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VOLUME 6 June 2009
INDIA ANALYTICAL SCIENCES

Dear customers,

It is our immense pleasure that we are receiving your feed backs and suggestions through this FRESH communications. We are trying our level best to incorporate different products and applications related to its usage in various industries and market segments. Our efforts are towards to keep you updated about the solutions available from PerkinElmer.

The newer techniques of analysis to get the precise and accurate results through the methodology used by experts and users worldwide are being synopsized for your quick glance. Your particular interest in products or solutions can be directed to us by email any time. We shall be pleased to interact with you for any techno commercial assistance possible from our end.

Team Marketing-India

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Gaining high resolution measurements of optical filters in the NIR range with the LAMBDA 1050 UV/Vis/NIR

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The most common measurement for optical filter quality testing is done in transmission mode with the results typically displayed in %T. The measurement may be performed at any angle of incidence required by the filter design. The angle of incidence light used can range from normal to greater than 60˚ with 45˚ being one of the most common requirements. When measurements are to be performed at angles other than normal, typically considered to be between 5˚- 8˚, polarization affects need to be considered.

Author Christopher Lynch PerkinElmer Life and Analytical Sciences 710 Bridgeport Avenue Shelton, CT 06484 USA. All modern high performance spectrophotometers use reflective gratings to disperse the source energy into specific wavelengths. This process will create strongly polarized light which will be used for sample analysis.For this reason, to achieve the truest results a depolarizer should be used at angles higher than 8˚ to measure a sample under random polarization conditions. If specific angles of polarization are required by the analysis, a polarizer should be added to allow the specific angle of polarization to be measured. This could range from S to P polarization or any angle between.

Optical filter types and design requirements vary widely from simple neutral density filters to the most complex multi layer bandpass designs. All have specific design requirements and all require spectroscopic analysis to validate designs and for quality control in manufacturing.

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Typical design requirements of several common types of filters are shown in Figures 1 and 2

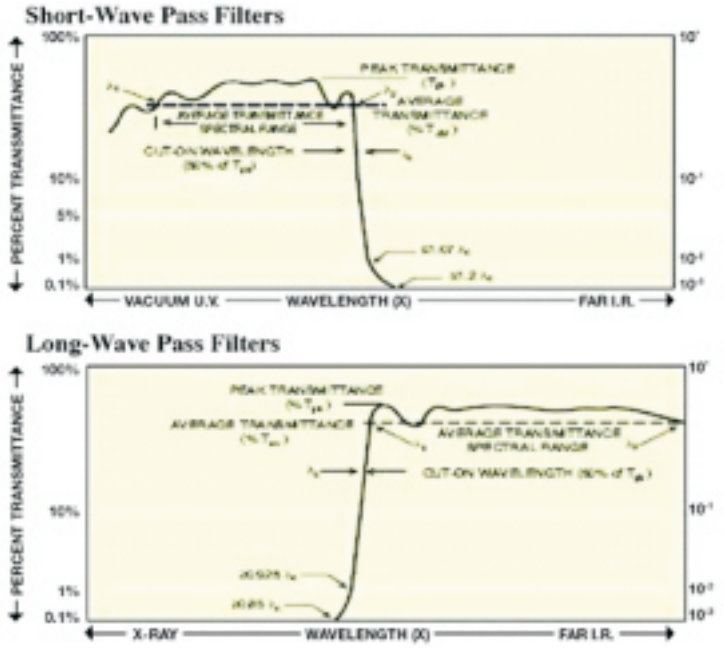

Figure 1. Typical filter design requirements.

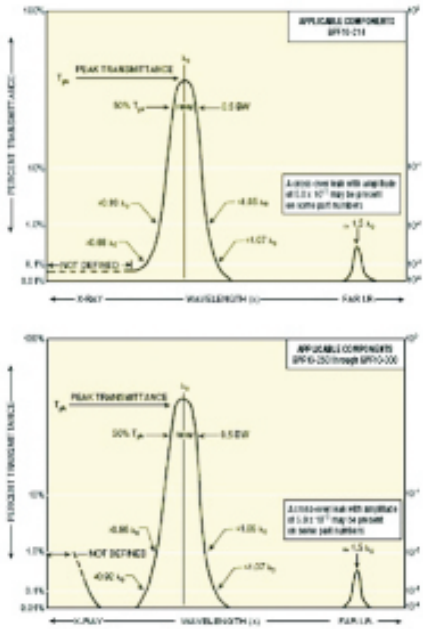

Figure 2. Typical bandpass filter response characteristics.

Optical filters have many functions, including color correction, used to improve color balance in many optical systems, to neutral density filters that produce specific reduction in the level of transmitted light. Thin film filters used in laser based systems have more demanding requirements typically requiring very narrow bandpass and very high out-of-band blocking. The characterization of these diverse optical filter types is critical from design through manufacturing, providing validation of a specific design and the necessary means to perform QC/QA of finished products.

The LAMBDA™ 1050 utilizes three detectors for optimum energy detection across the entire wavelength range of the instrument, 175-3300 nm: A gridless PMT for detec- tion in the UV/Vis, a high sensitivity Peltier cooled InGaAs detector for use in the 800-2600 nm region and a Peltier cooled PbS detector for the range from 2500-3300 nm. This offers the best combination of scanning speed and photodynamic range that can be achieved on a given sample type.

In determining the best method for the analysis of a specific filter several factors need to be considered. The design specification will provide the majority of the information necessary for a spectroscopic analysis. Wavelength range, bandpass, transmission and out of band blocking will all be defined and need to be taken into consideration.

Key to a successful analysis is translating the specifica- tion requirements into the method setup parameter used by the spectrophotometer. Choosing high resolution and a very small data interval for a filter type which has no spectral features will simply waste valuable time. In general the resolution needs to be 4-10 times smaller than the bandpass of a given component to provide sufficient data to validate a component.

Photodynamic range is another important consideration. The level of absorbance of a given material will impact how a method is developed and how fast a sample can be scanned. A notch filter with very high absorbing re- quirements may require reference

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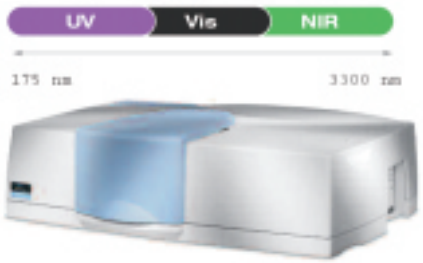
beam attenuation to be used to achieve accurate data on the true performance of a filter. Simply knowing the design goal of the component will determine the method details for a successful analysis to be performed.



Lambda 1050

Measuring a laser bandpass filter with a center wave- length of 1064 nm and a bandpass of 1 nm is a common requirement. A filter of this type will require slit resolution of 0.25 nm to achieve sufficient resolution to determine its characteristics. The LAMBDA 1050 using an InGaAs detector in the NIR region allows both high resolution and wider photodynamic range improving both signal- to-noise and speed of analysis over a conventional PbS detector.

With the proper selection of operating parameters completed and baseline data acquired, sample measurement can now be performed. Figure 3 shows the typical response of a laser line filter. Excellent signal-to-noise is seen in the spectra even at very high resolution.

Figure 4 shows the same filter measured on a typical spectrophotometer using a PbS (Lead Sulfide) detector in the NIR region. Poor signal-to-noise is due to low sensitivity of the detector at very high resolution.

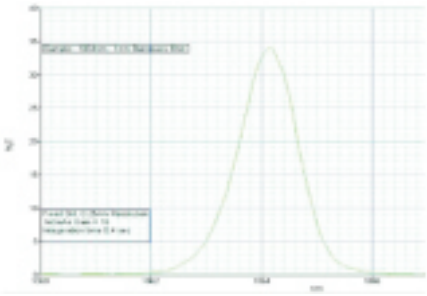


Figure 3. 1 nm Laser line filter measured with 0.25 nm resolution on a LAMBDA 1050 using an InGaAs detector

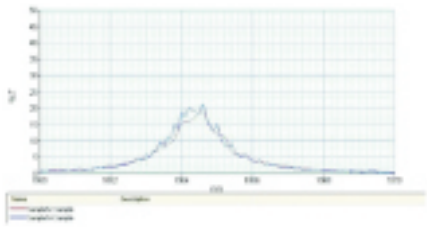


Figure 4. 1 nm Laser line filter measured with 0.25 nm resolution on a LAMBDA 950 using a PbS detector.

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Verification of Tartrates used in the wine industry by FTNIR

Introduction

Tartrates are used extensively in the wine industry to clear the 'muddiness' or sediment before bottling (materials used in this process are called 'finings'). This application example describes the use of NIR to discriminate between two typical samples, based on calcium tartrate and potassium bitartrate.

Why choose NIR for this application?

There are many different types of finings used in the beer, cider and wine making industries. These range from aluminosilicates to gelatin, egg white, fish glue and polyvinyl-polypyrrolidone or polyclar. Since many of the finings are the bi-products of other processes, they are often of extremely variable quality. For example, particle size distribu- tion varied widely from sample to sample. NIR reflectance is an ideal method for finings analysis since no sample preparation is involved (other than transferring a few grams of sample to a glass vial), and both chemical and physical property information is available.

Experimental

Four samples were provided for analysis. Two of type 1 (calcium tartrate based) and two of type 2 (potassium bitartrate/calcium tartrate mixture). The samples were prepared for analysis by placing a few grams of sample into a glass vial. NIR spectra of the four samples were generated using a PerkinElmer FT-NIR System equipped with in-board Reflection Accessory. Typical scan conditions:– 11000-3800 cm^{-1}, 8 cm^{-1} resolution, 16 scans.

Results

Type 1 sample spectra (based on calcium tartrate) are shown in Figure 1. The spectra are similar; the variation in baseline indicating a difference in particle size distribution between the two samples which can be removed by converting to a second derivative as shown in Figure 2. It is also easier to see the bands due to the calcium tartrate itself in the second derivative rather than the water bands which dominate Figure 1.
Type 2 samples (based on a potassium bitartrate/calcium tartrate mixture) are shown in Figure 3. Note the strong band at around 4700 cm-1 indicative of tartrate.

Analysis

It is a simple task to create a short COMPARE™ library and use it to verify the identification of test samples. This was done for the finings samples, see table 1. The results for a test sample of type 1 (batch 5412) and type 2 (batch 5431) indicate that the task of separating type 1 and type 2 samples is easily accom- plished. Resolution, intensity, noise and water blanking COMPARE filters were switched on to minimize unwanted spectral and sample interferences.

Spectrum 400 FTIR/FTNIR

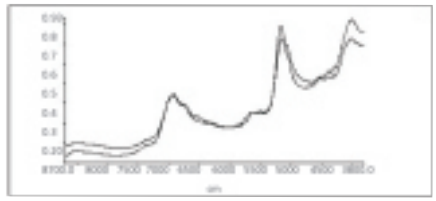

Figure 1: Sample spectra based on calcium tartrate.

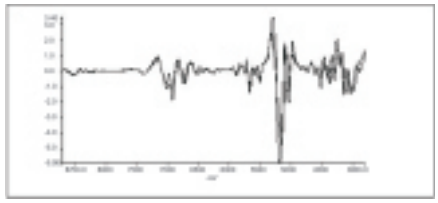

Figure 2: Second derivative of sample spectra based on calcium tartrate.

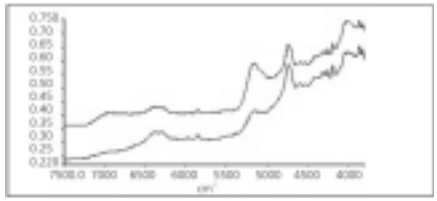

Figure 3: Sample spectra based on potassium bitartrate/calcium tartrate.

Compare – 5412.SP			
File	Correlation	Factor	Comments
type1.sp	0.9980	0.9714	Batch 5412
type1a.sp	0.9615	0.8213	Batch 5396
type2a.sp	0.1151	0.1506	Batch 5430
type2.sp	0.0679	-0.0754	Batch 5431

Compare – 5431.SP			
File	Correlation	Factor	Comments
type2.sp	0.9976	0.9330	Batch 5431
type2a.sp	0.9588	1.0889	Batch 5430
type1a.sp	0.1000	-0.0693	Batch 5396
type1.sp	0.0718	-0.0575	Batch 5412

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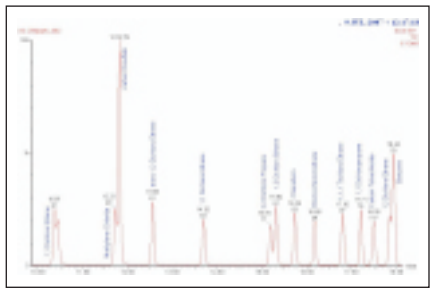
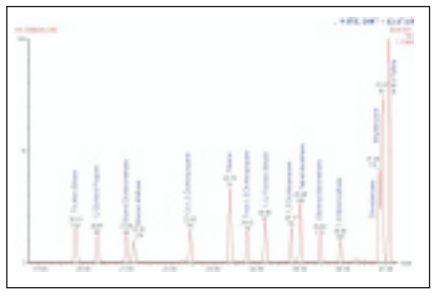
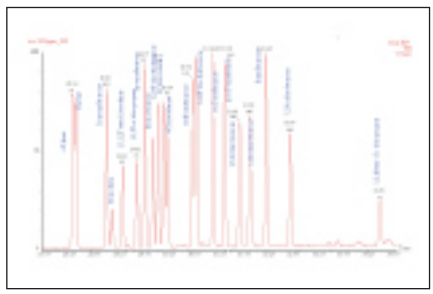
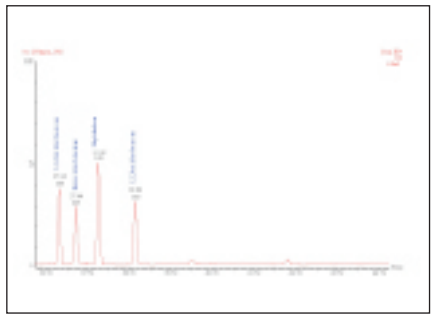
Analysis of Volatile Organic Compounds by ATD-GCMS









Introduction

Automated thermal desorber (ATD) has been widely used for air quality monitoring in a confined places and for outdoor. Volatile organic compounds (VOC) are common compounds analyzed using ATD hyphenated with gas chromatograph (GC) and gas chromatograph-mass spectrometer (GCMS). This brief paper outlines the conditions of the VOC analysis with ATD. TurboMatrix Thermal Desorbers can solve problems and provide answers for difficult applications in many industries. Thermal desorption is instrumental for monitoring environmental levels of organic pollutants and simplifies many routine determinations of volatiles in products such as disc drives and wafers in semiconductor manufacture, polymers, pharmaceuticals, foods and paints.

Air toxics analysis

The PerkinElmer Air Toxics Analyzer integrates several analytical techniques into a single, unified system solution, performing tube-based sampling in accordance with established methodologies such as U.S. EPA Method TO-17. Tube-based sampling offers greater convenience as well as some analytical advantages over traditional canister-based analysis. Comprised of a TurboMatrix Thermal Desorber, Clarus Gas Chromatograph and Clarus Mass Spectrometer (MS), the system provides outstanding analytical performance as well as several unique features to simplify and speed analysis. Thermal desorbers with PPC incorporate a powerful dry-purge technique that allows for analysis under extreme levels of humidity/moisture, while a unique capability of the Clarus GC/MS offers the benefits of both full scan and single ion monitoring simultaneously within a single analytical run.

Experiments

Instrument used was Clarus 600 GCMS with direct injection with Helium as carrier from ATD model Turbomatrix 650 with cold trap and NIOSH 2549 mix bed tube. Chromatograms in sequence of time shown below:

Observation/Conclusion

ATD and GCMS were demonstrated to be capable of analyzing volatile organic substances for environmental emission, occupational hazardous chemical exposure and indoor air quality monitoring.

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Determination of metal impurities in pharmaceutical samples by Inductive Coupled Plasma Optical Emission Spectrometer (ICP-OES)

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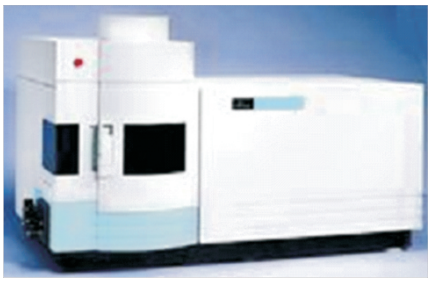

Introduction

The study carried out to find out impurities in some of the pharmaceutical samples The samples selected for the analysis are:

1. Omeprazole: Omeprazole , used for short-term treatment of active duodenal ulcer in adults.

2. Esomeprazole: It is used in the treatment of dyspepsia, peptic ulcer disease (PUD), gastroesophageal reflux disease (GORD/GERD) and Zollinger-Ellison syndrome. Esomeprazole provides improved efficacy, in terms of stomach acid control, over racemic omeprazole.

3. Sevelamer Hydrochloride: Sevelamer Hydrochloride is indicated for the control of hyperphosphataemia in adult patients receiving haemodialysis or peritoneal dialysis.

4. Albendazole: Albendazole is a member of the benzimidazole compounds used as a drug indicated for the treatment of a variety of worm infestations. The suitable method for the analysis of these samples was defined by the PerkinElmer Optima 7000 ICP spectrometer. The sample preparation involved digestion procedure. The convention method of digestion was causing the loss of elements due to open vessel. PerkinElmer Multiwave 3000 microwave digestion enabled the faster digestion without loss of element in matrix.

Standard and sample preparation:

1.Standard solutions are prepared in 2% Nitric Acid solution.
2.Sample preparation: Samples were digested by using Microwave digestion system (Multiwave 3000) as per the method mentioned in table 1. The multiwave represents a microwave based sample preparation system allows the rapid and complete wet-chemical decomposition of organic and inorganic sample materials in closed TFM or quartz glass tube reaction vessels at high pressure and high temperature.

Sample	Weight taken	Reagents used
Omeprazole	0.1519 g	4 mL HNO_3, 1 mL $HClO_4$
Esomeprazole	0.0853 g	4 mL HNO_3, 1 mL $HClO_4$
Sevelamer Hydrochloride	0.1736 g	4 mL HNO_3, 1 mL $HClO_4$
Albendazole	0.1009 g	4 mL HNO_3, 1 mL $HClO_4$

Table 1

Fig. 2 Multiave 3000

Fig. 3 vessels

After digestion program is complete, clear solutions obtained were transferred in standard volumetric flasks, volume was made up to 25 ml and analysis was carried out.

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Results:

Recovery study: 0.5 mg/l of mix standard solution spiked in a 0.10 gm of Albendazole sample and results found are showing following recovery.

Sr. No.	Element	Recovery %
1	As	84.6
2	Cd	98.4
3	Co	94
4	Cr	100.2
5	Cu	102
6	Fe	99.2
7	Mn	98.8
8	Mo	96.2
9	Ni	100.4
10	Pb	93.2
11	Se	89.8
12	Zn	94.8
13	Be	89.6
14	Sb	87.6

Conclusion:

1. Complete digestion of pharmaceutical samples can be carried by Multiwave digestion system without any loss of elements. It is programmable, safe and easy to operate.
2. From the observed results it is concluded that the analysis of pharmaceutical sample can be carried out by the ICP-OES for most of the metals at very low level concentration.

Sr. No.	Element	Sample 1 Omeprazole cap	Sample 2 Esomeprazole	Sample 3 Renagel sevelamer HCl	Sample 4 Albendazole	Unit
1	As	<0.64	<0.64	<0.64	<0.64	ppm
2	Cd	<0.06	<0.06	<0.06	0.08	ppm
3	Co	<0.08	<0.08	<0.08	<0.08	ppm
4	Cr	<0.09	<0.09	<0.09	<0.09	ppm
5	Cu	<0.04	<0.04	<0.04	<0.04	ppm
6	Fe	9.54	28.42	9.42	14.90	ppm
7	Mn	0.66	5.86	<0.03	<0.03	ppm
8	Mo	<0.08	<0.08	<0.08	<0.08	ppm
9	Ni	<0.14	<0.14	<0.14	<0.14	ppm
10	Pb	<0.34	<0.34	<0.34	<0.34	ppm
11	Se	<1.59	<1.59	<1.59	<1.59	ppm
12	Zn	<0.19	1.46	1.98	0.49	ppm
13	Be	<0.005	<0.005	<0.005	<0.005	ppm
14	Sb	<1.27	<1.27	<1.27	<1.27	ppm