

Presentation to the MCERTS 2007 Conference, Bretby, England.

FTIR MONITORING OF PROCESS EMISSIONS – OVERCOMING THE UNIQUE PROBLEMS OF THE CHEMICAL INDUSTRY

**Steve Hall
Protea Limited
First Avenue, Crewe, Cheshire, CW1 6BG
Tel:01270 256256, mail@protea.ltd.uk**

OUTLINE

Typical Chemical Process

Introduction to the problems of the chemical industry

Including:

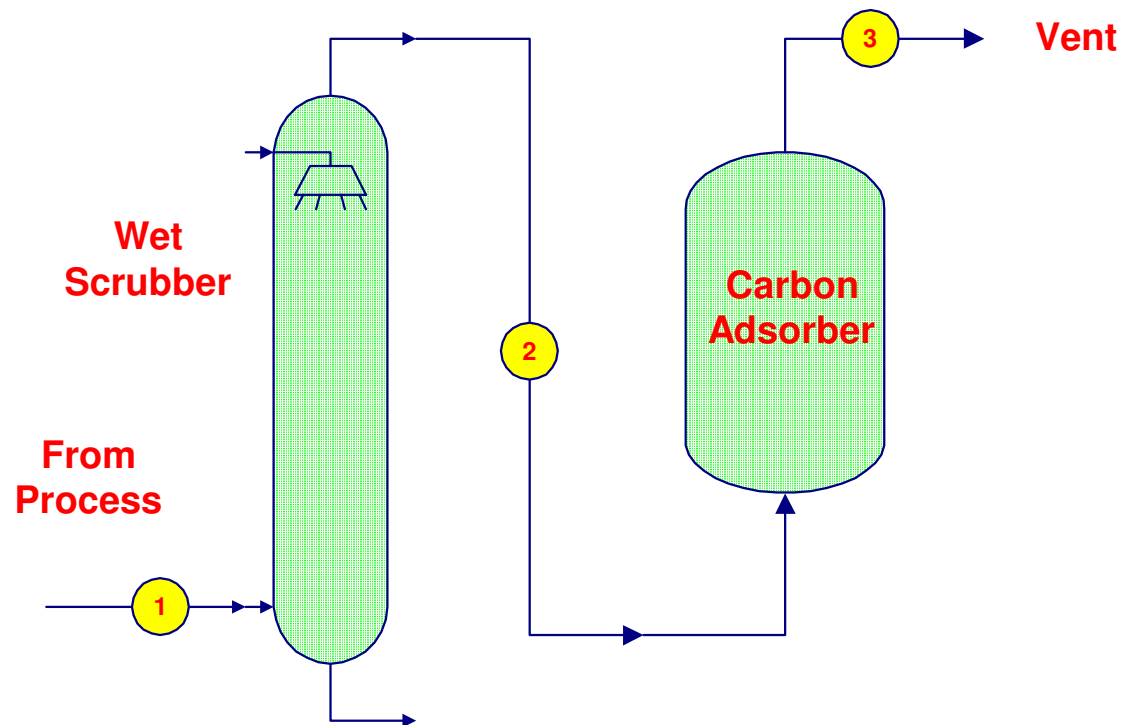
FTIR technology

The ASTM D6348 method

Parts of an FTIR analyser

Conclusions and closing comments

PART OF TYPICAL CHEMICAL PROCESS:



TYPICAL PROBLEMS:

Complex solvent mixtures

Unknown components

Speed of a monitored event

Rapidly changing concentrations

Moisture

Concentration ranges

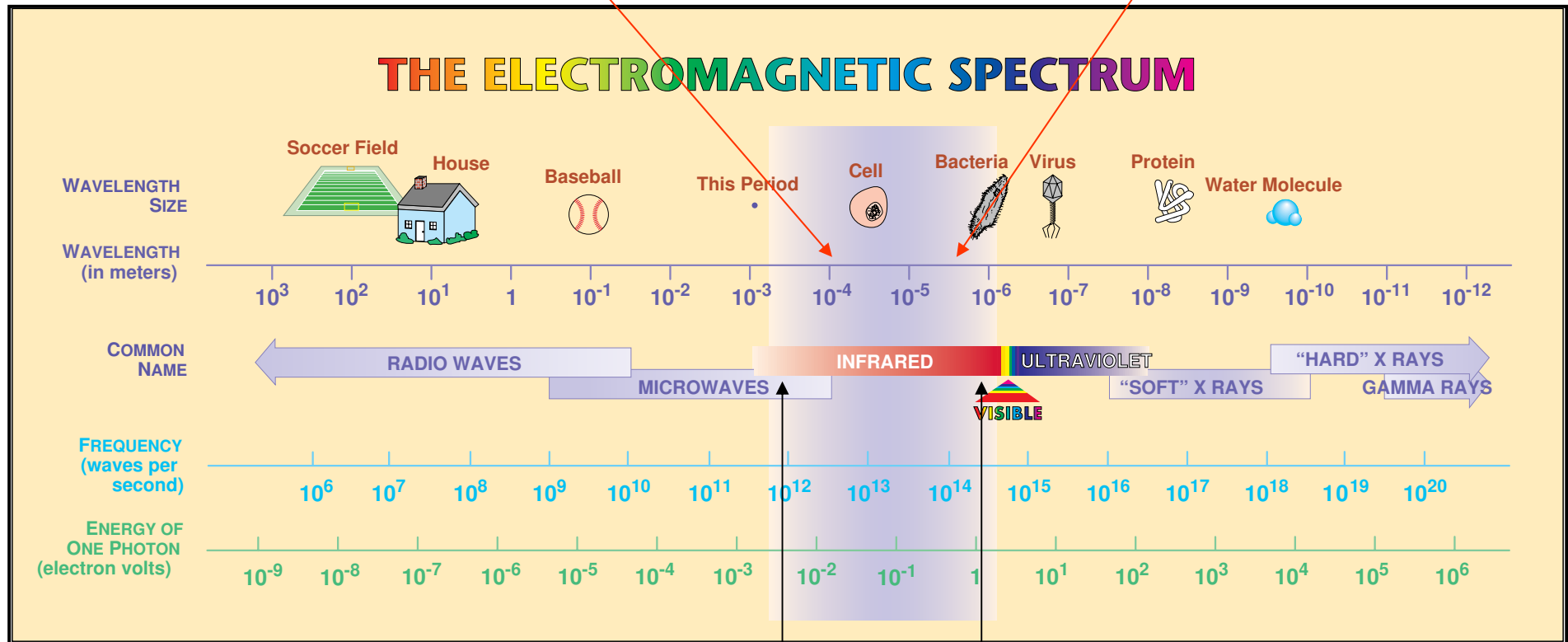
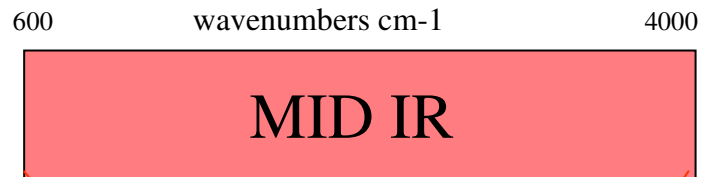
Intrinsic safety

Extended periods of monitoring

Complex solvent mixtures and Unknown components

FTIR is a great technique for solvents...

Mid IR detects
chemical bonds

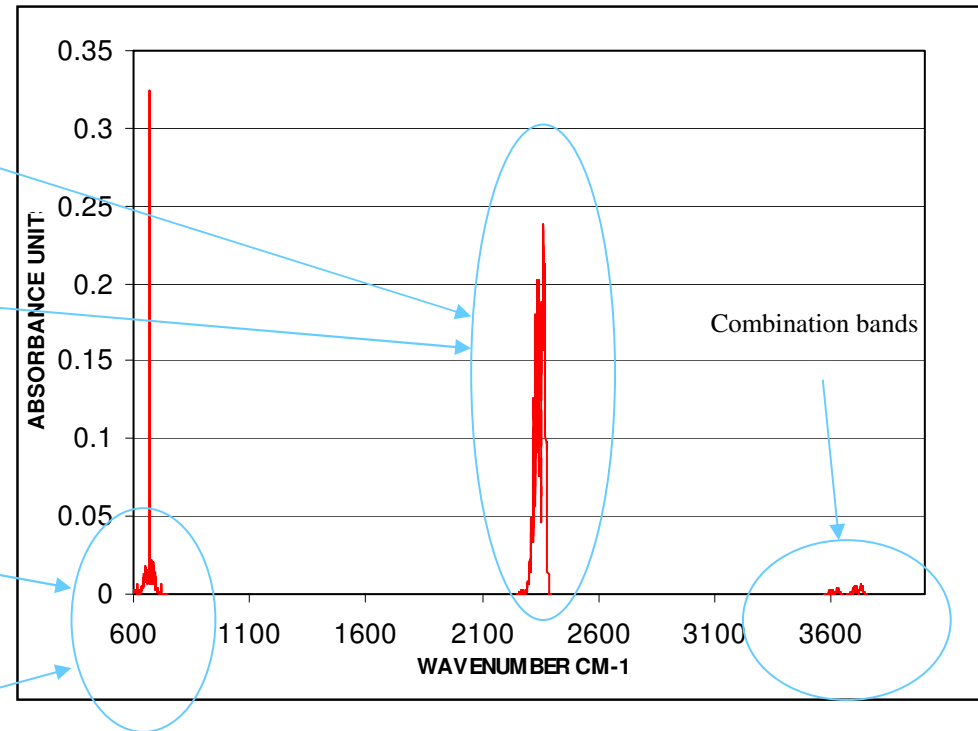
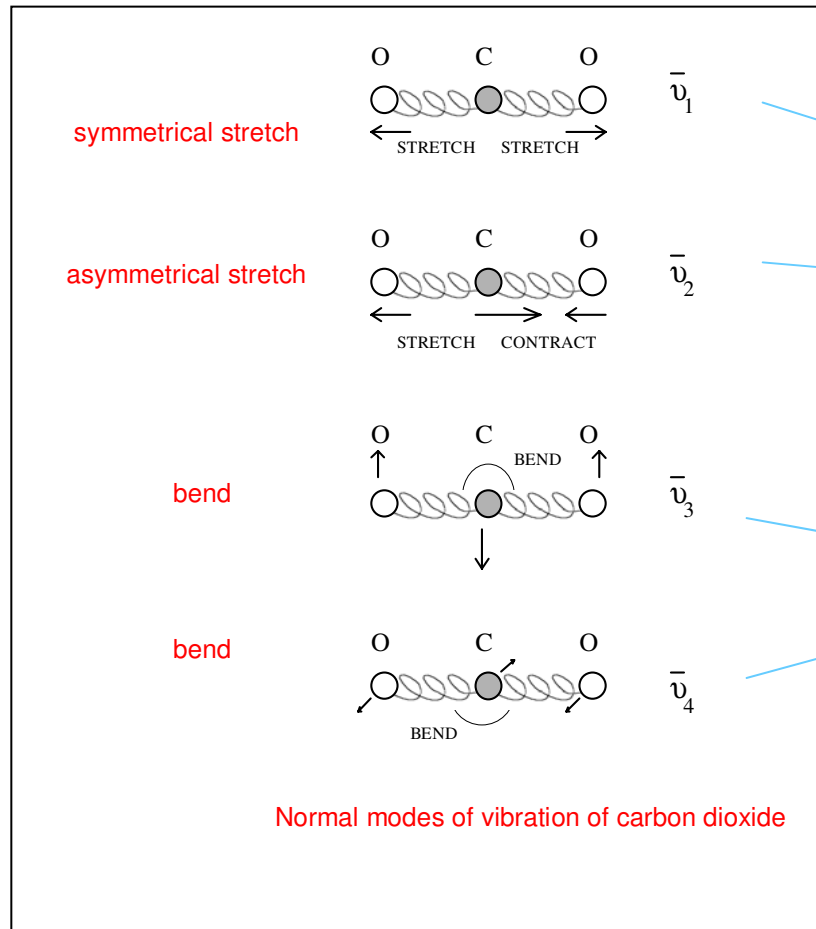


IR units: wavenumbers (cm^{-1})
 10^{-5} metres wavelength = 1000 cm^{-1}

Near-IR: $4000 - 14000 \text{ cm}^{-1}$

Far-IR: $5 - 500 \text{ cm}^{-1}$

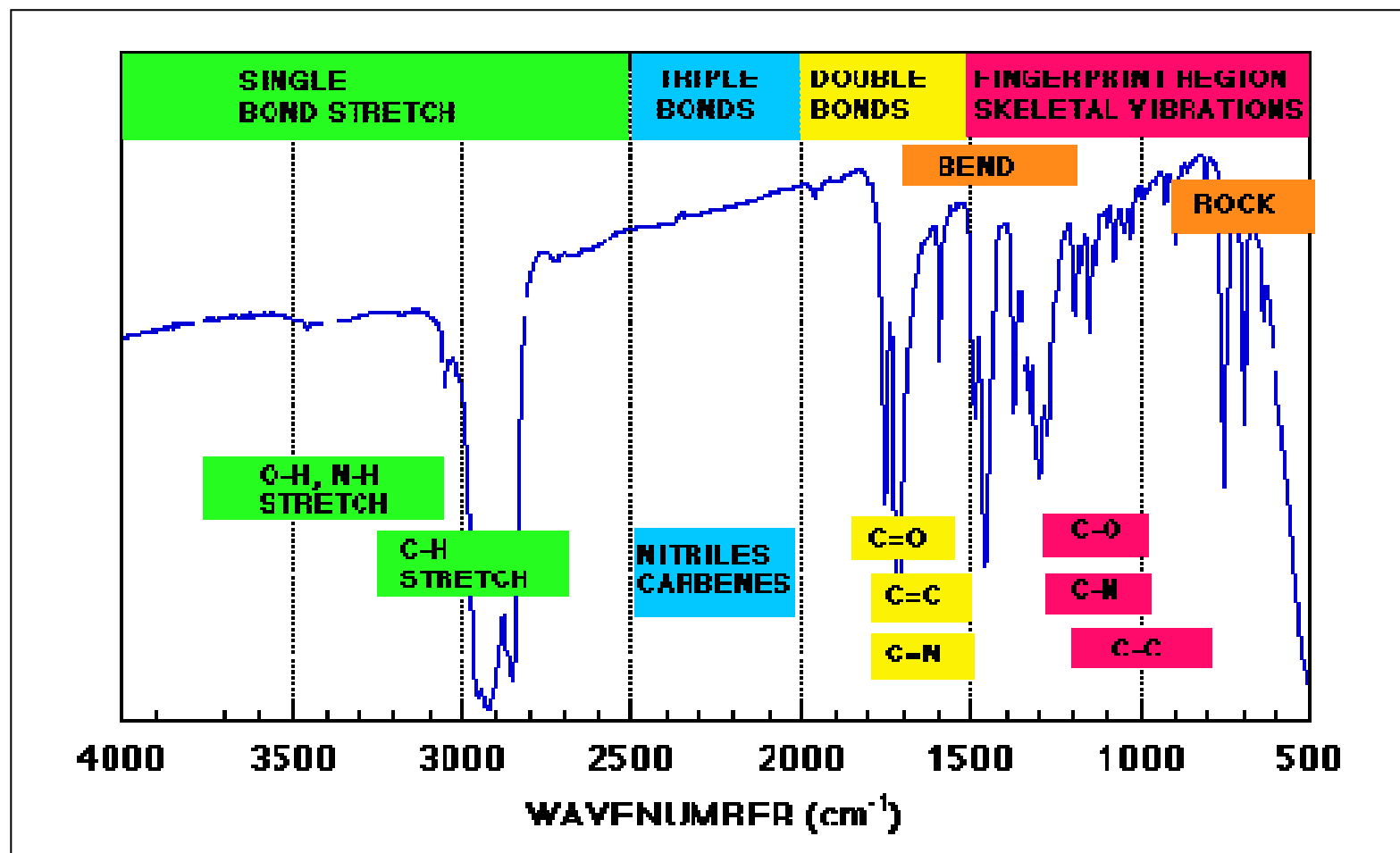
- Molecules have natural vibration frequencies as bonds vibrate
- IR frequencies matching these are absorbed



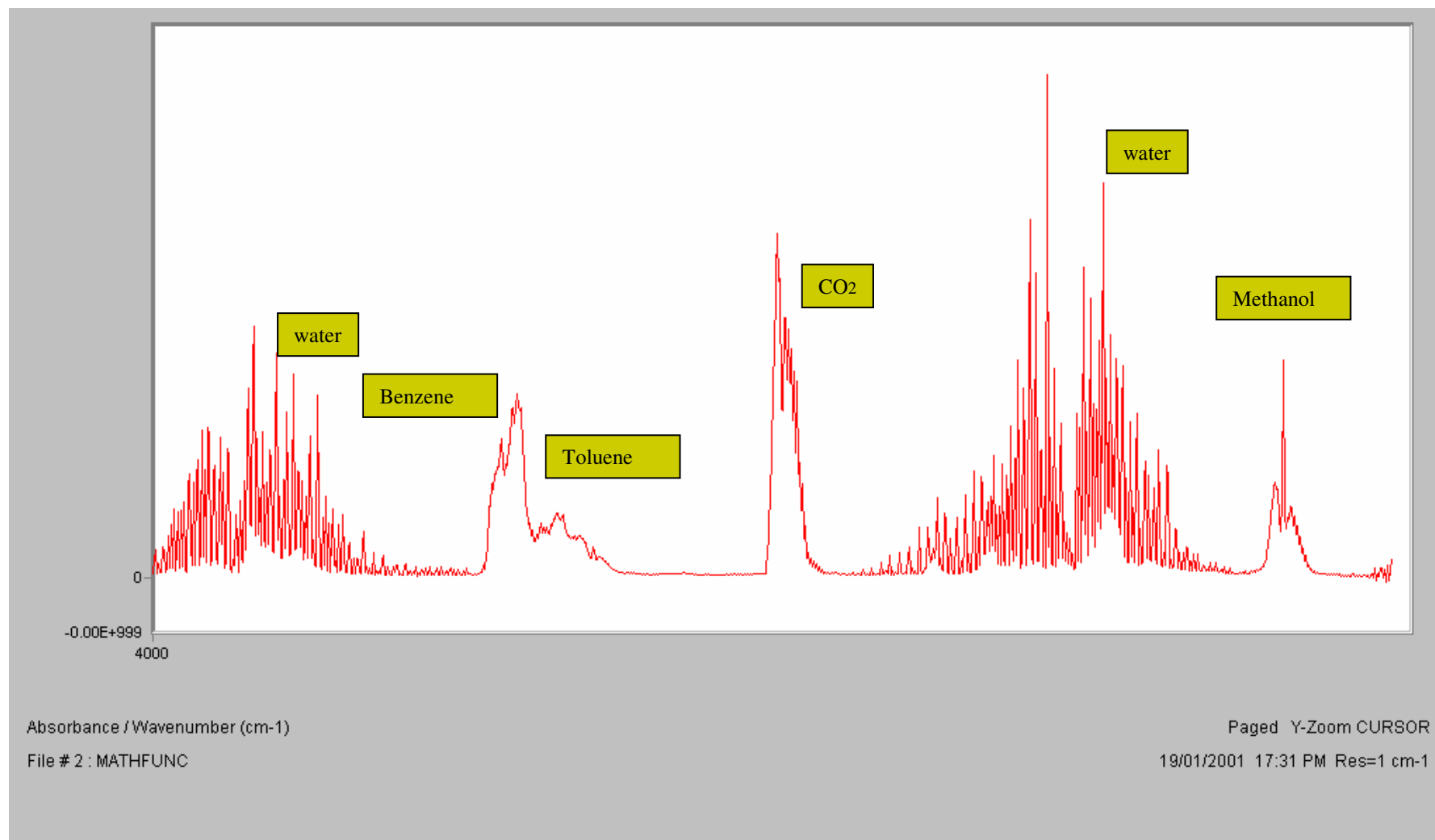
CO₂ Spectrum

Different bonds give
different absorption frequencies

**ABSORBANCES CHARACTERISTIC OF BOND TYPE
CAN BE USED FOR SIMPLE IDENTIFICATION
SMALL VARIATIONS DEPEND ON 'ENVIRONMENT' SURROUNDING BOND**



IR SPECTRUM



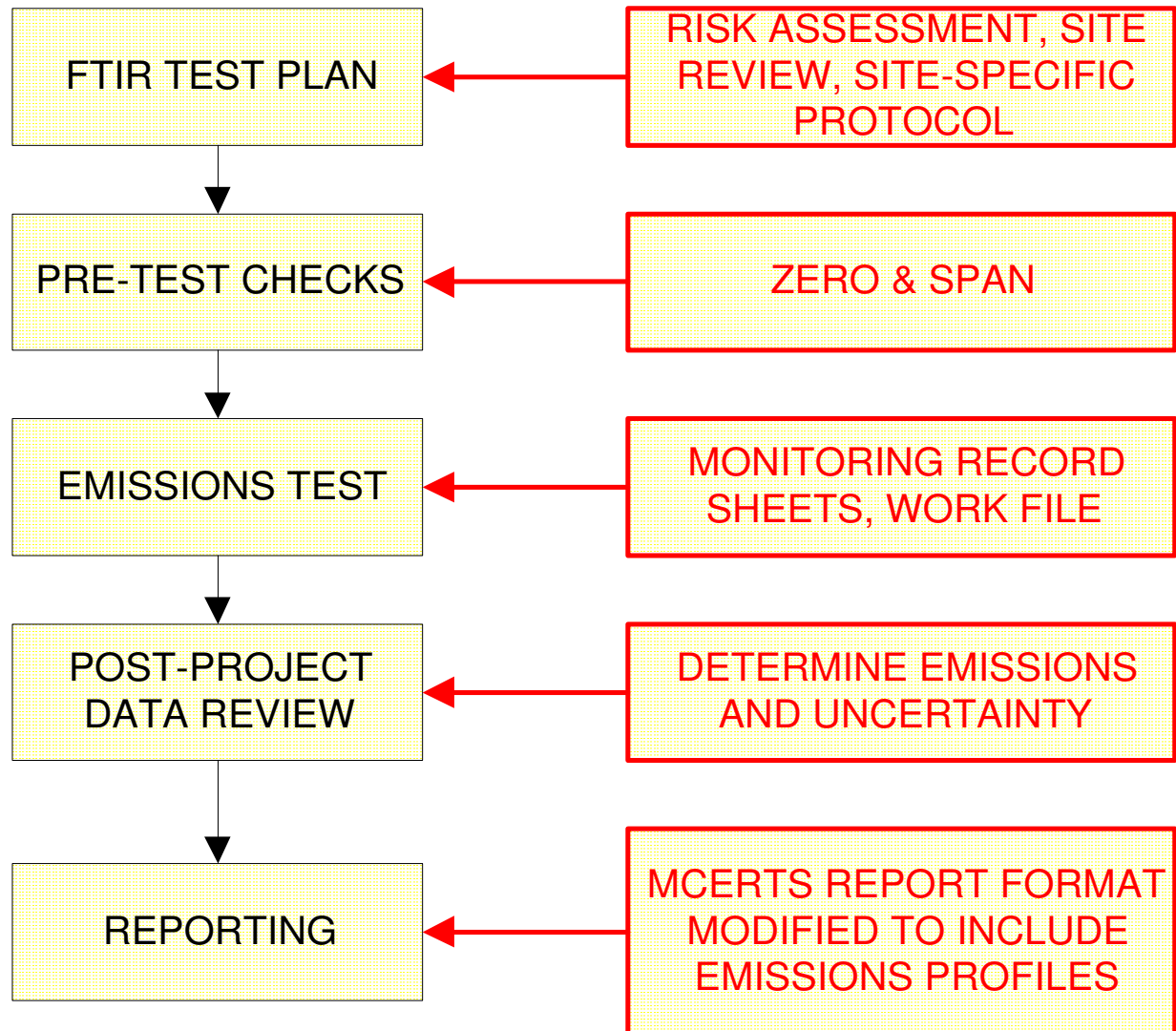
- Concentration proportional to height (generally)

THE ASTM D6348 METHOD

- A quick review

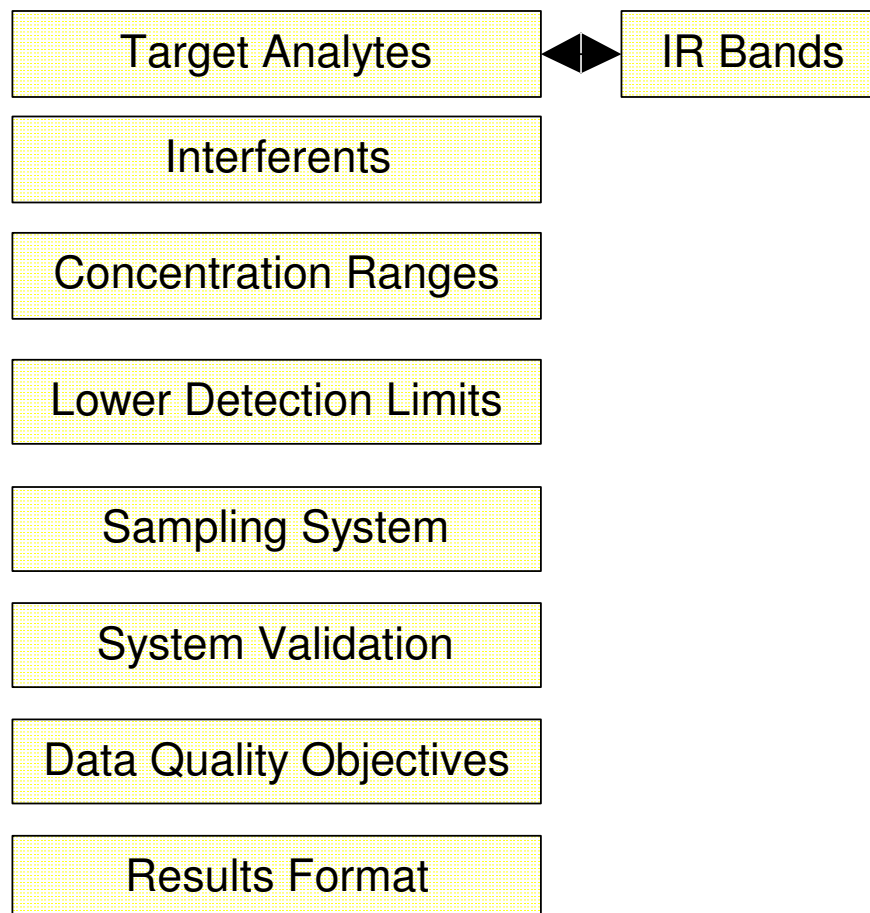
ASTM METHOD

MCERTS

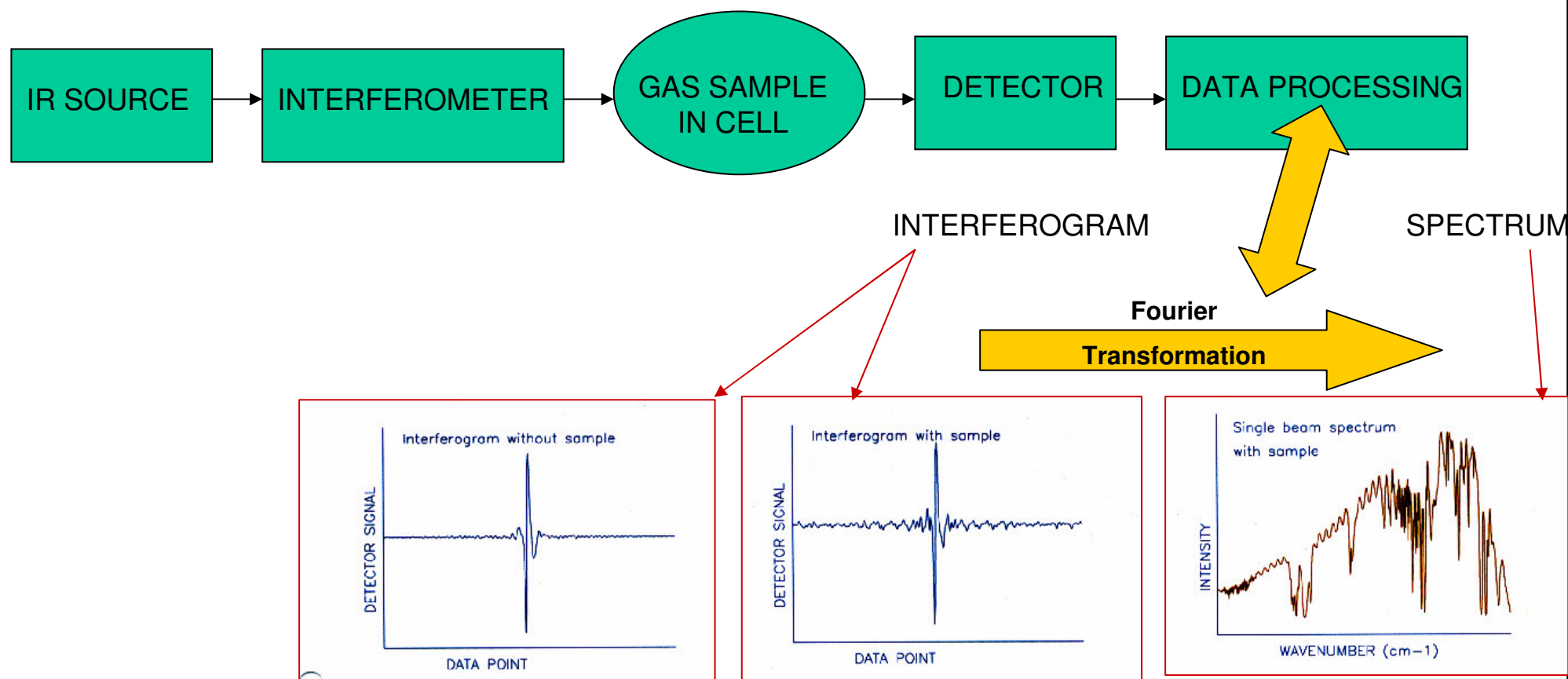


FTIR TEST PLAN:

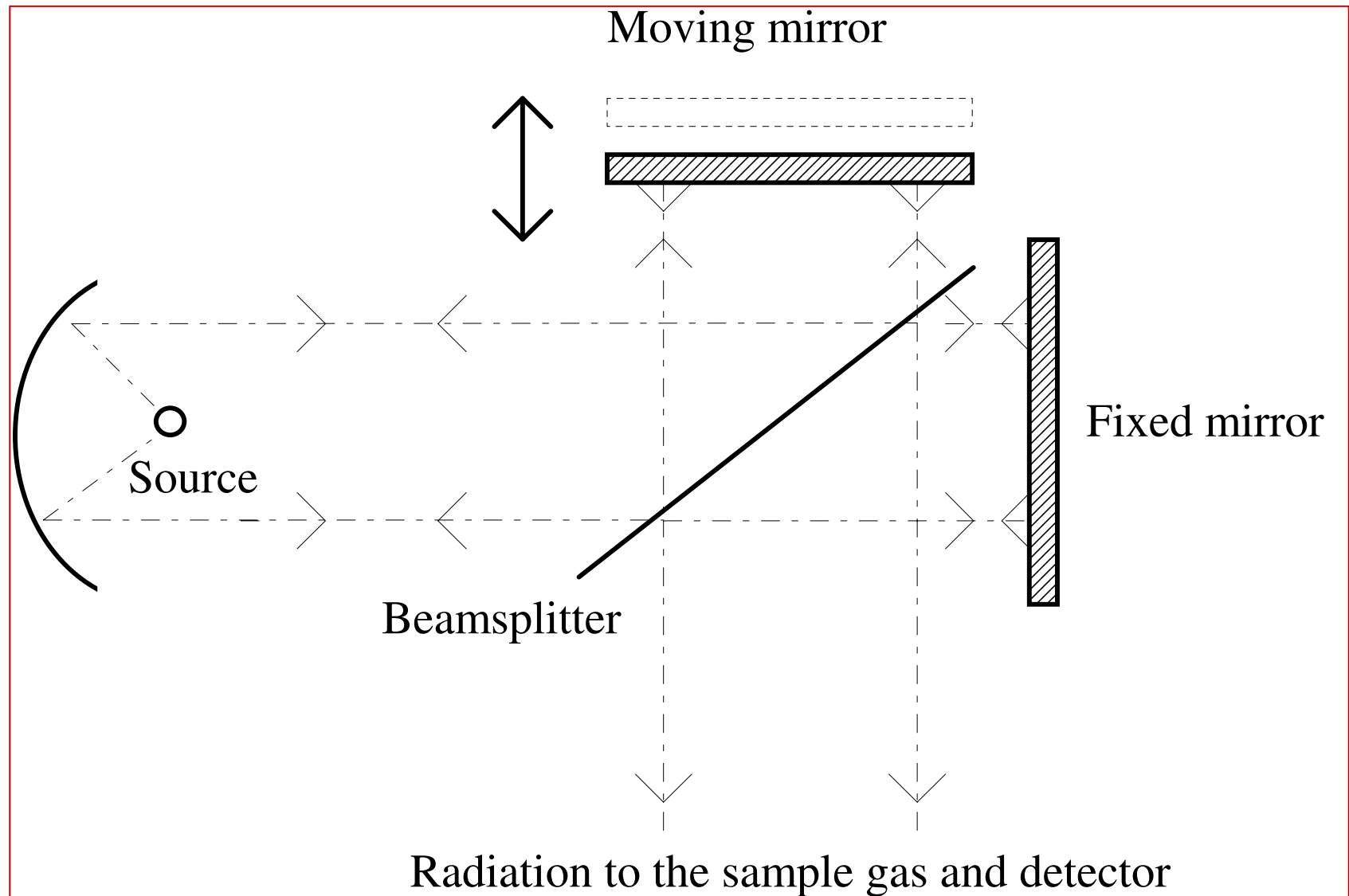
FTIR TEST PLAN



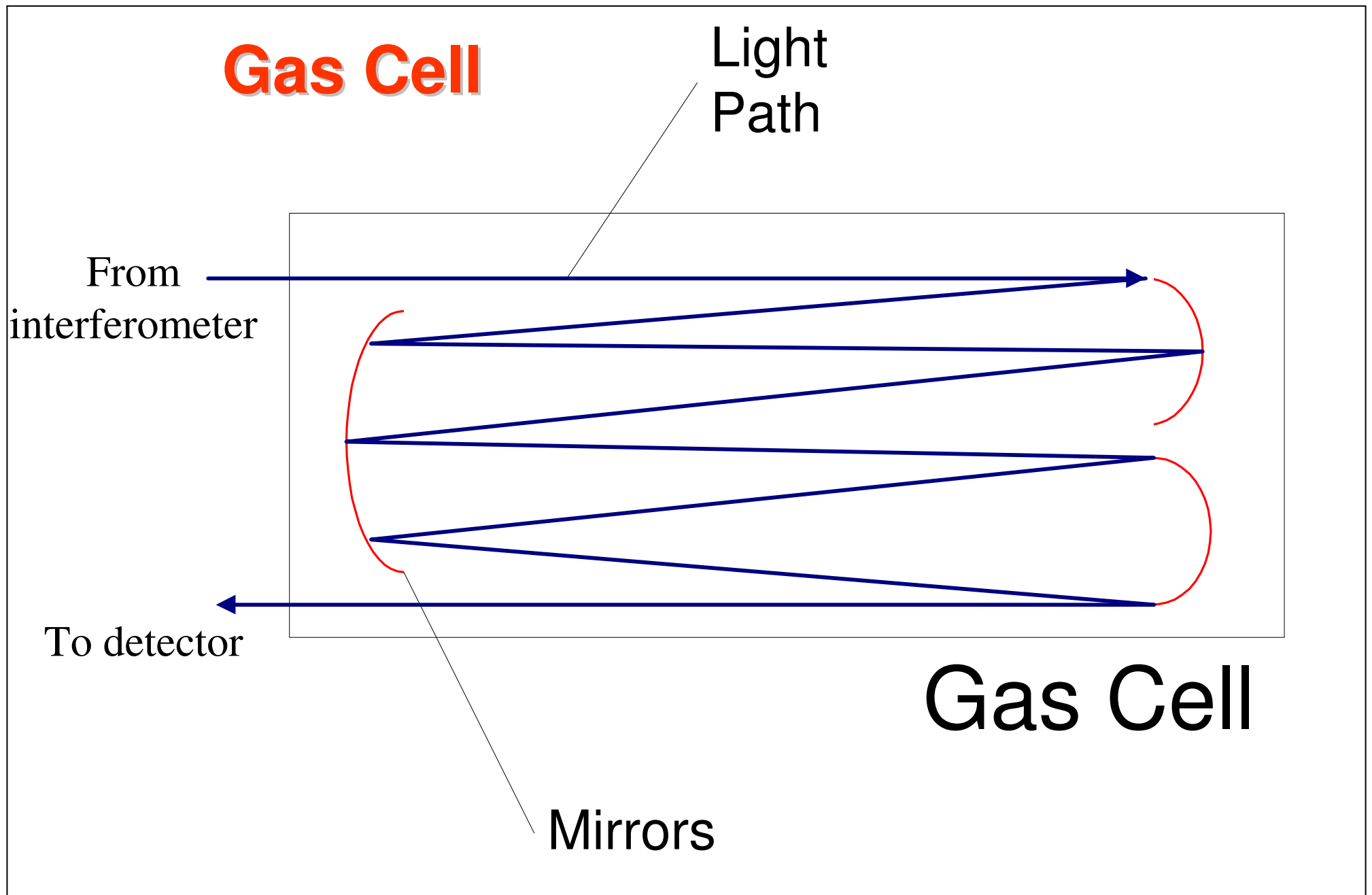
COMPONENTS OF FTIR SPECTROMETER



Michelson Interferometer



FTIR Analyser

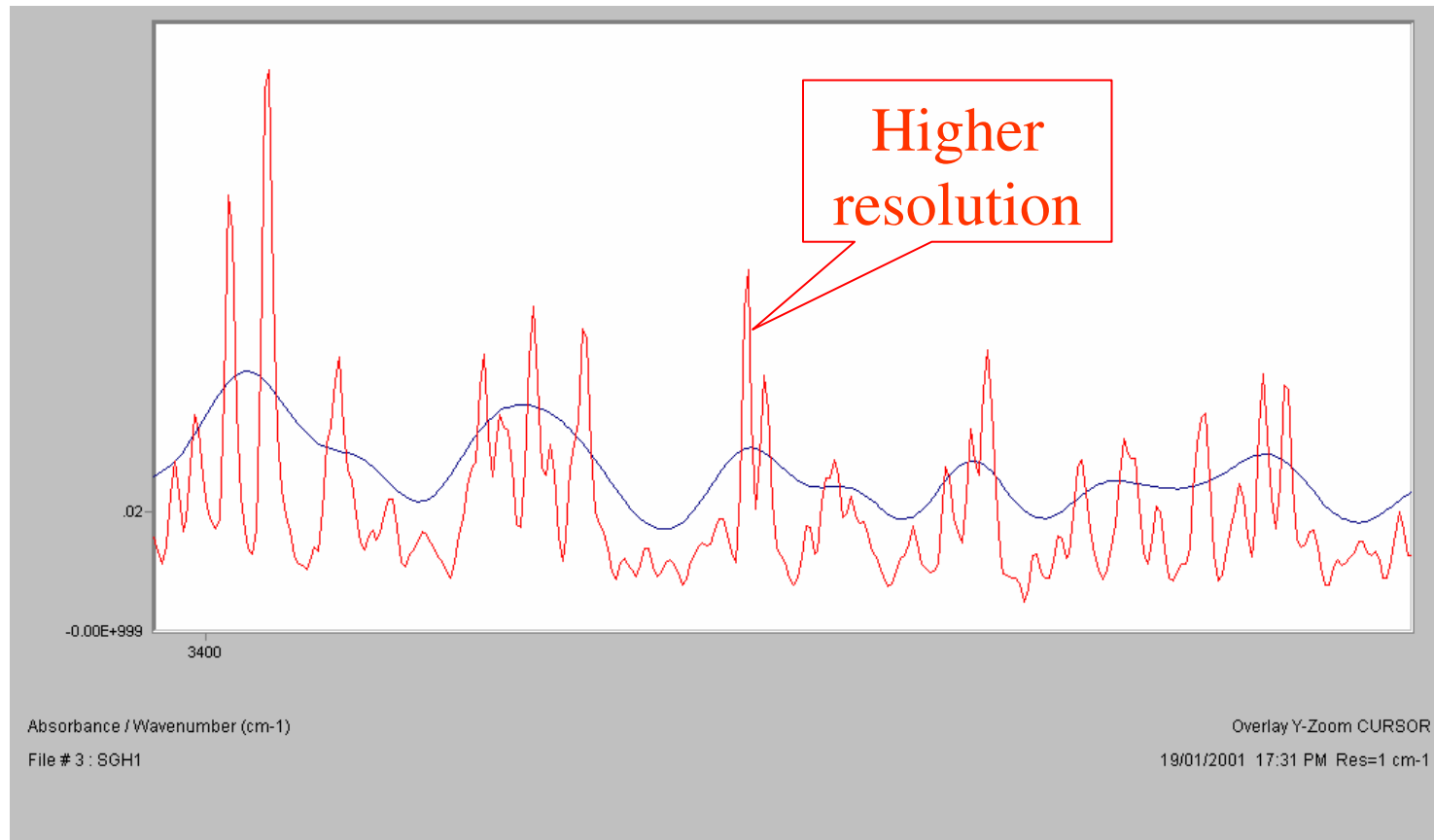


UNKNOWN COMPONENTS

- Sample FTIR spectra are saved during a project and can be checked later
- ‘Chemometric’ techniques can be used to identify and quantify unknown components using library spectra

UNKNOWN COMPONENTS

- May need to vary instrument resolution for complex mixtures to obtain fine spectral details



SPEED OF AN EVENT

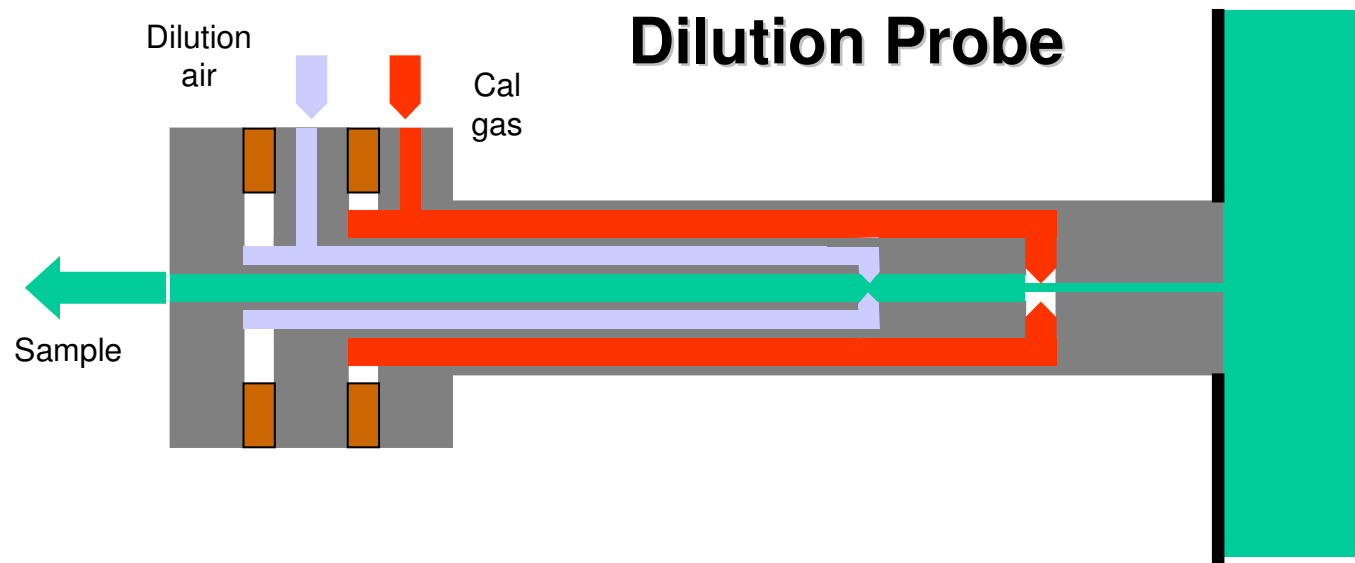
- **Certain process events can happen very quickly eg vessel venting**
- **Sample spectrum may be an average of many spectral scans**
- **Change scan number of interferometer but watch out for signal to noise (fewer scans means more noise)**

Rapidly Changing Concentrations

- May need to alter size of gas cell (mixing volume)
(if possible)

Moisture

- Ensure sample system and gas cell is heated
- Use dilution probe



Concentration Ranges

- High Concentrations
 - Reduce light path length in gas cell
 - Use dilution probe
- Low concentrations
 - Increase path length
 - Possibly increase resolution

Intrinsic Safety

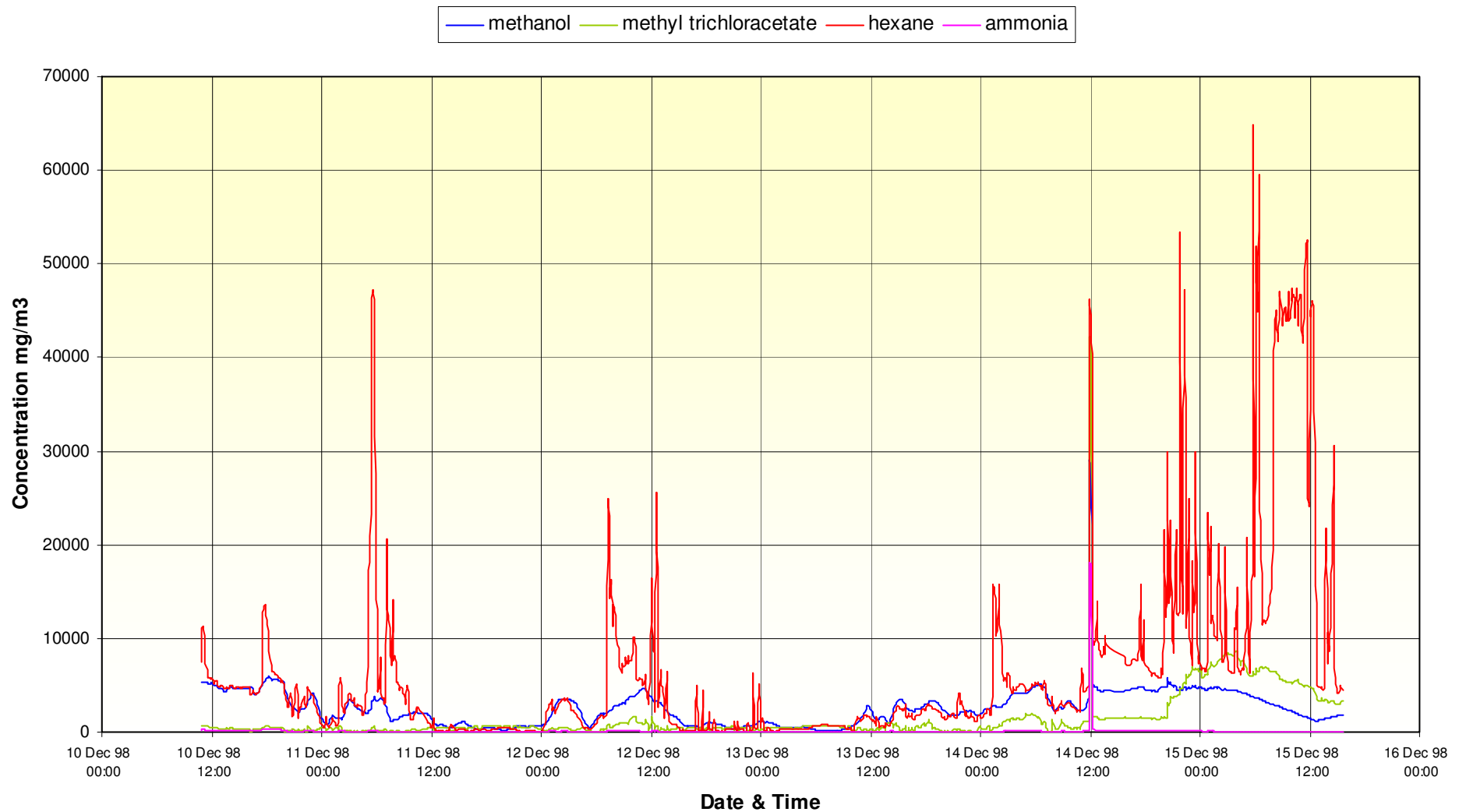
- Heated lines may not be possible
- Possibly use cold lines but check sampling system is fit for purpose
- Use dilution probe but watch out for lower detection limit

Extended Periods of Monitoring

- **Regular zeros and spans**
- **Regular system purges**
- **Regular leak checks**

Process Profiling - concentration profile

Concentration Profile



CONCLUSIONS

- **Infra red monitoring techniques are very powerful and there are many system parameters which can be changed to ensure good quality results.**
- **Selecting the right parameters may require some practical expertise**

FTIR Working Party

- **Developing best practice in FTIR use**
- **Focusing on the needs of the monitoring industry**
- **Looking forward to develop a MID document and maybe more**