CARATTERIZZAZIONE DEGLI OLI USATI MEDIANTE TECNICA FTIR

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INNOVHUB STAZIONI SPERIMENTALI PER L'INDUSTRIA

innovazione e ricerca


Anton Paar
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ANTON PAAR DEVELOPS, PRODUCES, AND SELLS HIGH-PRECISION MEASURING INSTRUMENTS AND CUSTOMIZED AUTOMATION AND ROBOTIC SOLUTIONS.

Anton Paar

MEASUREMENT TECHNOLOGY

- DENSITY AND CONCENTRATION MEASUREMENT
- VISCOMETRY
- POLARIMETRY AND REFRACTOMETRY
- DETERMINATION OF PETROLEUM PROPERTIES
- RHEOMETRY
- PARTICLE, PORE, AND SURFACE CHARACTERIZATION
- MECHANICAL SURFACE CHARACTERIZATION
- ANALYTICAL AND SYNTHETIC CHEMISTRY
- X-RAY STRUCTURE ANALYSIS
- FTIR AND RAMAN SPECTROSCOPY
- PROCESS INSTRUMENTATION
- AUTOMATION AND ROBOTICS
- KNEADING AND EXTRUSION TECHNOLOGY

HISTORY

1922

Anton Paar, a machinist, establishes the company in Graz.

1932

Margareta Platzer, Anton Paar's daughter, begins with **high-precision manufacturing.**

1957

The **first scientific instrument** is produced: the Kratky Small-Angle X-Ray Camera.

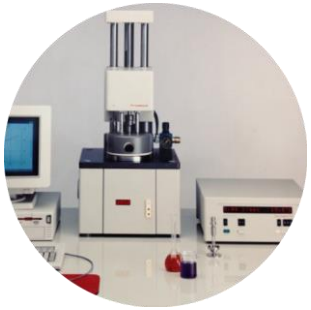
1963

Ulrich Santner, Margareta Platzer's son-in-law, pushes **cooperation with external research institutes** forward**.**

1967

The **first digital density meter** for laboratories is presented: DMA 02C.

HISTORY



1989

The first steps in **rheometry, viscometry,** and **digestion technologies** are taken.

2002

Friedrich Santner, Ulrich Santner's son-in-law, takes over sole management and pushes the company's **internationalization** forward.

SINCE 2007

Anton Paar has been growing. This has also been happening through **acquisitions** and the **expansion of measuring technologies**.

2016-2017

Instruments for the **characterization of mechanical properties and surfaces.**

2020

Dominik and Jakob Santner, Friedrich and Maria Santner's sons, become part of **management**.

FACTS & FIGURES

ESTABLISHED IN
1922

HEADQUARTERS
IN GRAZ / AUSTRIA

4,500+
EMPLOYEES

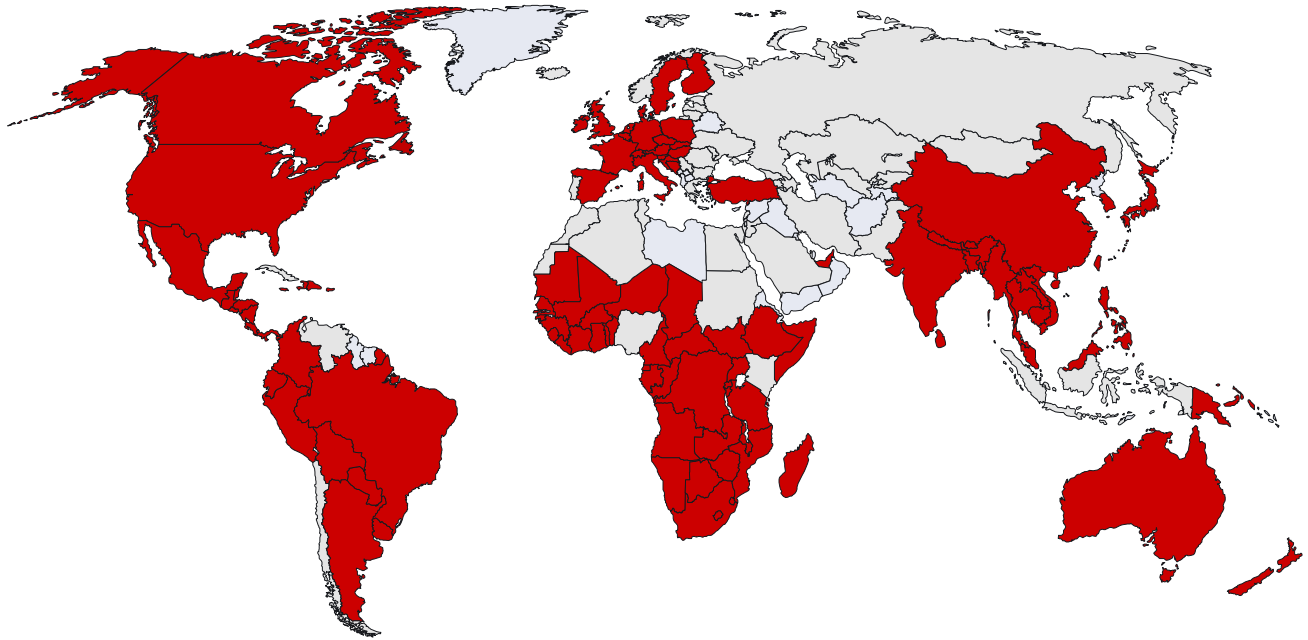
AS PART OF THE
ANTON PAAR GROUP AG
OWNED BY THE CHARITABLE
SANTNER FOUNDATION

14.5 % INVESTMENT
IN RESEARCH AND DEVELOPMENT

FROM ANNUAL TURNOVER
ANTON PAAR GMBH

ALL CRITICAL COMPONENTS
MANUFACTURED IN-HOUSE

OPERATING WORLDWIDE



8
PRODUCTION COMPANIES

39
SALES SUBSIDIARIES

32
DISTRIBUTION PARTNERS

OPERATING IN ITALY

28
SALES

6
APPLICATION SPECIALIST

18
SERVICE

15
STAFF

MEASUREMENT TECHNOLOGY

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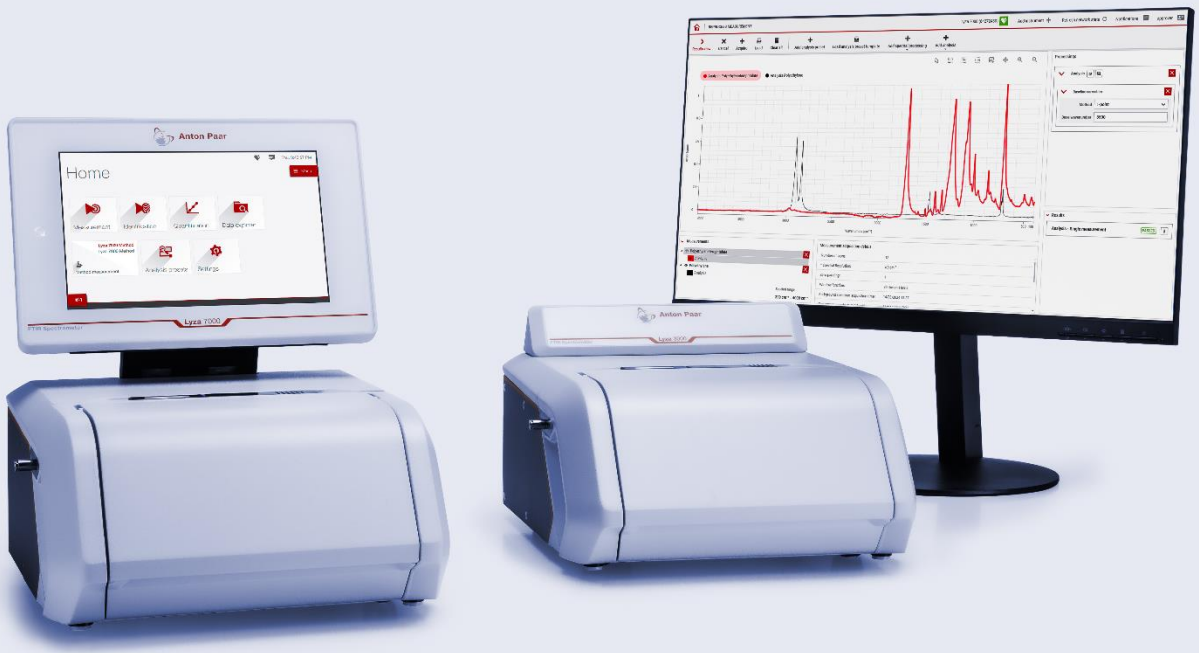
I

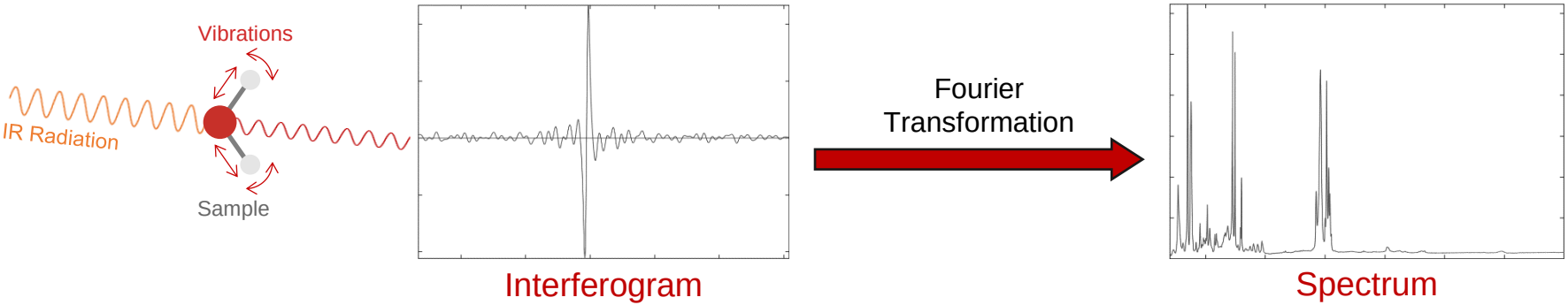
II



FTIR SPECTROMETER

LYZA SERIES

FTIR Spectroscopy

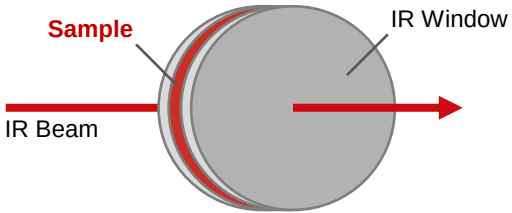


Principle

- Sample is irradiated with **infrared (IR) radiation**
- Some radiation is **absorbed** by the sample molecules → **vibration** of molecular bonds
- The remaining, **attenuated** (=weakened) light is detected → **interferogram**
- Fourier transformation converts the interferogram to a **spectrum**
- **Peaks** correspond to **functional groups** in the molecule
- Spectrum = "**fingerprint**" → identify / verify substances

FTIR Spectroscopy
Most Common techniques

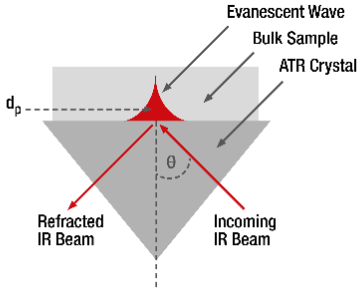
Transmission



- Sample is placed between two **IR transparent plates**
- Radiation passes through **plates** and **sample**
- Radiation **interacts** with sample

- Plate materials: CaF_2, KBr, ZnSe, and more

ATR (Attenuated Total Reflectance)



- Sample is placed on **crystal surface**
- IR radiation travels through crystal and is **reflected** at the crystal-sample interface
- IR light **interacts** with sample at reflection point
- Single or multiple reflections possible

- Crystal materials: Diamond, Ge, ZnSe, and more

LYZA SERIES

APPLICATIONS

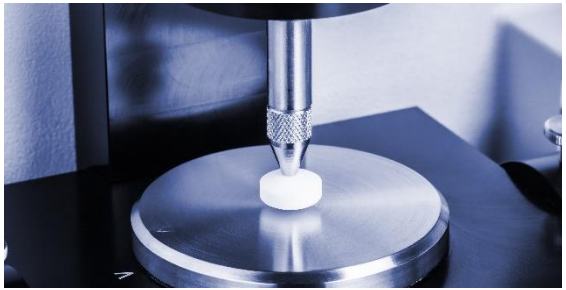
Application overview

Verification

Ensure a sample's chemical composition by comparing its FTIR spectrum with known references.

Examples:

- **Incoming goods inspection** : Ensure correct raw material properties
- **Final product QC**
  Pass/Fail analysis gives clear results



Identification

Identify unknown substances by comparing FTIR spectra to reference databases

Examples:

- **Contaminants**: Detecting impurities or adulterants raw materials & products
- **Forensics**: unknown forensic samples (e.g. drugs, explosives, or fibers)
- **Research:** unknown reaction products or intermediates in synthesis processes

Quantification

Determine the concentration of compounds in a sample through the analysis of peak intensities.

Examples:

- **Oil condition monitoring**: ASTM / DIN methods to evaluate used oil properties
- **Fuels**: Analysis of FAME content
- **Research**: Popular standard method. Predefined workflows ensure consistent results.

Application Reports

Quality control of polymers using Lyza 7000 FTIR spectrometer

Analysis of biodiesel/FAME in diesel blends with Lyza 7000 FTIR spectrometer

Differentiation between pure and PFA-blended PTFE via FTIR

Monitoring of oxidation in lubrication oil

Analysis of bituminous binder samples using FTIR spectrometer Lyza 7000

Find more at
www.anton-paar.com

Application Report

Monitoring of oxidation in lubrication oil

- Oxidation is predominant in every in-service lubrication oil
- Negative impact on oil properties
- Monitoring allows to determine the oils suitability for use
- ASTM E2412, D7214, D7414, D7418

Features

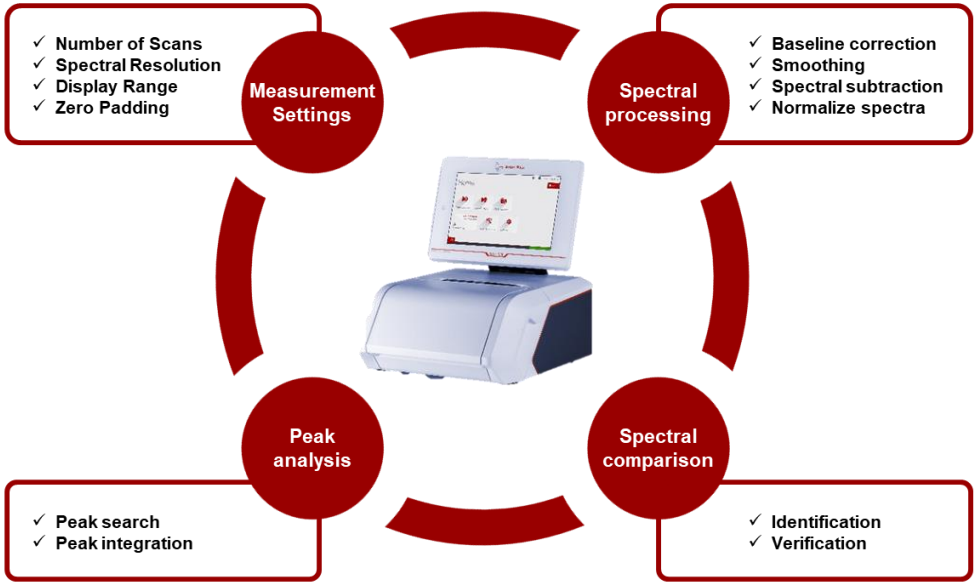
- Automated oxidation peak integration
- Correct measurement cell available with the instrument
- Touchscreen and small footprint

Benefits

- Can be measured by inexperienced user
- No worries about compliance of measurement cells
- Save space in your lab

Application Report

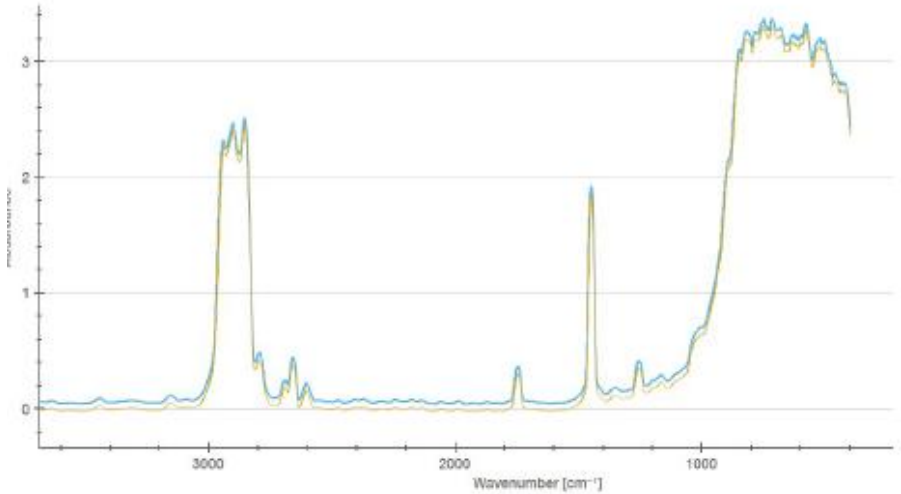
Monitoring of oxidation in lubrication oil → Spectral Processing

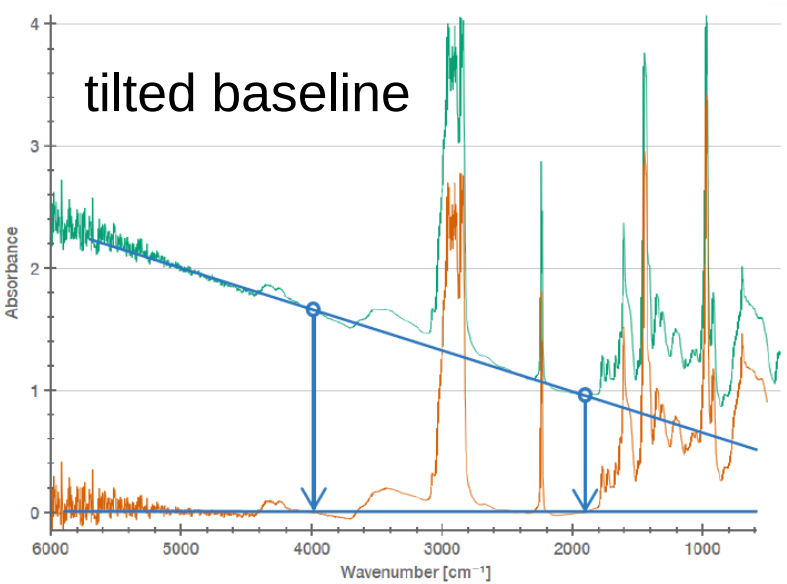


Group	Operation
Edit spectral range	Edit spectral range
Baseline correction	Baseline correction 1-point
	Baseline correction 2-point
	Baseline correction 2-point minima
Smoothing	Smoothing
Subtraction	Spectral subtraction
Normalization	Normalization maximum peak
	Normalization specific wavenumber
	Normalization peak integral
	Pathlength correction
Fringe reduction	Fringe reduction

Application Report

Spectral Processing - Baseline correction





1-point baseline correction. → *2-point baseline correction.*

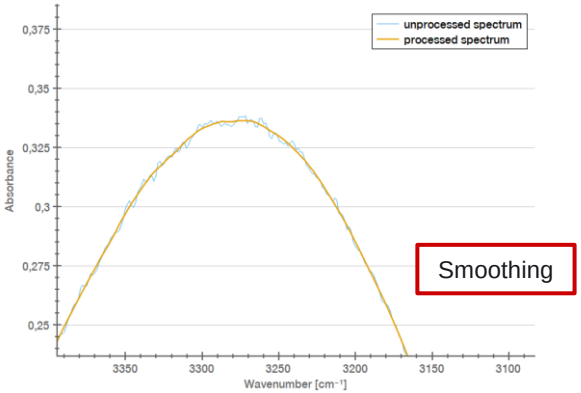
Baseline correction 2-point minima

(Required by ASTM)

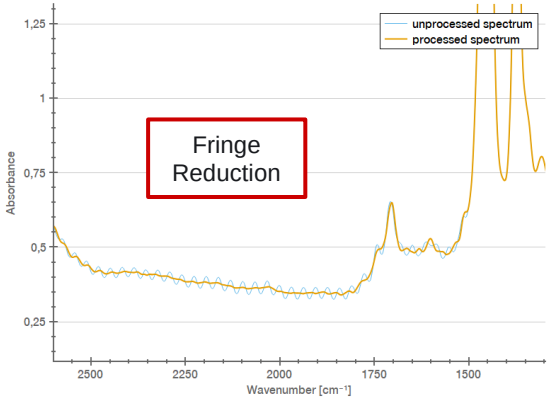
not a specific wavenumbers but a wavenumber range

Application Report

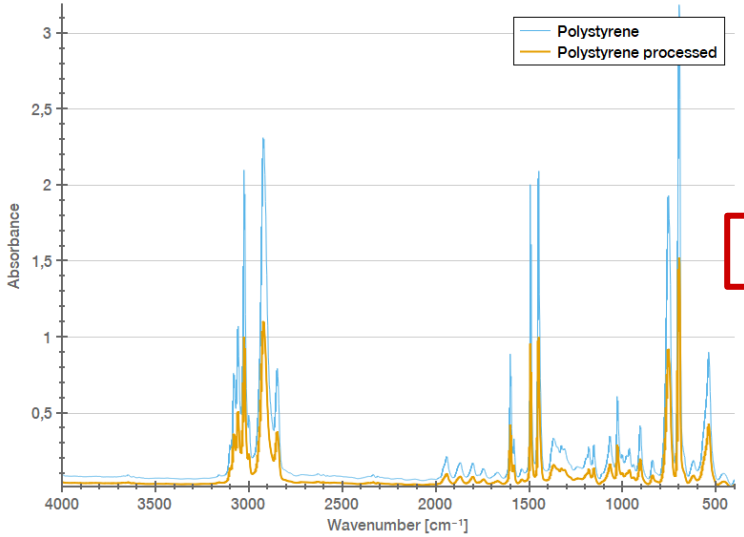
Spectral Processing



Smoothing



Fringe Reduction



Normalization

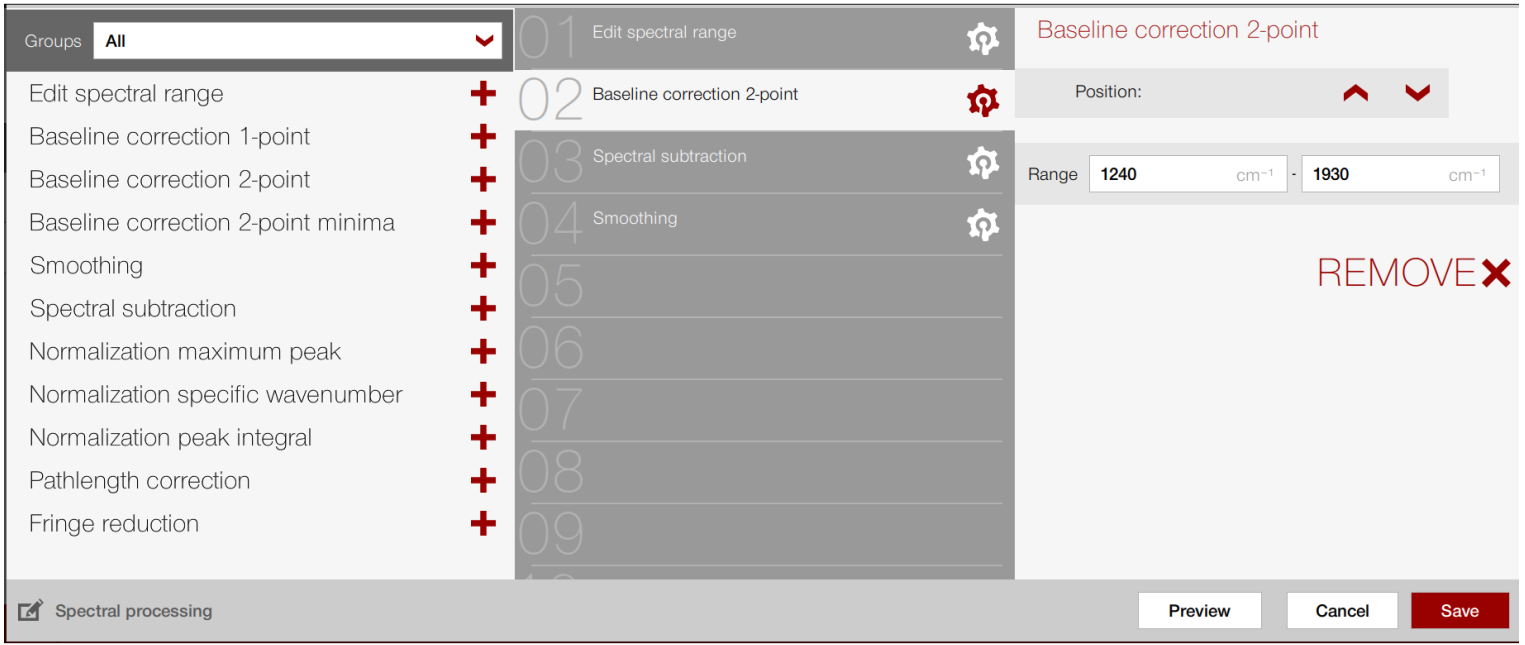
Pathlength correction

Normalize to a **specific pathlength** of a transmission cell.

Transmission cells have a pathlength that is given by the manufacturer (e.g. 0.100 mm) but most cells deviate from that nominal pathlength (e.g. 0.112 mm). Therefore, it is necessary to correct that deviation in the spectral processing to have comparable values. The spectrum is multiplied by the ratio of the actual to the given pathlength

Application Report

Spectral Processing



Application Report

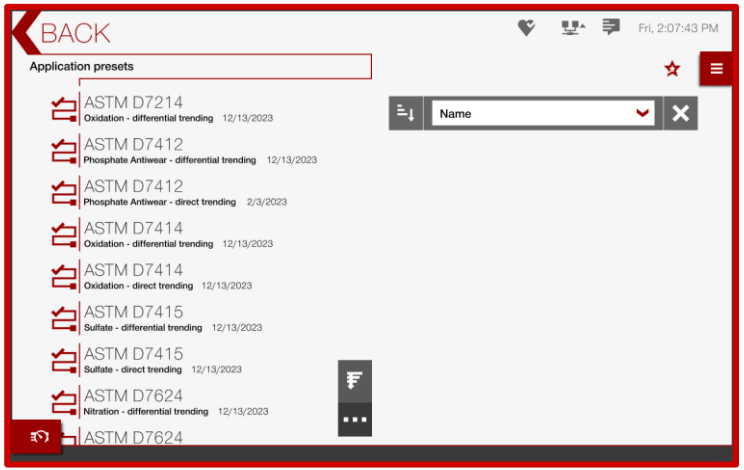
Lyza 7000: Used Oil Package

ASTM compliance

The package and its features make the instrument compliant to ASTM E1421 for instrument performance, D7418 for setup and operation of the FTIR spectrometer and several standards for different parameters:

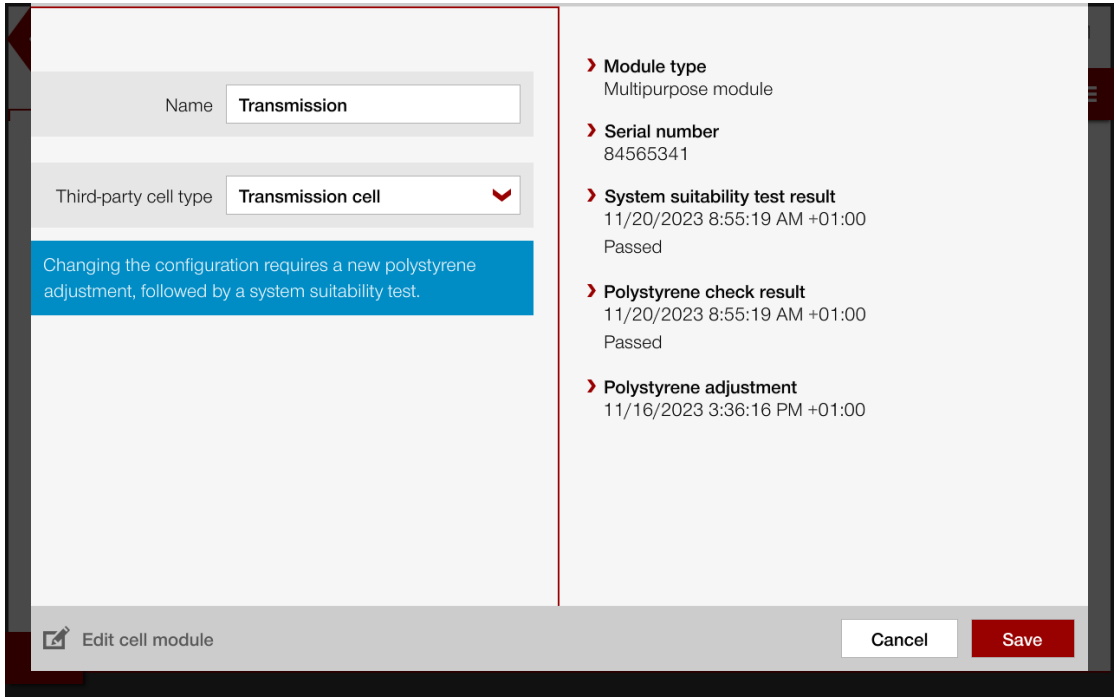
ASTM standard	Parameter
E2412 Direct trending Petroleum lubricants	Water
	Oxidation
	Nitration
	Gasoline
	Diesel
	Sulfate by-products
	Ethylene glycol
	Soot
E2412 Spectral subtraction Petroleum lubricants	Water A*
	Water B*
	Soot A
	Soot B*
	Oxidation A
	Oxidation B
	Nitration
	Phosphate antiwear additives
	Gasoline*
	Diesel*
	Sulfate by-products
	Ethylene glycol*
E2412 Direct trending Extreme pressure petroleum lubricants	Water
	Oxidation
E2412 Direct trending Polyol ester	Water
	Ester-Base-Stock Breakdown 1
	Ester-Base-Stock Breakdown 2
	Antiwear components
	Fuel
	Other contaminants
D7412 Direct trending	Phosphate antiwear additives
D7412 Differential trending	Phosphate antiwear additives
D7414 Direct trending	Oxidation
D7414 Differential trending	Oxidation
D7415 Direct trending	Sulfate by-products
D7415 Differential trending	Sulfate by-products
D7624 Direct trending	Nitration
D7624 Differential trending	Nitration
D7844 Direct trending	Soot
D7844 Differential trending	Soot
D7214 Peak area increase	Oxidation

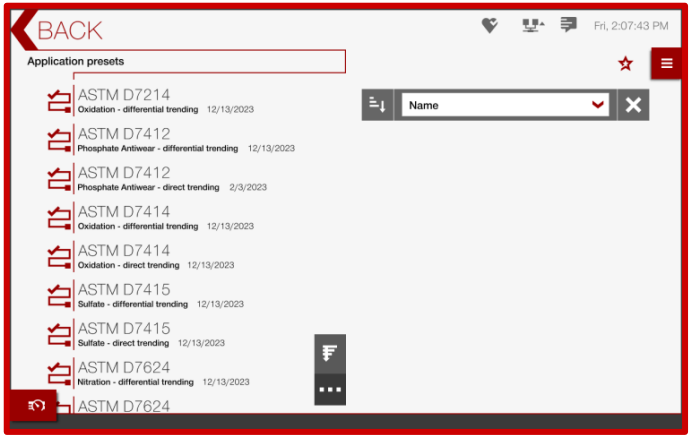
* requires additional calibration



Application Report

Lyza 7000: Used Oil Package



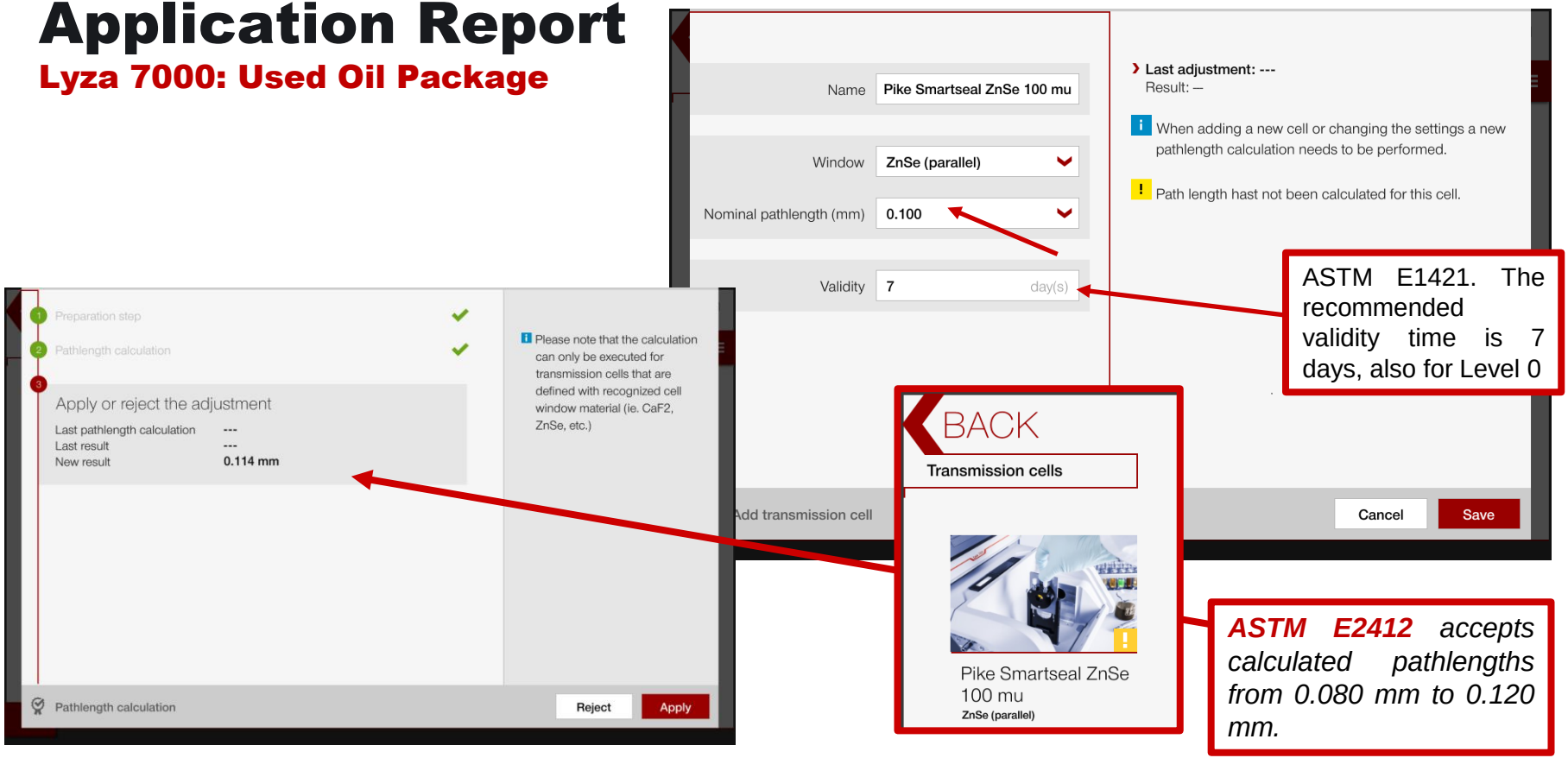


ASTM E1421 – References, Level 0 and Level 1 tests

If the instrument should measure according to ASTM E1421 it is necessary to start a few tests as a next step. Before you can start a Level 0 test, two measurements must be made as a reference

Application Report

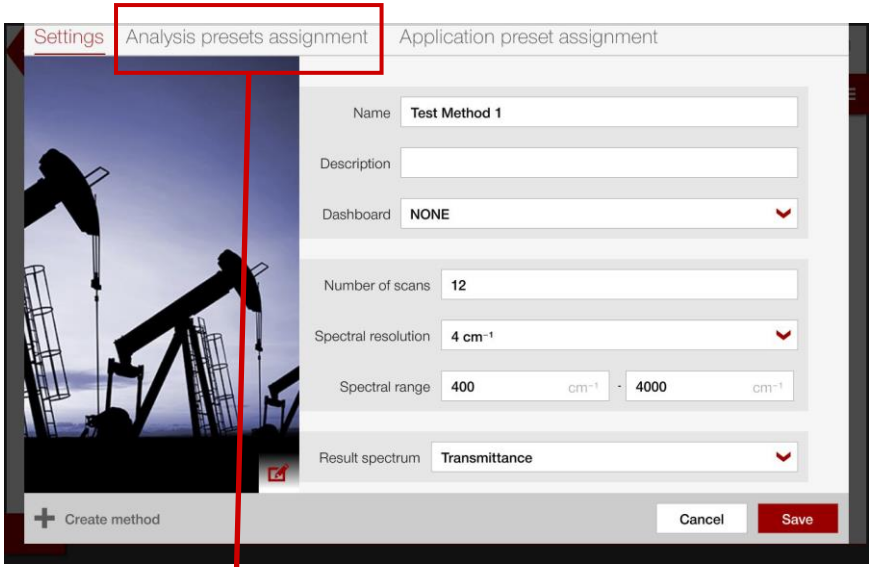
Lyza 7000: Used Oil Package



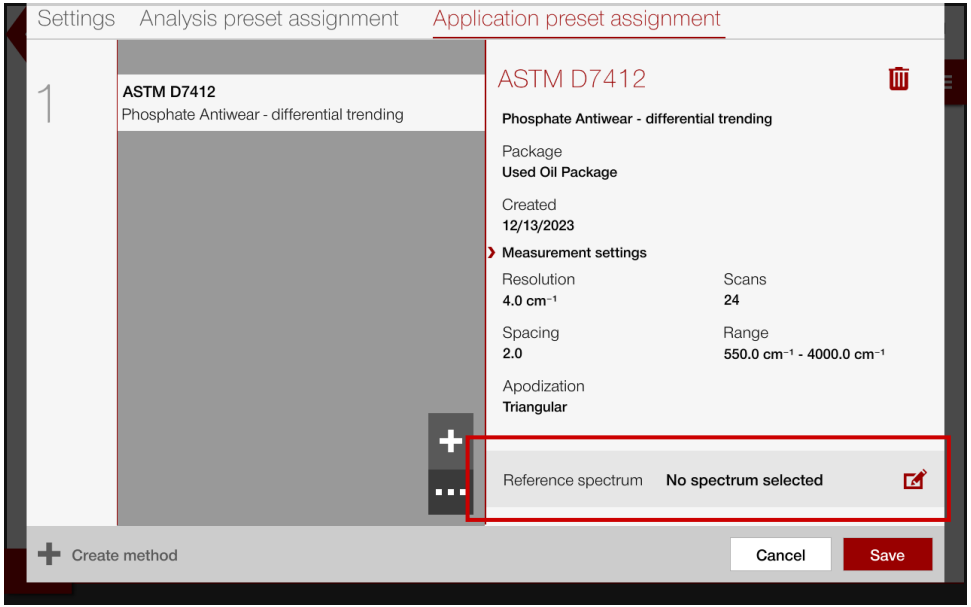
Application Report

Lyza 7000: Used Oil Package

AST E2412 *accepts calculated pathlengths from 0.080 mm to 0.120 mm.*



Spectral Processing



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Thank you for your attention

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FTIR Spectroscopy Vs. Raman Spectroscopy

- Both techniques are based on the interaction of light with the chemical bonds in sample molecules
- Complementary and confirmatory techniques: MANY substances are both IR & Raman active.

FTIR:

- Based on absorption of light
- Black body radiation in mid-IR region

Raman:

- Based on inelastic light scattering
- Laser with specific wavelength in VIS-NIR region

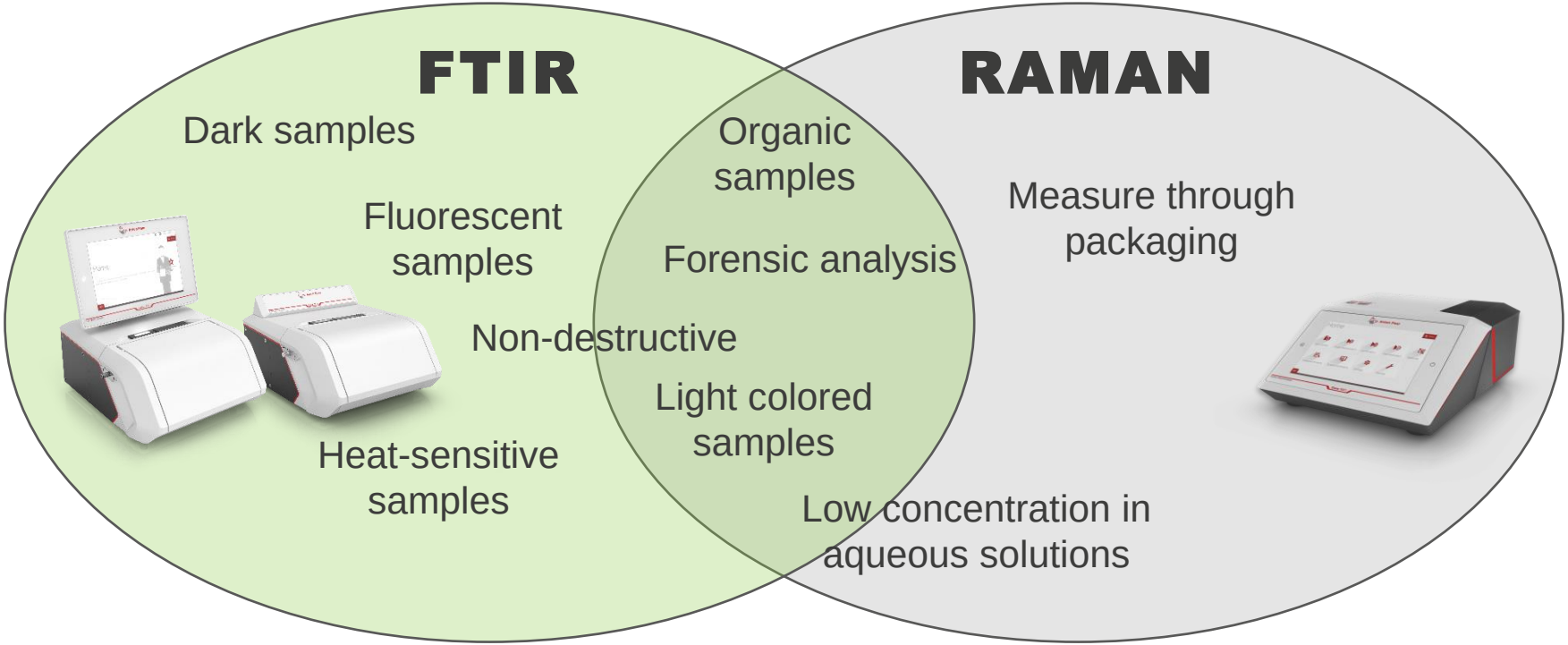
Primary applications:

- Colored samples
- Fluorescent samples
- Heat-sensitive samples
- Low concentrations (→ quantitative analysis)

Primary applications:

- Packaged samples
- Amorphous samples
- White powders
- Minerals
- Aqueous solutions

FTIR Spectroscopy
Vs. Raman Spectroscopy



Lyza Series - Features

Methods

Modular cell concept

Spectral libraries

System health

Quantification

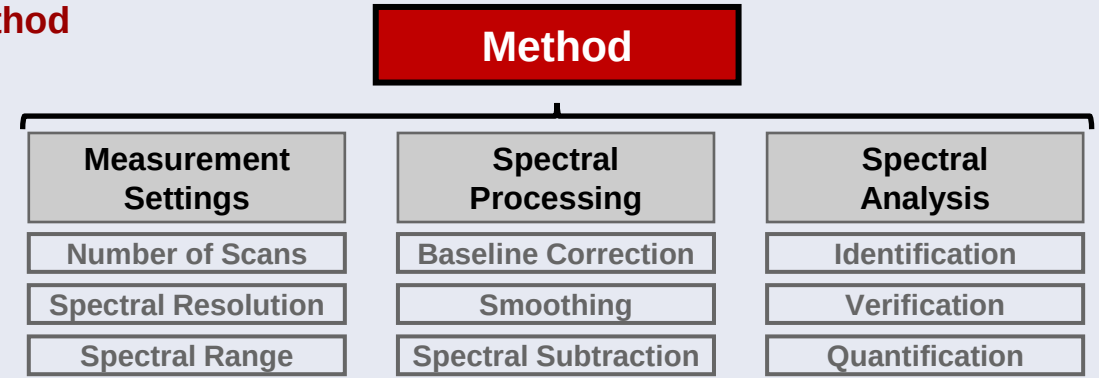
Powerful automated analysis: Easy as 1-2-3

- Perform complex measurements at the push of a button
- Combine multiple analyses in a single method with a customizable report

1. Choose Method

2. Press Start

3. Result



Lyza Series - Features

- Methods
- **Modular cell concept**
- Spectral libraries
- System health
- Quantification

Easily exchangeable cell modules

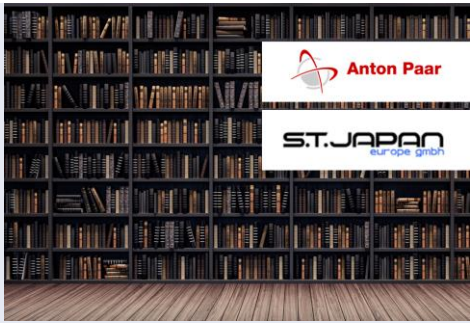
- Compatible with a variety of ATR and transmission cells
- Automatic calibrations for optimal performance with every cell
- Examples of compatible cells:
  - **ATR**
    -Pike IRIS *(Diamond, Germanium)*
  - **Horizontal ATR**
    -Pike HATR *(ZnSe)*
  - **Transmission**
    -SmartSeal Liquid Transmission *(CaF_2, KBr, ZnSe)*

Lyza Series - Features

- **Methods**
- **Modular cell concept**
- **Spectral libraries**
- **System health**
- **Quantification**

Versatile spectral libraries

- **Identify** any unknown substance
- **Verify** your product
- **Create** your own specialized library
- **Choose** from a variety of industry-standard libraries
- Anton Paar factory library **included**



Lyza Series - Features

- **Methods**
- **Modular cell concept**
- **Spectral libraries**
- **System health**
- **Quantification**

The most reliable spectrometer

- Innovative internal **system monitoring** technology (incl. humidity control)
- Ensures the instrument is calibrated via **self calibration workflows**
- Constant monitoring of all **critical components** in your system
- The **status LED** shows you the measuring progress wherever you are in the lab
- All you need to know, you know at a glance

Lyza Series - Features

- **Methods**
- **Modular cell concept**
- **Spectral libraries**
- **System health**
- **Quantification**

FTIR Quantification* Anyone Can Master

- Create your calibration in **less than 3 minutes**
- **Step-by-step** setup process to minimize errors
- **Seamless integration** into QC methods

**Lyza 7000 only*

back

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Application Report

Quick analysis of polymers using Lyza 7000

- Polymers are used for a wide range of applications
- The molecular composition dictates their chemical and mechanical properties
- FTIR is ideal for polymer analysis as well as quick verification or identification

Features

- Quick and comprehensive analysis
- Minimal to no sample preparation
- Several measurement cells available
- Touchscreen and small footprint

Benefits

- Simple pass/fail analysis or extensive characterization
- Save time and reduce errors
- Use the ideal cell for your application
- Save space in your lab

back

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Application Report

Differentiation between pure and PFA-blended PTFE via FTIR

- Small changes in polymer blend influence the processability
- Transmission measurements have a high sensitivity for the detection of contaminants
- FTIR provides quick method to test blend composition

Features

- Identification of low concentration compound
- Modular cell allows different approaches
- Film creation with Pike Pixie press

Benefits

- Quick check before processing
- Flexible approach to analytical problem
- Straightforward sample preparation

back

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Application Report

Analysis of bituminous binder samples using FTIR spectrometer Lyza 7000

- Bitumen can be modified with the addition of various additives
- Bitumen binder composition influences mechanical properties
- FTIR allows for chemical identification of additives

Features

- Optimized characterization method
- Horizontal ATR cell for high signal intensity
- Sample preparation with minimal solvent waste

Benefits

- Quick method for bitumen characterization
- Minimal sample preparation
- Cost-effective and environmentally friendly solvent usage

back

Anton Paar

Application Report

Analysis of biodiesel/FAME in diesel blends with Lyza 7000

- Regular diesel is often blended with biodiesel (=fatty acid methyl esters or FAME)
- Blend ratio is tightly regulated
- FTIR analysis provides quick FAME quantification
- DIN EN14078 or ASTM D7371

Features

- Quick and accurate quantification
- Standard-compliant analysis (DIN EN14078 or ASTM D7371)
- Touchscreen and small footprint

Benefits

- Easy analysis in a matter of minutes
- Measure with correct settings to fulfill your requirements
- Save space in your lab

back

Anton Paar

Application Report

Monitoring of oxidation in lubrication oil

- Oxidation is predominant in every in-service lubrication oil
- Negative impact on oil properties
- Monitoring allows to determine the oils suitability for use
- ASTM E2412, D7214, D7414, D7418

Features

- Automated oxidation peak integration
- Correct measurement cell available with the instrument
- Touchscreen and small footprint

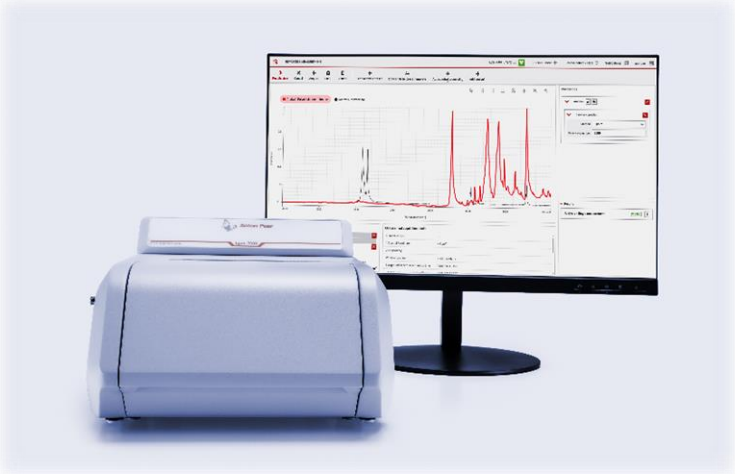
Benefits

- Can be measured by inexperienced user
- No worries about compliance of measurement cells
- Save space in your lab

Lyza Series with Spectroscopy Suite: Convenient. Compliant.

Benchtop Multipurpose FTIR Spectrometer with PC Software

- **Full instrument control** via PC
- **Pharma Compliance** on “excellence” level
  21 CFR part 11, EU Vol. 4 GMP Annex 11
- **IT compliance:** IT-qualified PC hardware, customer IT-security infrastructure and backup routines, Active Directory
- **UI/UX convenience:** Instrument operation, spectral analysis and method development on large PC screen
- Connection to **AP Connect**



Lyza Series with Spectroscopy Suite

Set up methods for measurement routines

- Run day-to-day samples **safe and efficient.**
- **No measurement settings** can be defined during the workflow.
- Automatic analysis of spectra with clear **pass/fail answer**.
- Only measure with **signed and approved settings.**
- **Never worry about data**: no deletion, lifetime retention, safe export.
- The electronic signing process for **traceable** submission, review, and approval of data.