APPLICATION NOTE

FT-IR Spectroscopy

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Tri-Range Applications of the Spectrum 3 Infrared Spectrometer

Introduction

The PerkinElmer Spectrum 3™ infrared spectrometer (Figure 1) gives the user the opportunity to measure across the Near, Mid and Far-infrared regions with one high-performance instrument. Using automatic switching of optical components, users are able to obtain high quality spectra in all three ranges. This allows one instrument to fulfil a wide assortment of applications in multiple areas of analysis.

Figure 1. Spectrum 3 FT-IR spectrometer.

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Glass Analysis

Spectroscopic analysis of glass used in both construction and the automotive industry is required to determine several very important parameters, a key example of which is the emissivity of glass. This is a key property in investigating the energy-saving capability of glass and the measurement is carried out in the Mid- and Far-infrared regions. The standard procedure for the determination of the emissivity of glass involves single beam measurements of the glass sample, a reference mirror and an empty background spectrum at 30 standard wavelengths. These wavelengths range from 5.5 µm to 50 µm (1818 – 200 cm^{-1}). The selected wavelengths are specified by three international standards; EN 673[1], EN 12898[2] and ISO 10292[3]. The ability of the PerkinElmer Spectrum 3 to measure in multiple regions of the infrared spectrum gives the user the potential to not only determine the emissivity of glass, but investigate other properties demonstrated in the near-infrared region.

Experimental

A 3 mm thick sample of float glass was measured using the PerkinElmer Spectrum 3 FT-IR spectrometer with the OMT Specular Reflectance set. For each of the glass, reference mirror and empty background, three measurements were made. The three spectral ranges used are shown in Table 1.

Table 1. Spectral Ranges used for sample measurement.

Range Mode	Spectral Range (cm^{-1})
Far-Infrared	700 – 100
Mid-Infrared	5000 – 450
Near-Infrared	10000 - 5000

The Spectrum 3 has the ability to scan the entire infrared range from Near-infrared to Far-infrared with automatic switching of optical components. The new 'Merge' function in Spectrum 10 may be used to combine spectra from different ranges to produce a single spectrum which covers the entire infrared region.

Results and Discussion

The merged spectra of the glass sample, the reference mirror and the background spectrum are shown in Figure 2.

The emissivity of glass is determined from the reflectance at room temperature using equation

$$\varepsilon_n = 1 - R_n \qquad \text{Equation 1}$$

Where ε_n is the emissivity at normal incidence and R_n is the reflectance at normal incidence. The reflectance is calculated as an average of 30 spectral reflectance values measured at specified wavelengths between 5.5 and 50 µm (1818.8 – 200.0 cm^{-1}).

First, both the glass and mirror measurements are corrected using the empty background spectrum. The reflectance is then determined at each wavelength by dividing the corrected glass value by the corrected mirror value. The overall reflectance is calculated by finding the mean of the reflectance at each of the 30 specified wavelengths. This may be carried out using the 'equations' function of Spectrum 10 software or with an external data analysis application.

Using this calculation, the emissivity of the 3 mm float glass sample was found to be approximately 0.95, in agreement with the commonly accepted values for glass emissivity at room temperature.[4]

In addition to emissivity calculations, the near-infrared region of the spectrum can also provide information regarding thermal transmittance. Glass designed specifically for absorbing heat will generally have a higher reflectance in the near-infrared region. For example, the standard 3 mm float glass shows low reflectance in the near-infrared region and thus is not heat absorbing glass.

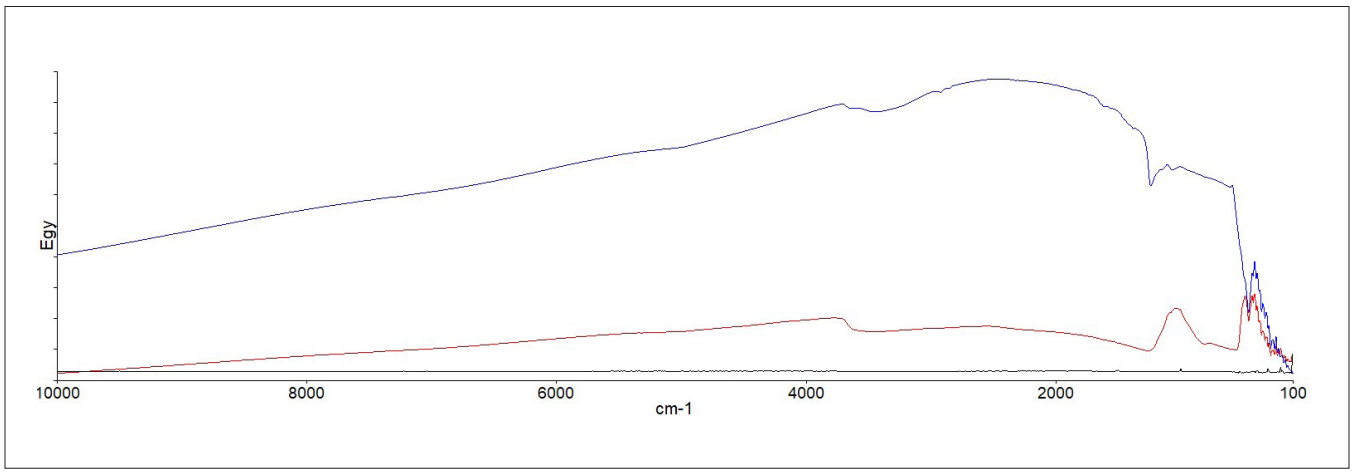

Figure 2. Tri-range spectra of glass (Red), a reference mirror (Blue) and no sample (Black).

Pharmaceutical Analysis

Infrared spectroscopy has become a standard method in the analysis of pharmaceutical materials with the advantage of being fast and non-destructive. The near and mid-infrared regions are the most commonly used as they can provide detailed information about chemical structure (mid-infrared) and physicochemical properties (near-infrared). However, there are also some research applications of far-infrared spectroscopy such as the determination of solid-state structural properties and polymorphism studies.

A pharmaceutical product which demonstrates strong spectral features in all three regions is clotrimazole, an anti-fungal medication, the chemical structure of which is shown in Figure 3.

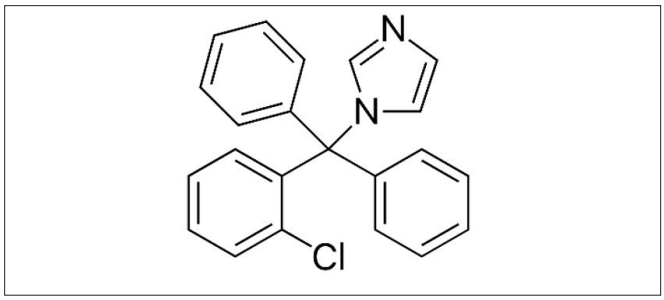

Figure 3. Chemical structure of clotrimazole.

Experimental

A sample of clotrimazole was measured in all three regions of the infrared spectrum, using automatic switching of optical components, and the spectral parameters shown in Table 2. Measurements in the mid and far-infrared regions were carried out using an ATR accessory, while measurements in the near-infrared region were carried out using the NIRAll accessory.

Results and Discussion

The tri-range spectrum of clotrimazole is shown in Figure 4.

Having the ability to measure a pharmaceutical sample in all three ranges of the infrared region allows analysts to simultaneously investigate multiple properties and obtain more significant information on the sample. The Spectrum 3 tri-range infrared spectrometer provides an all-in-one solution for pharmaceutical analysis with the Spectrum 10 enhanced security (ES) software allowing users to collect and store data according to the FDA 21 CFR part 11 regulations.

Industrial Antioxidant Analysis

Analysis of materials used in industrial settings is of utmost importance to limit downtime of machinery and ensure supply chain efficiency. One example of a material used across a wide range of industries is butylated hydroxytoluene (BHT). This is used as an antioxidant in transformer oil, fuels, rubber, pharmaceuticals and some food products. The chemical structure of BHT is shown in Figure 5.

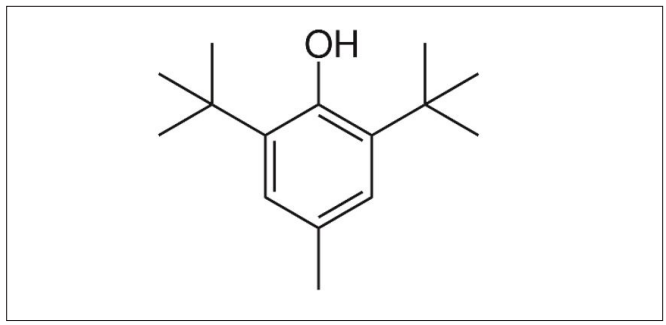

Figure 5. Chemical structure of BHT.

Table 2. Spectral parameters used for the measurements of clotrimazole.

Infrared Region	Parameter	Value
Far-infrared	Range	700 – 30 cm^{-1}
	Resolution	8 cm^{-1}
	Number of Scans	128
Mid-infrared	Range	5000 – 450 cm^{-1}
	Resolution	4 cm^{-1}
	Number of Scans	16
Near-infrared	Range	10000 – 4000 cm^{-1}
	Resolution	4 cm-1
	Number of Scans	64

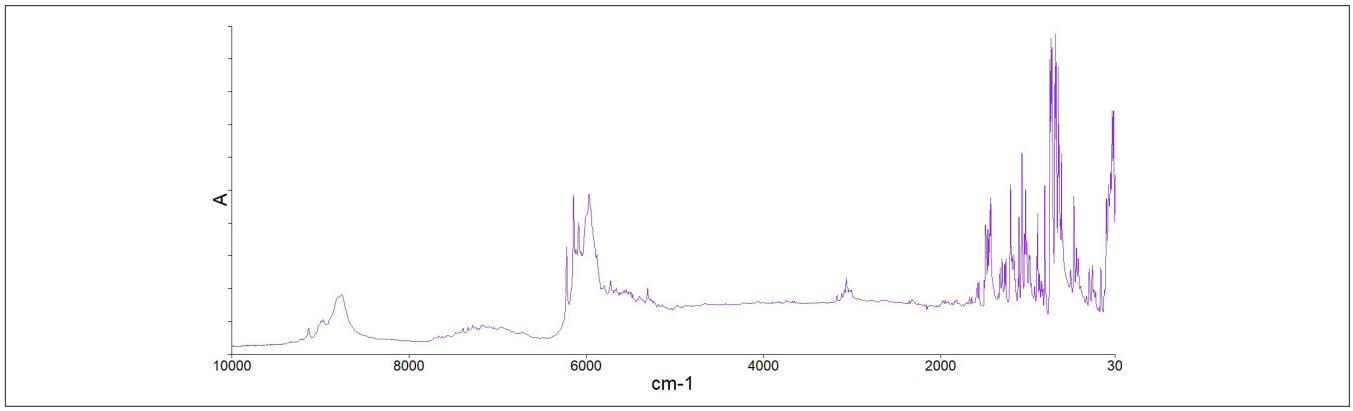

Figure 4. Tri-range spectrum of clotrimazole.

Experimental

The Spectrum 3 was used to measure a sample of BHT in all three regions of the infrared spectrum using the spectral parameters shown in Table 3. Measurements in the mid and far-infrared regions were carried out using an ATR accessory, while measurements in the near-infrared region were carried out using the NIRAll accessory.

Results

A tri-range spectrum of BHT is shown in Figure 6.

As with other materials, different areas of the infrared spectrum of BHT serve different purposes. For example, the sharp peak at around 3700 cm^{-1} has been used for quantitative determination of BHT in food samples such as edible oils.[5]

One previously mentioned use of BHT is as an antioxidant in rubber and other polymers. Far-infrared provides an incredibly useful tool for measurement of polymers due to strong intermolecular vibrations and weak intramolecular vibrations falling in this range. This region has been used to successfully quantify BHT as an additive in polymer samples as lattice vibrations of some polymers fall between 50 – 100 cm^{-1}.[6]

Summary

The Spectrum 3 FT-IR spectrometer provides an all-in-one infrared solution for a wide variety of markets. Automatic switching of optical components allows users to seamlessly collect data in the near, mid and far-infrared regions.

For pharmaceutical samples, Spectrum 10 enhanced security (ES) software includes features such as audit trails and electronic signature points for compliance with FDA 21 CFR part 11 regulations.

The large selection of sampling accessories and software features makes the Spectrum 3 an incredibly versatile tool in any analytical laboratory.

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	Resolution	4 cm^{-1}
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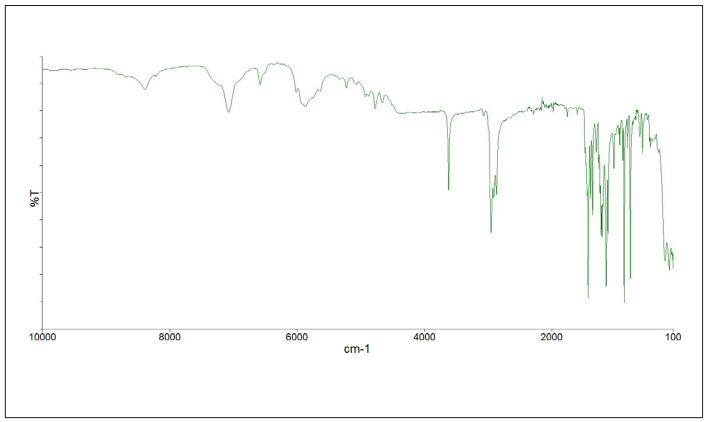

Figure 6. Tri-range spectrum of BHT.

References

1. European Standard 673:2011 – Determination of Thermal Transmittance Value: Glass in Building
2. European Standard 12898:2019 – Determination of the Emissivity: Glass in Building
3. ISO Standard 10292 (1994) – Glass in Building – Determination of steady-state U values (Thermal Transmittance) of Multiple Glazing.
4. R. Gardon, The Emissivity of Transparent Materials, J. Am. Ceram. Soc., 1956, 39, 278-87
5. W. Ammawath, Y. B. Che Man, R. B. Abdul Rahman, B. S. Baharin, A Fourier Transform Infrared Spectroscopic Method for Determining Butylated Hydroxytoluene in Palm Olein and Palm Oil, J. Am. Oil Chem. Soc., 2006, 83, 187-191
6. T. Ogishima, C. Kuroda, N. Hirai, Y. Ohki, Identification and quantification of phenol-type antioxidants in low-density polyethylene by broadband far-infrared spectroscopy, Polym. Test., 2019, 76, 10-18

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