Technical Overview

Agilent
Trusted Answers

ATR Sampling Accessories for the Agilent Cary 630 FTIR Spectrometer

An easy and versatile way to perform FTIR measurements

Introduction

Attenuated total reflectance (ATR) is the most widely used sampling methodology for Fourier transform infrared (FTIR) spectroscopy. This popularity is due to its ability to quickly and easily measure a broad range of sample types, including liquids, solids, powders, semisolids, and pastes. The Agilent Cary 630 FTIR spectrometer uses a versatile modular concept, where precisely optimized sampling modules are attached to the front of the Cary 630 FTIR engine. Depending on the specific application or sample, different ATR sensors are used. Permanently aligned optics allow a wide range of modules to be swapped in seconds, with no user alignment. The Cary 630 FTIR accommodates a wide selection of ATR sensors, and features the ability to switch from one ATR sensor to another instantaneously.

For most applications, single reflection zinc selenide (ZnSe), diamond, and germanium (Ge) ATR sampling modules are available for the Cary 630 FTIR. These modules are used with a sampling press, and are excellent for analyzing solid materials, as well as liquids, pastes, and gels. The diamond sensor is highly durable, and the best choice for harder materials. The ZnSe sensor is a good choice for softer solids. The Ge sensor, with a shorter pathlength, is the best choice for highly absorbing samples. In addition, the multireflection ZnSe ATR sampling module is a great option for the analysis of liquids, pastes, and gels, where extra sensitivity is needed.

Because these sampling technologies are custom engineered for the Cary 630 FTIR, the analyst can have confidence in the results, whichever ATR sensor is chosen for their application. This custom engineering translates into the highest performance, sensitivity, and ease-of-use of any spectrometer in its class.

This technical overview reviews the ATR sensors available for the Cary 630 FTIR spectrometer (Figure 1). It describes which sensor should be selected for a specific sample type and summarizes typical applications for ATR FTIR spectroscopy with the Cary 630 FTIR.

Figure 1. The Agilent Cary 630 FTIR spectrometer can be equipped with the right sampling modules to cover all analytical needs. The diamond ATR sampling module is ideal for the FTIR analysis of solids, liquids, gels, and powders.

Operation

In ATR sampling, the infrared (IR) light travels through a crystal, is totally internally reflected at least once at the crystal-sample interface; and the reflected light travels to the FTIR detector. During the internal reflection, a part of the IR light travels into the sample, where it can be absorbed. The portion of light that enters the sample is called the evanescent wave. The penetration depth of the evanescent wave into the sample is defined by the refractive index difference between the sample and the ATR crystal. To account for different sample types and different pathlength requirements, several materials with different refractive indices are used as ATR sensors.

Single reflection ATR

Single reflection measurements allow for the light beam to be internally reflected within the crystal one time (Figure 2).

Figure 2. Single reflection ATR accessory for the Agilent Cary 630 FTIR spectrometer.

Multireflection ATR

In contrast, multireflection ATR sensors use a longer crystal, which allows the light beam to be totally reflected off the sample surface multiple times before detection (Figure 3). As a result, the IR light interacts with the sample at each of the reflection spots, providing a longer effective pathlength. This increases the sensitivity of the measurement, ideal for challenging applications that require a lower limit of detection and faster data collection.

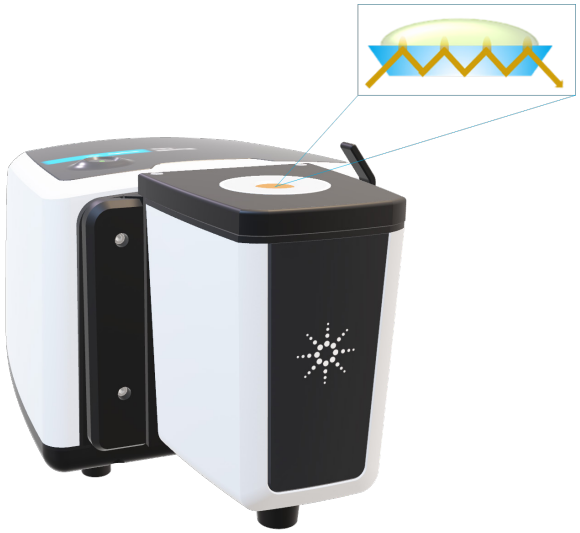

Figure 3. Multireflection ATR accessory for the Agilent Cary 630 FTIR spectrometer.

Sample measurements

To analyze liquid samples or pastes using the ATR modules, a small drop of the sample is placed onto the ATR crystal. The measurement is taken, and, after completion, the crystal can be wiped clean using a light solvent, if necessary.

To analyze powders, thin films, or other solid samples, the sample is placed onto the ATR crystal and pressed down using the swivel press to ensure optimal contact between sample and crystal. After the measurement, the sample can then be recollected, ideal for low-volume or expensive samples. The crystal can then be wiped clean using a light solvent, if necessary.

The swivel press on the single reflection ATR modules applies the optimal pressure for the given ATR sensor material. The swivel press can be rotated 360 degrees for easy access to the ATR crystal for sampling or cleaning. The press can also be detached when not required (e.g., when only liquid samples are analyzed). The multireflection ATR module is used for liquid samples only, and hence does not need a press.

ATR sensors for the Cary 630 FTIR

Whether your applications require the single reflection ZnSe, diamond or Ge, or the multireflection ZnSe, the Cary 630 FTIR delivers class-leading performance. Unlike other FTIR spectrometers that use third-party accessories, sampling technology for the Cary 630 FTIR is custom designed by Agilent engineers, and perfectly matched to the optical characteristics of the spectrometer. The rugged, no-alignment design permits instantaneous exchange of these ATR sampling technologies, so that any sample type you might face will readily be accommodated. The different ATR modules for the Cary 630 FTIR spectrometer are outlined in Table 1.

Table 1. ATR modules for the Agilent Cary 630 FTIR spectrometer.

ATR Module	Wavelength Range	Effective Pathlength	Sample Type	Swivel Press
Single Reflection ZnSe ATR Module	5,100 to 600 cm^{-1}	1.1 µm at 4,000 cm^{-1} 2.6 µm at 1,700 cm^{-1} 7.3 µm at 600 cm^{-1}	Neat or concentrated; soft solids, pastes, gels, liquids; not strong acids or bases	Yes
Multireflection ZnSe ATR Module	5,100 to 600 cm^{-1}	5.5 µm at 4,000 cm^{-1} 13.0 µm at 1,700 cm^{-1} 36.5 µm at 600 cm^{-1}	Lower-level components, more dilute solutions; pastes, gels, liquids; not strong acids or bases	No
Single Reflection Di ATR Module	6,300 to 350 cm^{-1} [a] 5,100 to 600 cm^{-1} [b]	1.1 µm at 4,000 cm^{-1} 2.6 µm at 1,700 cm^{-1} 7.3 µm at 600 cm^{-1}	Hard solids, particulates, polymers, pastes, liquids; all pH ranges	Yes
Single Reflection Ge ATR Module	5,100 to 600 cm^{-1}	0.15 µm at 4,000 cm^{-1} 0.36 µm at 1,700 cm^{-1} 1.02 µm at 600 cm^{-1}	Carbon black-filled polymers	Yes

[a] Cary 630 FTIR engine with KBr optics
[b] Cary 630 FTIR engine with ZnSe optics

Zinc selenide (ZnSe) ATR modules

ZnSe is a semiconductor-type material that has been used for many years as an ATR sensing element. It is relatively hard, has a broad wavelength range, and is insoluble in water. For this reason, it is an excellent choice for pliable solids, pastes, gels, and liquids. It can be used to analyze aqueous solutions in the pH 5 to 9 range. The Cary 630 FTIR has two variations of ZnSe sampling technology available: multireflection and single reflection sensors.

Single reflection ZnSe ATR module

Because of the relatively short pathlength of infrared light into the sample, this sensor is ideal for neat samples; that is, samples that are relatively concentrated or pure. The single reflection ZnSe module is a good choice for identification of softer materials, as well as viscous liquids. When measuring solids, such as polymer films, it can be used with the Cary 630 FTIR. The ZnSe ATR module is depicted in Figure 4.

Figure 4. Zinc selenide ATR module for the Agilent Cary 630 FTIR spectrometer.

Multireflection ZnSe ATR module

The exceptional performance of the Cary 630 FTIR, combined with the increased pathlength of the multireflection ATR sensor, demonstrates a spectrometer system of unsurpassed sensitivity. Lower concentration components in pastes, gels and liquids can be measured. Solutes in diluted or concentrated aqueous solutions in the pH range of 5 to 9 can also be analyzed. Since the multireflection sensor is recessed slightly in its stainless steel holder, it is ideal for analyzing nonviscous liquid samples. This sensor is a superior choice when qualitative or quantitative measurements are required. It is not recommended for use with solid materials, since it is not used with the pressure clamp. The multireflection ZnSe ATR module is depicted in Figure 5.

Figure 5. Multireflection zinc selenide ATR accessory for the Agilent Cary 630 FTIR spectrometer.

Single reflection diamond (Di) ATR module

The use of diamond as an ATR material has revolutionized FTIR sampling. Harder samples, such as minerals and harder polymers, can be readily analyzed with this sensor because it cannot be scratched. The diamond ATR is also resistant to strong acids and bases, and is excellent for measuring high- or low-pH aqueous solutions. The diamond ATR for the Cary 630 FTIR is a single reflection sensor used with the sample press to enable good contact between the material to be analyzed and the diamond surface. As a single reflection sensor, it is ideal for neat or pure substances that are particulates, powders, or other hard materials. The unique design of the diamond ATR for the Cary 630 FTIR results in high-energy throughput, and the combination of the spectrometer and diamond sampling technology typically outperforms other routine FTIR systems. The single reflection diamond ATR module is depicted in Figure 6.

Figure 6. Single reflection diamond ATR module for the Agilent Cary 630 FTIR spectrometer.

Single reflection germanium (Ge) ATR module

Ge is a brittle, hard, semimetallic element with a high index of refraction, which yields a shorter depth of penetration for infrared radiation. The Cary 630 FTIR uses a single reflection Ge ATR element, which is an excellent choice for analyzing materials that are highly absorbing, or have highly scattering components. Samples such as polymers containing carbon black are often analyzed with the Ge ATR. O-rings, gaskets, and black rubber tires are all examples of materials that are well suited to analysis by the Cary 630 FTIR Ge ATR sampling module. The single reflection germanium ATR module is depicted in Figure 7.

Figure 7. Single reflection germanium ATR module for the Agilent Cary 630 FTIR spectrometer.

Application examples

ATR analysis is used extensively throughout industry and academia in a wide range of applications. Agilent provides many application notes demonstrating the use of the ATR modules and providing comparisons to other available sampling techniques. A summary of some of the currently available application notes follow. A full suite of applications can be found on the Agilent website.

Pharmaceutical Packaging Materials Quality Control and USP Chapter <661.1> Compliance

This study focuses on the use of FTIR spectroscopy for the analysis of polymers used in pharmaceutical packaging. A diamond ATR module was used to determine the differences between name brand and generic packaging, as well as to demonstrate the detection of counterfeit pharmaceutical products. The application of the Cary 630 FTIR to USP pharmaceutical packaging regulations, outlined in chapter <661.1> Plastic Materials of Construction, has also been demonstrated.

Download the full application note.

Quick and Real-Time Potency Determination of Cannabinoids Using FTIR Spectroscopy

In this study, the potency of different cannabis products by their respective THC content was determined. A Cary 630 FTIR with the diamond ATR module was used to analyze multiple cannabis sample types, such as extracts, concentrates, and distillates, with no sample preparation required. The use of the Cary 630 FTIR with the diamond ATR module offers fast and nondestructive analysis in production workflows, for example, increasing productivity and throughput in sample analysis.

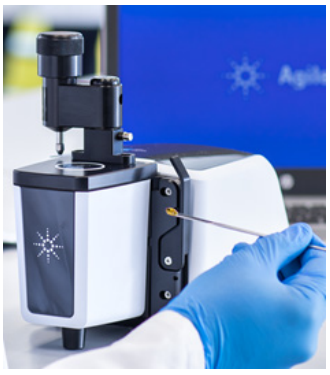

Figure 8. Spectra of cannabis concentrates and distillates were recorded without any sample preparation, using an Agilent Cary 630 FTIR spectrometer with Di ATR module.

Download the full application note.

Determination of Sucrose Levels in Infant Cereals Using the Agilent Cary 630 ATR-FTIR Spectrometer

This study determined the amount of sugar present in multiple brands of breakfast cereal marketed towards children. The cereal samples were ground into a powder and directly placed onto the diamond ATR module to quantify the sucrose concentrations. The results obtained with the Cary 630 FTIR yielded very good correlation with HPLC data from the same samples.

Download the full application note.

The Agilent Cary 630 FTIR Spectrometer Quickly Identifies and Qualifies Pharmaceuticals

This study highlights the sensitivity of ATR-FTIR analysis of pharmaceutical products when classifying and assessing the purity of specific raw ingredients. A Cary 630 FTIR with the diamond ATR sampling module was used to analyze samples of pure and contaminated acetylsalicylic acid. The unique, logic-setting capabilities in the Agilent MicroLab software allow identification, qualification and easy distinction between different qualities of raw materials and ingredients in pharmaceutical applications.

Download the full application note.

Fuel Blend Analysis Using the Cary 630 Spectrometer and 5 Bounce ZnSe ATR Accessory

In this study, the composition of various fuel blends was determined. A Cary 630 FTIR with the multireflection ZnSe ATR module was used to reproducibly analyze diesel/butanol blends, with no need for sample preparation. This provided information about quantity and mixing phenomena at the molecular level.

Download the full application note.

Analysis of Alcohol Levels in Hand Sanitizer

A Cary 630 FTIR with the diamond ATR module was used to quantify the alcohol content in hand sanitizers. The MicroLab software was used to develop a routine quality control (QC) method that automatically identified the type of alcohol, and accurately determined the alcohol concentration.

Figure 9. The neat hand sanitizer sample is placed onto the ATR accessory, fitted to the Agilent Cary 630 FTIR. The Agilent MicroLab software uses picture-guided workflows to help users follow each step of the measurement including cleaning and sampling.

Download application flyer.

Detection of Counterfeit Pharmaceuticals

In this study, the Cary 630 FTIR with the diamond ATR module was used to collect IR spectra of authentic pharmaceutical tablets and placebo samples. The spectra were library matched against a comprehensive IR library using the MicroLab software. For the important and frequently counterfeited pharmaceuticals, ethambutol hydrochloride and cefuroxime axetil, it was found that the Cary 630 FTIR reliably differentiated authentic samples from counterfeit samples.

Download the full application note.

Automated FT-IR Screening Method for Cocaine Identification in Seized Drug Samples

A Cary 630 FTIR with the diamond ATR module was used to collect IR spectra of seized cocaine samples. Together with an automated method in the MicroLab software, the Cary 630 was able to identify the presence of cocaine, showing good correlation with cocaine concentration determined by HPLC. The instrument-software configuration proved to be suitable as a nondestructive way to prescreen seized drugs suspected to contain cocaine.

Download the full application note.

www.agilent.com/chem/cary630

DE74554071

Printed in the USA, January 19, 2022
5991-6858EN

Agilent
Trusted Answers