FTIR for GreenTech Applications

Dr. Karolina Haupa

What is GreenTech?

Definition: Technology and innovations aimed at reducing environmental impact.

Focus: Promoting sustainability and enhancing resource efficiency.

Key Areas:

- Renewable energy (solar, wind, hydrogen)
- Energy storage
- Carbon capture
- Environmental monitoring
- Heat management
- Sustainable agriculture

Objective: Combat climate change, pollution, and resource depletion.

FTIR in GreenTech

Heat management

Catalysis

Battery research

Microplastics

Energy storage

Renewable fuels

Fuel cells

Green House Gas (GHG) monitoring

Sustainable agriculture

SpectroElectroChemistry

Electrochemistry: studies the relationship between electricity and chemical change

IR spectroscopy: gives a characteristic spectrum for each substance (except for homonuclear diatomic gases and pure metals)

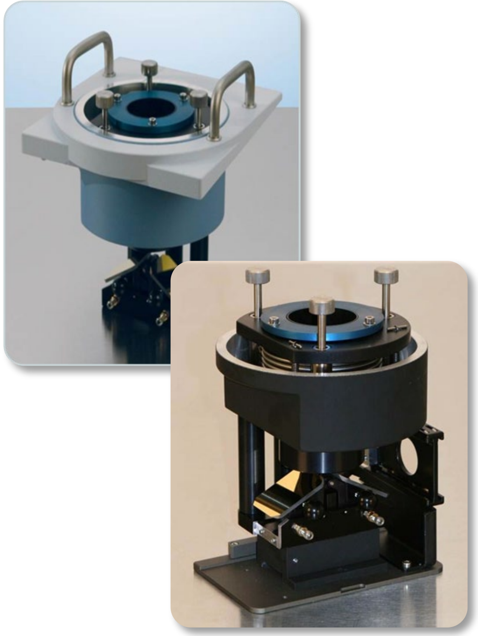
IR SpectroElectroChemistry:
offers insights into reaction mechanisms and may permit the study of intermediate/short-lived species.

IR SpectroElectroChemistry can be applied for investigations of the electrolytes, solutes, and/or the electrode surfaces; and is useful for different studies:

- Electrochemical reversible reactions
- Oxidation processes
- Electrosynthesis
- Reaction of Biomolecules

Typical instrumental Setup for FT-IR Spectroelectrochemistry

FT-IR reflection accessory for electrochemical investigations combined with FT-IR spectrometer:



INVENIO –Purged spectrometer

VERTEX - Vacuum spectrometer, removes atmospheric disturbance (better sensitivity) and enables quantification of CO_2 and H_2O

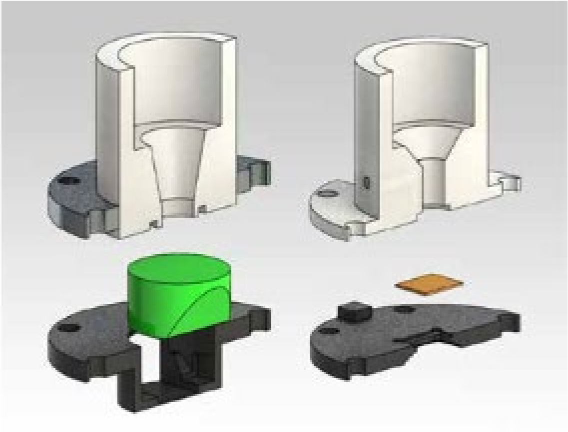
- Different techniques can be realized, **ATR and reflection measurements at electrode surfaces** through a thin electrolyte layer (<50 µm)
- **Time resolution up to ms** range to follow fast process and enabling kinetic studies

[Click here to watch an Application Video on this topic]

FT-IR Spectroelectrochemistry: ATR cells (surface enhanced)

PIKE Jackfish SEC cell in combination with Veemax III (purged version)

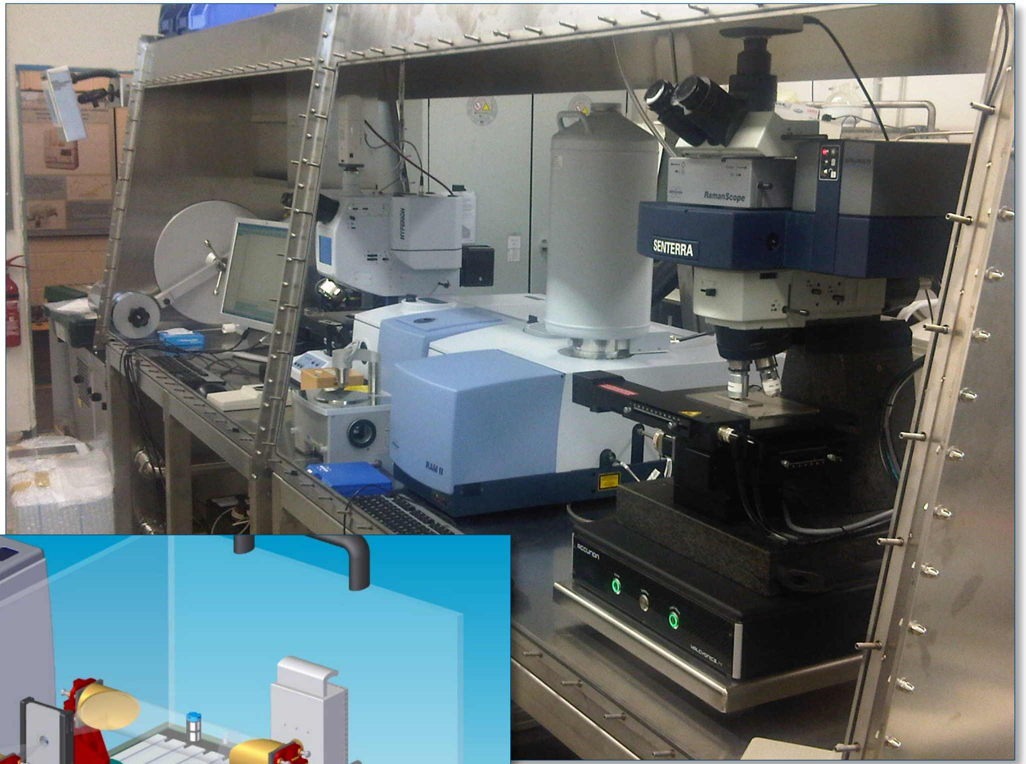
- designed for surface-sensitive electrochemical ATR-SEIRAS
- Easy removal of the crystal for metal layer deposition, the metal layer acts as working electrode
- Crystal materials: Si, Ge, ZnSe, ZnS. ATR face-angled crystal with 60° , alternatively, Si wafers
- PTFE or PEEK holders
- Ready to use cell with different cell and electrode options

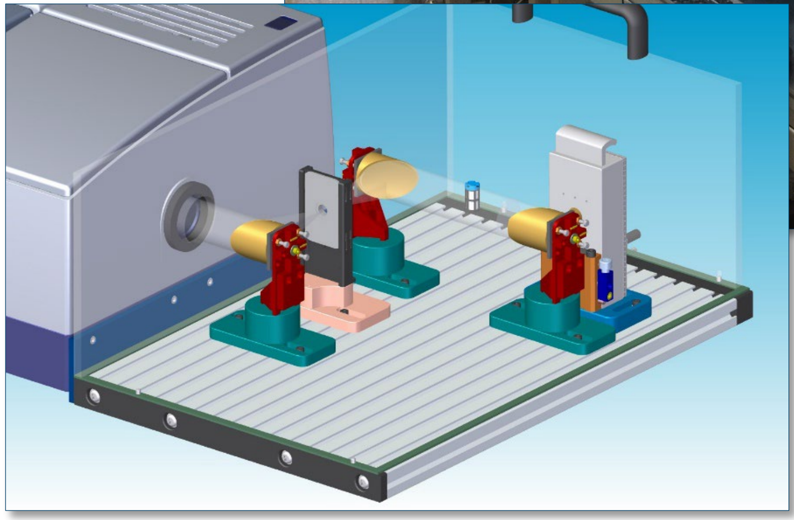


Instrumental setup for battery research in R&D lab

Two options for analyzing air-sensitive components and process:

1. An external configurable sampling platform can be placed into a glove box, with the spectrometer outside
   - ✓ A viewport on the side of the glove box is required.
   - ✓ Compatible with all kinds of sampling accessories.
2. The whole FT-IR working station with FT-Raman, IR and Raman microscopes, and in-situ spectroelectrochemistry accessory can be placed in a glove box





Application example: Li battery research

Battery research:

Li-AQ (anthraquinone) battery, ATR, test cell

Inert pouch/coin cell design, protected from the atmosphere.

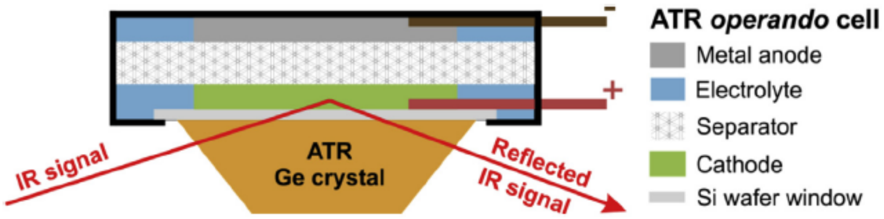
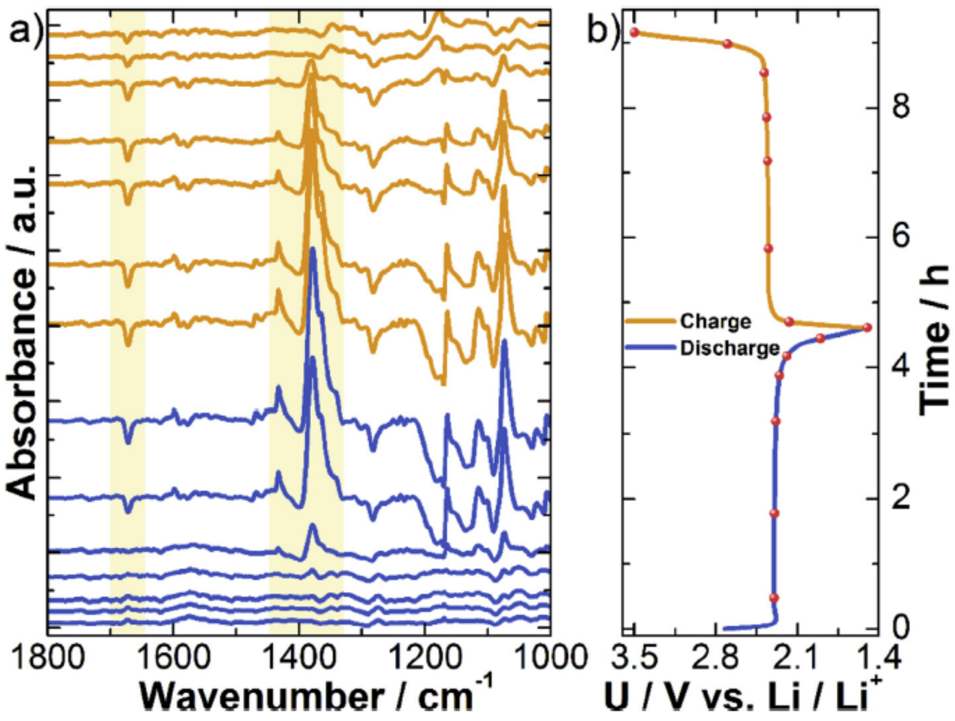


Fig. 1. Schematic representation of ATR-IR pouch cell on Ge ATR crystal.



Operando ATR-IR characterization of Li-AQ (anthraquinone) battery during first cycle.

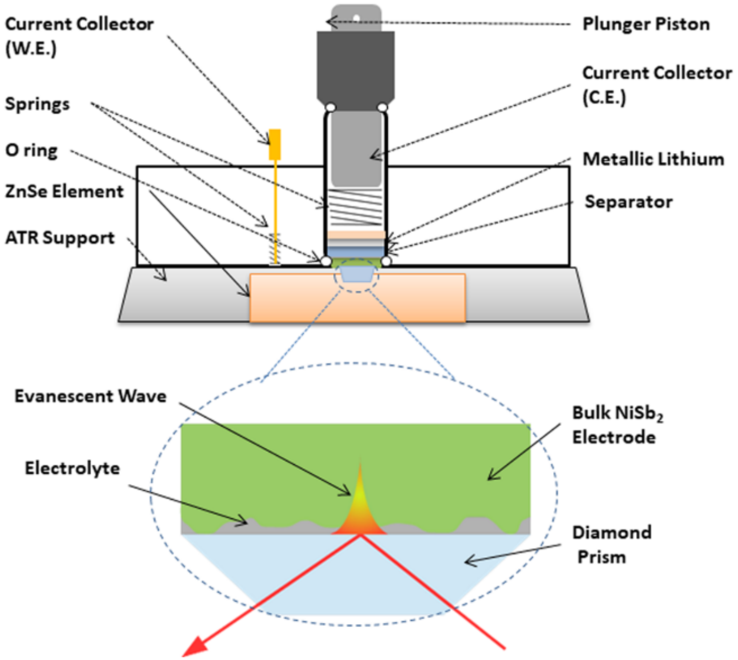
J. Bitenc et al. Energy Storage Materials 21 (2019) 347–353

Application example: Li battery research

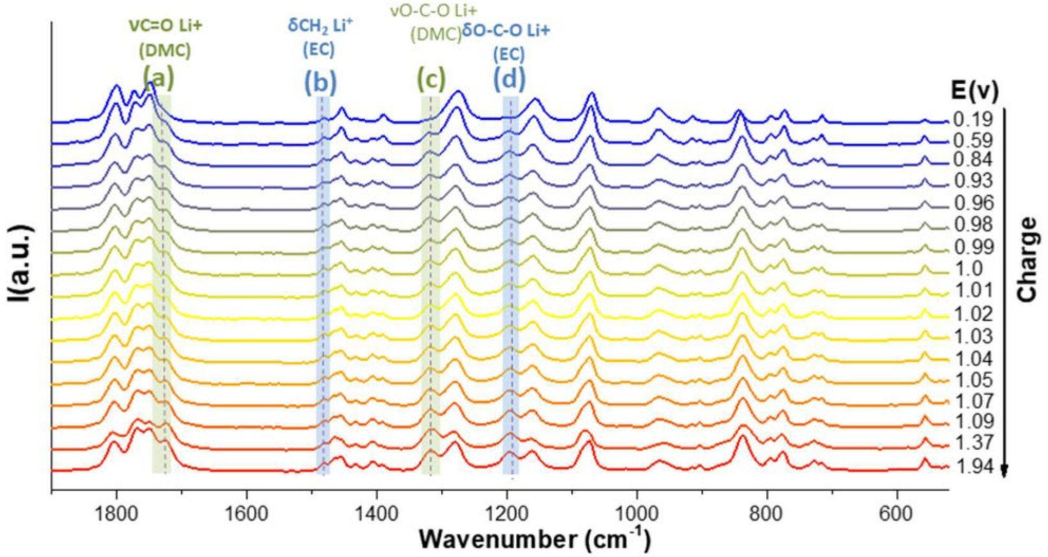
Battery research:

$NiSb_2$ electrode, ATR, test cell

In-situ cell, protected from the atmosphere (metallic lithium).



Scheme of the *in-situ* electrochemical cell used for the operando ATR-FTIR

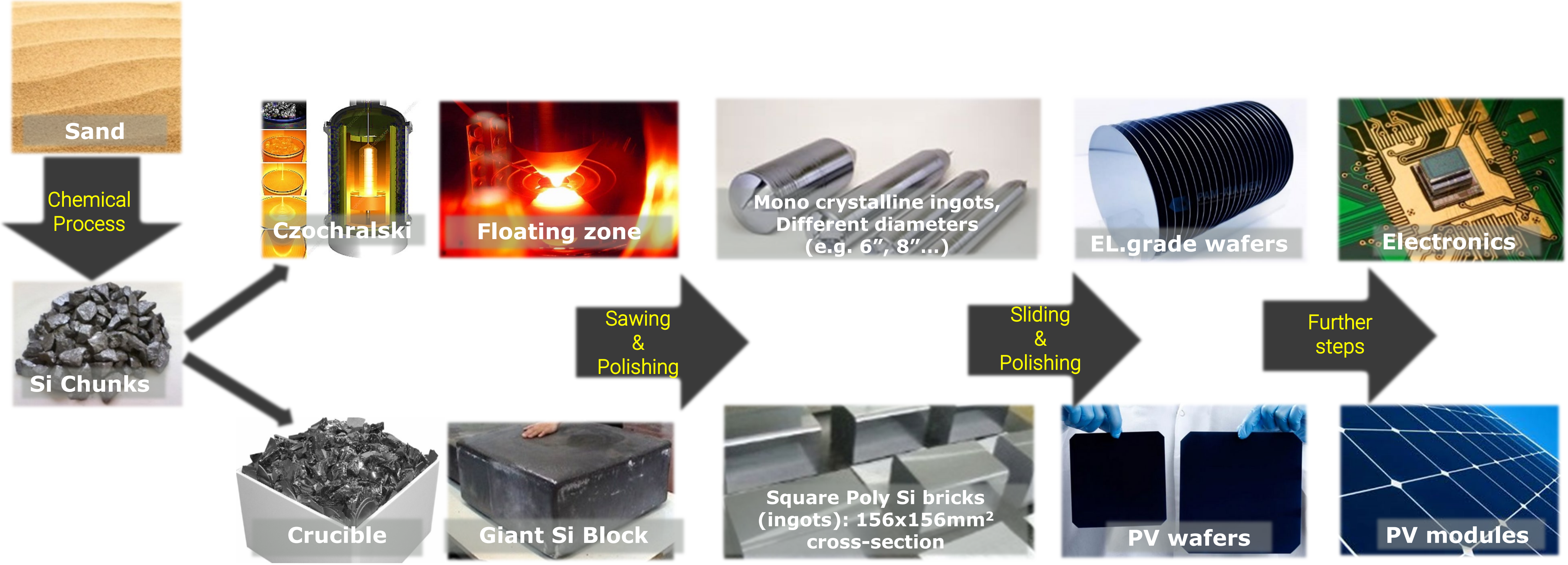


1st charge of $NiSb_2$ electrode at 1C rate with 1 M LiPF 6 in EC:DMC (1:1)

Observation of the Li^+ solvation

L. Monconduit et al., *J. Phys. Chem. C* 2017, 121, 48, 26598–26606

From sand to PV modules…



Semiconductors & Solar Panels: Si QC & Epilayer analysis

- Ultrathin layers with thickness in nm scale
- Half-quantification of chemical residuals after different bake/expose process.
- Non-expose, Expose, PAB, PEB, HB.

- Epilayer(s) with thickness in micron scale
- Single- or Multi-layer
- Mapping on wafer

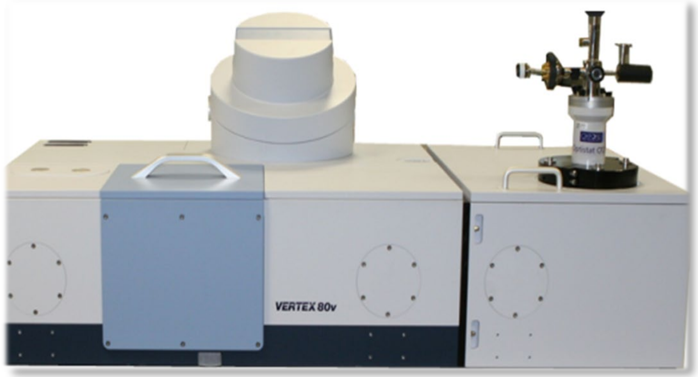
- Substitutional Carbon
- Interstitial Oxygen
- Detection limits @ several 100ppba
- Single- or Poly-crystals
- ASTM/SEMI MF1391 (Carbon)
- ASTM/SEMI MF1188 (Oxygen)

- Ultrathin layers with thickness in nm scale
- SiN, BPSG, PSG, etc.
- Quantification of B, P and -H

Semiconductors & Solar Panels: Photoluminescence

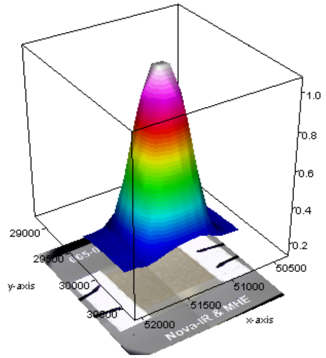
NIR & MIR PL studies (with amplitude-modulated step scan)

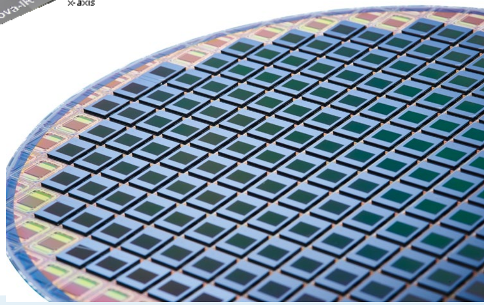
- Room temperature & low temperature



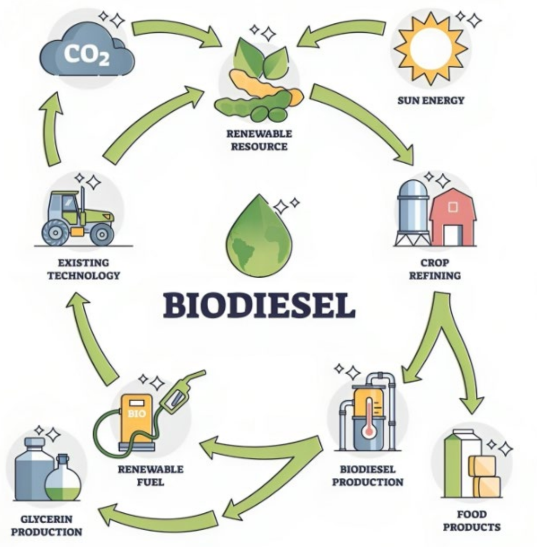
Micro photoluminescence emission with Hyperion emission option

Click here to watch an Application Video on this topic





Re-Fuels, Bio-Fuels, Bio-diesel, SAF…



- Methods: Transmission or ATR, liquid gas, TGA-FTIR…
- Focus: qualitive and quantitive
- Many norms: e.g., EN14078
- Sometimes more exotic composition
- MIR / NIR

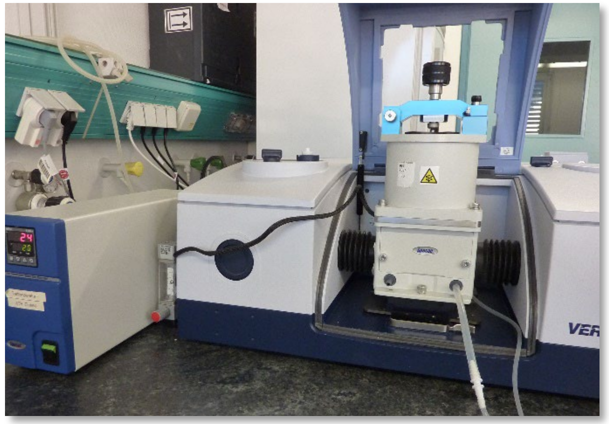
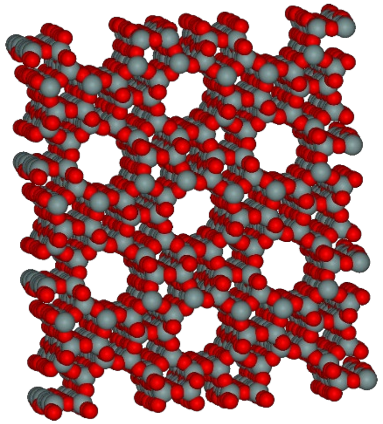


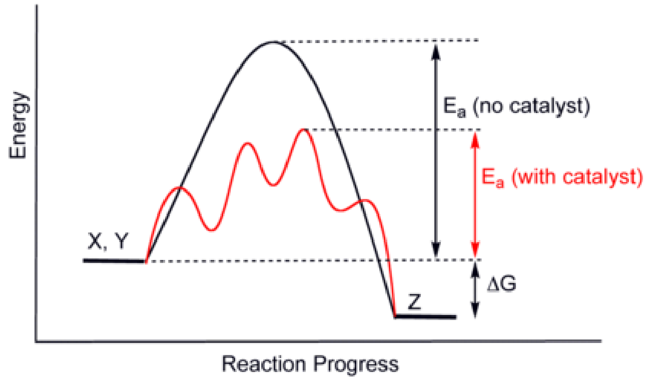
Fig. 2 Pearl™ Horizontal Type Liquid FTIR Transmission Accessory

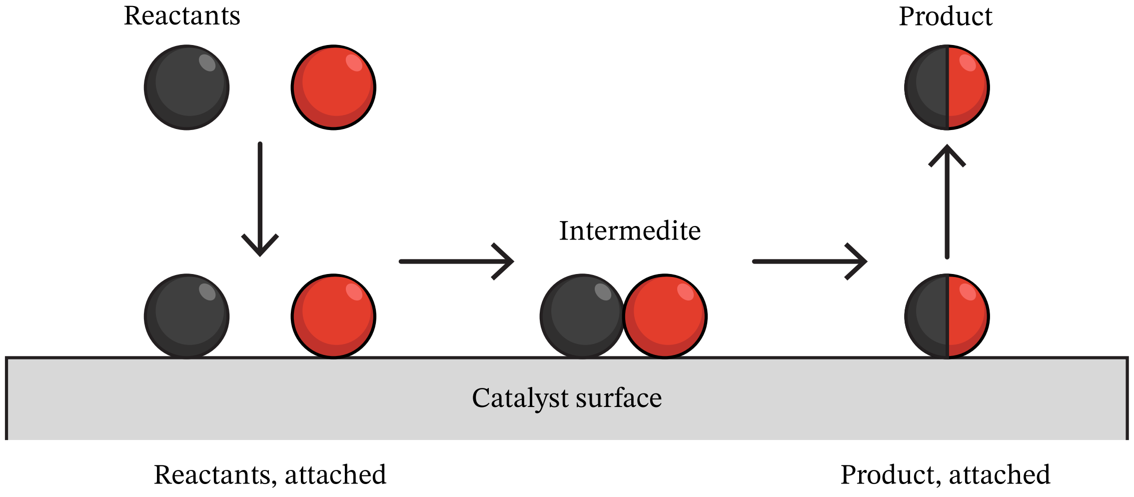
Catalysis

- Catalysis (in chemistry) - **the modification of the rate of a chemical reaction, usually an acceleration, by addition of a substance not consumed during the reaction**
  - Homogeneous or **heterogeneous catalysis**
  - Heterogeneous / solid catalysts are often porous materials supported by porous materials
  - Photolatalysis

- Model systems in lab scale to understand catalysis process: adsorption and surface reaction on crystalline catalysis
  - Close to real catalysis process in industry: adsorption and surface reaction on powdered catalysis
  - In-situ reaction monitoring using FT-IR spectroscopy



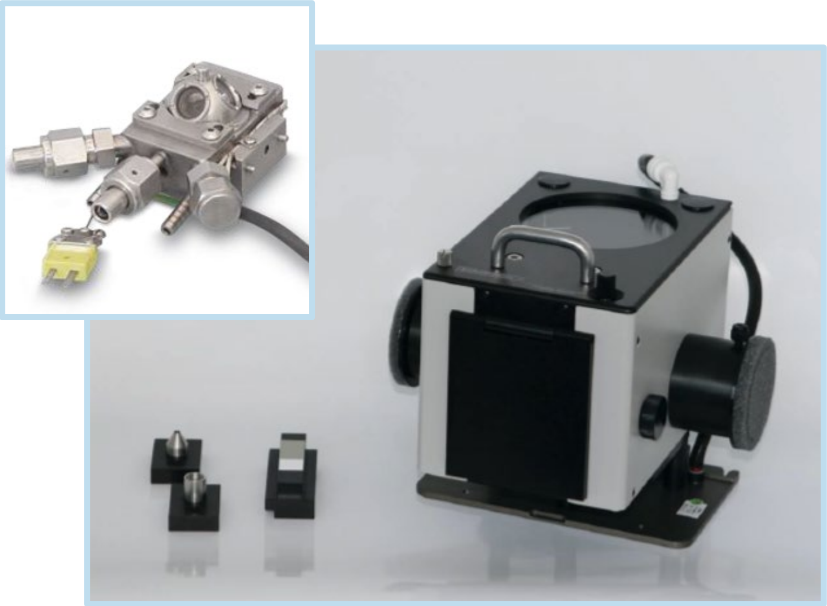




Accessories for in-situ Reaction Monitoring

Origin of power for catalytic reactions:

- Temperature
- Pressure
- Photon (e.g. UV light)
- Electric



High Temperature Reaction Chamber

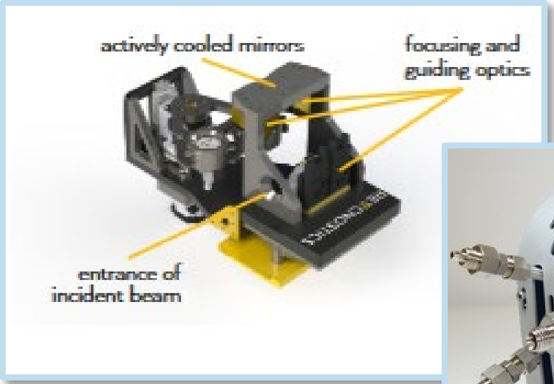
133 mPa to 133 kPa
Up to 910°C (under vacuum)
Up to 3.44 Mpa with optional high pressure dome

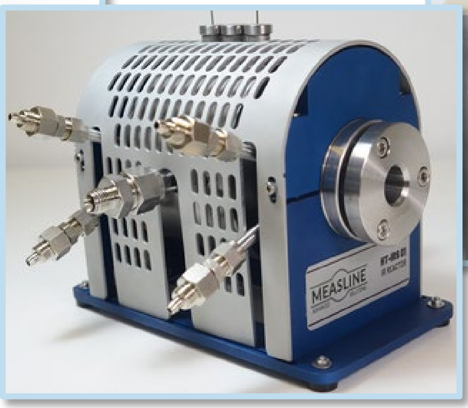
Low Temperature Reaction Chamber

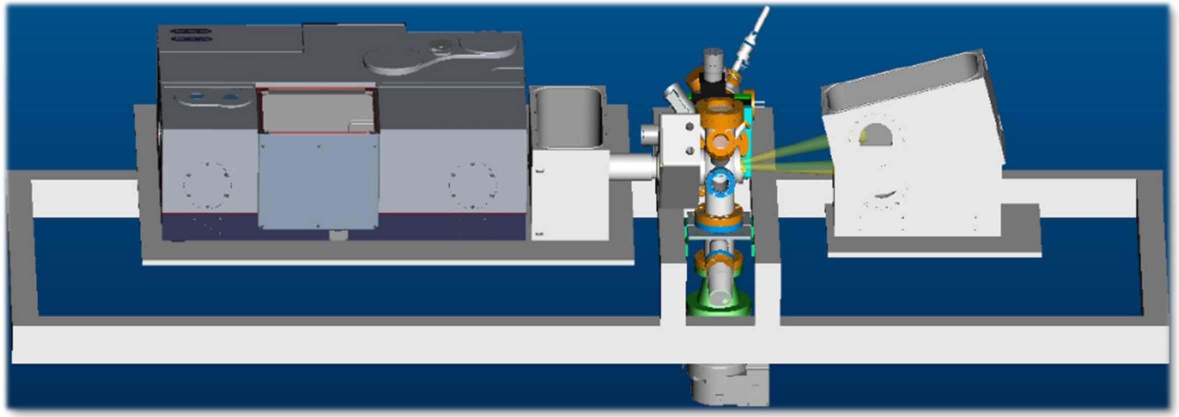
133 mPa to 133 kPa
-150˚ C to 600˚ C
(under vacuum)

More advanced cases:

- Transmission
- Photocatalysis
- Plasma
- Vacuum adaptations
- Iso-potential-DRITFS







Heat Management

Stay warm inside OR Keep heat outside

Examples:

- Construction
  - paints, glass, coatings, tapes, etc.
- Clothes
- Solar thermal plants & collectors
- Outdoor Heat Shelters

Emissivity

- Emission intensity (I) depends on angle and temperature and wavenumber: ε (ϑ, Φ, ν, T)
- Close to room temperature (~300K) main thermal radiation occurs in mid infrared (MIR)
  - Engineering: defined in mid infrared
- Emission measurement? No!
  - Weak sample emission & strong interference of 300K background radiation

Kirchhoff's law of radiation:

→ **Reflectance Measurement!**

A = Absorptance
R = Reflectance
T = Transmittance
→ **A+R+T = 1**

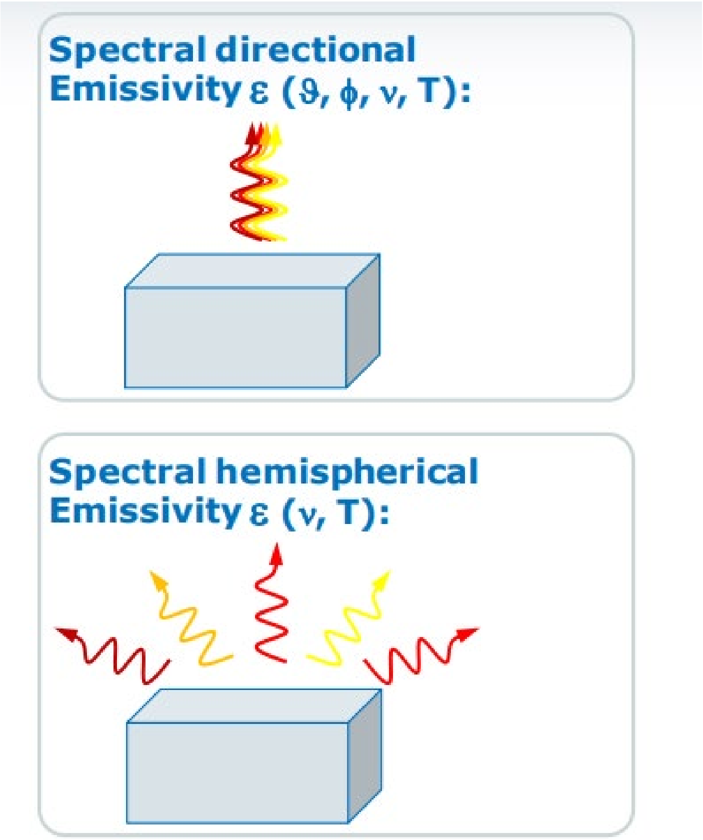
- Mid infrared: for many materials T=0
- Kirchhoff's 2nd Law: A = ε

→ **ε(ν) = 1-R(ν)**

→ **Non-transparent materials:**
ε can be determined from reflectance!

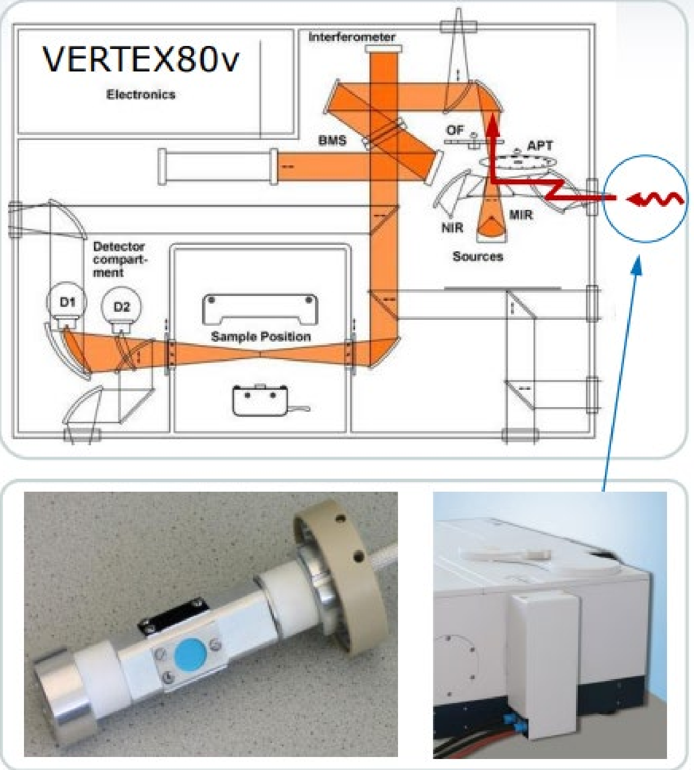
Definition of Emissivity ε

$= \text{Intensity}_{\text{Sample Emission}} / \text{Intensity}_{\text{BB Emission}}$



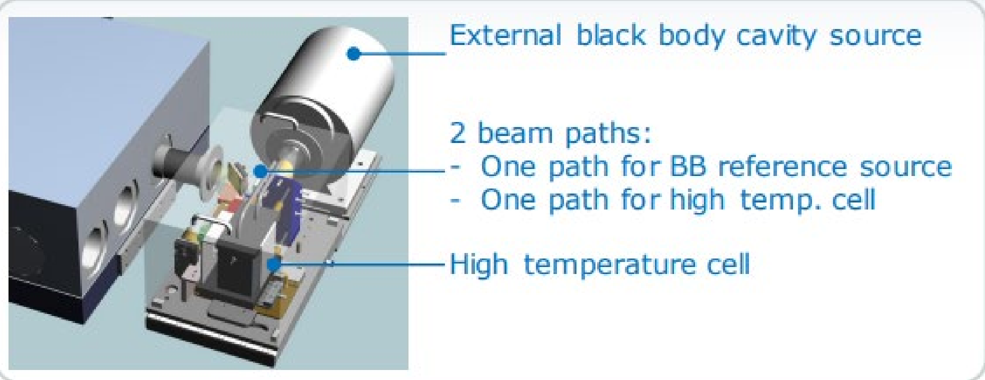
ν: wavenumber, T: temperature,
ϑ, ϕ: emission angle

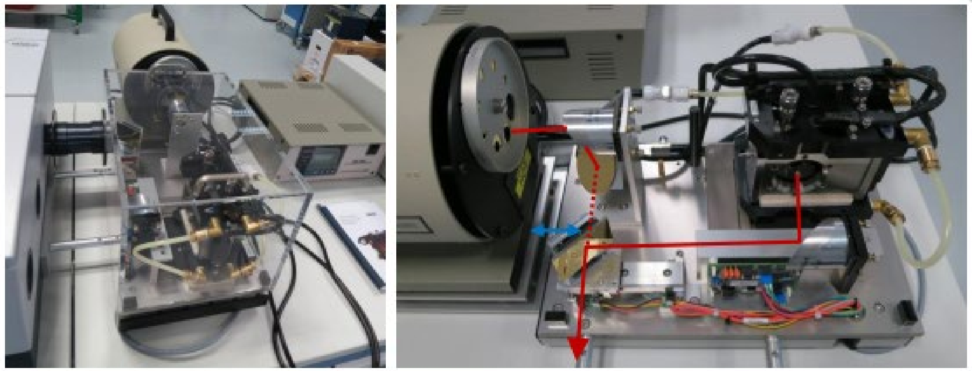
Emissivity – direct measurment of thermal emssivity



Emission adapter A540/3: up to 4 samples

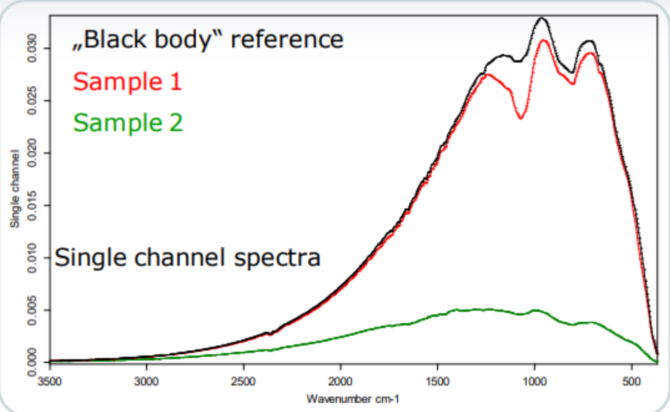
Suitable for 50-400°C





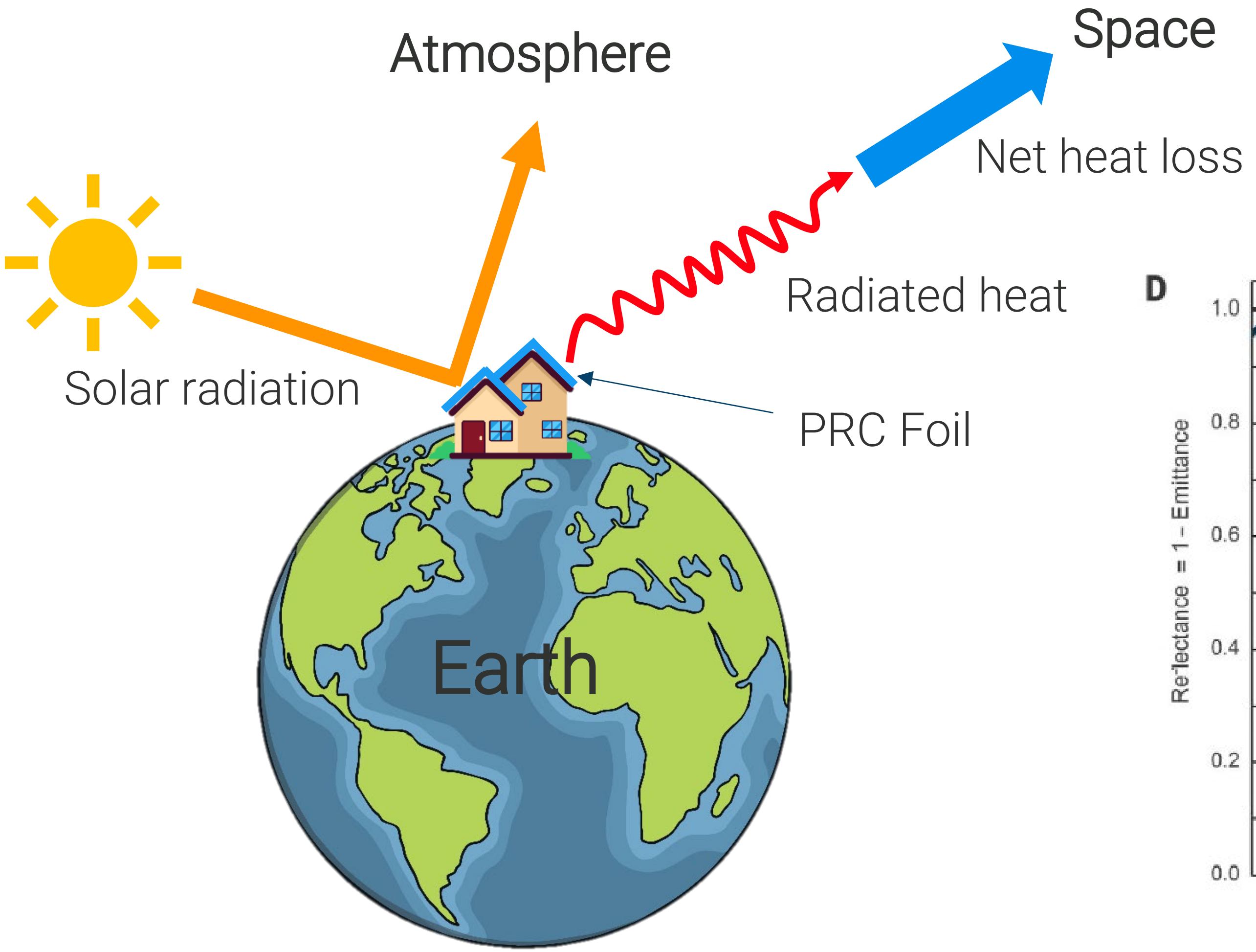
High temperature emission setup adapted to VERTEX80v

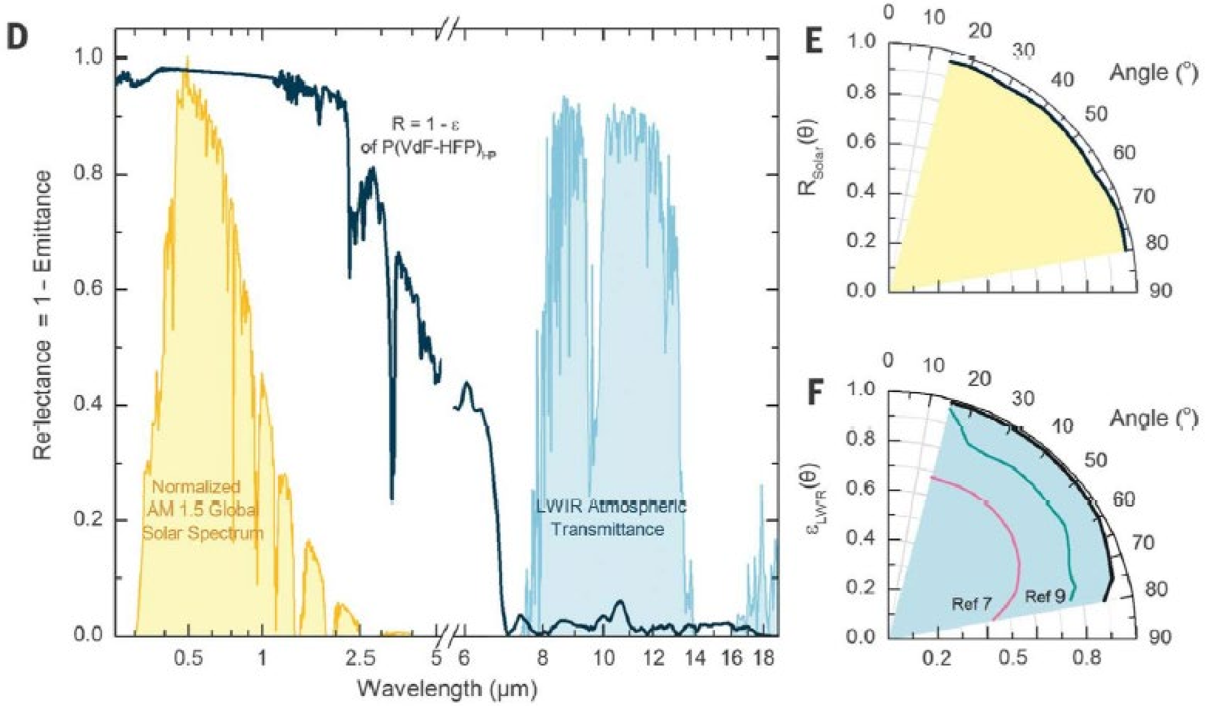
- **External emission setup:**
  - Black body cavity source, tuneable from 50-1050° C
  - High temperature cell, up to 800° C (alternatively A540)
  - Double beam path for reference and sample emission with automated switching.



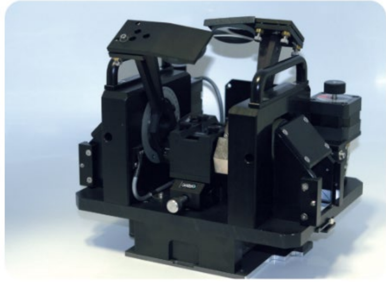
Thermal emission, T=200° C: VERTEX70v, DTGS, A540

Passive-radiative cooling



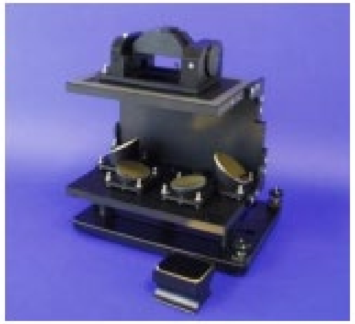


Directional emissivity for Passive Radiative Cooling

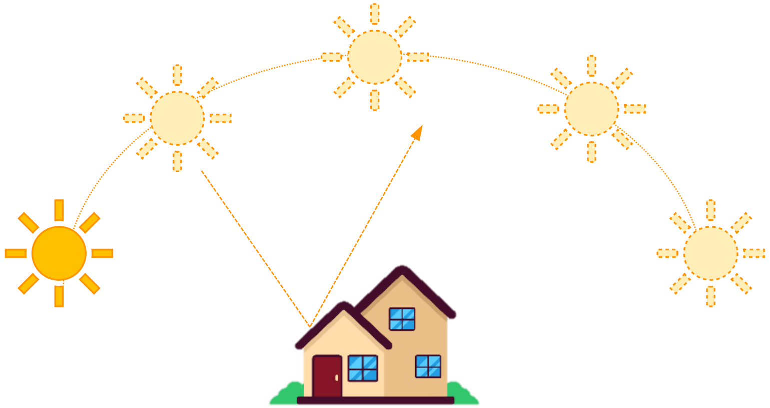


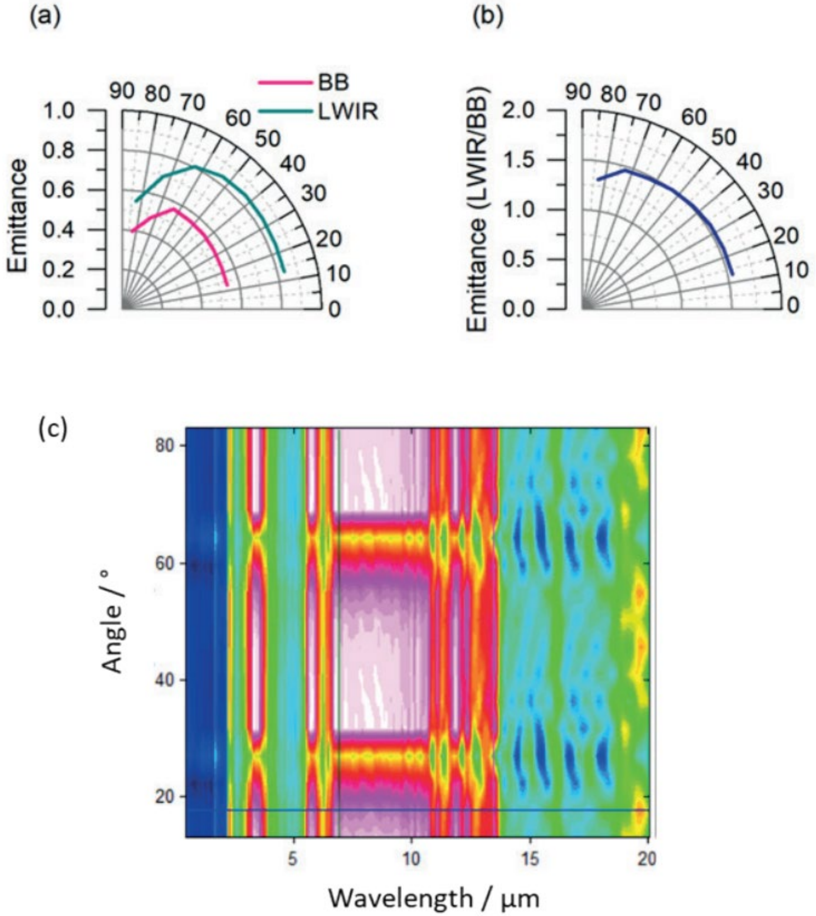
A513/Q-A

Bruker A519:
absolute reflectance with small angle of incidence.



10Spec accessory:
relative reflectance with small angle of incidence





Hemispherical emissivity

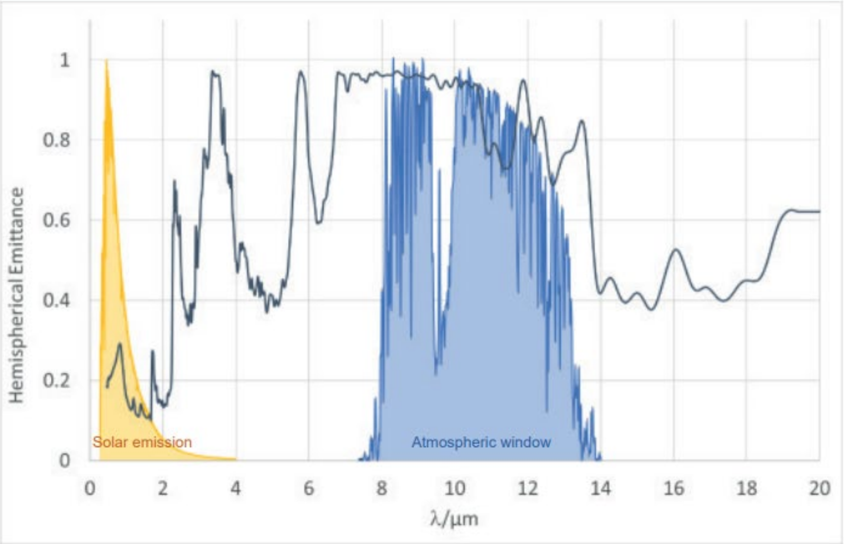
Integrating spheres A562

Gold (MIR)

PTFE (NIR-Vis)

Analysis of:

- Hemispherical reflectance
- Diffuse reflectance
- Diffuse transmittance
- Directional Refl.

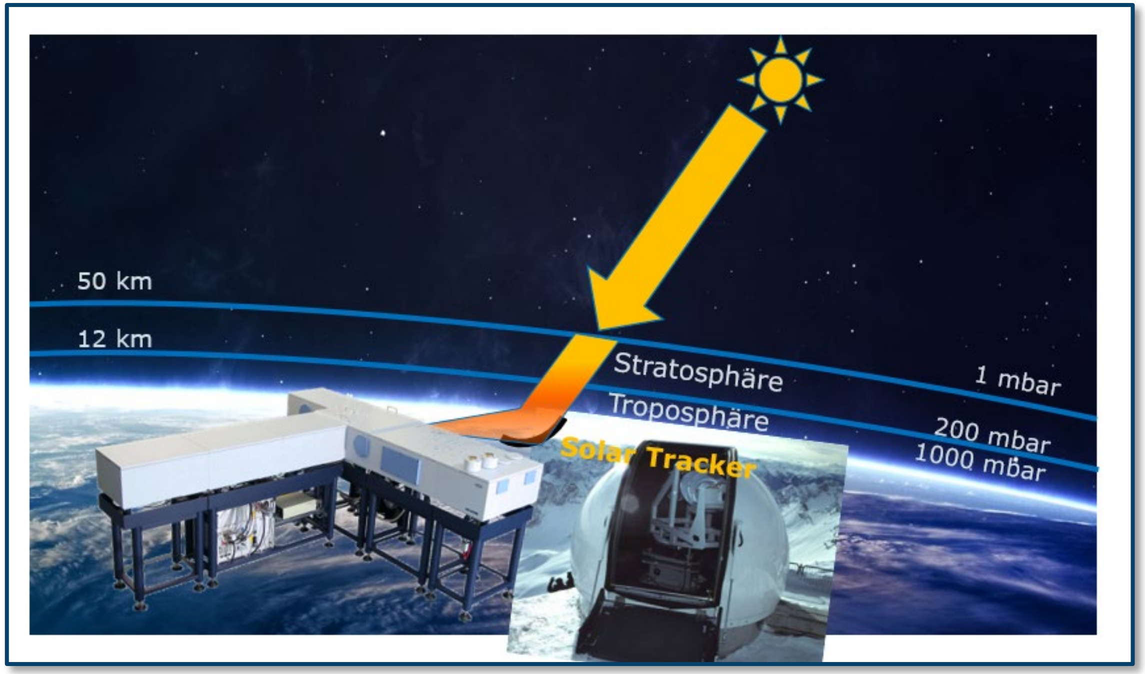


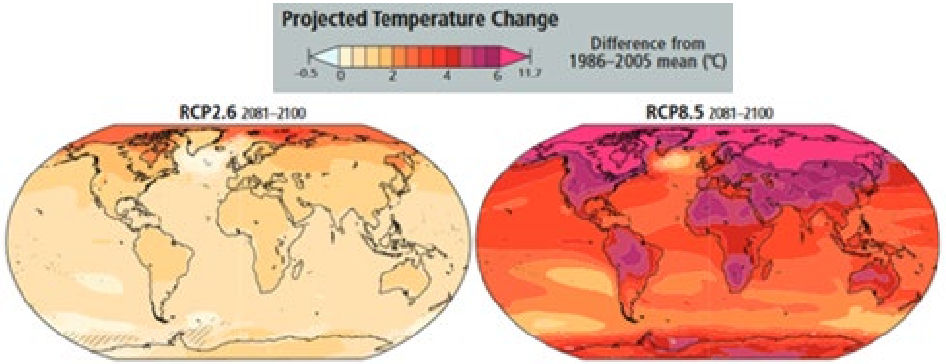
The ε^{LWIR}(HS) was calculated to be 0.85 and hemispherical emittance selectivity S^{HS} = 1.36. These values are comparable to 0.83 and 1.32 obtained by Huang, Mandal, and Raman in their experiment[15].

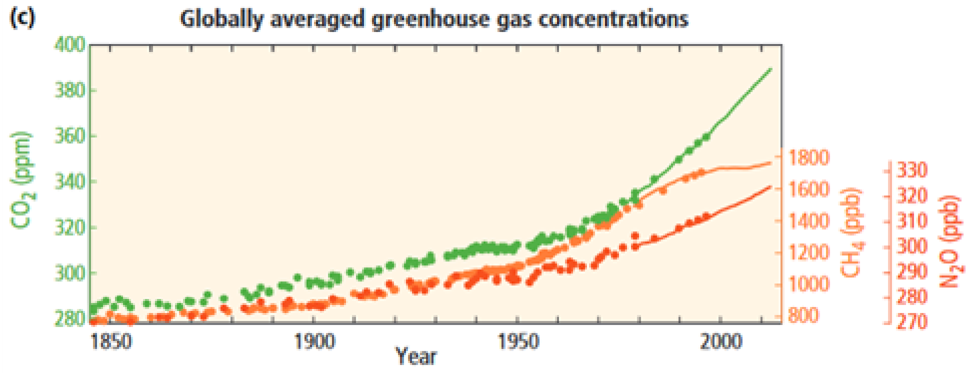
Environmental Monitoring – Green House Gases

GHG monitoring with the IFS 125 HR

- Measurement of greenhouse gases (GHGs):
  source = sun; sample = atmospheric column





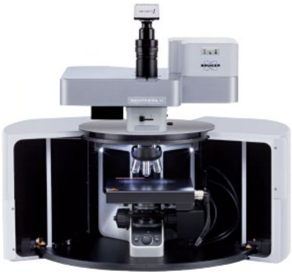


- Total Carbon Column Observing Network: TCCON
- Network for the Detection of Atmospheric Composition Change: NDACC

Environmental monitoring - Microplastic

ALPHA II

LUMOS II



SENTERRA II

Analytical Method	Minimum Particle Size	Filter Requirements	Degree of Automation	Acquisition Speed
ATR FT-IR Spectroscopy	> 500 µm	not applicable	low	slow
FT-IR Microscopy	> 10 µm	IR transparent	high	fast
ATR FT-IR Microscopy	> 5 µm	any filter any substrate	high	medium
FT-IR Imaging	> 5 µm	IR transparent	very high	very fast
ATR FT-IR Imaging	> 2 µm	any filter any substrate	high	medium
Raman Imaging	> 0.5 µm	non fluorescent	very high	fast

Microplastic analysis can also be integrated with Invenio or Vertex setup

VERTEX NEO R With Vacuum ATR + HYPERION II ILIM

CO_2, VOC, biomass, environmental studies with TGA-FTIR

ORIGINAL ARTICLE

Production and characterization of waste nutshells derived biocarbon through slow pyrolysis: an investigation on the effects of pyrolysis temperature

Kikaoseh Agweh[1] · Michael R. Snowdon[1] · Ranjeet Kumar Mishra[1,2] · Guowei Chen[1] · Singaravelu Vivekanandhan[3] · Amar K. Mohanty[1,4] · Manjusri Misra[1,4]

Received: 31 August 2022 / Revised: 19 January 2023 / Accepted: 22 January 202

scientific reports

OPEN **Thermal degradation and flame spread characteristics of epoxy polymer composites incorporating mycelium**

Nattanan Chulikavit[1], Tien Huynh[2], Akbar Khatibi[1], Raj Das[1] & Everson Kandare[1]

Open Access Article

TGA-FTIR Analysis of Biomass Samples Based on the Thermal Decomposition Behavior of Hemicellulose, Cellulose, and Lignin

by Esin Apaydın Varol * and Ülker Mutlu

Department of Chemical Engineering, Faculty of Engineering, Eskisehir Technical University, 26555 Eskişehir, Turkey

* Author to whom correspondence should be addressed.

Energies **2023**, *16*(9), 3674; https://doi.org/10.3390/en16093674

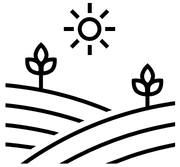
ELSEVIER
Infrared Physics & Technology
Volume 72, September 2015, Pages 52-57

Novel use of TGA–FTIR technique to predict the pollution degree in marine sediments

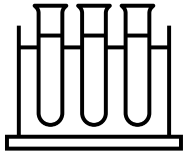
Fatiha Oudghiri, José Luis García-Morales, María Rocío Rodríguez-Barroso

Sustainable agriculture – soil analysis

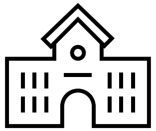
Easy – Rapid – Precise – Eco-Friendly



Field studies



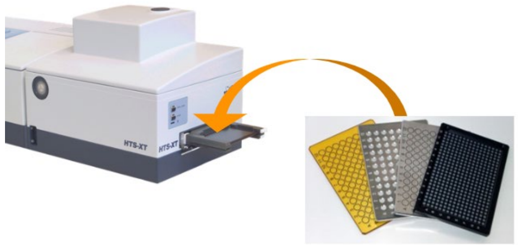
Central Laboratory



Academia

Portable & On-Site with ALPHA II

- Compact system for field-ready soil analysis.
- Rapid and accurate detection of pesticides, heavy metals, and microplastics.



Fast & Automated High-Throughput Analysis with HTS-XT

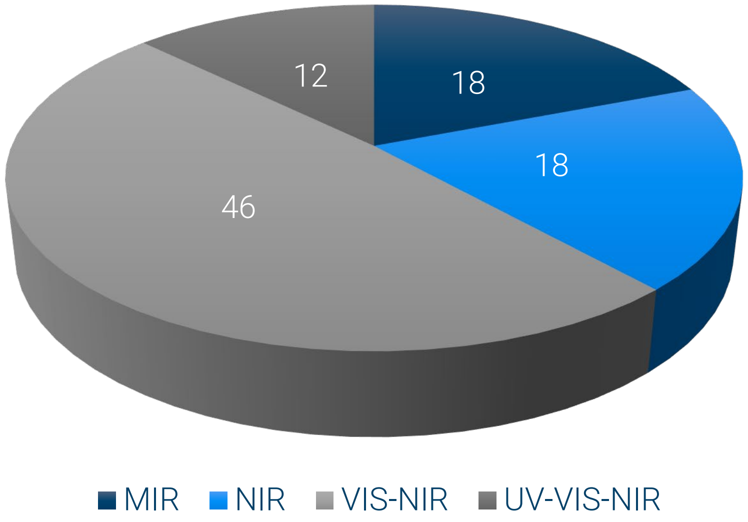
- No KBr pellets, no chemicals - just dry, grind, and measure for effortless screening.
- **Multi-sample DRIFTS automation** for rapid identification, classification, and quantification.

High-End Research with INVENIO X

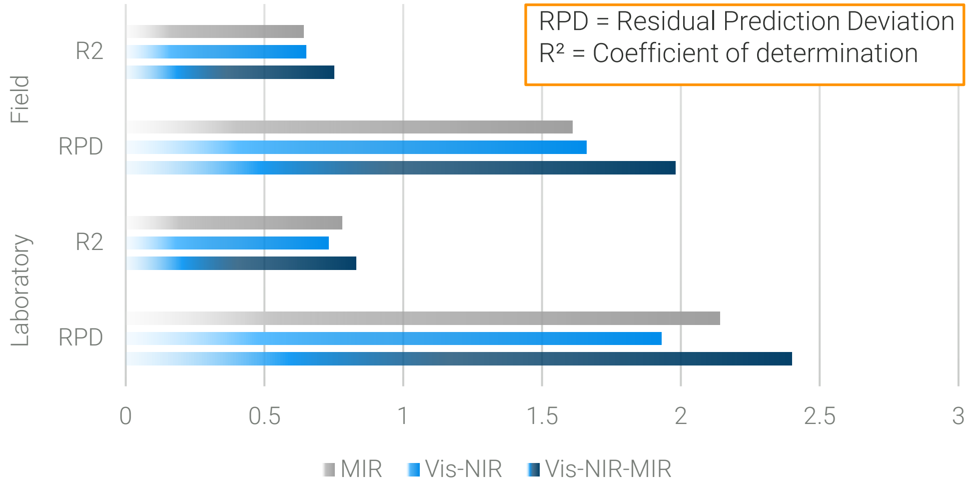
- No compromises: broadest spectral range in one go (MIR-NIR-VIS) for advanced soil studies.
- Unmatched precision in physical and chemical soil characterization
- Ideal for comprehensive data acquisition in **advanced studies and AI-supported analysis**.

Spectral soil analysis

Established methods



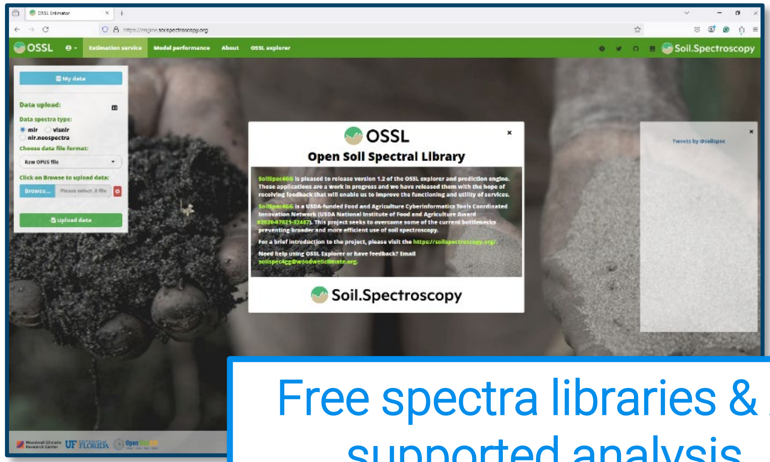
	Laboratory		Field	
	RPD	R²	RPD	R²
MIR	2,14	0,78	1,61	0,64
Vis-NIR	1,93	0,73	1,66	0,65
Vis-NIR-MIR	2,4	0,83	1,98	0,75



R.A. Viscarra Rossel, D.J.J. Walvoort, A.B. McBratney, L.J. Janik, J.O. Skjemstad. (2006) Visible, near infrared, mid infrared or combined diffuse reflectance spectroscopy for simultaneous assessment of various soil properties. Geoderma, 131, 59-75.

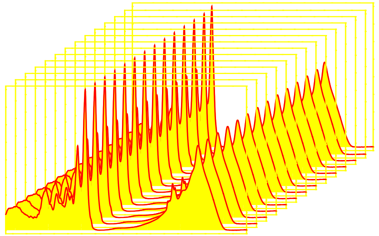
M. Vohland, B. Ludwig, M. Seidel, C. Hutengs, Quantification of soil organic carbon at regional scale: Benefits of fusing vis-NIR and MIR diffuse reflectance data are greater for in situ than for laboratory-based modelling approaches, Geoderma 405, 2022, 115426

Principle of soil spectra evaluation

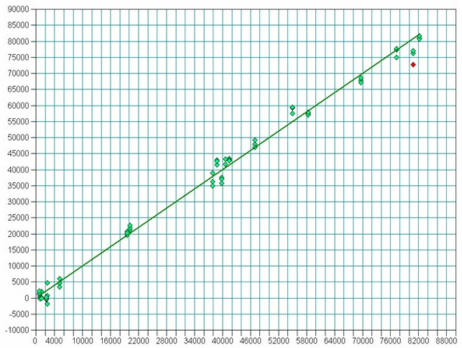


Free spectra libraries & AI supported analysis

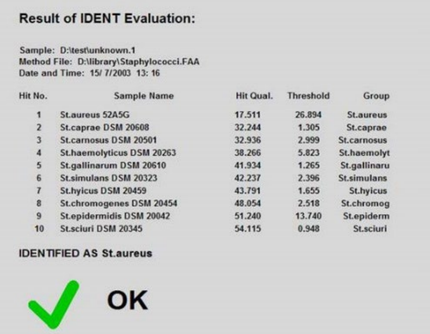
FTIR-Spectra



Quantification
(based on calibrations)



Identification
(based on libraries)



Result of IDENT Evaluation:

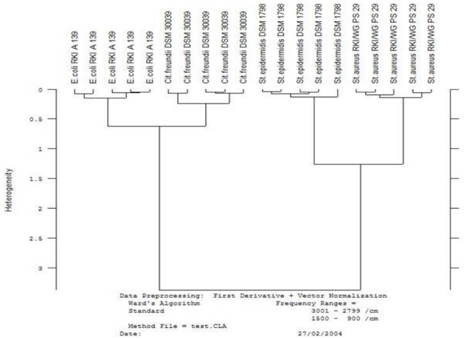
Sample: D:\test\unknown.1
Method File: D:\library\Staphylococci.FAA
Date and Time: 15/ 7/2003 13: 16

Hit No.	Sample Name	Hit Qual.	Threshold	Group
1	St.aureus 52A5G	17.511	26.894	St.aureus
2	St.caprae DSM 20608	32.244	1.305	St.caprae
3	St.carnosus DSM 20501	32.936	2.999	St.carnosus
4	St.haemolyticus DSM 20263	38.266	5.823	St.haemolyt
5	St.gallinarum DSM 20610	41.934	1.265	St.gallinaru
6	St.simulans DSM 20323	42.237	2.396	St.simulans
7	St.hyicus DSM 20459	43.791	1.655	St.hyicus
8	St.chromogenes DSM 20454	48.054	2.518	St.chromog
9	St.epidermidis DSM 20042	51.240	13.740	St.epiderm
10	St.sciuri DSM 20345	54.115	0.948	St.sciuri

IDENTIFIED AS St.aureus

OK

Classification
(based on data set)



OPUS QUANT 2

OPUS IDENT

Innovation with Integrity