

Agilent Cary 630 FTIR sample interfaces — combining high performance and versatility

Technical Overview

Introduction

The Agilent Cary 630 is a robust, easy to use, superior performing FTIR in a compact package. In addition to the sensitive and stable interferometer engine, the Agilent Cary 630 FTIR offers a series of customized sample interfaces, which are rapidly interchangeable, while at the same time, engineered for optimum performance and ease of use. The innovative sampling technology is designed specifically for the Agilent Cary 630 spectrometer, affording both sensitivity and versatility to handle the widest array of samples.

This overview details the design and use of sample interfaces available on the Agilent Cary 630 FTIR.





Transmission module

The transmission module provides the classic infrared sample interface (Figure 1), and allows for the measurement of solids, liquids or gases. Solids are analyzed in a pressed KBr pellet or a nujol mull, liquids are measured with a traditional sealed or demountable cell, and gases can be measured by using the Agilent Cary 630 FTIR gas cell. The purgeable transmission module is also useful for measuring thin polymer films. In each case, proper sample preparation is required, in order to obtain an acceptable measurement.

Use the transmission module for:

- Quantitative analysis
- Qualitative analysis, library search
- Transmission measurements
- Analysis of solids, liquids and gases (with appropriate sampling preparation such as KBr pellets, nujol mulls, evaporation onto salt plates, sealed and demountable liquid cells, and so forth)



Figure 1. Transmission module

Gas cell

The Agilent Cary 630 FTIR's 50 mm pathlength gas cell (Figure 3) is designed to be used with the transmission mount and provides gas phase transmission measurements. The gas cell's stainless steel body and

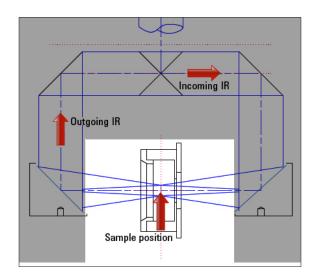


Figure 2. Transmission module optical diagram

KBr windows make it suitable for the analysis of a variety of samples across a broad spectral range.



Figure 3. Gas cell

Use the gas cell for:

- Quantitative analysis
- Qualitative analysis, library search
- · Transmission measurements

TumbIIR single pathlength transmission

The TumbIIR (Figure 4 and 5) is Agilent's unique liquid transmission sample interface. Especially suited to oil and fuel analysis, the TumbIIR delivers the sensitivity and reproducibility of a conventional sealed liquid cell, and is as easy to use and clean as an ATR. The fixed 100 µm pathlength provides quantitative analysis down to 50 ppm in liquid solutions with no sample preparation. The TumbIIR is ideal for rapid analysis of both highly viscous and volatile liquid samples.



Figure 4. TumbIIR

TumbIIR specifications:

- Quantitative analysis
 - LOD ~50 ppm (sample dependant)
- Transmission measurements
- Liquids and thin polymer films (< 50 µm thick)
- Fixed pathlength = 100 μm factory calibrated
- Repeatability = ± 0.25 μm
- · ZnSe windows
- Spectral range: 5,100–600 cm⁻¹

DialPath multipath liquid transmission

Unique DialPath technology facilitates long pathlength FTIR transmission measurement of liquids, without the inconvenience of cumbersome infrared transmission cells—no spacers, windows, or syringes needed. The DialPath interface (Figure 6) provides the same

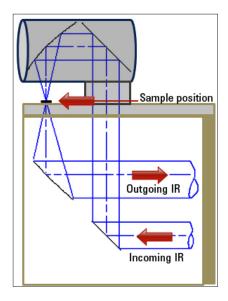


Figure 5. TumbIIR and DialPath optical diagram

sensitivity, easy to use features and repeatability of the

TumbIIR, in a design that enables you to instantaneously select any of three factory-set pathlengths. The three pathlengths deliver the versatility to handle both qualitative library matching, as well as different levels of quantitative analysis. Compared to the TumbIIR, the DialPath also has the added advantage of customizing the sensitivity to the application.



Figure 6. DialPath

DialPath specifications:

- Quantitative analysis
 - LOD ~25 ppm (sample dependant with 250 μm pathlength cell)
- Qualitative analysis, library search
- Transmission measurements

- Liquids and thin polymer films (< 50 µm thick)
- Fixed pathlengths—set of three factory-calibrated
 - \circ 30, 50, 100 µm
 - 50, 100, 250 μm
- Repeatability = ± 0.25 μm
- · ZnSe windows
- Spectral range: 5,100–600 cm⁻¹

Diamond ATR

Diamond attenuated total reflectance (ATR) (Figure 7 and 8) is the most common sample interface used in infrared spectroscopy, because it is easy to use and provides high-quality spectra with no sample preparation. ATR can be used to measure solids, liquids or gels, and the diamond ATR element is impervious to scratching and corrosion. This surface technique provides a short pathlength, and is particularly suited to experiments such as sample identification, polymer identification, co-polymer percentage determination, raw material verification, unknown sample identification and concentration determinations. It is widely used in food and beverage analysis, chemicals, pharmaceuticals, fuel and oil analysis. The Agilent Cary 630 FTIR diamond ATR interface snaps in and out in seconds with no alignment required and has a built-in pressure clamp to ensure optimum and reproducible pressure.



Figure 7. Diamond ATR

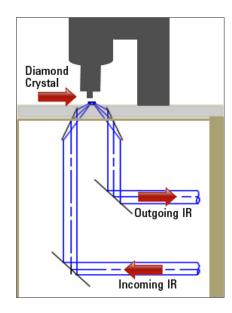


Figure 8. Diamond ATR optical diagram

Diamond ATR specifications:

- Qualitative analysis, library search
- Quantitative analysis
 - LOD ~1% (sample dependant)
- Crystal type and pressure clamp
 - Single reflection, nominal angle = 45°
 - Diamond
 - Slip clutch press provides consistent pressure
- Pathlength = $1.2 \mu m @ 1,700 \text{ cm}^{-1}$
- Analysis of solids, liquids and gels
- The Agilent Cary 630 FTIR ZnSe engine
 - Spectral range: 5,100−600 cm⁻¹
- · The Agilent Cary 630 FTIR KBr engine
 - Spectral range: 7,000–350 cm⁻¹

Diffuse reflection

The diffuse reflection sample interface (Figures 9 and 10) provides quantitative and qualitative information from powdered or solid samples. This technique delivers more sensitive quantitative analysis than ATR, while still being easy to use. Powdered samples are commonly mixed in KBr powder; the loading of the sample in KBr can be adjusted to obtain the required sensitivity. The diffuse reflection sample interface is optimized for use with the Agilent Cary 630 FTIR optical system, providing the greatest sensitivity available for fast, accurate measurements. The multi-position sample plate includes both a diffuse gold reference standard and a diffuse polystyrene standard.



Figure 9. Diffuse reflection

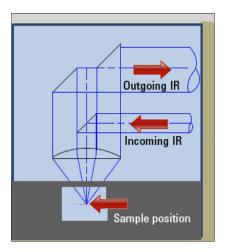


Figure 10. Diffuse reflection optical diagram

Diffuse reflection interface specifications:

- · Quantitative analysis
- · Qualitative analysis, library search
- Normal incidence, diffuse reflectance
- Sample tray
 - Gold and polystyrene reflectance standards
 - 3 sample cups
 - 10 mm sample cup size
- · Ideal for analysis of powders, tablets and solids
- ZnSe windows
 - Spectral range: 5,100–600 cm⁻¹

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