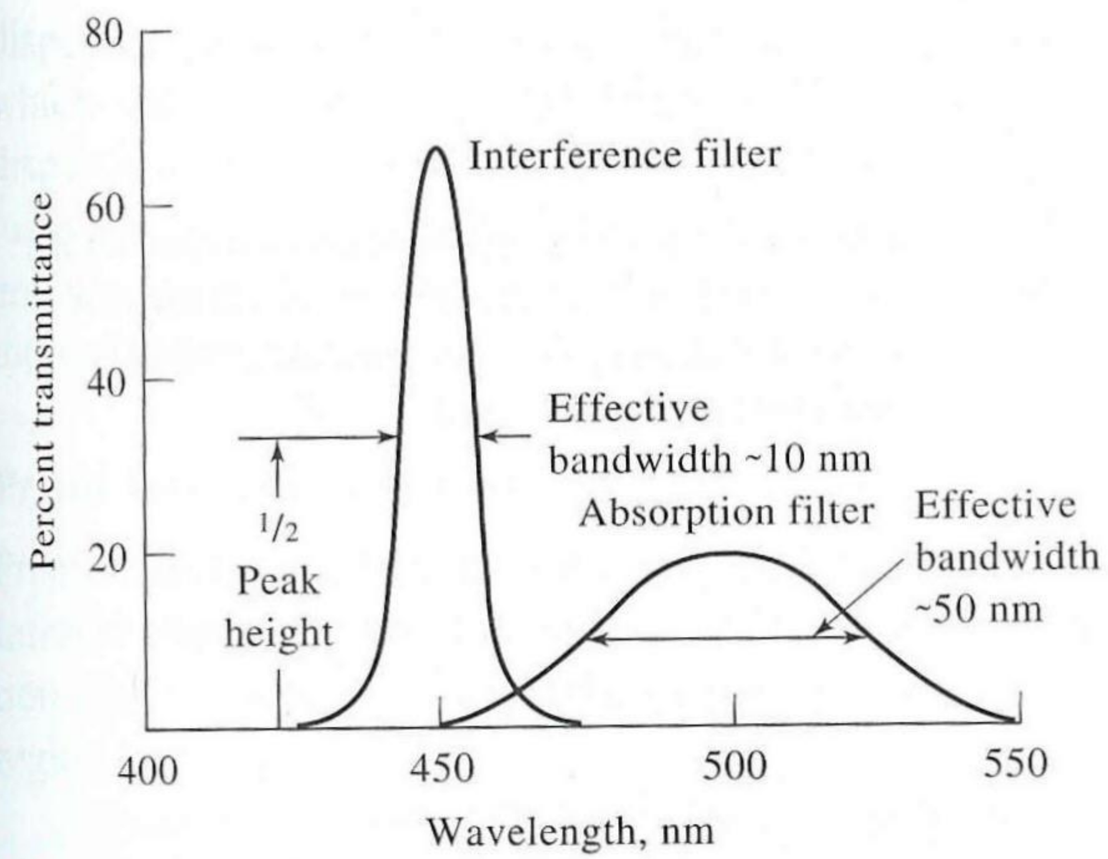


Figure 7-14 Effective bandwidths for two types of filters.

Components of Optical Instruments

Monochromaters

- Vary the wavelength of radiation over a selected range. (Scan a spectrum)

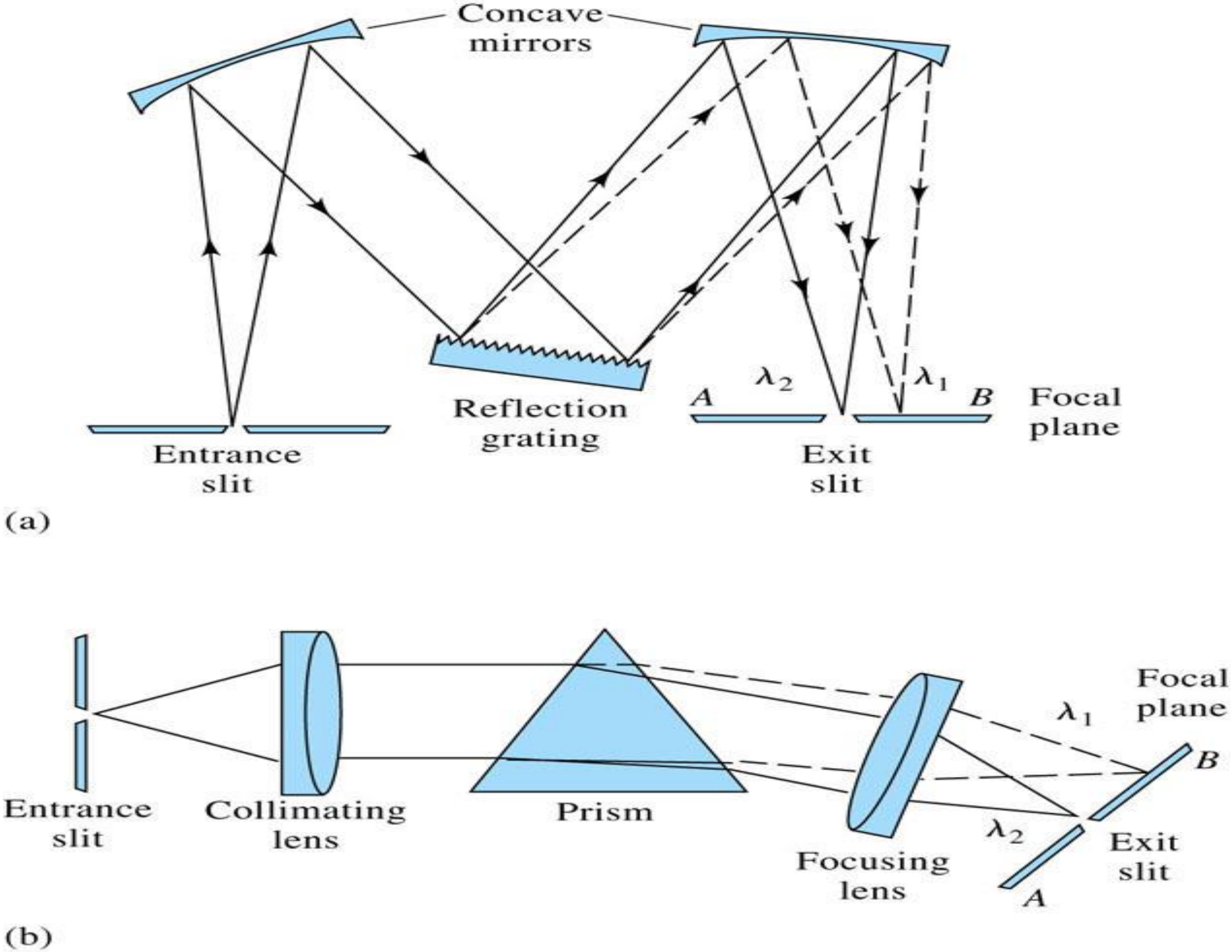
Components of Optical Instruments

Components of the Monochromater

Include:

- Entrance slit (allows a rectangular optical image)
- Collimating lens or mirror (produces a parallel beam of radiation)
- Prism or grating (disperses radiation into component wavelengths)
- Focusing element (focuses image on a focal plane)
- Exit slit (isolates desired spectral band)

Components of Optical Instruments



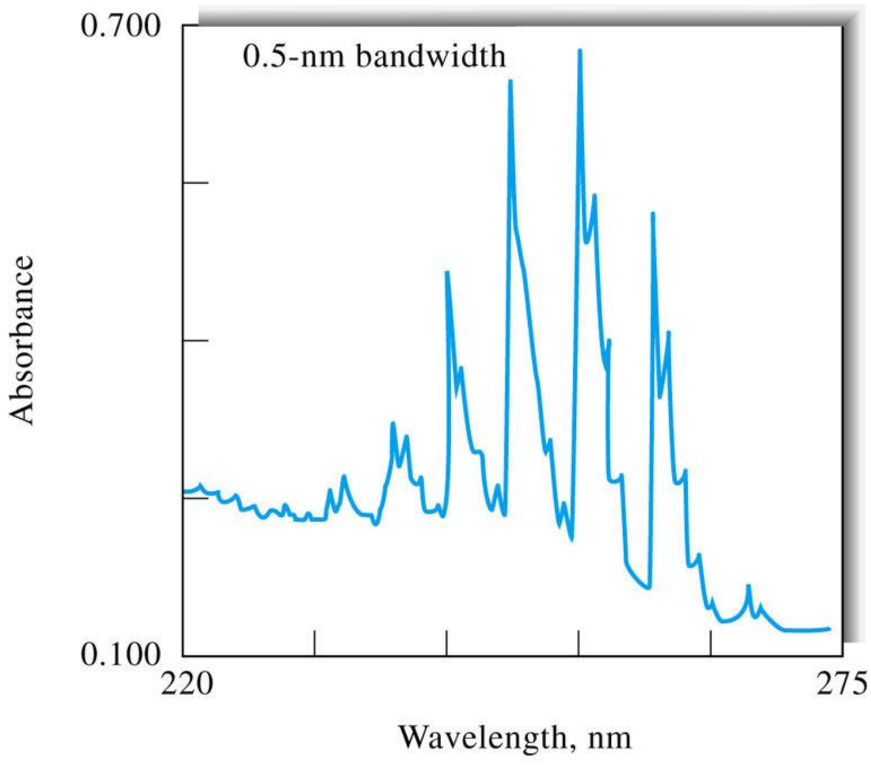
Components of Optical Instruments

Monochromator Slits

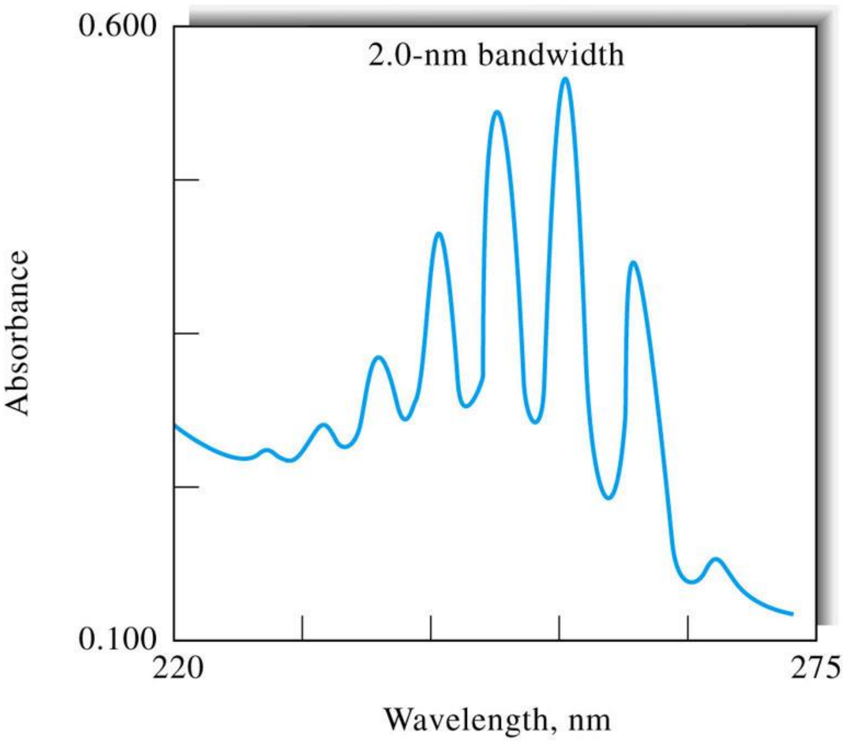
- Two pieces of metal with sharp edges.
- Must be exactly parallel to each other and lie on the same plane.
- The opening of the slit can be fixed or adjusted mechanically.

Components of Optical Instruments

Monochromator Slits



(a)



(c)

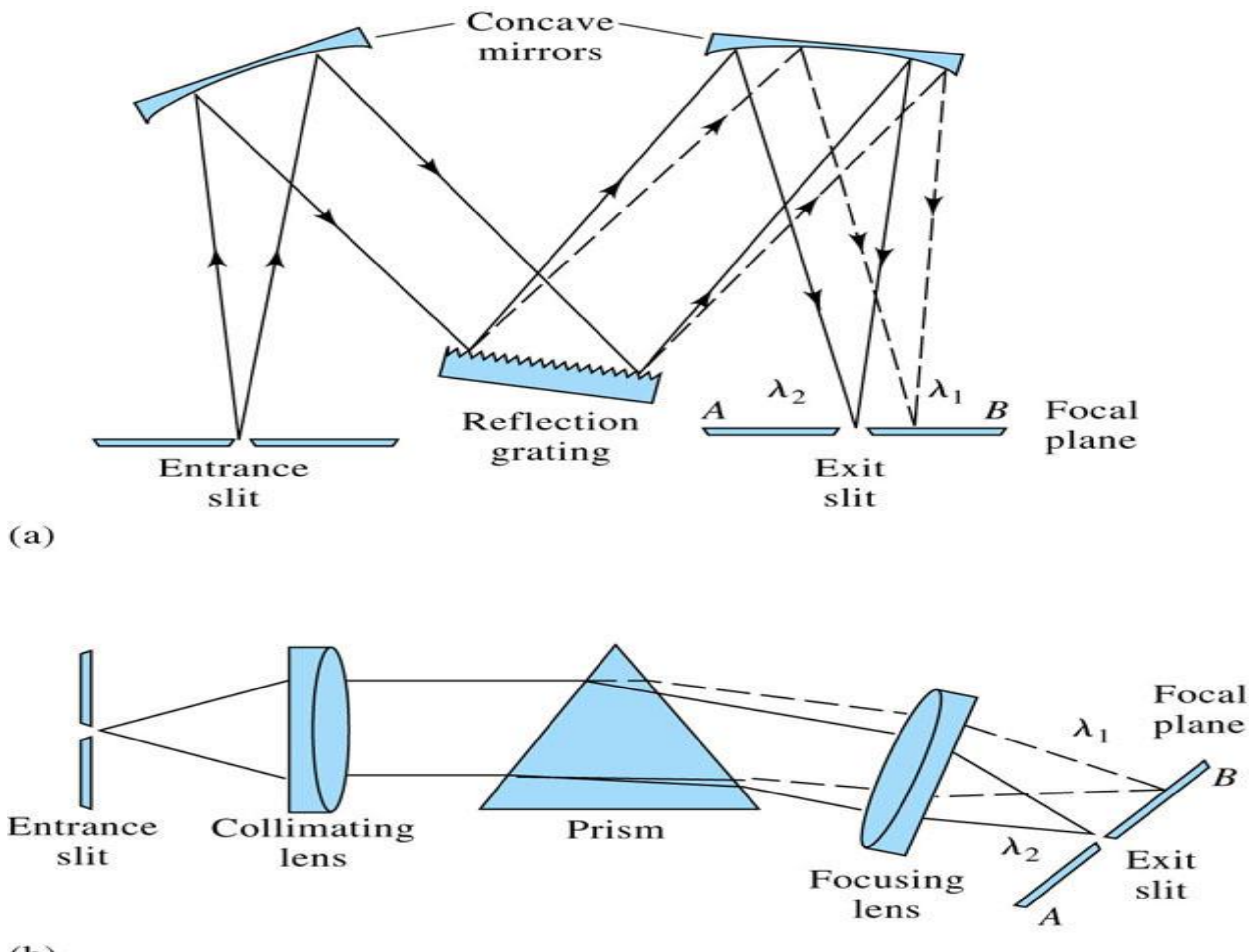
Components of Optical Instruments

Effect of Slit Width

Wider slits give poor resolution

A decrease in slit width gives power reduction in radiant energy and becomes a problem with low signal-to-noise ratios.

Components of Optical Instruments



Components of Optical Instruments

Historically, monochromators were prism instruments, however today nearly all commercial monchromators use reflection gratings.

Components of Optical Instruments

Grating Monochromators

Replica gratings

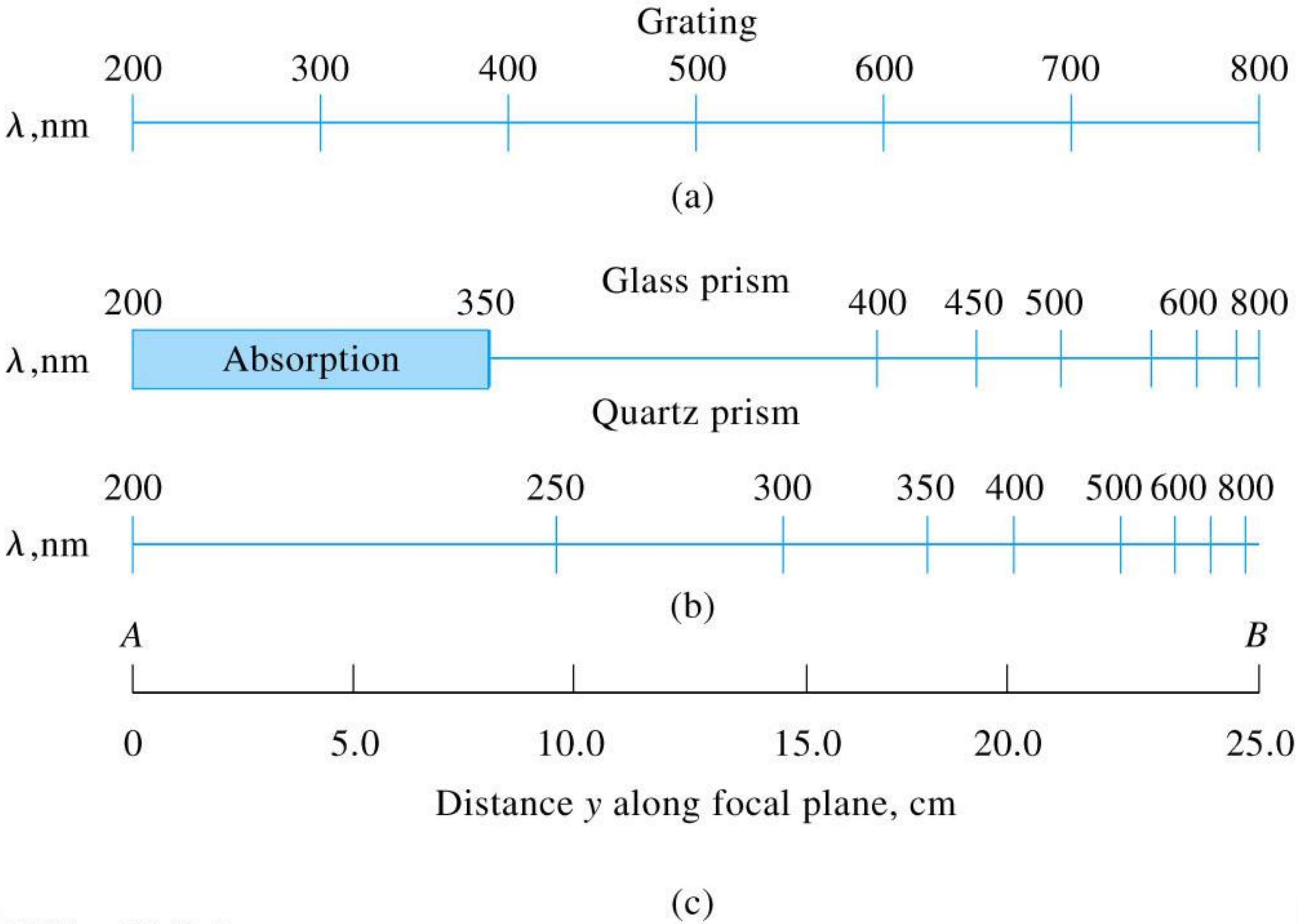
- Manufactured from a master grating consisting of a hard, optically flat, polished surface rules with a diamond tool.
- These gratings typically contain 10-200 groves/mm for IR region and 300-2000 groves/mm for UV-vis region.
- Replica gratings are formed from master gratings by a liquid resin casting process.

Components of Optical Instruments

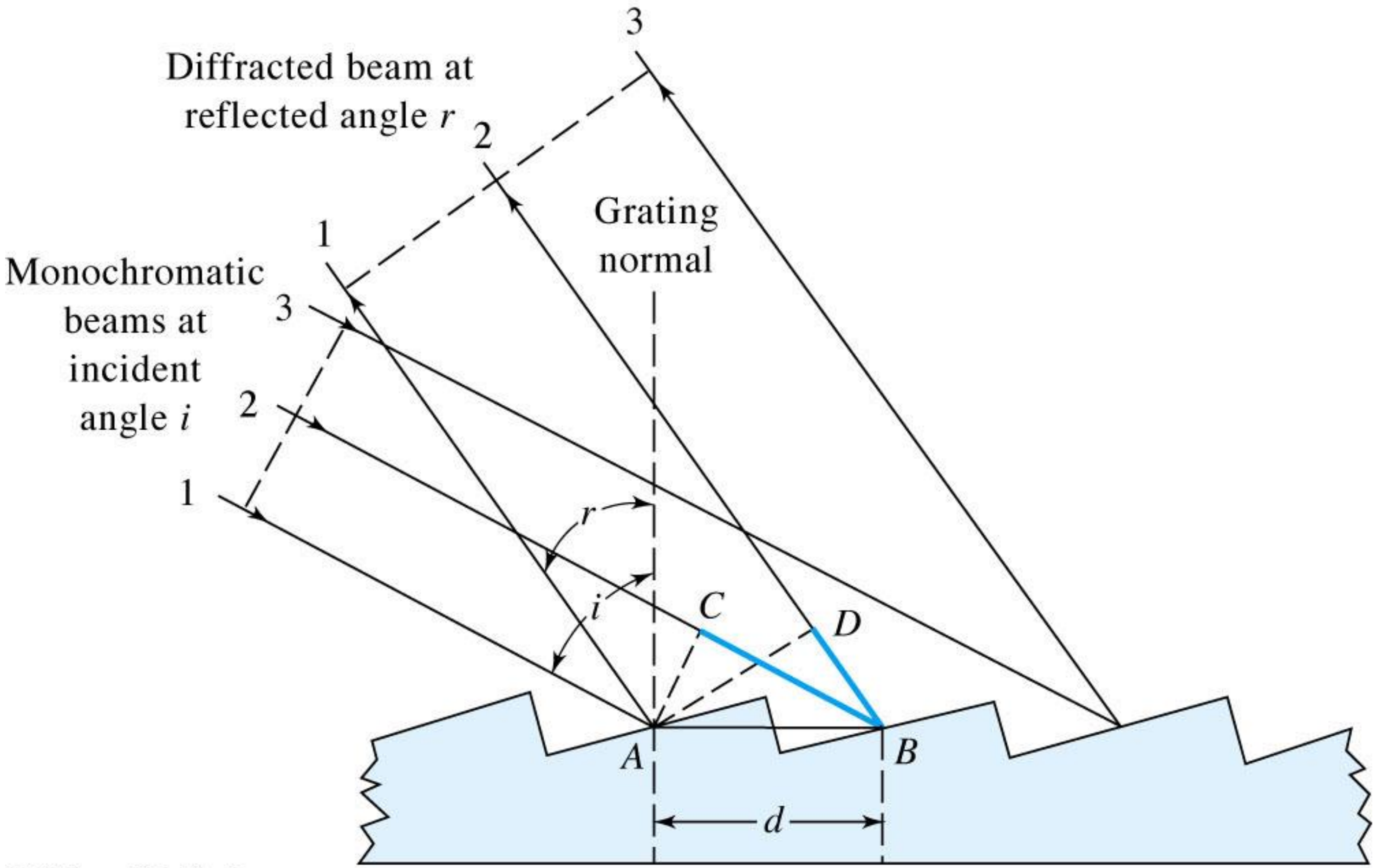
Advantages of gratings:

- Cheaper
- Give better wavelength separation
- Disperse radiation linearly along the focal plane

Components of Optical Instruments



Components of Optical Instruments



Components of Optical Instruments

Echellette Grating

Grooved with broad faces for reflection and narrow faces away from reflection.

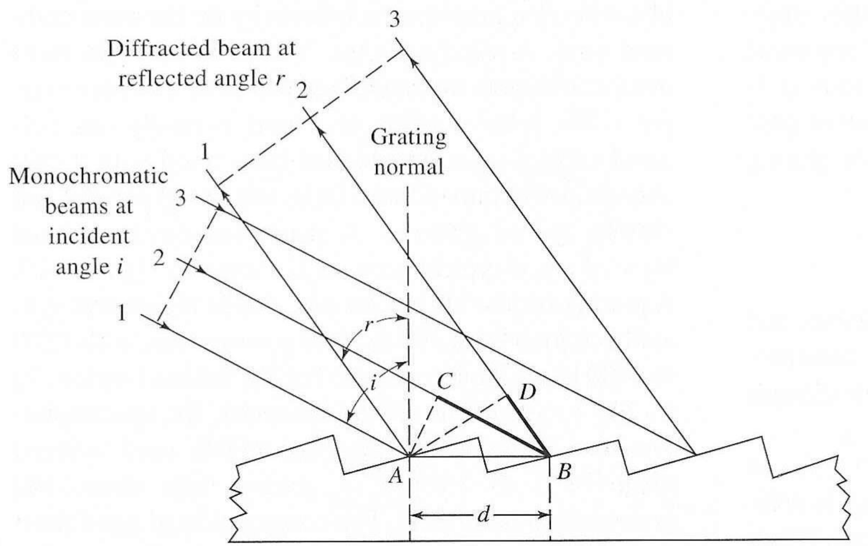


Figure 7-19 Mechanisms of diffraction from an echellette-type grating.

Components of Optical Instruments

Echellette Grating

Common geometry used to cause constructive interference so that

$n\lambda = (\overline{CB} + \overline{BD})$

n – whole number – diffraction order

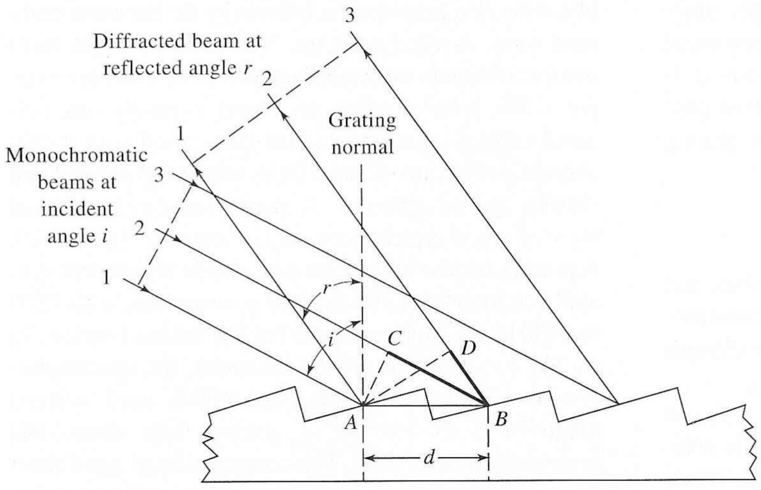


Figure 7-19 Mechanisms of diffraction from an echellette-type grating.

Components of Optical Instruments

Echellette Grating

CAB = i   and   DAB = r

$\overline{CB}$ = d sini and $\overline{BD}$ = d sinr

d-spacing between reflecting surfaces

$n\lambda = d(\sin i + \sin r)$

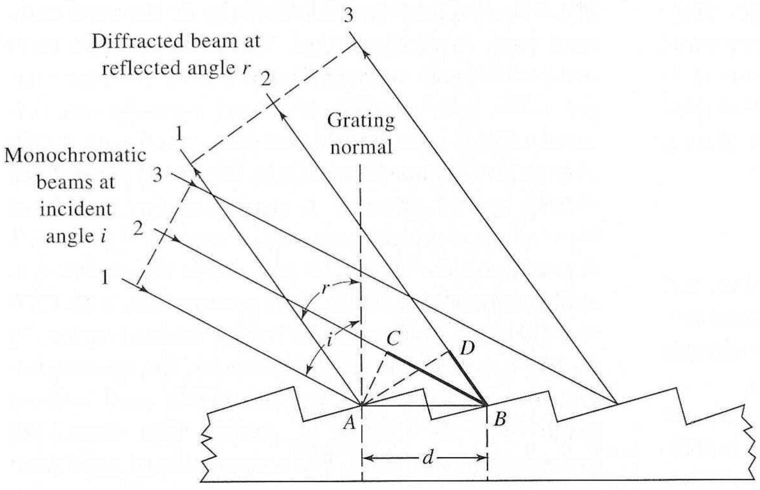


Figure 7-19 Mechanisms of diffraction from an echellette-type grating.

Components of Optical Instruments

Echelle Grating

Angle of reflection, r, is close to the angle of incidence, i.

$r = i = \beta$

$n\lambda = 2d\sin\beta$

Components of Optical Instruments

Echelle Monochromator

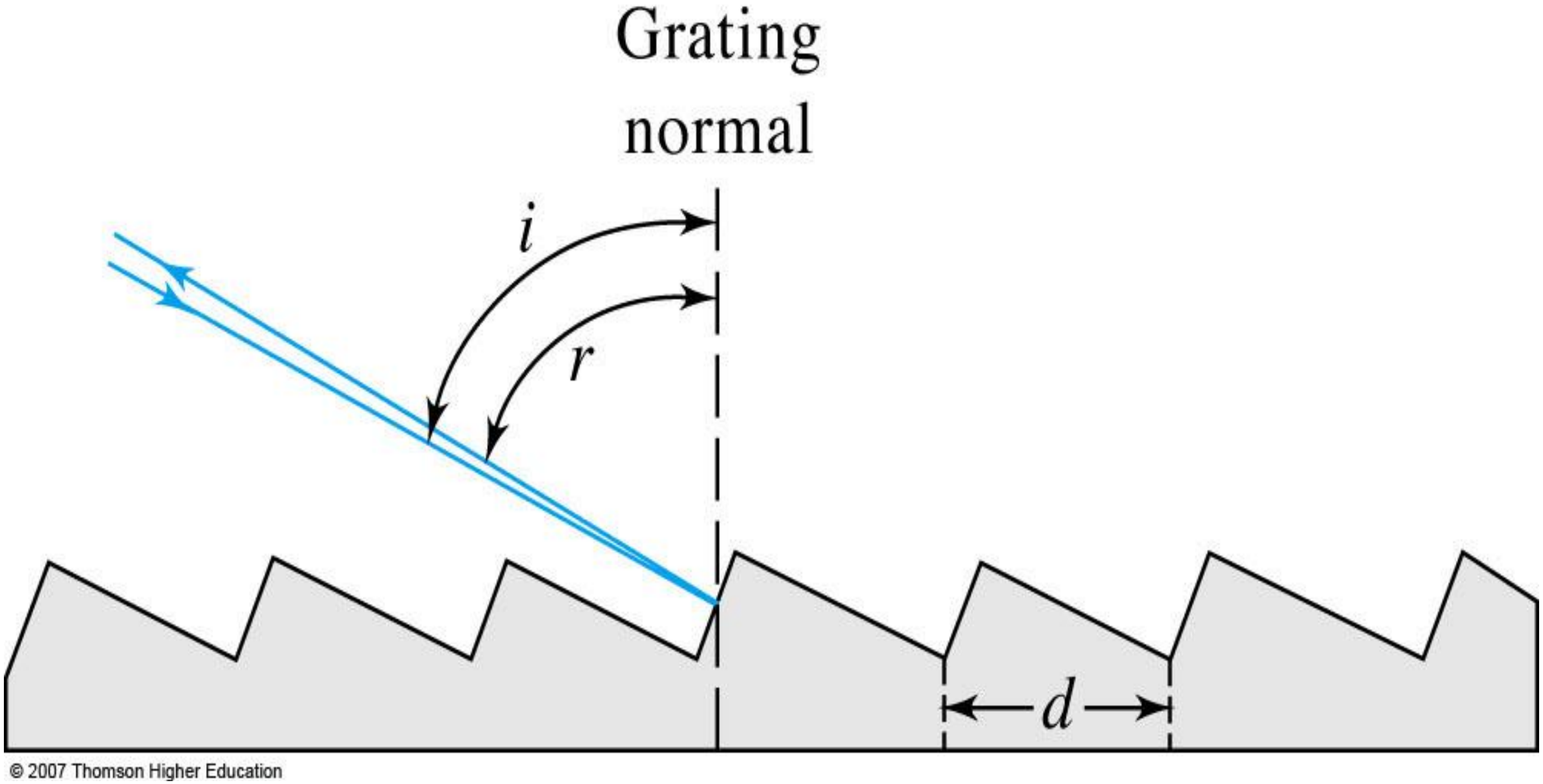
Contains two dispersing elements arranged in a series.

First element is the echelle grating.

This type of grating has reflection from the short face at a steep angle.

Components of Optical Instruments

Echelle Monochromator



Angle of reflection, r, is close to the angle of incidence, i. **$r = i = \beta$**

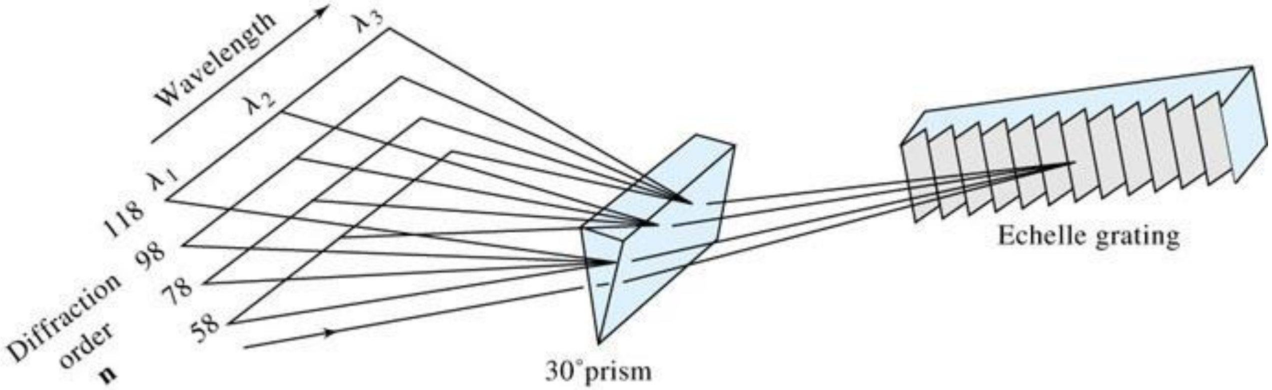
Components of Optical Instruments

Echelle Monochromator

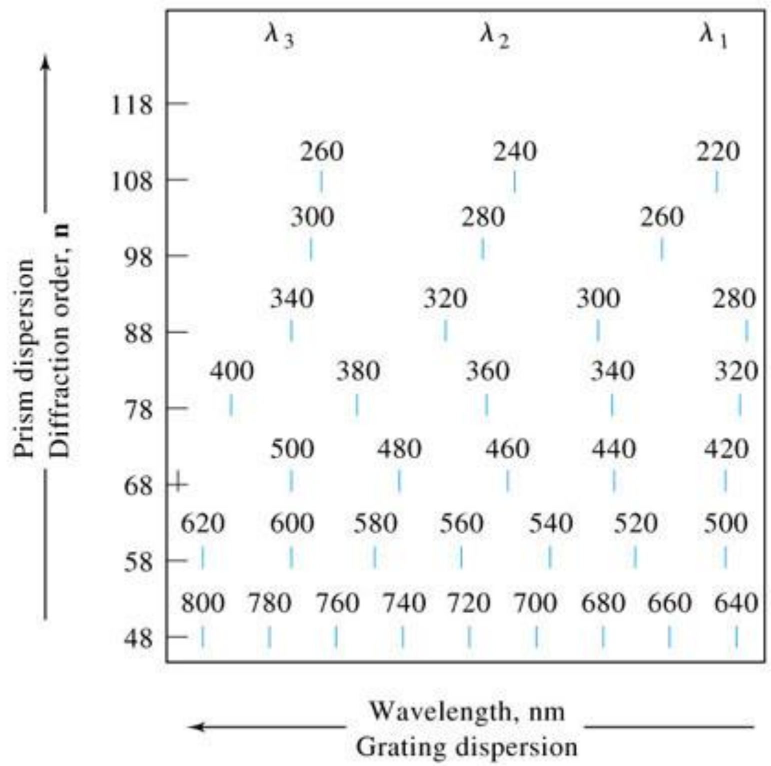
Second element is a low dispersion prism.

Advantage of an echelle grating:

- **higher dispersion**
- **higher resolution**


Wavelength
λ_3
λ_2
λ_1
118
98
78
58
Diffraction order
n
30°prism
Echelle grating

(a)


λ_3 λ_2 λ_1
118
260 240 220
108
300 280 260
98
340 320 300 280
88
400 380 360 340 320
78
500 480 460 440 420
68
620 600 580 560 540 520 500
58
800 780 760 740 720 700 680 660 640
48
Prism dispersion
Diffraction order, n
Wavelength, nm
Grating dispersion

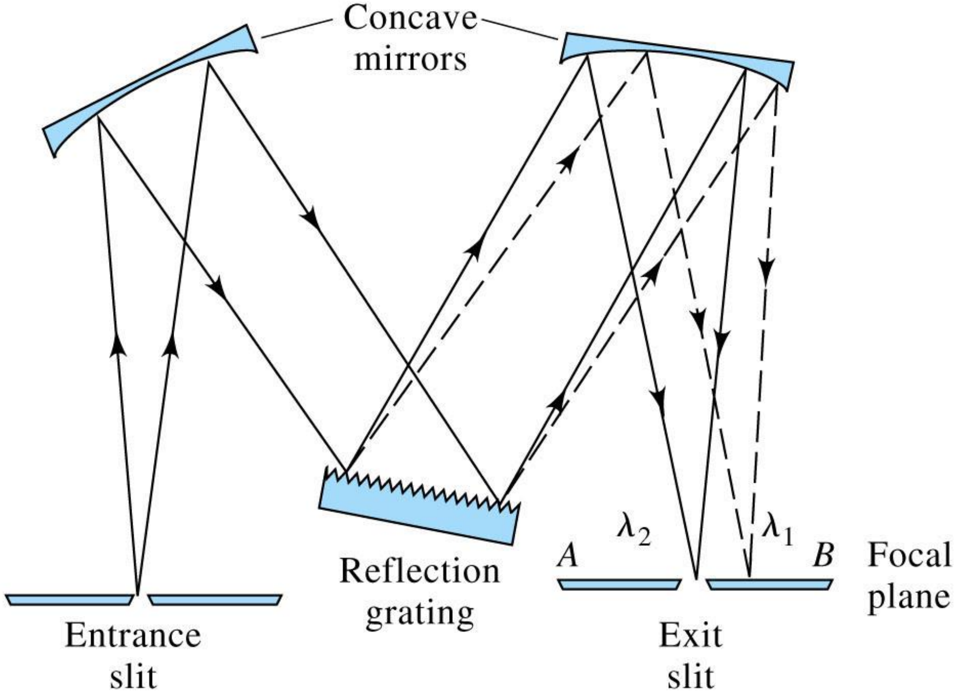
(b)

Components of Optical Instruments

TABLE 7-1 Comparison of Performance Characteristics of a Conventional and an Echelle Monochromator

	Conventional	Echelle
Focal length	0.5 m	0.5 m
Groove density	1200/mm	79/mm
Diffraction angle, β	10°22′	63°26′
Order **n** (at 300 nm)	1	75
Resolution (at 300 nm), $\lambda/\Delta\lambda$	62,400	763,000
Reciprocal linear dispersion, D^{-1}	16 Å/mm	1.5 Å/mm
Light-gathering power, F	$f/9.8$	$f/8.8$

Components of Optical Instruments



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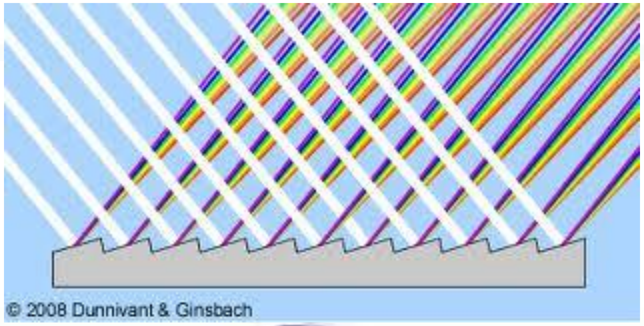


Components of Optical Instruments

Holographic Gratings

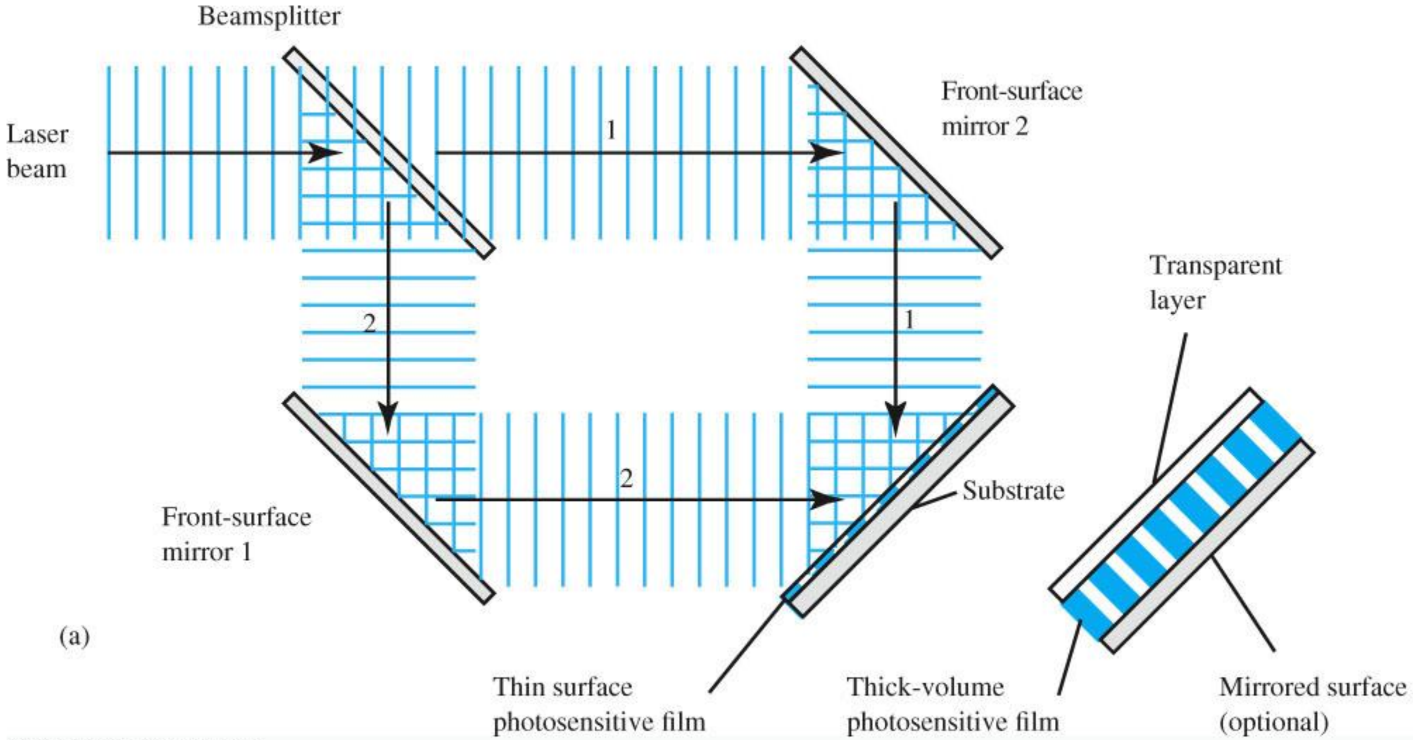
Gratings formed from an optical technique (lasers) on a plane or concave glass. (semiconductor industry)

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

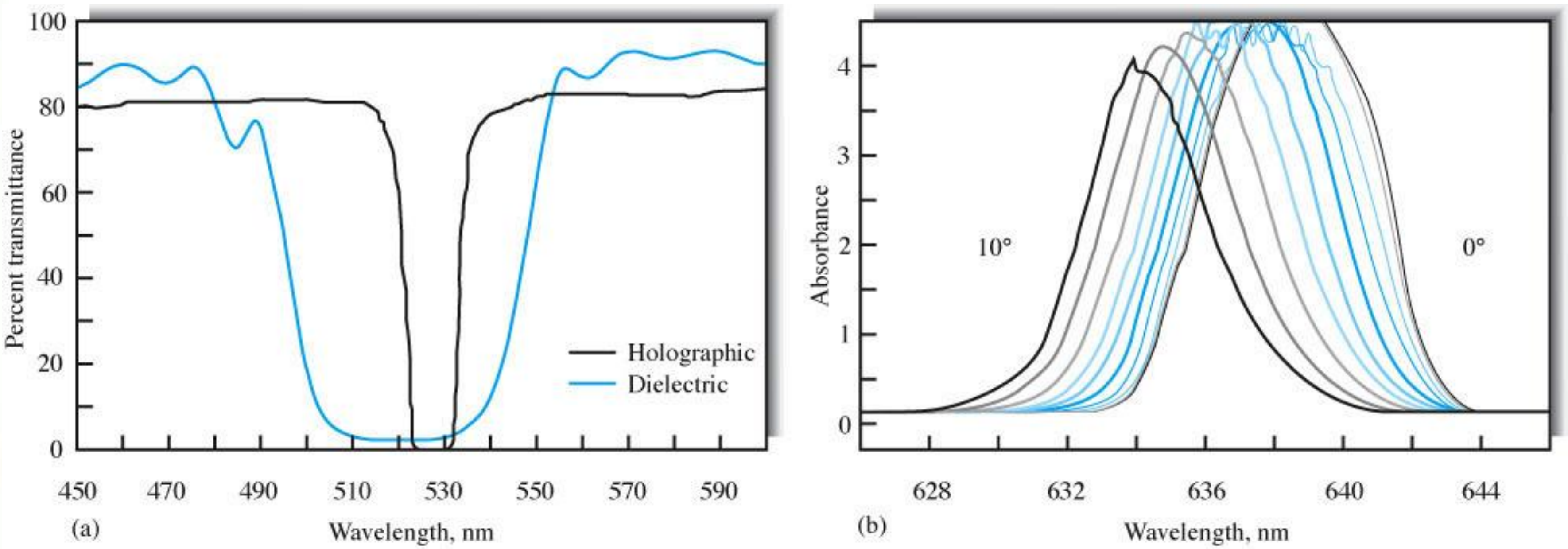
Advantages
- Greater perfection
- Less stray radiation and ghost
- Low cost

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

Components of Optical Instruments

Filters

Holographic Filters

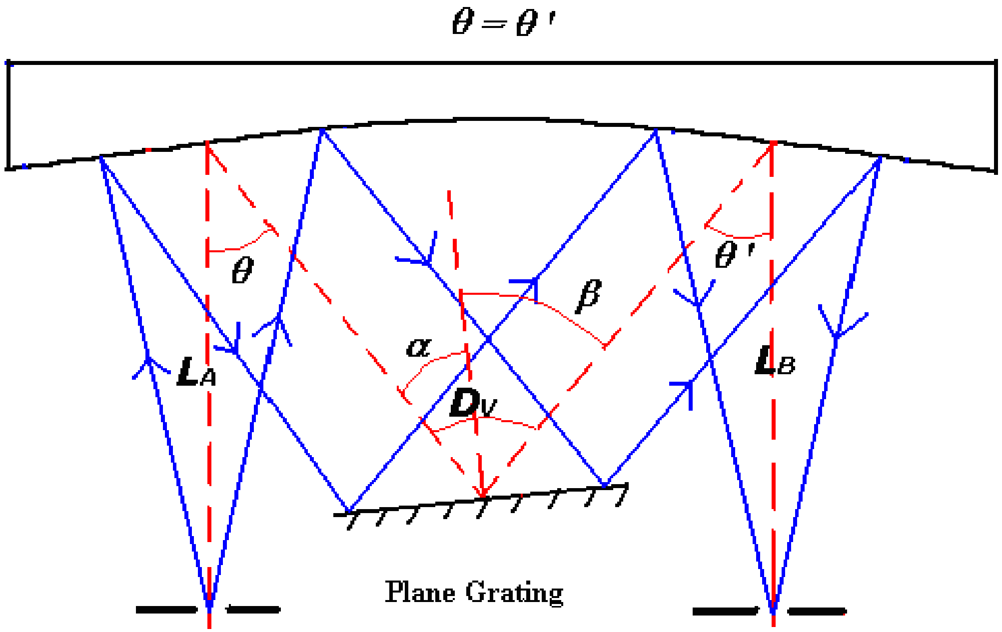


Components of Optical Instruments

Monochromator Configuration

Fastie-Ebert

One large spherical mirror and one plane diffraction grating.
Inexpensive but image quality low offaxis due to system aberrations.

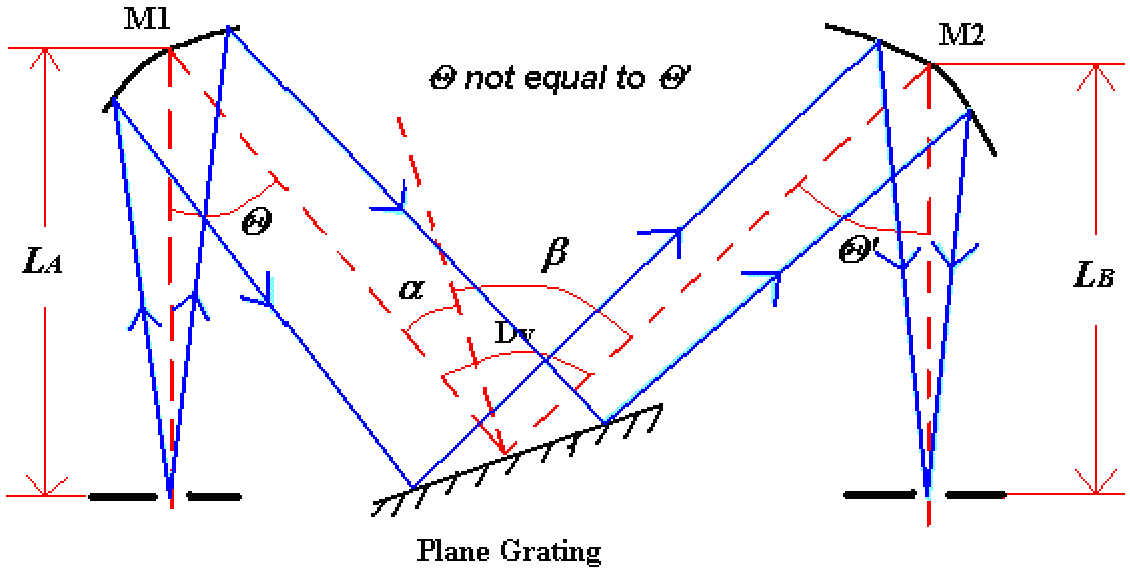


Components of Optical Instruments

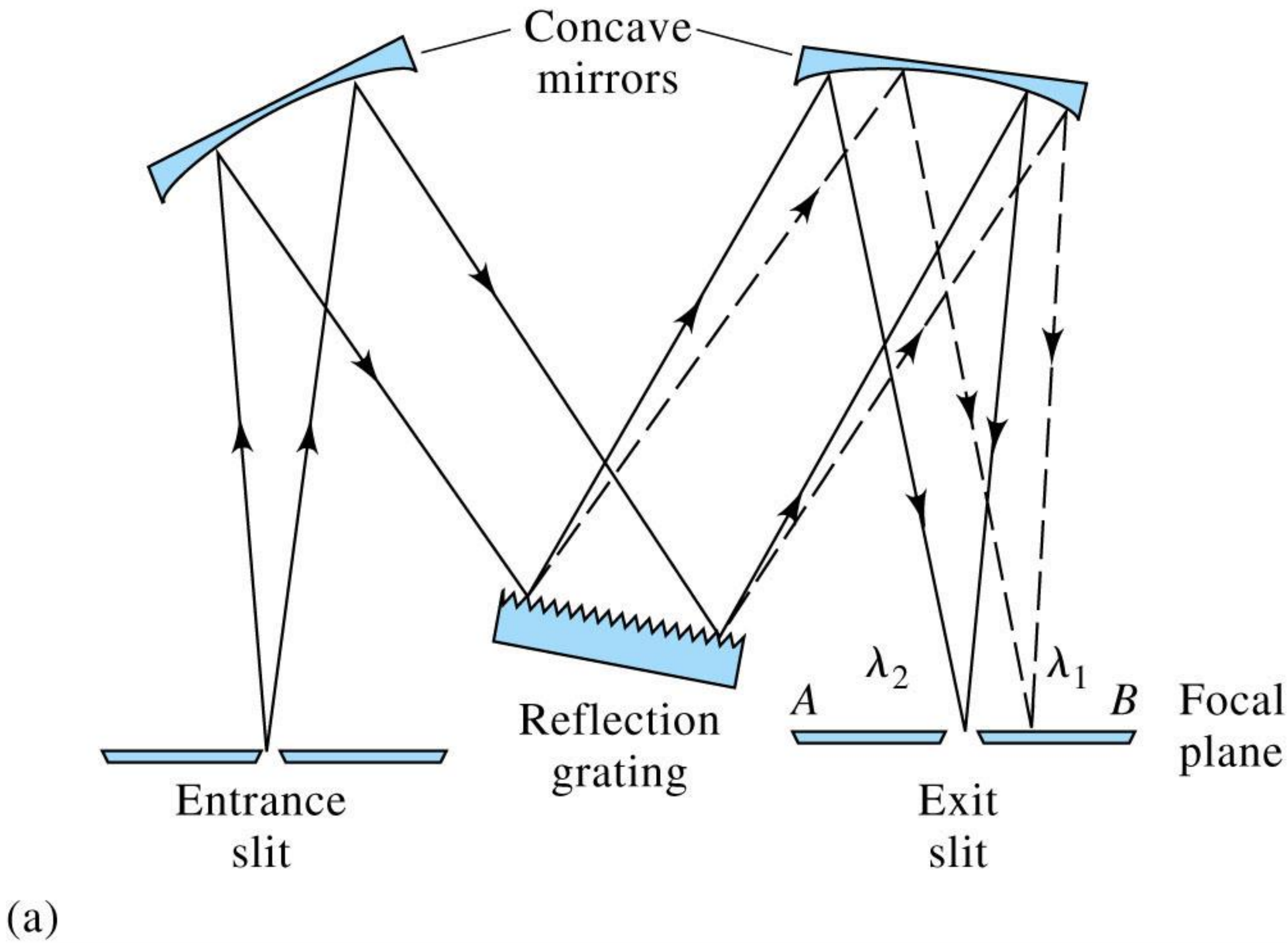
Monochromator Configuration

Czerny-Turner

Two concave mirrors and one plane diffraction grating. Flexible and good image quality, can accommodate very large optics.



Components of Optical Instruments



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

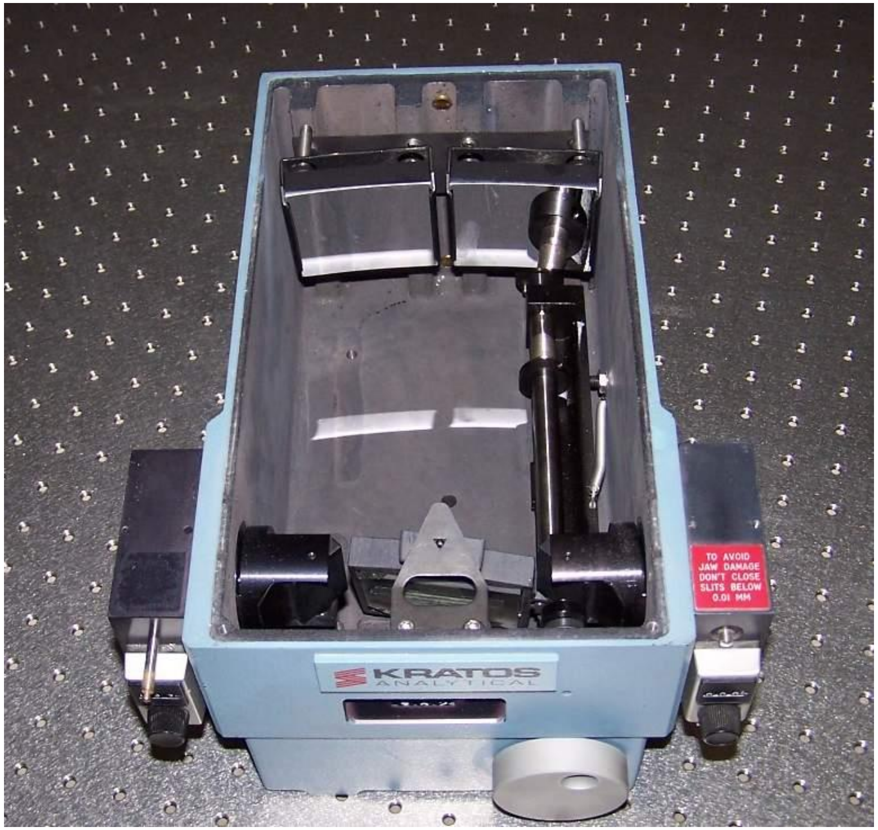
Scattered and stray radiation at other wavelengths interfere with measurements.

Source of unwanted radiation

- Reflection from monochromator housing
- Surface imperfections
- Dust particles

Components of Optical Instruments

Source of unwanted radiation



Components of Optical Instruments

Minimize unwanted radiation by:

- **Baffles**
- **Coating interior surface with flat black paint**
- **Seal monochromator with windows**

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

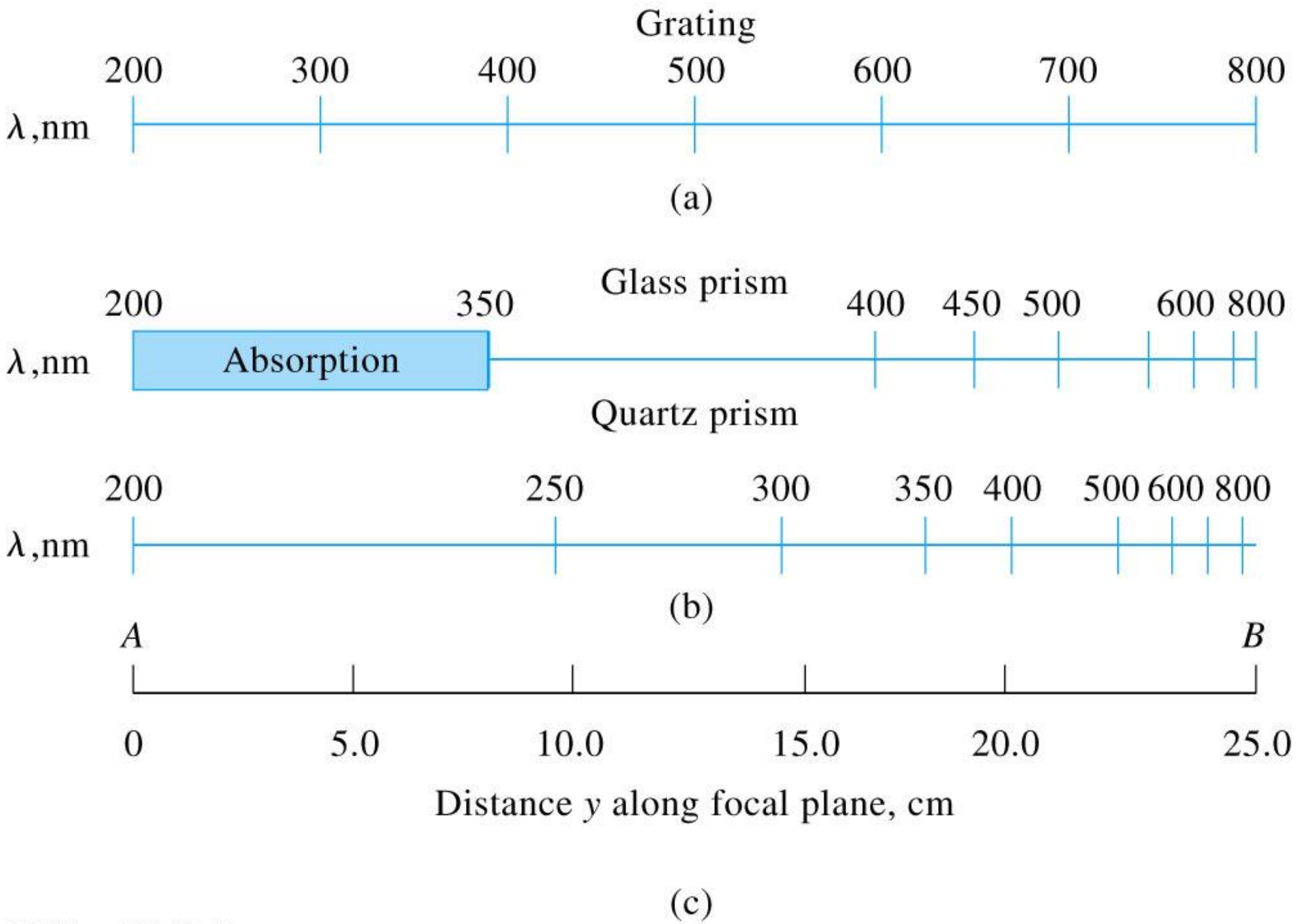
$d\lambda$ – change in wavelength

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

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

Resolving Power, R

The ability to separate adjacent images that have a slight difference in wavelength.

$R = \lambda/\Delta\lambda$

λ – average wavelength of the two images

$\Delta\lambda$ – difference of two images

$R = \lambda/\Delta\lambda = nN$ (n-diffraction order, N - # of grating blazes illuminated by radiation coming through the entrance slit)

Typically R ranges from 10^3 to 10^4

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

$F = f/d$

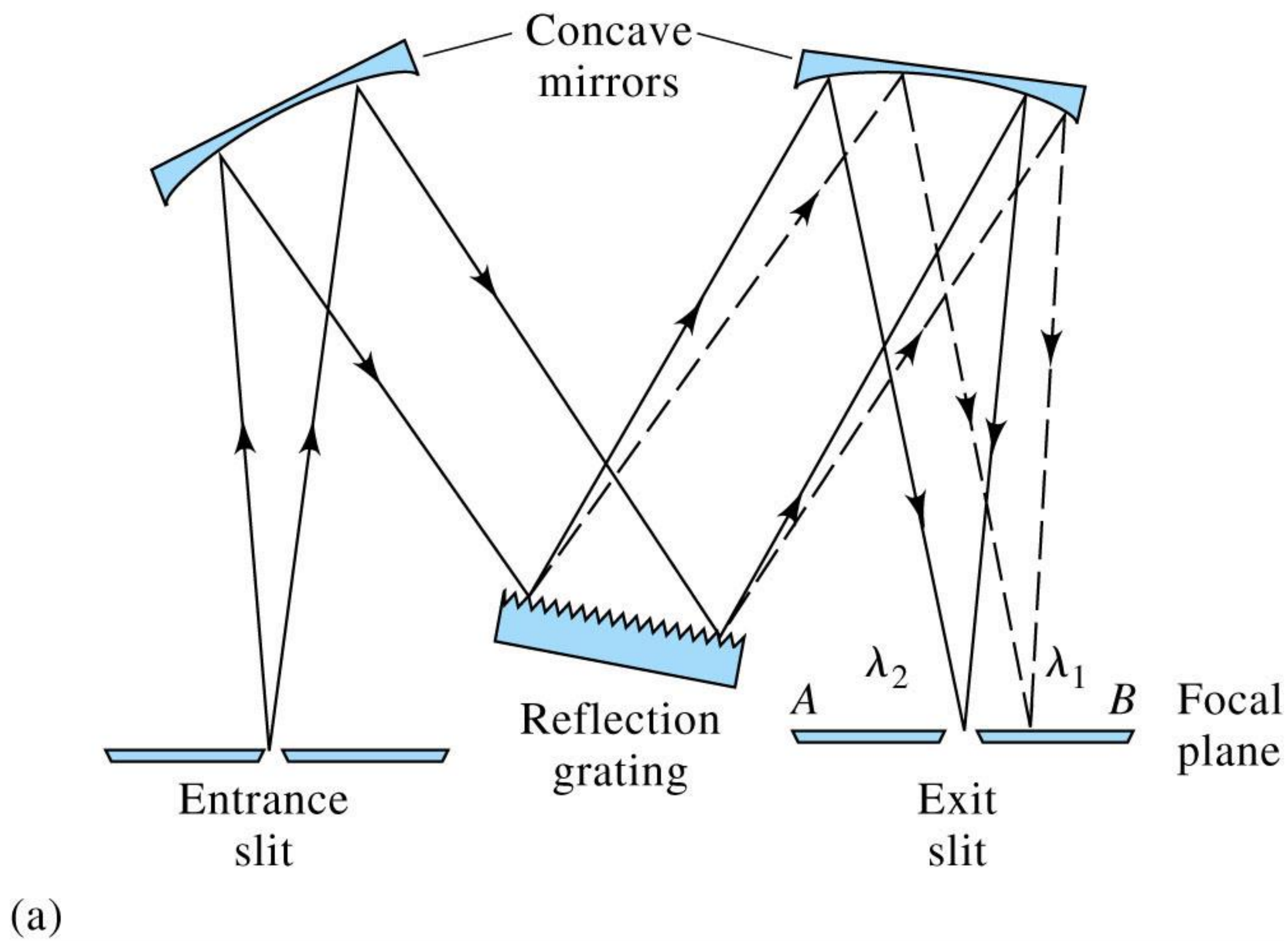
f- focal length of collimating mirror

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



Assignment

- Read Chapter 6 & 7 & 13
- Read Chapter 15
- Read Chapter 16 & 17
- HW 3: Ch. 16: 7, 8, 11 and Ch. 17: 2, 4, 5 (Due Today)
- HW4: Ch. 15: 1, 2, 4, 5, 9, 13 (Due 2-02)
- HW5: Ch. 7: 2-4, 8-13, and 16 (Due 2-05)
- Test 1- possible dates?