

In-situ materials characterization for planetary exploration

Eng Keong Chua
echua@andrew.cmu.edu

Outline

- ▶ *Motivation*
- ▶ *Instruments on lander*
 - ◆ *Working principle*
 - ◆ *Recent development*
 - XRD/XRF (CheMin)
 - Portable XRD/XRF (Terra)
- ▶ *Instruments on orbiter*
 - ◆ *Working principle*
 - ◆ *Recent development*
 - Portable FT-IR



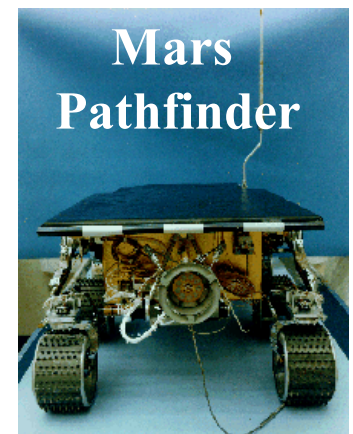
Motivation

- ▶ *In situ analyses of surface rocks and soils*
 - ◆ Investigate the structure and composition at the site by *analysis tools integrated into tele-operated robot*
- ▶ *Benefits:*
 - ◆ Survey before planning human to a site of interest
 - ◆ Acquire information for decisions in real time at the site with remote control

Instruments deployed by space agency

► Lander

- ◆ Gas Chromatography Mass Spectrometry
- ◆ X-Ray Fluorescence Spectrometry
- ◆ Alpha Proton X-Ray Spectrometry
- ◆ X-Ray Diffraction Spectrometry

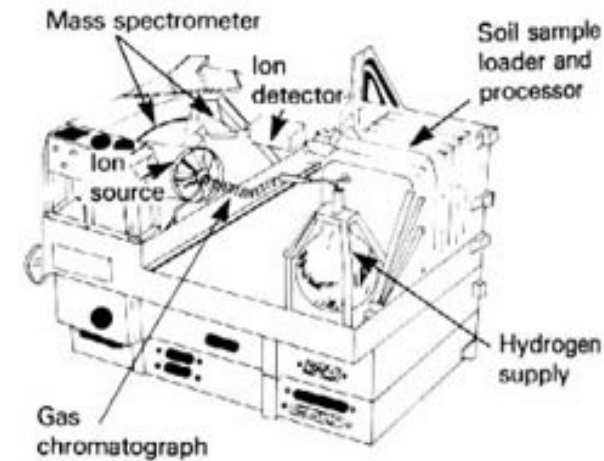


Gas Chromatography Mass Spectrometry

► Identify organic molecule or atmosphere composition (NASA Viking 1 - 1976)

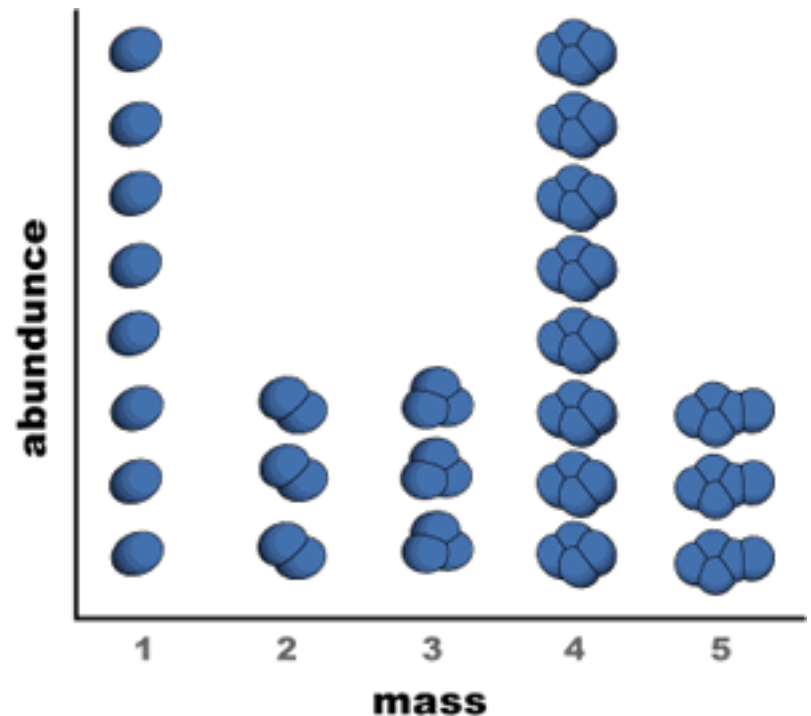
► GCMS setup

- ◆ Gas chromatography (GC) separates chemical mixture into pure chemicals
- ◆ Mass spectrometer (MS) identifies and quantifies the chemicals
- ◆ Data from mass spectrometer is sent to a computer to plot mass spectrum



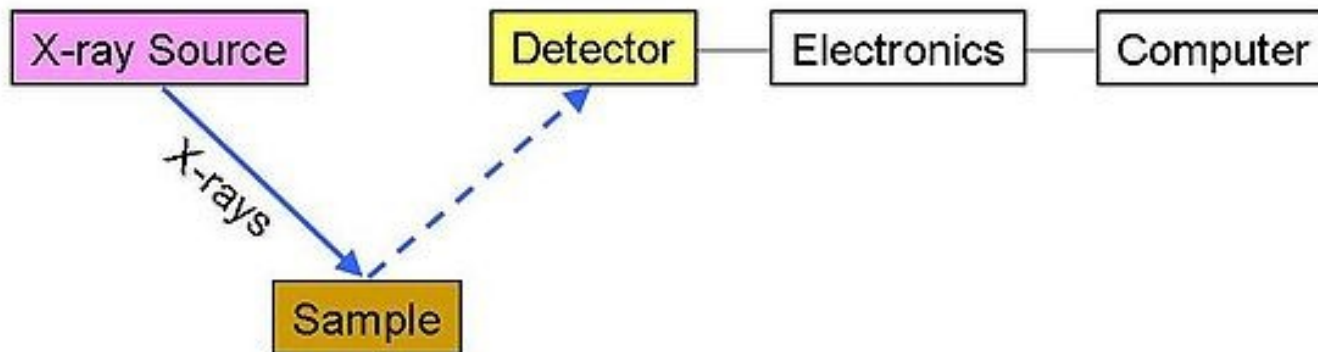
Principle of GCMS

- ▶ Decomposition of the pure molecules in MS, data is plot as mass spectrum
 - ◆ This observed spectrum is compared with published patterns of known decomposition that is stored in a fragmentation library to identify the organic molecule



X-Ray Fluorescence Spectrometry

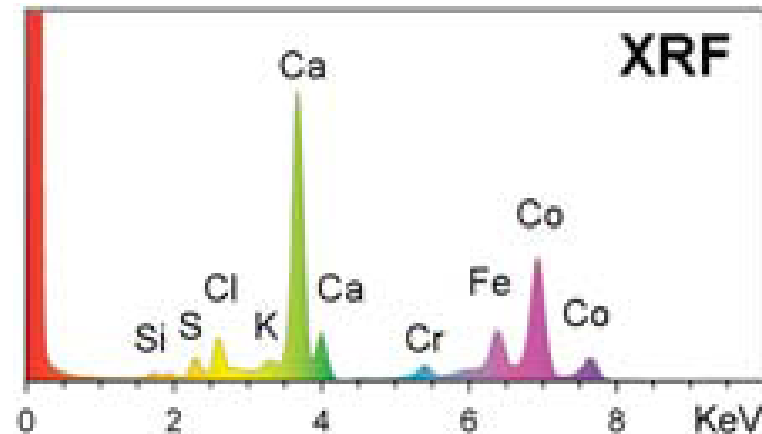
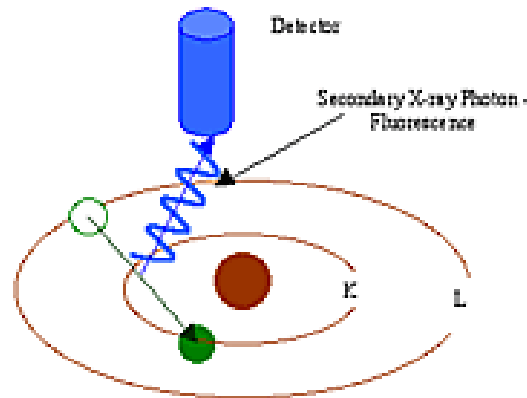
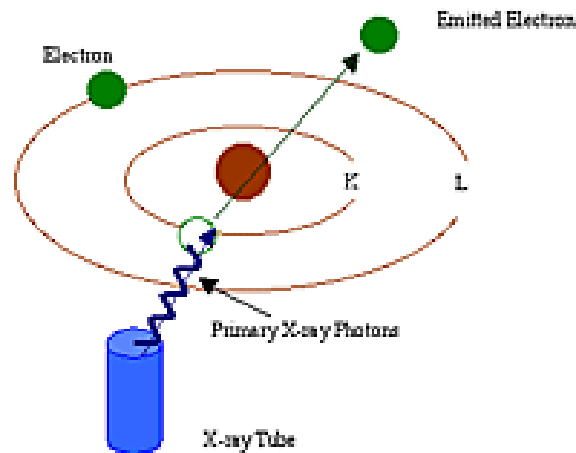
- ▶ Identify and determined concentration of elements: (NASA Viking 1 - 1976)
 - ◆ solid, powdered and liquid samples
- ▶ XRF setup



Principle of XRF

- ▶ Measures characteristics X-rays of fluorescent emission produced by a irradiated sample

$$\lambda = h \cdot c / E$$



Alpha Proton X-Ray Spectrometry

- ▶ Analyses the chemical element composition of a sample (NASA Mars Pathfinder- 1996)
- ▶ APXS set up
 - ◆ Alpha particles sources irradiated on sample
 - ◆ Detector:
 - Scattered alpha particles
 - Emitted protons
 - Fluorescent X-rays



Principle of APXS

▶ Principle:

◆ Based on interactions of alpha particles with matter:

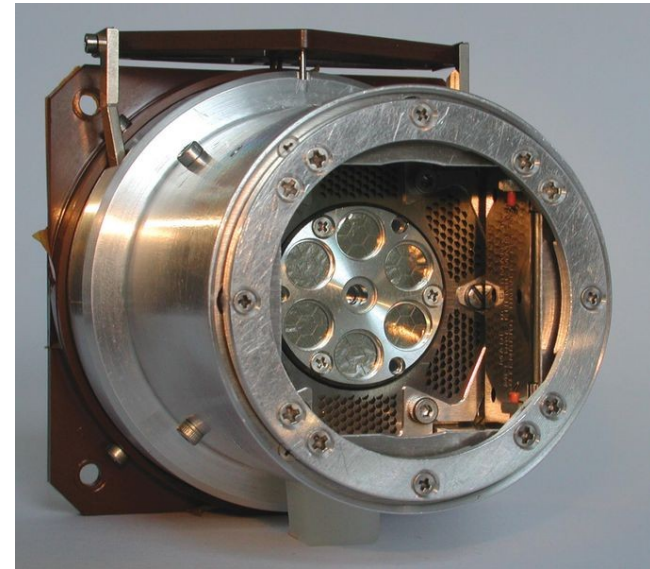
■ Collision of alpha particles with nuclei

- Elastic scattering of alpha particles by nuclei

- Alpha particles absorbed by nuclei produce proton of certain energy by alpha-proton nuclear reactions

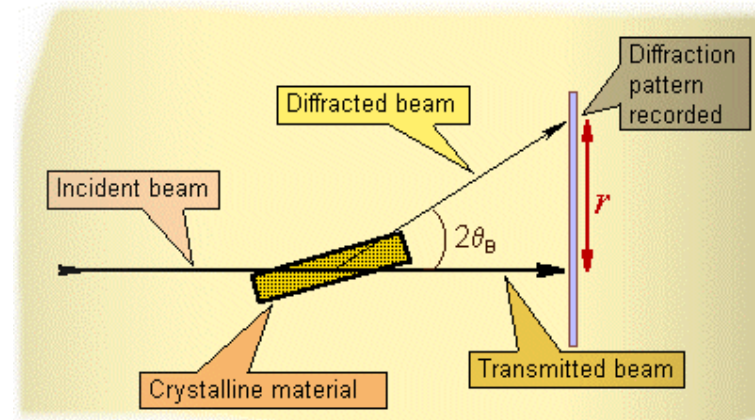
■ Collision of alpha particles with electrons of the atoms

- Excitation of the atomic structure of atoms leading to the emission of characteristic X-rays

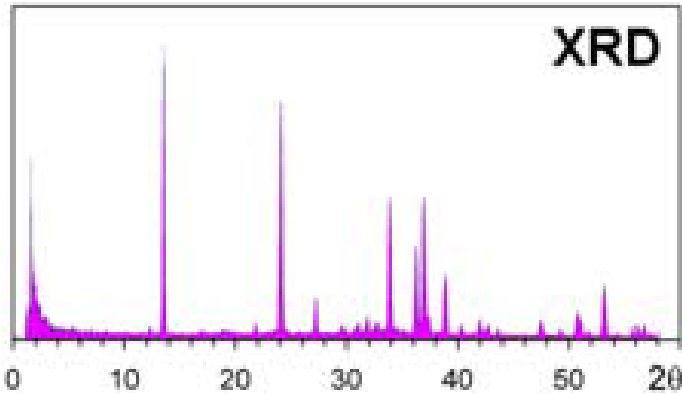


X-Ray Diffraction Spectrometry

- ▶ XRD the most comprehensive tool to identify crystal structure (ESA Beagle 2 - 2003)
 - ◆ Examine reflection of X-rays from a material at various angles.
 - X-rays were passed through slits to produce narrow beam, which fell on a material at centre of the spectrometer
 - Reflected (Diffracted) beam was measured in an charge coupled device (CCD)

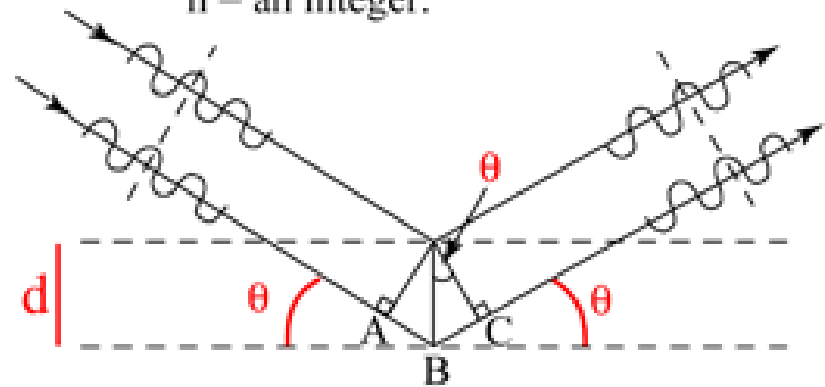
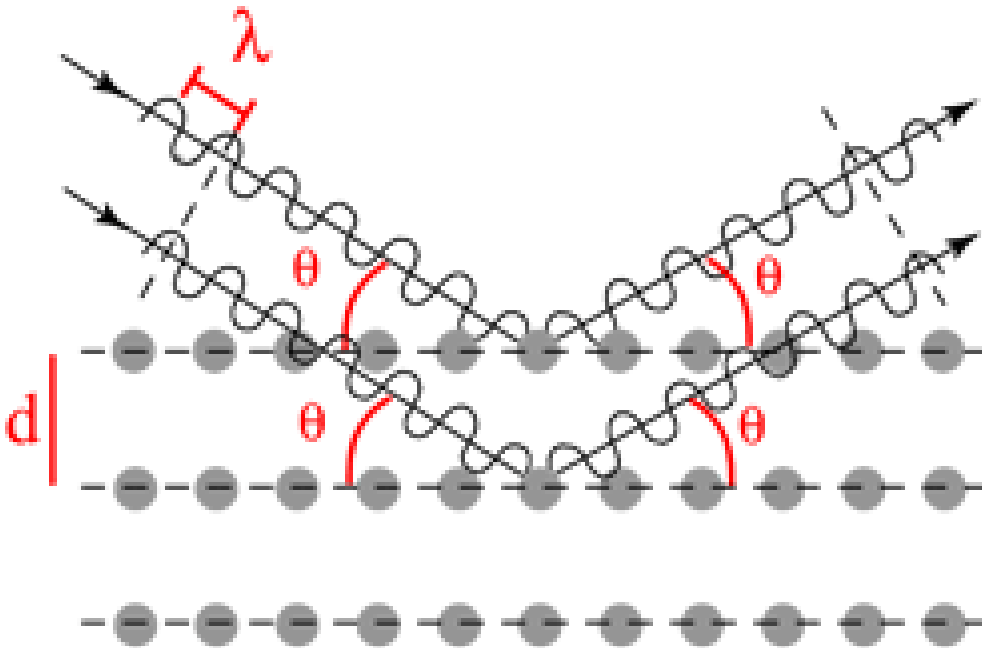


Principle of XRD



$$\text{Bragg's law: } n \lambda = 2 d \sin \theta$$

where λ = wavelength of the X-ray,
 d = interplanar spacing of the crystal,
 θ = angle between the lattice plane
and the X-rays,
 n = an integer.



$$\sin \theta = \frac{BC}{d} \quad \text{or} \quad d \sin \theta = BC$$

If $2d \sin \theta = (AB + BC) = n \lambda$,
then constructive interference

Recent Development

- ▶ To characterize a material completely, need to identify the composition and structure of a material
 - ◆ GCMS, XRF and APXS can determine the composition of the element
 - ◆ XRD can determine the structure of the material
- ▶ Integration of XRD and XRF
 - ◆ XRD/XRF (CheMin)
 - ◆ Portable XRD/XRF (Terra)

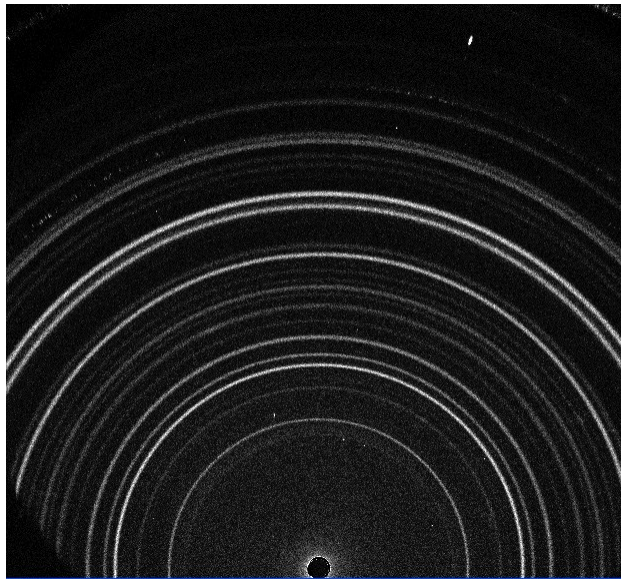
XRD/XRF (CheMin)

- ▶ CheMin developed (NASA Mars Science Laboratory rover to be launched in 2011)
 - ◆ Capable of getting the composition and the structure of the sample



XRD/XRF (CheMin)

- ▶ Enabler for CheMin integration
 - ◆ Same source (X-ray)
 - ◆ An electronic area array detector
 - Charge coupled device (CCD) sensitive to X-ray
 - Records fluorescent X-rays (characteristic X-rays)
 - Records diffracted X-rays that are scattered from sample

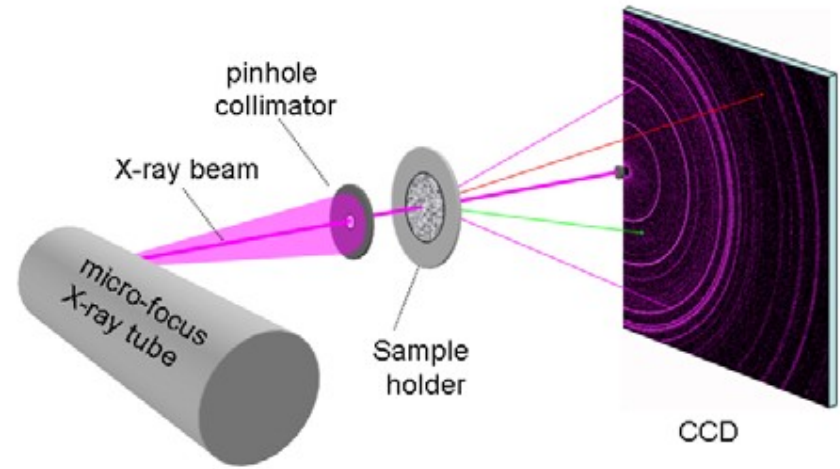


Bright rings correspond to diffraction bands; fluoresced pixels have no spatial correlation.

XRD/XRF (CheMin)

► Architecture

- ◆ X-ray generated by microfocus X-ray tube
- ◆ Combined with pinhole collimator
- ◆ Material loaded in sample holder
- ◆ CCD detector collects the X-ray signal of sample in term of energy and position
- ◆ CCD is cooled to -100°C to limit dark current
- ◆ Weighs 30 kg



XRD/XRF (CheMin)

▶ Parties involves:

- ◆ *inXitu (USA)*
- ◆ *NASA Ames Research Center (USA)*
- ◆ *Los Alamos National Laboratory (USA)*
- ◆ **Detector Advanced Development, Jet Propulsion Laboratory (USA)**
- ◆ *Indiana Univ. (USA)*

Portable XRD/XRF (Terra)

- ▶ Terra developed based on CheMin technology to provide a field deployable instrument
 - ◆ Temperature or humidity sensitive minerals can experience phase transitions during transport to the laboratory.
 - ◆ Capability to analyze these materials *in-situ* allows *determination* of native mineralogical compositions



Portable XRD/XRF (Terra)

- ▶ Architecture developed based on CheMin
 - ◆ Redesigned around a smaller CCD to save cost, mass and power.
 - CCD is cooled to -45°C using a Peltier cooler.
 - Collimation with miniature slits in place of pinhole to maximize throughput.
 - System includes an embedded computer to control the instrument, acquire and process data in real time, and offer a graphical user interface through a wireless link.
 - Li-ion batteries ~4 hrs of autonomous operation.
 - Complete instrument weights less than 15 kg

Portable XRD/XRF (Terra)

▶ Parties involves:

- ◆ InXitu (USA)
- ◆ Chesapeake Energy Corporation (USA)
- ◆ NASA Ames Research Center (USA)
- ◆ Earth & Env. Sciences, Los Alamos National Laboratory (USA)
- ◆ NASA Johnson Space Center (USA)
- ◆ TECSEN, Univ. Paul Cézanne (France)
- ◆ Dept. of Geological Sciences, Indiana Univ. (USA)
- ◆ Dept. of Geosciences, Univ. of Oslo (Norway)
- ◆ Earth and Planetary Exploration Services, Univ. of Oslo (Norway)
- ◆ Dept. of Geological Sciences and Geological Engineering, Queen's Univ. (Canada)

Instruments deployed by space agency

▶ Orbiter

◆ Infrared Spectrometry

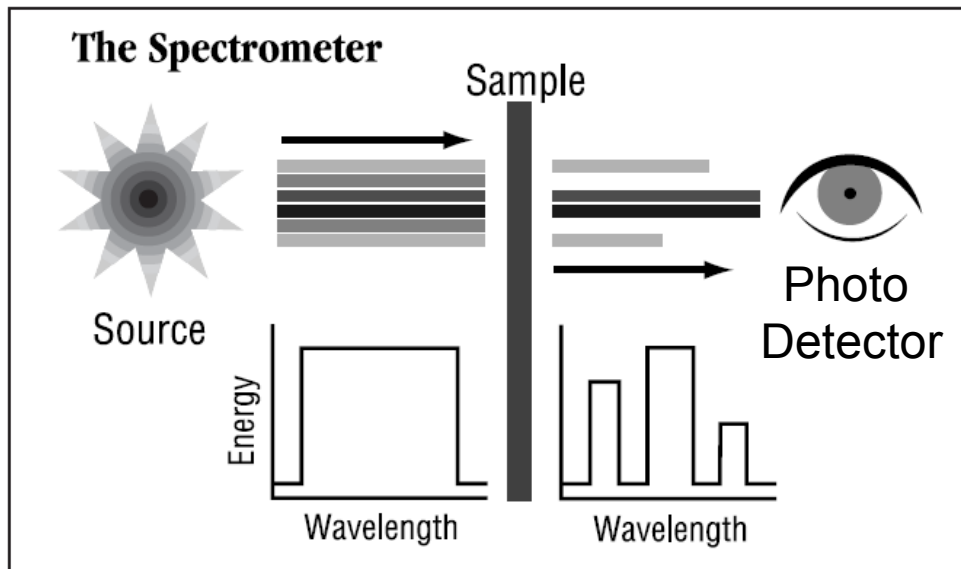


Infrared Spectrometry

▶ SPICAM-IR Atmospheric Spectrometer (ESA Mars Express Orbiter)

- ◆ Determining composition of atmosphere from wavelengths absorbed by constituent gases

▶ SPICAM-IR setup



Record amount of energy absorbed when the frequency of the infrared light is varied to form infrared spectrum

Principle of IR

- ▶ Infrared spectrum represents sample's fingerprint
 - ◆ Absorption peaks correspond to frequencies of vibrations between bonds of atoms
 - ◆ Different material has unique combination of atoms, no two compounds produce same infrared spectrum.
 - ◆ Range of this photo detector (1.0-1.7 μm)
 - Carbon dioxide (absorption at 1.43 μm and 1.57-1.6 μm bands)
 - Water vapor (absorption at 1.38 μm)

Recent Development

▶ Portable FT-IR

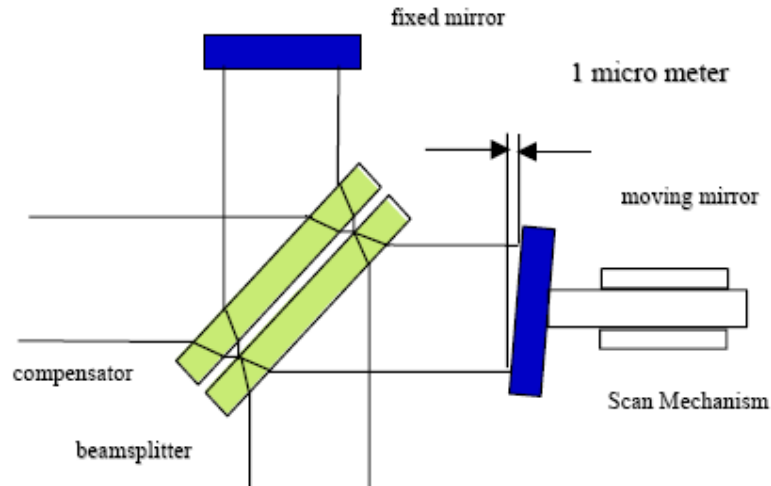
- ◆ Advantages of Portable FT-IR
- ◆ Disadvantages of Michelson interferometer
 - Solution with Rotary scanning interferometer

Advantages of Portable FT-IR

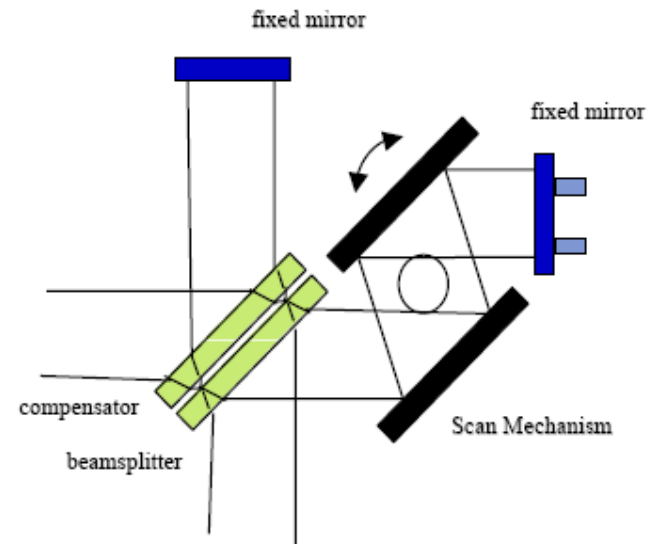
- ▶ Provide a rapid on-site spectroscopic information to identify the composition of sample
 - ◆ Faster measurement technique for collecting infrared spectra
 - IR light is guided through an [interferometer](#) (Michelson) and pass through the sample, the measured signal is interferogram
 - Performing a mathematical Fourier transform on this signal results in a spectrum
 - ◆ Portable instrument allows an analyst to take the lab to the field

Disadvantages of Michelson interferometer

Michelson interferometer



Rotary scanning interferometer



- ▶ Mirror alignment sensitive to vibration, shock, temperature, and component fatigue
 - ◆ Tilt of 1 micrometer will change interferogram by >10%
- ▶ Maintaining constant alignment is a routine and costly process

- ▶ Mirrors are fixed
- ▶ Stable performance
- ▶ Repeatable rotary scan mechanism

Portable FT-IR

► Specifications of portable FT-IR

◆ Resolution:

- From 4 cm^{-1} for routine analysis up to 0.5 cm^{-1} for high resolution work such as gas and multi-component analysis
- Higher energy near-IR ($0.8\text{-}2.5 \mu\text{m}$), wave number $\sim 14000\text{-}4000 \text{ cm}^{-1}$

◆ Dimensions: 49x39x19 cm

◆ Power consumption: 40 W

◆ Input voltage: 12 V

◆ Weight: 18 kg



Portable FT-IR

- ▶ Parties involves:
 - ◆ Tallinn Tech. Univ. (Estonia)
 - ◆ Monash Univ. (Sydney)
 - ◆ Interspectrum (Estonia)
 - ◆ Thermo Scientific (USA)
 - ◆ ABB (Germany)
 - ◆ A2 Technologies (USA)
 - ◆ Bruker AXS (Germany)

assignment

▶ Name:

- ◆ 2 types of wave that has heat effect
- ◆ 3 types of radiation that has reaction with a photographic film
- ◆ 2 types of radiation that has ionisation and kill living cell

assignment

- ▶ **Determine the 4 characteristics that all the radiation share with the 4 hints given:**
 - **Speed of all the radiation?**
 - **What do all the radiation carry from place to place?**
 - **Could all the radiations travel from the Sun to Venus? Is there a need for a medium to enable travelling?**
 - **Is all the radiation longitudinal or transverse type of wave?**

References

- ▶ http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20050205037_2005206851.pdf
- ▶ http://www.esa.int/SPECIALS/Mars_Express/SEMUC75V9ED_0.html
- ▶ http://en.wikipedia.org/wiki/Viking_1
- ▶ http://www.unsolvedmysteries.oregonstate.edu/MS_05
- ▶ http://en.wikipedia.org/wiki/Mass_spectrometry#Gas_chromatography
- ▶ <http://cmapsnasacmex.ihmc.us>
- ▶ <http://www.daviddarling.info/encyclopedia/V/VikingGCMS>
- ▶ <http://marstech.jpl.nasa.gov/content/detail.cfm?Sect=MTP&Cat=base&subCat=SSA&subSubCat=&TaskID=2256>
- ▶ <http://www.innovx.com/themes/IX/graphics/xrfspectrometry/spectrometry.html>

References

- ▶ http://serc.carleton.edu/research_education/geochemsheets/techniques/XRF.html
- ▶ <http://en.wikipedia.org/wiki/APXS>
- ▶ http://mpfwww.jpl.nasa.gov/MPF/mpf/sci_desc.html
- ▶ <http://www.nasaimages.org/luna/servlet/view/all/what/Alpha+Proton+X-ray+Spectrometer>
- ▶ <http://www.physics.pdx.edu/~pmoeck/phy381/Topic5a-XRD.pdf>
- ▶ <http://www.wwnorton.com/college/chemistry/gilbert/tutorials/ch10.htm>
- ▶ http://en.wikipedia.org/wiki/Scintillation_counter
- ▶ <http://en.wikipedia.org/wiki/Photomultiplier>
- ▶ http://www.mcswiggen.com/FAQs/FAQ_EF-6.htm
- ▶ http://en.wikipedia.org/wiki/Mars_Science_Laboratory

References

- ▶ P. Sarrazin, et. al., International Centre for Diffraction Data, Advances in X-ray Analysis, Volume 48 (2005)
- ▶ P. Sarrazin et. al., 39th Lunar and Planetary Science Conference (2008)
- ▶ S. M. Chemtob et. al., 40th Lunar and Planetary Science Conference (2009)
- ▶ http://en.wikipedia.org/wiki/Infrared_spectroscopy
- ▶ http://www.wooster.edu/chemistry/is/brubaker/ir/ir_works_modern.html
- ▶ T. Tonnisson, IEEE Electronics Conference, 2008
- ▶ <http://www.thermo.com/>
- ▶ <http://www.abb.com/>
- ▶ <http://www.a2technologies.com/>
- ▶ <http://www.bruker-axs.de/>

THE END