NIR SPECTROSCOPY
Instruments

Umeå 2006-04-10 Bo Karlberg

What is needed to construct a NIR instrument?

- A light source
- A dispersive unit (monochromator)
- A detector
- (Fibres)
- (Absorbance/reflectance-standard)

The light source

The tungsten (W) lamp is the most common light source

Relatively long life-time

A lamp change should not change instrument performance

The light source

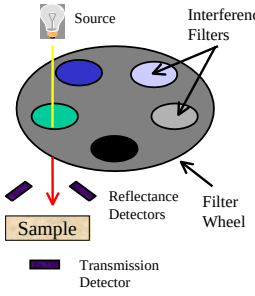
Light emitting diodes (LEDs) have been proposed as NIR light sources = an ideal concept!

Not ready for “real” applications yet……

Some “dispersive principles”

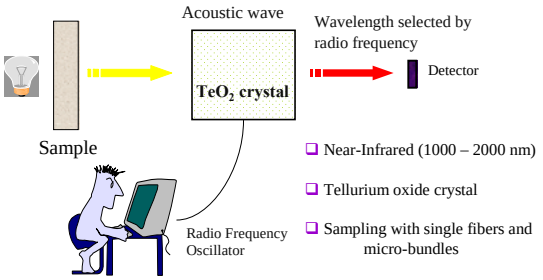
- Filter: two types (minimum)
  a) Fabry-Perot interference filter
  b) AOTF (acousto-optical tunable filters)
- Holografic grating
- The interferometer principle (FT-NIR)

Discrete filter systems



- Single and double beam configurations
- UV/visible, Near-Infrared, mid-Infrared filters
- 1 - 44 Filters
- Non-contact for solids
- Sample cells, single fibers, fiber bundles for liquids
- Fast, rugged, inexpensive

Acoustic Optical Tunable Filter (AOTF)



- Near-Infrared (1000 – 2000 nm)
- Tellurium oxide crystal
- Sampling with single fibers and micro-bundles

AOTF

Tellurium oxide, birefringent crystal

Acoustic waves change the refractive index of the material

Polychromatic light radiated onto one side of the crystal comes out as two monochromatic beams on the other side

AOTF

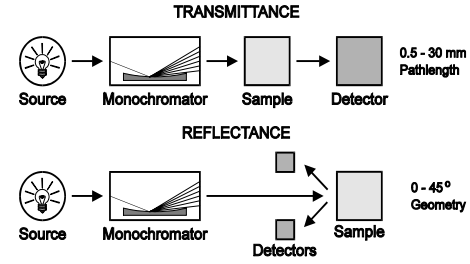
Advantages:

- no moving parts
- adjustable intensity
- narrow beams

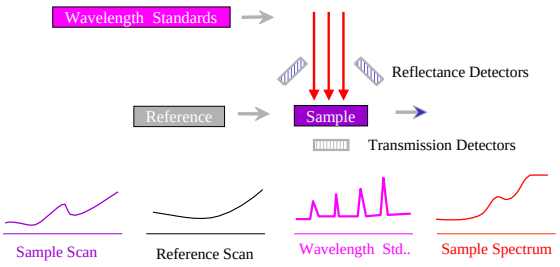
Disadvantages:

- difficulties when measuring highly absorbing samples
- limited wavelength range

Monochromator NIR

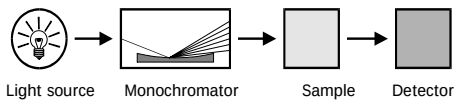


Digitally synchronous holographic grating system



We distinguish between pre-dispersive and post-dispersive configurations

PRE-DISPERSIVE



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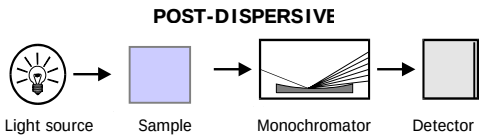


FT-NIR

FT = Fourier Transform

How does it work?

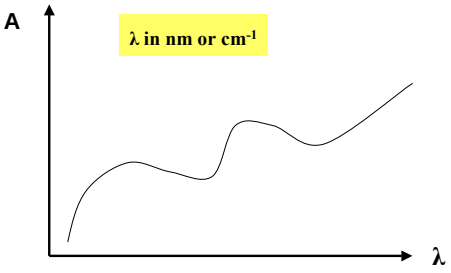
First, we have to distuingish between:

- Frequency domain spectroscopy

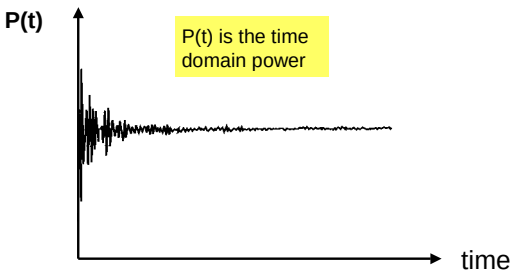
  and

- Time domain spectroscopy

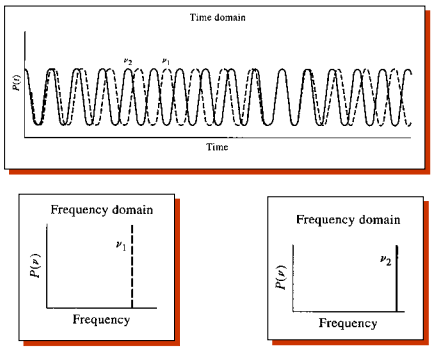
Frequency domain spectroscopy



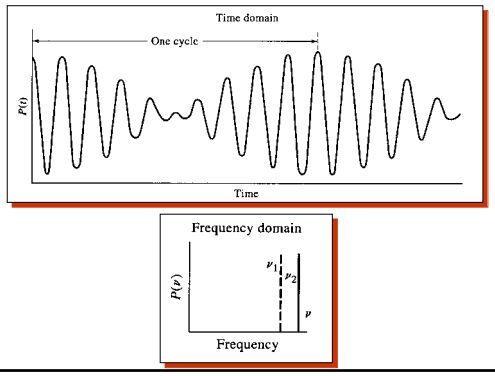
Time domain spectroscopy



Interferogram



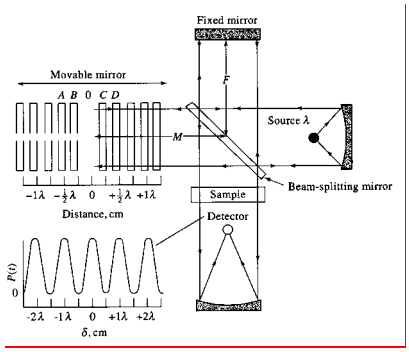
Interferogram



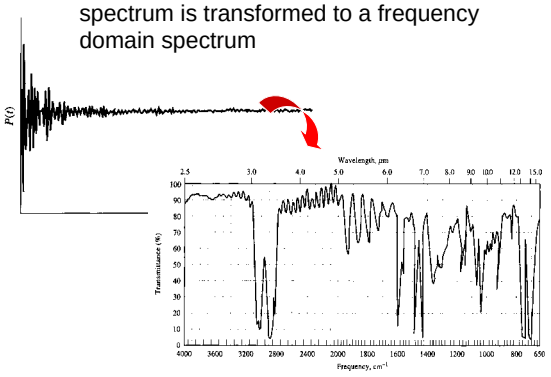
No detector can register waves at the speed of light… however:

time domain spectra can be created through application of interferometric approaches

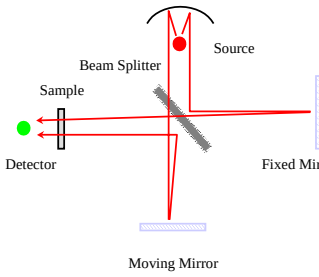
The interferometer principle



Fourier transform = the time domain spectrum is transformed to a frequency domain spectrum

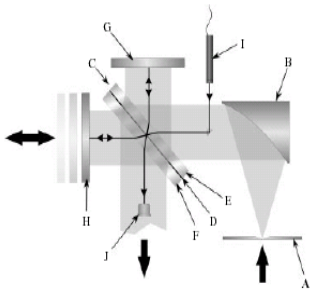


The Michelson interferometer



- Non-dispersive system with white light illumination
- Mechanical or magnetic drive mechanism
- Single beam rapid scanning of reference and sample
- Co-addition of spectra to improve signal-to-noise
- Sampling attachment

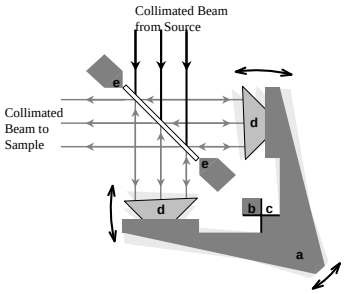
The Michelson interferometer



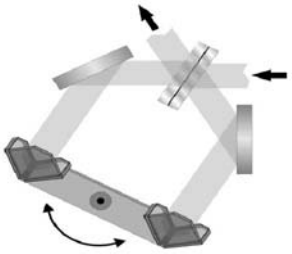
SAMPLE

The “wishbone” interferometer

a “Wishbone”
b Anchor
c Flex pivot (2)
d Cube corner mirrors
e Beam Splitter

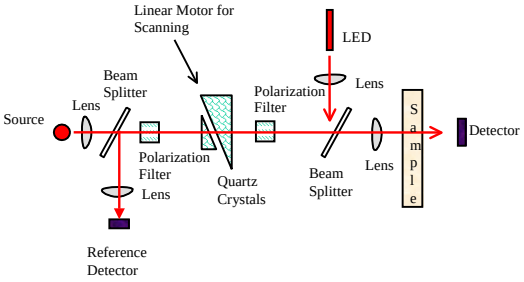


The wishbone design



Double pendulum

The crystal interferometer



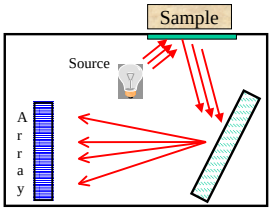
Dispersion principles, summary

- NIR: filter, grating and FT instruments are equally common on the market (roughly)
- mid-IR: total domination of FT instruments

Detectors, NIR

- Silicon detector, up to 1100 nm, stable, rapid, reliable, inexpensive
- Lead sulphide, 900-2600 nm, a common NIR detector, established, a little slow response
- InGaAs (indium gallium arsenide), 800-1700 nm, 1300-2200 nm, 1500-2500 nm, expensive

The diode-array design



Fixed Grating

- UV/visible, Near-Infrared
  - Silicon Array (Si) 400-1100 nm
  - Lead Sulfide (PbS) 1100-2500 nm
  - Indium Gallium Arsenide (InGaAs) 800-1600 nm
- Sample cells, fiber optic bundles
- Resolution is determined by the number of elements in the array

Scanning NIR systems

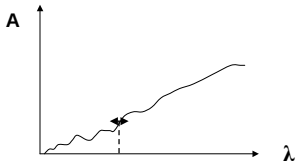
System	Advantages	Disadvantages
Holographic Gratings	Rapid Scanning High Dynamic Range Rugged (Digital) Extend Scan Ranges	Moving Parts
Interferometers	Rapid Scanning Large Aperture	Moving Parts Environmentally Sensitive (Varies) Bandpass Variation
AOTF	Fast Scanning Fast Stepping	Moving Parts Spectral Artifacts Bandpass Variation RF & Temperature Sensitive Unique Components (Crystal)
Diode Arrays	No Moving Parts Rapid Scanning Rugged	Limited Wavelength (Cost) Limited Dynamic Range Temperature Sensitive Pixel Variations

Qualitative analysis

- Identification of various substances (often very pure)
- Classification

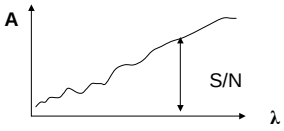
Qualitative analysis

In this case the ***spectral resolution*** is of large importance

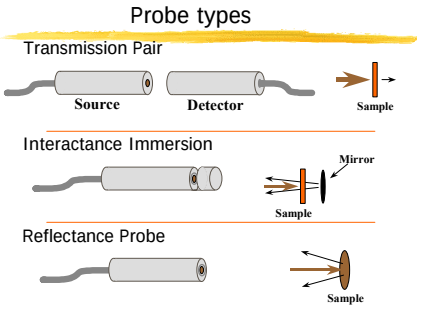


Quantitative analysis

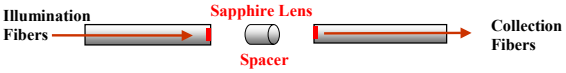
In this case the ***signal-to-noise ratio*** is of large importance



Sample presentation, NIR



Contact Probes: Transmission

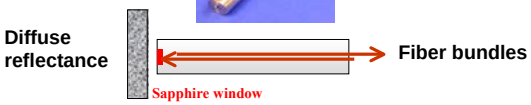


- Spacers for fixed path lengths (1 to 40 mm)
- 316 Stainless steel or other metals
- Ratings: 300C at 5000 psi (350 atm)
- Metal to sapphire seals

Contact probes: Immersion

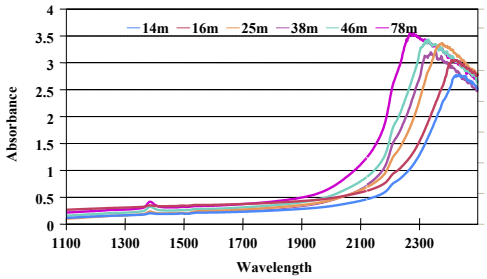
- **0 to 15% Total solids**
- **316 Stainless steel or other metals**
- **Ratings: 300C at 5000 psi (350 atm)**
- **Metal to sapphire seals**

Contact probes: Reflectance

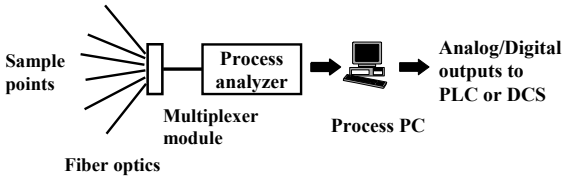


- **316 Stainless steel or other metals**
- **Ratings: 300C at 5000 psi (350 atm)**
- **Metal to sapphire seals**

Fiber optics vs. length



Multiplexer systems



- **Multiple sample points per instrument**
- **Sequential analysis**
- **Reduced cost per measurement point**

Side-stream sampling

- Transmission, Interactance & Reflectance Probes
- 316 Stainless Steel Construction
- 1 in. Swagelok Fittings for Probes
- 0.5 & 1.0 in. NPT for Sample Stream