

HUMAN HEALTH

ENVIRONMENTAL HEALTH

Fresh

VOLUME 47 | Summer Edition, 2015 | Environmental Health

FOR A BETTER
FUTURE

Helping customers accelerate insights to better protect our environment,
our food supply and the health of our families

We will continue to build upon our over 75-year legacy of scientific
innovation, continue to anticipate challenges in human and environmental
health and help customers navigate these challenges through our solutions.



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A message from the President, Environment Health



Srinivas Addepalli

Dear readers,

This year, we witness launch of wide range of new products, application notes and recognition from reputed world bodies, which we would like to share with you.

We introduce products in the Inorganic, Material Characterization and Chromatography areas of analytical instrumentation.

Inorganic:

PinAAcle 500 AAS, World's first Flame AA Spectrometer with complete corrosive resistant sample introduction system.

Material Characterization:

Spotlight 150i and 200i FTIR-Microscopy, intelligently automated with the compact Spectrum-2 FTIR and Frontier FTIR/NIR for routine analysis to challenging applications in single point mode, line scan and mapping mode.

Lambda 265, 365 and 465 UV-Vis Spectrometers, an up gradation of our best-selling robust Lambda UV-Vis systems with cutting edge technology.

Chromatography:

We have collaborated with Waters' for Empower software solution with 21 CFR compliance to provide a seamless connectivity for our GC, HS and GCMS. To serve our applied customers (non-pharma) better and provide end to end solution, we have launched Altus™ HPLC and UPLC in collaboration with Waters Inc.

Thanks once again for your support and making us your research partner.

I solicit your feedback on this edition.

Sincerely

Srinivas Addepalli

President, Environment Health
PerkinElmer, India



New Technologies

Altus™ HPLC & UHPLC

Today's laboratories want more from their liquid chromatography system: Higher performance. Better reliability. More consistent and comprehensive analytical workflows. And most of all, more predictable and reproducible results. At the same time, you want less complexity, hassle, and guesswork from those you count on for service and support. That's the Altus™ HPLC platform. Based on proven technology at work in thousands of labs worldwide, the Altus HPLC delivers integrated fluidics in a low dispersion design, with excellent reproducibility, peak capacity and resolution, and exceptional results. Optimized detection choices deliver extremely low detection limits for all the applications you need most. Automation features – everything from simple system startup to automated sample management to tool-free maintenance – ensure that everyone in your lab can run analyses quickly and easily, taking full advantage of the Altus HPLC system's much strength. Put that together with the leading chromatography data software in the business and the industry's best, most knowledgeable service and support organization, and you can now run your chromatography with the utmost confidence.

Front Panel Display

- Informative one touch display
- Fast walkup operation
- Automated system preparation function

Autosampler

- 120 sample capacity
- Alphabetically indexed and color-coded carousels for multiple users
- High precision sample delivery
- Programmable variable volume injection
- Tool-free replacement of needle wash frit
- Programmable needle height in 1 mm increments
- Programmable dynamic needle wash minimizes sample carryover

Bigger sample capacity

The Altus HPLC system can handle up to 120 standard-size vials in five separate sample carousels. So whether you're running a quick one-off sample or a series of methods from different analysts, your sample queues are simple to set up and run. And the sample environment can be temperature controlled and is ideal for light-sensitive samples as well.



Detectors - UV/VIS, PDA, RI, FL

- System delivers a full range of sensitive detection options
- Analyzer compounds with flexibility
- Whether you use each singly or in combination with other methods
- You'll be able to glean more

Column Oven

- Forced air recirculation peltier based column heater
- Integral mounting clips for column(s) and tubing's

Solvent Manager

- Degassing with four channel high efficiency vacuum in-line degasser
- Low volume degassing chambers enhance rapid solvent changeovers
- Pulse free solvent delivery
- Eleven available gradient curve profiles
- Automated, continuous solvent compressibility compensation
- Automated, continuous plunger seal wash
- Tool-free, simple access to plungers, plunger seals, plunger wash seals

Easy to use

The Altus HPLC system's large built-in, easy-to-navigate user interface enables your scientists to take full advantage of its fast system setup procedures with SystemPREP™, for quick, one touch walkup functionality. SystemPREP automatically readies the system to run samples ideal for daily startups, when

changing solvents, or after prolonged periods of system inactivity. The system's solvent management feature also degasses and accurately blends up to four chromatographic solvents for pulse-free, effortless delivery. Performance PLUS™ check-valve technology maximizes system uptime.

Built-in, integrated fluidics

The Altus HPLC system's integrated solvent and sample management functions deliver consistent performance from system to system, for highly reproducible results. Automatic, continuous solvent compressibility provides accurate and precise solvent delivery and reproducible retention times, regardless of solvent. The system's fluidics design ensures that accidental leakage and spills can be managed quickly and safely. Flexibility in programmable needle wash – making sample-carryover issues a thing of the past.

Thermal management

The column heating option uses integrated forced-air recirculation, making for an exceptionally stable column environment. With the passive column heating option, you have the confidence that your sample will be at the same temperature as the column.

UV/Vis detector

A sensitive, flexible dual-wavelength absorbance detector, with the ability to identify and quantify low-level impurities in the same run as the analyte of interest.

Photodiode Array (PDA) Detector

Advanced optical detection with exceptional chromatographic and spectral sensitivity – perfect for performing peak-purity analysis, even under extreme conditions.

Refractive index detector

An effective tool for isocratic quantification of components with little or no UV absorption – just the thing for analyzing and quantifying food sugars.

Fluorescence detector

A great solution for environmental monitoring applications in which increased sensitivity and maximum selectivity are required.

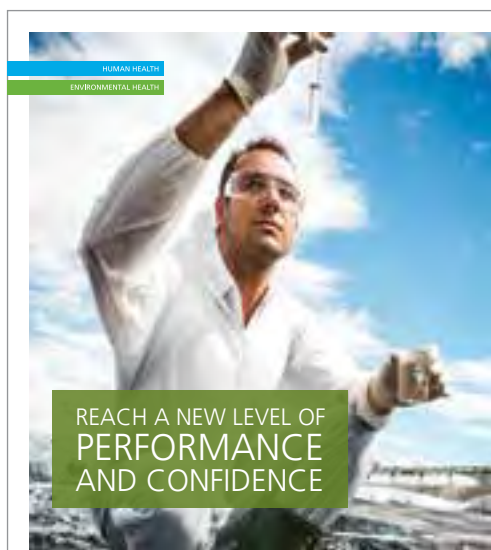
Workflow-driven, user-centric QuickStart™ interface, empower chromatography data software:

- Instrument control and data acquisition
- Peak detection (including the Apex-Track™ peak detection algorithm for consistent results)

- Quantitation
- GPC processing and calculations
- Data review
- Data searching
- Mass spectrometry
- Custom reporting

Chromatography data software (CDS) deliver the highest degree of confidence in your results. The most versatile, easiest to use, delivering extraordinary results with minimal training. Empower®3 software also provides customizable reports, integrated custom fields and calculations, and online help, while providing industry-leading security and data integrity.

The single-window Empower® 3 QuickStart™ interface allows all your users to perform tasks that match their skill level, with no confusion. You can develop new methods, including PDA and MS data extraction, customize reports, manage data, and receive results and notifications by email. The QuickStart™ interface streamlines the collection, processing, reviewing, and reporting of chromatographic results. Our comprehensive portfolio of solutions is designed to ensure you receive accurate, repeatable results on time, every time throughout the lifetime of your instrument.



Want more from your chromatography? Then you're in good company. Our new Altus™ LC technology running Empower® 3 software delivers integrated fluidics in a low-dispersion design, with excellent reproducibility, peak capacity and resolution, and exceptional results. And our multivendor service and support sets the standard for the industry – and then raises it. Altus technology: Now you can expect more from your chromatography – and get it.

Find out more at www.perkinelmer.com/altusuplc



Altus™ UPLC

New Technologies

PinAAcle 500 Flame atomic absorption spectrometer



PinAAcle™ 500 AA: fully-integrated, flame-only atomic absorption (AA) spectrometer ideal for labs needing an easy-to-use, high-performance flame AA for detecting metals and metalloids in environmental samples. With a touch screen interface with the flexibility to operate via its Syngistix Touch™ or Syngistix™ for AA Software, the PinAAcle 500 spectrometer can be coupled with a new FAST Flame sample automation accessory, providing the lowest cost-per-element flame AA.

Fast, simple operation with exceptional sensitivity and precision

- The PinAAcle 500 offers superior stability, longer life, lower maintenance costs, and the fastest return on investment of any Flame AA.
- Syngistix Touch™ or Syngistix™ for AA bringing a new level of flexibility and unparalleled ease-of-use to flame AA. Intuitive touch screen is easy-to-use, no training required. New methods can easily be created and stored. If your lab uses the same method each day, then all the user has to do is recall the stored method and the instrument will automatically be ready to run.

- Analyze lower concentrations with the accuracy of higher-end AA systems and achieve excellent precision.
- The sampling compartment is allowing easy access when you need to change burner heads or nebulizers. The burner system uses an innovative quick-lock design.
- Fully contained, maintenance-free fiber optical technology for high light throughput for best detection limits. It's provides fast start up and long-term stability.
- Excellent signal-to-noise ratio using solid-state detector.

The harshest environments and corrosive samples

- The world's first flame AA system with completely corrosive resistant sample introduction system which provides superior performance for analysis of corrosive and high solid matrices.
- Discover the new benchmark for durability and reliability.
- Peace of mind with fast return on investment.

Minimum maintenance and maximum speed

- Reduce operating costs with a rug-

ged design that virtually eliminates maintenance. The entire instrument is not only easy to use and maintain, it also includes safety features normally found only on top of-the-line AAS.

- Optimize your investment with a longer-lasting, corrosion-resistant platform.

Sample automation solutions:

The accessories simply plug in and are automatically recognized by the system when you turn it on.

AutoPrep 50 automatic dilution system

- Precise online dilution for faster, more accurate analyses
- Fully automated sample introduction when paired with a PerkinElmer autosampler

S10 Autosampler

For automated operation, add the S10 autosampler. PerkinElmer autosamplers come with a self-rinsing sampling probe and the flexibility to select from three sample tray types.

- Rugged design and corrosion-resistant components ensure long-term reliability and reproducible, precise results

Additional sample preparation solutions

Flow Injection for Atomic Spectroscopy (FIAS)

- The Hydride system can adapt your AA for the determination of Hg and hydride-forming elements with exceptional detection limits.

Titan MPS™ Microwave Sample Preparation System

- Microwave sample preparation systems that can deliver complete, uncontaminated samples – each and every time.
- Complete control of real-time temperature and pressure for the mineralization of a broad range of sample types.
- Cylindrical, pressure-resistant, PFA-coated stainless steel oven chamber and optical temperature and pressure reaction monitoring systems ensure the most reproducible results. Deliver a simple, safe, cost-effective microwave digestion – one that provides the quick return on investment that labs are looking for in these times of constrained budgets

Real-time, true double-beam optics

- High light through put detection limits available
- Fully contained, maintenance-free fiber optic technology
- Automatically adjusts to changes in lamp intensity for stable baselines and compensates for drift multiple times per second
- Fast start-up and exceptional long-term stability without recalibration

Real-time, true double-beam optics

- Accurately measures even the most difficult elements (including arsenic and barium)
- Excellent signal-to-noise ratios
- No need for expensive photomultiplier tubes

Syngistix touch software

- Quickly and easily save and share all methods and results
- Large, easy-to-use, full-color touchscreen
- Flexibility to mount touchscreen on either side of instrument

Corrosion-resistant design

- Industry's most completely corrosion-resistant system featuring
- conformal-coated circuit boards
- Polymer-coated flame shield
- Polymeric sample introduction module

Small footprint

- 26" (W) X 25" (D) X 25" (H)
- Saves valuable bench space
- Upgradable on-board computer



Real-time, true double-beam optics

- Coded 2-inch Lumina™ cableless Hollow cathode Lamps (HCLs) provide exceptional performance and stability
- Electrodeless Discharge Lamps (EDLs) ensure improved sensitivity and extended lamp life

Quick change modular sample introduction

- Simplifies routine maintenance/Cleaning
- Corrosion-resistant, durable design

HUMAN HEALTH
ENVIRONMENTAL HEALTH

IMPROVES BOTH WORKFLOWS & CASHFLOWS



NEW PinAAcle 500 Flame AA

Reliability, sensitivity, affordability-together at last in a Flame AA.

With the new PinAAcle™ 500, you can experience an uncompromising level of performance for an unbeatable price. The world's first Flame AA system engineered for complete corrosion resistance, the PinAAcle 500 offers superior durability, longer life, lower maintenance costs, and the fastest return on investment of any Flame AA.

Discover an instrument designed to outlast and outperform. And take your laboratory to a new PinAAcle of productivity and profitability.

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New Technologies

Spotlight 150i and spotlight 200i



In every laboratory in which analytical IR and microscopy are factors – in food packaging and other advanced materials, forensics, pharmaceuticals, biomaterials, academia, and a host of other disciplines – the trend is toward less specialization and more emphasis on learning a variety of instrumentation. As lab functions expand and become more centralized, bigger challenges – and a wider array of samples and sample sizes – are being tested by users who are being asked to do more than ever before. It's the new normal, and you have to adapt to a changing, and more challenging, landscape.

Helping you meet those challenges, both large and small, is what the Spotlight™ IR microscope systems are designed to do. With simple operation that's easy enough for novices to perform. Clear, common software controls for all sample types – from smallest to large. And streamlined reporting tools that let all your people concentrate on their core responsibility: moving your science forward.

It's intelligent technology, and it's simple to use: Smart region-of-interest search. Batch analysis and reporting for mul-

tipoint and multicomponent measurement. Auto-ATR optimization for fast, accurate results. And so much more. Best of all, it's high-performance IR microscopy with full-featured FT-IR included, the most flexible and versatile solution of its kind. The Spotlight 150i and Spotlight 200i systems: Taking on today's challenges – and your challenges to come.

The Spotlight microscope systems are designed for the lab scientist faced with increasingly challenging samples – and who needs higher sensitivity and simpler work flows to meet those challenges. That means faster, more intelligent automation, state-of-the-art technologies, easy-to-use software, plus simple tools for everything from setup to method development to data analysis. The result? The highest sensitivity and sophisticated analysis capabilities for even the most challenging samples.

Very intelligent automation:

With the Spotlight systems, everything is designed to speed you to high-quality results, with automatic features and functions that are simply unprecedented on any IR microscope. Its advanced technol-

ogy performs a variety of tasks to provide everything from automated setup to complete characterization – in record time.

For example, intelligent region of interest (ROI) finding makes time-consuming manual setup for analysis of multiple particles and layers a thing of the past, so it's perfect for finding contaminant specks and analyzing powder samples. At the same time, automated laminate analysis routines quickly locate features and set optimum scanning conditions for the sample viewed. Plus, you can combine analyses with point scanning for multiple sample points – so you can deliver results, not spectra, for a multitude of operations.

Nearly everything else on the system is automated, too:

- Automatic ATR performs multiple sampling modes, including single point, line scans, and maps, in a single experiment – with minimum sample preparation compared with transmission analysis, while maintaining spectral integrity and quality.
- Configurable validation routines speed instrument performance validation tests, so you're always ready for operation.

- The capability of combining random markers and line scans across boundaries and 2D maps enables more complete, reproducible sample characterization – even in unattended mode.
- When configured with the Frontier FT-IR platform, an automatic beamsplitter change can quickly reconfigure the system for multispectral range operation.



Microsampling:

Multiple microscope collection modes provide optimum configurations for sub-100-micron samples, enabling the microscope to measure the greatest

range of samples with the standard instrument. And additional spectral-range options are available for specialized sample types.



HUMAN HEALTH
ENVIRONMENTAL HEALTH

COMPREHENSIVE
SERVICE SOLUTIONS
TO MAXIMIZE YOUR LAB
PRODUCTIVITY

PerkinElmer OneSource Service Contracts provide dependable coverage to enhance your productivity.

Avoid unexpected service costs

Unexpected instrument failure not only disrupts laboratory productivity, but can be expensive to fix. Choosing a service contract that provides breakdown cover insures against unexpected costs, saving your laboratory money.

Minimize instrument downtime

Having instruments under a service contract

will guarantee a response time from our dedicated engineering team. Highly skilled, they deliver >90% first time fix rate ensuring your laboratory gets up and running as soon as possible.

Extend instrument life

Regular preventive maintenance will ensure your instrumentation is fully optimized, increasing the efficiency of your laboratory.

Choose the plan that's right for your laboratory

Labor

Enhance laboratory productivity by maintaining instrument efficiency; with a priority breakdown response and technical support.

Comprehensive

For laboratories where maximizing instrument uptime is critical to performance

New Technologies

Lambda... Innovation ahead...!!!



We are stepping ahead with a new series of UV/Vis instruments Lambda 265, 365 and 465 for users performing routine UV/Vis testing in teaching, biochemistry, environmental, R & D, pharmaceutical QA/QC and materials testing laboratories.

New materials testing, research and development, analytical testing – the challenges in these and other key areas of manufacturing and academics are becoming more complex all the time. And so are most labs' operations, with the analysis of nanomaterials, metamaterials, and other industrial materials development requiring global alignment on an unprecedented scale. The new lambda line is compiled of compact and easy to use UV/Vis systems having best performance to meet the requirements of any labs. These instruments and a new version of software which drives them provide simple operation for easy training and learning, a small footprint for more efficient use of lab space, and have a full range of accessories and software to support established, standard UV test methods. Best of all, these systems' advanced design packs all these global capabilities into a compact footprint that fits into any lab. Put that together with industry-leading expertise in UV/Vis, and

you have systems you can count on for a long time to come.

The new line will be having two diode array (PDA) systems (Lambda 265 and 465) and a traditional double beam spectrometer (Lambda 365). In diode array technology the detector can detect as many wavelengths simultaneously as number of individual diodes, pixels or elements of resolution. Thus with Photodiode array detector, it is possible to measure multiple wavelengths simultaneously. Diode array technology is known for its minimum stray light effect, fast scan speed and high signal to noise ratio. With PDA technology fast scan speed; kinetics studies are faster never before.

Lambda 265: Fast, accurate, affordable results With ultrafast data processing and maximum reliability, the LAMBDA 265 is the ideal system for a wide range of R&D and QA/QC applications, all while taking up minimal bench space. Its photodiode array (PDA) detector enables data to be acquired simultaneously across the full wavelength range – from 190 nm to 1100 nm. In seconds, your processing is complete and ready for you to act on. Plus, the LAMBDA 265 system's robust modular design, with no moving parts,

is ideal for any busy lab. The high energy Xenon flash lamp is active only when a spectrum is being acquired and provides years of worry-free operation. And the system's compact size makes it simple to move it to any location.



Lambda 265:

Lambda 365: Compact, versatile high-performance double-beam UV/Vis The LAMBDA 365 delivers state-of-the-art UV/Vis performance that meets the needs of pharmaceuticals, analytic chemists, geneticists, and manufacturing QA/QC analysts everywhere. With 21 CFR part 11 software available, the Lambda 365 is ready to support all your needs – everything from standard methods and applications to those requiring regulatory compliance. The system delivers a variable spectral bandwidth capability from 0.5 nm to 20 nm, so your applications can benefit from a wide range of accessories. These accessories include multicell changers (both water and Peltier

temperature-controlled), solid sample accessories for transmission and reflectance, optical fiber probes for remote measurements, an integrating sphere for color and diffuse measurements, and a range of cuvette holders to meet your sampling needs. When high stability and low stray light are critical, the LAMBDA 365 double-beam technology is the ideal solution. The large sample compartment can easily accommodate more than 10 sampling accessory combinations. Easy-to-install accessories minimize setup time and effort, and multicell changers are autoaligned by the instrument software to optimize the sample position. This feature provides optimized results in a wide range of routine applications, including manufacturing and pharmaceutical QA/QC, environmental testing, academics, and more.



Lambda 365:

Lambda 465: High performance PDA that delivers reliability – and confidence. Designed specifically for high-end research as well as routine and high-throughput applications, the LAMBDA 465 is the innovative PDA solution that provides maximum reliability – for maximum confidence in your results. Its PDA technology allows the acquisition of a full spectrum – from 1100 nm to 190 nm – in as little as 20 msec. In addition, the system has 1-nm resolution, allowing it to meet the requirements of a number of pharmacopoeias. With 21 CFR part 11 compliant software, it's an ideal solution for dissolution, fast kinetics, and other applications where high-speed scanning and high resolution are required – and it's perfect for method development and sample analysis, too. For flexibility in sampling along with high resolution and low noise spectrum, a dual light source (tungsten and deuterium) in a see-through configuration is



Lambda 465:

unitized. This configuration provides the highest energy throughput possible – an important consideration with accessories that impact energy throughput.

Whatever the demands of your lab for material analysis, with the LAMBDA

family of UVV is instruments, we're delivering a new level of confidence. These benchtop-friendly systems help maximize your lab's efficiency, enabling you to handle your current sample workload, then expand as your lab's business changes and grows. And with their simple interfaces and intuitive software, training costs are minimized – and uniform global integration is an achievable goal.

Here, we are presenting benchtop UVs with results that say "confidence."



Though UV VIS spectrometry is an established technique; the sampling and optics are the keys to obtain meaningful and reliable results. Some of the accessories are mentioned below may be of great use for certain applications.

Applications	Segment
Ethnic skin characterization	Health care
In Vitro sun screen SPF measurement	Health care
Characterization of single walled CNT	Nano-materials
Band-Gap energy measurement of TiO2	Nano materials and PV
Reflectance measurement of solar materials	Solar and photo voltaic
High resolution measurements of optical filters	Glass and optics
Fiber optics sampling	Paints and surfaces
Measurement of UVA and UVB of lenses	Glass
Ultra micro volumes of nucleic acids	Life sciences
DNA melt studies	Life sciences
Characterization of light sources	Solar
Formaldehyde and Hexavalent chromium contents in toys	Health care
Olive oil purity measurement	Food
Transmission spectra of ultra-small gemstones	Gems
Formaldehyde analysis	Textiles
Bilirubin, Porphyrin Analysis	Clinical diagnostics



Food & Beverages

Analysis of Phenolic Antioxidants in Edible Oil/Shortening Using the PerkinElmer Altus UPLC System with PDA Detection

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 PerkinElmer, Inc.
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Introduction

Phenolic antioxidants are commonly used in food to prevent the oxidation of oils. Oxidized oil and fats cause foul odor and rancidity in food products, which is a major cause for concern to the food industry. Globally, regulations vary, but current maximum allowable levels are as low as 100 µg/g (100 ppm).

This application note presents a UHPLC method for the analysis of the ten most common phenolic antioxidants that may be found in such products. The application was carried out with minor modifications to the AOAC Official Method 983.15⁽¹⁾. This method applies to the analysis of finished food products. A 2.7-µm SPP (superficially porous particle) C18 column was used, allowing one to achieve very high throughput at a back-pressure considerably lower than that for UHPLC columns.

This method was then applied to a commercial vegetable shortening product, which per label claim, was reported to contain at least one of the antioxidants being analyzed.

Method conditions and performance data, including linearity and repeatability, are presented.

Experimental

Hardware/Software

For all chromatographic separations, a PerkinElmer® Altus™ UPLC® System was used, including the Altus A-30 Solvent delivery Module, Sampling Module, A-30h

Column Module and PDA (photodiode array) Detector with a 10-mm path-length flow cell. All instrument control, analysis and data processing was performed using the Waters® Empower® 3 Chromatography Data Software (CDS) platform.

Method parameters

The HPLC method parameters are shown in Table 1.

Table 1. UHPLC Method Parameters

HPLC Conditions								
Column:	PerkinElmer Brownlee™ 2.7 µm 2.1 x 100 mm C18 (Part# N9308404)							
Mobile Phase:	Solvent A: Water; Solvent B: Acetonitrile Solvent program:							
	Time (min)	Flow Rate (mL/min)	0%A	%B	%C	%D	Curve	
	1	Initial	0.600	60.0	40.0	0.0	0.0	Initial
	2	4.50	0.600	45.0	55.0	0.0	0.0	6
	3	7.00	0.600	18.0	82.0	0.0	0.0	6
	4	10.00	0.600	18.0	82.0	0.0	0.0	6
	5	10.10	0.600	60.0	40.0	0.0	0.0	11
	Equil. Time ("Next inj. Delay Time"): 3 minutes							
Analysis Time:	10 min.							
Flow Rate:	0.6 mL/min. (maximum pressure during run: 6600 psi)							
Oven Temp.:	35 °C							
Detection:	Altus A-30 PDA; wavelength channels: 280 and 220 nm							
Injection Volume:	1 µL							

Solvents, standards and samples all solvents and diluents used were HPLC grade and filtered via 0.45- μm filters.

The phenolic antioxidant standard kit #2 (catalog# 40048-U) was obtained from Supelco® (Irvine, CA). This included nordihydroguaiaretic acid (NDGA), propyl gallate (PG), octyl gallate (OG), lauryl gallate (dodecyl gallate (DG)), 2-tert-butyl-4-hydroxyanisole (BHA), 2,6-di-tert-butyl-4-hydroxymethylphenol (IonoX 100), tert-butylhydroquinone (TBHQ), 3,5-di-tert-butyl-4-hydroxytoluene (BHT) and ethoxyquin. In addition, a 2, 4, 5-trihydroxybutyrophenone standard (THBP; catalog# 2620¹-X9) was obtained from SynQuest® (Alachua, FL).

Using a 100-mL volumetric flask, a 100-ppm stock standard was made up by dissolving 10 mg of each of the ten antioxidant standards in methanol and then bringing the flask up to the mark with methanol. Individual calibrant standards were prepared using the 100-ppm stock solution.

The sample (Sample X) was a commercially available vegetable shortening purchased at a local food market. The sample was prepared by dissolving 3 grams of Sample X in 15 mL of hexane in a 50-mL centrifuge tube and vortexing for 5 minutes. The resulting solution was then extracted with three 30-mL portions of acetonitrile, combining the three extracts into a 250-mL evaporation dish. The combined extract was evaporated down to 1-2 mL and reconstituted to 6 mL with methanol.

Prior to injection, all calibrants and samples were filtered through 0.22- μm filters to remove small particles.

Results and Discussion

Figure 1 shows the chromatographic separation of the 10 phenolic antioxidants in under nine minutes. Figure 2 shows the overlay of 10 replicate 50-ppm standard injections, demonstrating excep-

tional reproducibility. Retention time % RSDs ranged from 0.10 (early eluters) to 0.03 (later eluters).

In a previous application note ⁽²⁾, it has been noted that ethoxyquin may not be well detected at 280 nm. However, we did not observe this, and we could easily detect the analyte at 5-ppm levels. The same injection was also captured on a separate channel, set to 220 nm, as shown in Figure 3. At this wavelength, it is evident that the ethoxyquin has approximately two times the signal intensity. However, this additional signal intensity was not really required here, as current maximum allowable concentrations for phenolic antioxidants only go down to 100 ppm, which was easily handled at 280 nm.

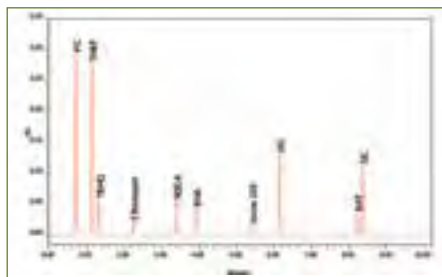


Figure 1. Chromatogram of 50-ppm phenolic antioxidant standard; wavelength = 280 nm.

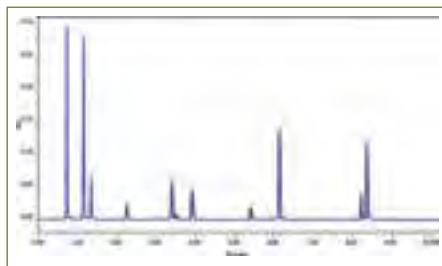


Figure 2. Overlay of 10 replicates of 50-ppm check standard; wavelength = 280 nm.

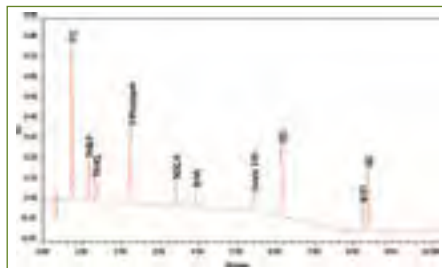


Figure 3. Chromatogram of 50-ppm phenolic antioxidant standard; wavelength = 220 nm.

Figure 4 shows three representative calibration results over a concentration range of 5 to 100 ppm. All ten components had linearity coefficients > 0.999 (n = 3 at each level).

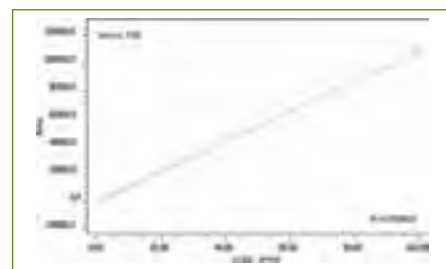
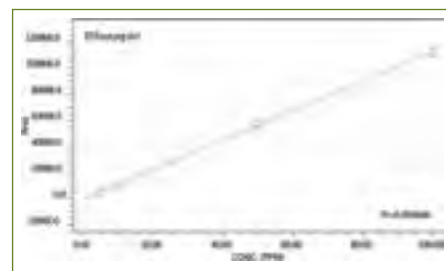
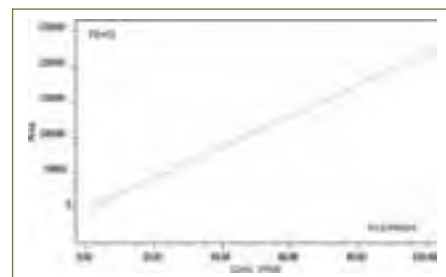


Figure 4. Three representative results of 5-level calibration sets for the phenolic antioxidants; wavelength = 280 nm.

Figure 5 shows the chromatographic results of Sample X overlaid with the 50-ppm standard. A peak eluting at exactly the time of TBHQ (tert-butylhydroquinone) was observed. This was consistent with the product label claim. By back-calculating the concentration in the original sample, it was determined that Sample X contained approximately 12-ppm of TBHQ. The actual concentration could not be verified as it was not provided in the product's label claim.

Per Figure 6, upon closer examination of the chromatogram of Sample X, a small peak at about 8.23 minutes was also observed. This matched the elution time

for DG (dodecyl gallate) in the standard mix. If this was indeed DG, its concentration was below the calibration curve, estimated to be <math><0.5\text{ ppm}</math>. Further verification of the identity of this peak was not pursued.

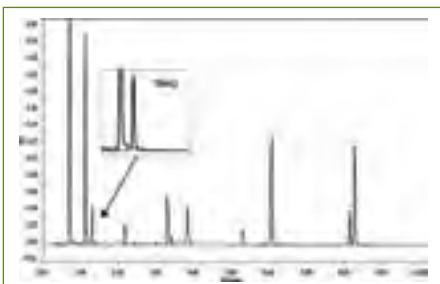


Figure 5. Chromatogram of Sample X (blue) overlaid with 50-ppm standard (black); wavelength = 280 nm.

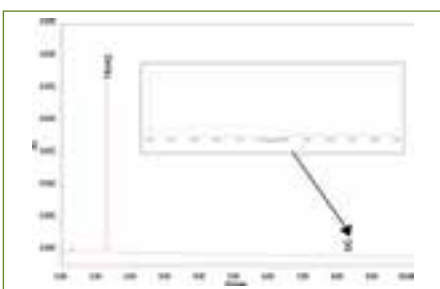


Figure 6. Chromatogram of Sample X with zoomed in area just after 8 minutes; wavelength = 280 nm.

Conclusion

This work has demonstrated the effective chromatographic separation of ten phenolic antioxidants using a PerkinElmer Altus UPLC[®] with a PDA detector and the Empower[®] 3 CDS system. The results exhibited excellent retention time repeatability as well as exceptional linearity over the tested concentration ranges. At an analytical wavelength of 280 nm, the sensitivity for all 10 phenolic antioxidants was found to be more than adequate to accommodate the current maximum allowable concentration limit of 100 ppm.

We were able to identify and quantitate the phenolic antioxidant content in a commercial vegetable shortening product and the results matched the label claim of the manufacturer.

From a food quality perspective, considering the ever growing emphasis on food monitoring, this application is intended to serve as a valuable guide for the monitoring of edible oils/shortening. It should be noted that in the U.S., per

label claims, only some of the vegetable shortenings reported any amount of phenolic antioxidant. None of the edible oils that were found in stores reported any phenolic antioxidants. However, although only edible vegetable shortening was tested for this study, the provided sample preparation procedure and chromatographic application easily lend themselves to the analysis of edible oils as well.

References

1. Official Methods of Analysis, Method 983.15, Association of Official Analytical Chemists (AOAC), Arlington, VA USA
2. Monitor Antioxidant Additives in Foods, Using HPLC (AOAC Method 983.15) or Capillary GC and a Supelco Reference Standards Kit, Application Note 78, Sigma-Aldrich/Supelco, 2004
3. Analysis of Common Antioxidants in Edible Oil with the PerkinElmer Flexar[™] FX-15 System Equipped with a PDA, Application Note, PerkinElmer, Inc.



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- Analytical services
- Multipurpose analysis



Food & Beverages

Analysis of Sugars in Honey Using the PerkinElmer Altus HPLC System with RI Detection

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Introduction

Honey consumption has grown significantly during the last few decades due to its high nutritional value and unique flavor. The price of natural bee honey is much higher than other sweeteners making it susceptible to adulteration with cheaper sweeteners, primarily sucrose. Besides lower levels of nonsugar ingredients, natural honey primarily consists of glucose and fructose and may contain low levels of sucrose and/or maltose.^{1,2} However, according to the international regulations, any commercially available “pure”-labeled honey products that are found to have in excess of 5% by weight of sucrose or maltose are considered to be adulterated.³

With the focus on possible honey adulteration, this application highlights the LC separation of various sugars found in honey and the analysis of these components in four storebought honey samples. Method conditions and performance data, including linearity and repeatability, are presented.

Experimental

Hardware/Software

For all chromatographic separations, a PerkinElmer Altus™ HPLC system was used, including the Altus A-10 Solvent and Sample Module, Column Module,

integrated vacuum degasser/column oven and an Altus A-10 RI Detector. All instrument control, analysis and data processing was performed using the Waters® Empower® 3 CDS platform.

Method Parameters

The HPLC method parameters are shown in Table 1

Table 1. HPLC Method Parameters.

HPLC Conditions							
Column:	PerkinElmer Brownlee™ Analytical Amino 3 μm, 4.6 x 150 mm (Part# N9303505)						
Mobile Phase:	Solvent A: 65:35 acetonitrile/water Solvent program:						
	Time (min)	Flow Rate (mL/min)	0%A	%B	%C	%D	Curve
	Initial	1.000	100.0	0.0	0.0	0.0	Initial
Analysis Time:	6 min.						
Flow Rate:	1.0 mL/min. (2300 psi)						
Oven Temp.:	25 °C						
Detection:	Altus A-10 RI; cell temp.: 35 °C						
Injection Volume:	5 μL						
Sampling (Data) Rate:	10 pts./sec						

Solvents, Standards and Samples

All solvents and diluents used were HPLC grade and filtered via 0.45- μ m filters.

The sugar standards were obtained from Supelco® (Irvine, CA) and consisted of fructose, glucose, maltose and sucrose. Stock sugar standards were made using 65:35 acetonitrile/water as diluent. For the 1333 μ g/mL (ppm) stock solution, the standards were first dissolved in 17.5 mL of water before adding 32.5 mL of acetonitrile. The lower level standards were then prepared from this stock solution.

All commercially available honey products were purchased at local stores. They were labeled Honey W, Honey X, Honey Y and Honey Z. Each honey was prepared by dissolving 2.5 g into 50 mL of 65:35 acetonitrile/water, followed by another 1:1 dilution using the same solvent.

Prior to injection, all calibrants and samples were filtered through 0.45- μ m filters to remove small particles.

Results and Discussion

Figure 1 shows the chromatographic separation of the 1333- μ g/mL (ppm) sugar standard containing the four target sugars using the optimized conditions described above. The analysis time was under six minutes.

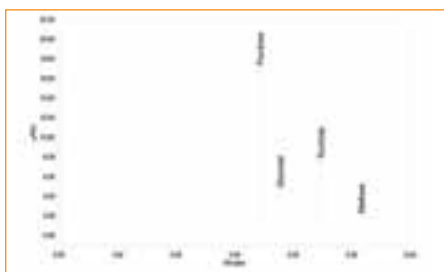


Figure 1. Chromatogram of the 1333 μ g/mL sugar standard.

Figure 2 shows the overlay of 12 replicate 667- μ g/mL sugar standard injections, demonstrating exceptional reproducibility. Retention time % RSDs

were also quite exceptional, exemplified by 0.026% RSD for fructose.

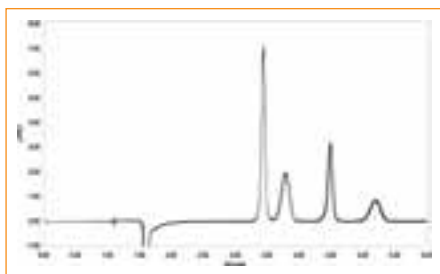


Figure 2. Overlay of 12 replicates of the 667 μ g/mL sugar standard.

Figure 3 shows the calibration results for all four sugars over a concentration range of 133 to 1333 μ g/mL. All four sugars followed a quadratic (2nd order) fit and had R² coefficients > 0.999 (n = 3 at each level).

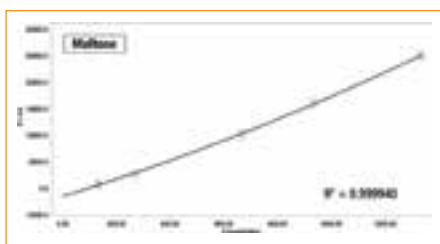
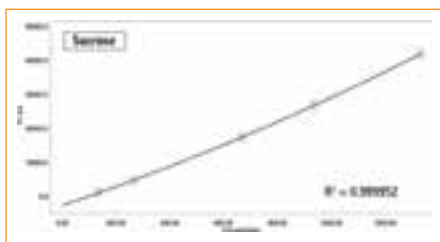
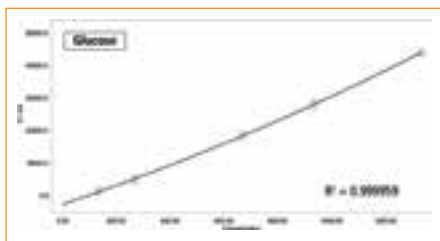
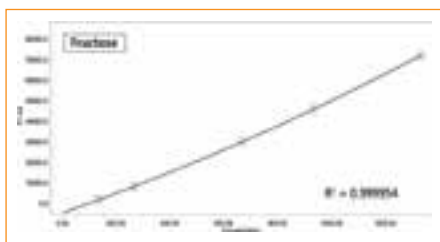


Figure 3. Results of 5-level calibration sets for fructose, glucose, maltose and sucrose.

Using the same chromatographic conditions, four honey samples were analyzed. The chromatographic results for Honey X, Honey Y and Honey Z are shown in Figure 4. Comparing the chromatograms of these honey samples with the sugar standards, it can be observed that all three honey samples contain the same three sugars: fructose, glucose and small amounts of sucrose.



Figure 4. Overlaid chromatograms of Honey X (green), Honey Y (black) and Honey Z (blue).

Based on standard calibration, the quantitative results for each honey sample are shown in Table 2. Combining the fructose and glucose percentages for each honey sample, the overall fructose and glucose content for Honey X, Y, and Z was determined to be 50.90%, 57.13%, and 53.60%, respectively. These results are consistent with the accepted overall content of fructose and glucose in honey, expected to be somewhere around 60%.¹ The sucrose content for each honey sample was determined to be 3.20%, 3.26% and 3.90%, respectively. These values are all below the 5% mass ratio limit for sucrose that is allowed in unadulterated honey. Based on the data presented, the three store-bought honey samples do not appear to be adulterated with cheaper sweeteners.

Upon closer examination of the chromatogram of Honey W, a smaller but significant peak was observed at about 5.10 minutes (Figure 5). This matched the elution time for maltose in the standard mix. The amount of maltose was calculated to be 43.85 mg, and the percent sugar was calculated to be 1.75% (w/w). Considering the 5% (by

weight) limit that is allowed in commercially available “pure”-labeled honeys, the resulting maltose level found in Honey W suggests it was not adulterated.

Table 2. Quantitative results.

Honey X:		
Component	Amount	Percent Sugar (W/W)
Fructose	556.05	22.24
Glucose	716.48	28.66
Sucrose	79.875	3.20

Honey Y:		
Component	Amount	Percent Sugar (W/W)
Fructose	610.23	24.41
Glucose	817.95	32.72
Sucrose	81.525	3.26

Honey Y:		
Component	Amount	Percent Sugar (W/W)
Fructose	602.30	24.09
Glucose	737.78	29.51
Sucrose	97.525	3.90

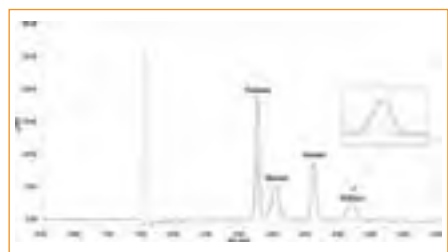


Figure 5. Overlay chromatograms of Honey W (red) and the 133 ppm sugar standard (black), zooming in on last eluting peak.

Conclusion

This work has demonstrated the effective chromatographic separation of four sugars using a PerkinElmer Altus HPLC System with RI detection. The results exhibited very good retention time repeatability as well as excellent linearity over the tested concentration ranges. From a food quality perspective, there is an ever growing emphasis on food monitoring. This is especially the case

pertaining to the adulteration of honey. With this in mind, this work focused on the sugar analysis of four store bought honeys, identifying the particular analytes contained in each of the honey samples, as well as comparing the sugar profiles, both chromatographically and quantitatively.

References

1. W. Guo, Y. Liu, X. Zhu and S. Wang, “Dielectric properties of honey adulterated with sucrose syrup”, Journal of Food Engineering, pp. 1-7, 2011.
2. A. Moussa, D. Noureddine, A. Saad and S. Douichene, “The Relationship between Fructose, Glucose and Maltose Content with Diastase Number and Anti-Pseudomonal Activity of Natural Honey Combined with Potato Starch”, Organic Chemistry Current Research, vol. 1, no. 5, pp. 1-5, 2012.
3. Codex Alimentarius Commission, 2001; GB18796-2005, 2005

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Food & Beverages

The Analysis of Copper, Iron, and Manganese in Wine with the PinAAcle 500

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Introduction

With the popularity of wine consumption continuing to grow, regulations are being implemented as to the metal content allowed in wines, such as those imposed by China for copper, iron, and manganese on imported wine¹ – the maximum permitted concentrations are shown in Table 1. These analyses can be easily accomplished using flame atomic absorption (AA) spectrometry. This work demonstrates the analysis of copper (Cu), iron (Fe), and manganese (Mn) in wine with the PinAAcle™ 500 flame atomic absorption spectrometer.

Table 1. Chinese limits on copper, iron, and manganese in imported wines.

Element	Limit (mg/L)
Copper (Cu)	1
Iron (Fe)	8
Manganese (Mn)	2

Experimental

All analyses were performed on the PerkinElmer PinAAcle 500 AA spectrometer operating with an air/acetylene flame with hollow cathode lamps, according to the conditions in Table 2. The standard nebulizer and spray chamber were used. All samples and standards were introduced manually using self-aspiration.

The wine samples included in this study are shown in Table 3 and were analyzed neat with no sample preparation – samples were just poured from the bottles into sample tubes. To assess accuracy, all samples were measured at two different wavelengths. In addition, spike recoveries at both the regulatory limits and half of the regulatory limits were performed. All analyses were made against external calibration curves with the standards being made in deionized water. The highest calibration standard exceeded the upper regulatory limit for each element.

Results and Discussion

Tables 4-6 show typical results for the analyses for copper, iron, and manganese in the wine samples. For clarity, results from multiple analyses of each sample are not shown. These results indicate that all elements in all the wines are under the regulatory limit, with the manganese level in the chardonnay being closest to the limits. Spike recoveries were within 15% for all wines, indicating a lack of matrix interference, corroborating the accuracy of the results. Repeated analyses of all wines and spikes produced results consistent with those shown in Tables 4-6, demonstrating the stability of the method.

All samples and spikes were also measured at a second wavelength, as

indicated in Table 2. The results of these analyses were consistent with those in Tables 4-6, providing further confidence that the results are accurate.

Table 2. PinAAcle 500 AA spectrometer instrumental conditions.

Parameter	Copper	Iron	Manganese
Primary Wavelength (nm)	324.75	248.33	279.48
Secondary Wavelength (nm)	327.40	302.06	279.83
Slit (nm)	0.7	0.2	0.2
Air flow (L/min)	2.5	2.66	2.66
Acetylene flow (L/min)	10	7.36	7.36
Calibration standards (mg/L)	1, 2, 3	1, 5, 12	1, 2, 5
Calibration curve type	Linear through zero	Non-linear through zero	Non-linear through zero

Table 3. Wines analyzed.

Type	Country of Origin
Chardonnay	Australia
Cabernet Sauvignon	France
Red	USA
White Zinfandel	USA

Table 4. Copper in wine results (regulated level = 1 mg/L).

Wine	Concentration (mg/L)	+ 0.5 mg/L (mg/L)	% Recovery	+ 1 mg/L (mg/L)	% Recovery
Chardonnay	0.29	0.80	102	1.30	101
Red	0.19	0.72	107	1.24	105
White Zinfandel	0.14	0.63	98	1.13	99
Cabernet Sauvignon	0.13	0.61	96	1.09	96

Table 5. Iron in wine results (regulated level = 8 mg/L).

Wine	Concentration (mg/L)	+ 0.5 mg/L (mg/L)	% Recovery	+ 1 mg/L (mg/L)	% Recovery
Wine	Concentration (mg/L)	+ 4 mg/L (mg/L)	% Recovery	+ 8 mg/L (mg/L)	% Recovery
Chardonnay	0.57	4.63	102	8.72	102
Red	1.56	5.99	111	10.1	107
White Zinfandel	2.02	6.03	100	10.2	102
Cabernet Sauvignon	3.33	7.33	100	11.1	97

Table 6. Manganese in wine results (regulated level = 2 mg/L).

Wine	Concentration (mg/L)	+ 1 mg/L (mg/L)	% Recovery	+ 2 mg/L (mg/L)	% Recovery
Chardonnay	1.70	2.60	90	3.49	90
Red	1.30	2.24	94	3.17	94
White Zinfandel	1.06	1.94	88	2.89	92
Cabernet Sauvignon	0.97	2.04	107	2.93	98
Cabernet Sauvignon	3.33	7.33	100	11.1	97

Conclusion

This work has clearly demonstrated the ability of the PinAAcle 500 AA spectrometer to accurately measure copper (Cu), iron (Fe), and manganese (Mn) in a variety of wine samples at levels which meet the regulations imposed by China for imported wine. The Syngistix Touch™ software operated from the PinAAcle's large touchscreen display allows for simple operation when analyzing samples. If more flexibility is desired, Syngistix™ for AA software can also be used, running from an on-board computer. For increased sample throughput when analyzing large batches, a FAST Flame sample automation system can be used with the PinAAcle 500. With the faster sample throughput, equivalent results can be obtained for the analysis of Cu,

Fe, and Mn in wine². The flexibility of operating mode and sample introduction systems, combined with its analytical capabilities, makes the PinAAcle 500 an excellent instrument for measuring metals in wines.

References

1. "Manganese Levels in China", Wine Australia (<http://www.wineaustralia.com/en/~media/0000Industry%20Site/Documents/News%20and%20Media/News/Media%20Releases/2014/Manganese%20Levels%20in%20China.ashx>).
2. Spivey N., Thompson P., "The Analysis of Copper, Iron, and Manganese in Wine with FAST Flame Atomic Absorption", PerkinElmer Application Note.

Consumables

Component	Part Number
Cu Hollow Cathode Lamp	N3050121
Fe Hollow Cathode Lamp	N3050126
Mn Hollow Cathode Lamp	N3050145
Cu 1000 mg/L Standard	N9300183 (125 mL) N9300114 (500 mL)
Fe 1000 mg/L Standard	N9303771 (125 mL) N9300126 (500 mL)
Mn 1000 mg/L Standard	N9303783 (125 mL) N9300132 (500 mL)
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)



Environment

Analysis of Minerals in Drinking Water with the PinAAcle 500 Atomic Absorption Spectrometer

Authors:

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Atomic Spectroscopy
Training and Consulting

Kenneth Neubauer
PerkinElmer, Inc.

Introduction

With water quality varying widely with geography and geology, as well as pollution considerations, it is important to know the metal content of waters, both for consumption and industrial use. Although a variety of techniques can measure minerals in water, one of the simplest, least expensive, and fastest is flame atomic absorption (AA) spectrometry. As a result, the technique continues to enjoy widespread use, despite the increasing popularity of ICP-OES and ICP-MS.

This work focuses on the determination of seven non-toxic elements usually found in drinking waters with the PerkinElmer PinAAcle™ 500 flame atomic absorption spectrometer. Although other lower-level elements can also be measured by flame AA, these are most commonly analyzed by either graphite furnace AA, ICP-OES, or ICP-MS.

Experimental

Samples consisted of municipal and well waters collected locally, spring waters purchased from a local grocery store, and a certified drinking water standard (Trace Metals in Drinking Water – High-Purity Standards™, Charleston, South Carolina, USA). Sample preparation consisted only of acidifying each water with 1% HNO₃ (v/v) and adding 0.1% lanthanum chloride as a releasing reagent for calcium (Ca) and magnesium (Mg) and as an ionization suppressant for sodium (Na) and potassium (K).

All analyses were carried out with the PinAAcle 500 flame AA spectrometer using the conditions in Tables 1 and 2. Due to the high mineral content, the burner was rotated 30 degrees to decrease the signal intensity for the analysis of the minerals. In addition, K and Na were analyzed in emission mode, which allowed the PinAAcle 500 to be auto-

configured in such a way to extend the analytical range so that even higher concentrations could be measured. This allowed minimal dilution for K and elimination of dilution for Na.

Samples were introduced via self-aspiration with a high-sensitivity nebulizer, which is standard on the PinAAcle 500 spectrometer. The nebulizer was used without the spacer (providing maximum sensitivity) for the determinations of copper (Cu), iron (Fe), and zinc (Zn). The spacer was inserted for the determinations of Na, K, Mg and Ca.

Table 1. PinAAcle 500 instrument and analytical conditions common to all elements.

Parameter	Value
Air Flow (L/min)	2.5
Acetylene Flow (L/min)	10
Read Time (sec)	3
Replicates	3

Table 2. PinAAcle 500 instrument and analytical conditions specific to each element.

Element	Wavelength (nm)	Slit (nm)	Mode	Burner Angle (degrees)	Calibration Standards (mg/L)	Calibration Curve
Ca	422.67	0.7	Absorption	30	0.5, 1.0, 2.0, 5.0, 10, 20, 40	Non-Linear through Zero
Cu	324.75	0.7	Absorption	0	0.05, 0.10, 0.25, 0.50	Linear Through Zero
Fe	248.33	0.2	Absorption	0	0.05, 0.10, 0.25, 0.50, 1.0	Linear Through Zero
Mg	285.21	0.7	Absorption	30	0.5, 1.0, 2.0, 5.0, 10	Non-Linear Through Zero
K	766.49	0.7	Emission	30	2, 5, 10, 20, 30, 40, 50	Non-Linear Through Zero
Na	589.00	0.2	Emission	30	2, 5, 10, 20, 30, 40, 50	Non-Linear Through Zero
Zn	213.86	0.7	Absorption	0	0.05, 0.10, 0.25, 0.50	Linear Through Zero

Results and Discussion

All calibrations yielded correlation coefficients of 0.999 or greater. The accuracy of the calibrations was assessed with an independent calibration verification (ICV) solution, which was diluted 100 times to fall within the range of the calibration curve. The results of the ICV appear in Table 3 and demonstrate the accuracy of the calibration curves.

Table 3. Results for independent calibration verification (ICV).

Element	Concentration (mg/L)	Experimental (mg/L)	% Recovery
Ca	5.00	4.86	97
Cu	0.25	0.26	104
Fe	1.00	1.00	100
Mg	5.00	4.88	98
K	5.00	4.78	96
Na	5.00	5.12	102
Zn	0.20	0.21	105

To validate the methodology, a reference material was first analyzed, with the results shown in Table 4. All recoveries are within 10% of the certified value, demonstrating the accuracy of the methodology.

Table 4. Results for reference material (all units in mg/L).

Element	Experimental (mg/L)	Certified (mg/L)	% Recovery
Ca	33.4	35.0	95
Cu	0.022	0.020	110
Fe	0.095	0.100	95
Mg	8.69	9.00	97
K	2.28	2.50	91
Na	5.90	6.0	98
Zn	0.070	0.070	100

Table 5. Results for samples (all units in mg/L).

Element	Municipal Water (mg/L)	Well Water-1 (mg/L)	Well Water-2	Well Water-3 (mg/L)	Spring Water-1 (mg/L)	Spring Water-2
Ca	17.7	0.148	35.3	32.4	3.43	19.2
Cu	0.048	< DL	0.052	0.017	< DL	< DL
Fe	< DL	< DL	0.019	< DL	< DL	< DL
Mg	6.43	0.026	4.90	5.12	0.799	6.09
K	< 0	233*	4.89	4.10	0.73	0.69
Na	38.4	3.63	10.9	42.9	6.60	7.25
Zn	0.008	0.043	0.010	0.023	< DL	< DL

* Sample required a 10x dilution

With the accuracy of the method established, several drinking water samples from various sources were analyzed. The municipal and well water samples were collected directly from a faucet, while the spring water samples were poured from the bottles in which they were purchased. The results appear in Table 5.

The presence of Cu and Zn in the four samples collected from the faucet is most likely due to leaching from copper pipes, fittings, and solder. Well Water-1 is interesting as it contains the lowest levels of all samples, except for an extraordinarily high level of K. Further investigation determined that this residence has a water softener installed which utilizes K as the counter-ion to remove high levels of Ca and Mg from the well water.

As expected, Cu and Zn are not detected in the spring waters; only the minerals are present. The variation in mineral concentration is indicative of the different geologies of the areas where these waters originate.

Finally, detection limits were determined for Cu, Fe, and Zn as three times the standard deviation of ten blank measurements (i.e. 1% HNO₃), as shown in Table 6. Because of their elevated

levels, detection limits were not determined for the mineral elements (i.e. Ca, K, Mg, Na). In addition, since these elements are usually present at high concentrations, the instrument was detuned for their analysis. Therefore, detection limits would be meaningless.

Table 6. Detection limits.

Element	Detection Limit (mg/L)
Cu	0.002
Fe	0.006
Zn	0.004

Conclusion

This work has demonstrated the ability of the PinAAcle 500 to successfully measure mineral elements in drinking water samples, including municipal, well, and spring waters. By taking advantage of the ability to rotate the burner and measure in emission mode, both trace and mineral elements could be measured. With Syngistix Touch™ software, the PinAAcle 500 AA spectrometer can be operated exclusively from a touchscreen interface. For greater flexibility, the ability to run Syngistix™ for AA software from an on-board computer is also available. This flexibility makes the PinAAcle 500 flame AA spectrometer an excellent choice for the analysis of drinking waters.

Consumables Used

Component Part	Number
High sensitivity nebulizer	N3160144
Autosampler tubes (15 mL)	B0193233
Autosampler tubes (50 mL)	B0193234
Ca Hollow cathode lamp	N3050114
Cu Hollow cathode lamp	N3050121
Fe Hollow cathode lamp	N3050126
Mg Hollow cathode lamp	N3050144
Zn Hollow cathode lamp	N3050191
Quality control standard, 21 elements	N9300281
Initial calibration verification standard	N9300224
Pure-Grade Ca Standard (1000 mg/L)	N9303763 (125 mL) N9300108 (500 mL)
Pure-Grade K standard (1000 mg/L)	N9303779 (125 mL) N9300141 (500 mL)
Pure-Grade Mg Standard (1000 mg/L)	N9300179 (125 mL) N9300131 (500 mL)
Pure-Grade Na standard (1000 mg/L)	N9303785 (125 mL) N9300152 (500 mL)

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Pharmaceuticals

Detection and Identification of Microplastic Particles in Cosmetic Formulations Using IR Microscopy

Author:
 Ian Robertson
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 Seer Green, UK

Introduction

It is estimated that there is in excess of 150 million tons of plastic materials in the world's oceans. Much of this pollution consists of large items such as discarded drink bottles and plastic bags. However, there is increasing research into the amount of much smaller materials, termed microplastics, in the river and ocean systems which present a different type of problem for marine life.

Many cosmetic products, such as facial scrubs, toothpastes, and shower gels, currently contain microplastic beads as abrasive materials. These microplastics, which are typically submillimetre in size, get washed down the sink and are too small to be filtered by sewage treatment plants consequently ending up in the river systems and ultimately in the oceans. These microplastics can be ingested by marine organisms and fish and end up in the human food chain.

In 2014 a number of U.S. states banned the use of microplastics in cosmetic formulations and most cosmetic companies are voluntarily phasing out their use. Infrared (IR) spectroscopy is the established technique for identifying polymer

materials and has been used extensively for identifying large (over 100 micrometer) polymer materials. The Spectrum Two™ is a portable FT-IR spectrometer that can operate from a battery pack and has been used on boats for immediate identification of these polymers.¹ For microplastics, down to a few micrometers in size, an IR microscope can be used for the detection and identification of these materials.

Two commercially available products were tested using the Spotlight™ 200i IR microscope system in order to determine whether microplastics were present as the exfoliant and to identify the types of plastics used.

Product 1 is a commercially available facial scrub. Product 2 is a commercially available body scrub. Each of these products was mixed with hot water in order to dissolve the soluble ingredients in the formulation. The resulting solution was filtered through a 50 micrometer mesh, capturing any insoluble components greater than 50 micrometers in size. The filter was then allowed to dry in air prior to IR microscopy measurements. The samples were measured

both directly on the mesh and also after transferring the residual particles onto an IR transmitting window on a microscope holder. Visible images of the collected microplastics are shown as Figures 1a and 1b.

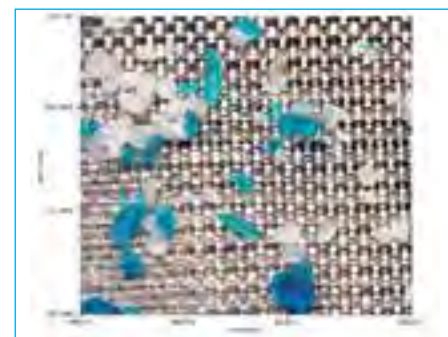


Figure 1a: Microplastics in Product 1 (facial scrub) collected on mesh.

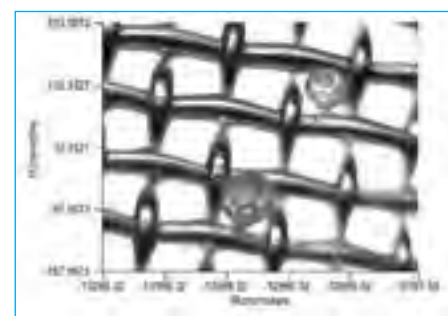


Figure 1b: Magnified view of microplastics collected from Product 2 (body scrub).

It is clear from these images that Product 1 has irregular-shaped microplastics with particles of two different colors. The particles from Product 2 are regular spheres with those visible in Figure 1b being approximately 50 and 80 micrometers in diameter. Infrared spectra of these materials can be measured in either transmission or reflectance on the IR microscope. Spectra measured on one of the particles in Figure 1a, in-situ on the mesh, are shown as Figure 2.

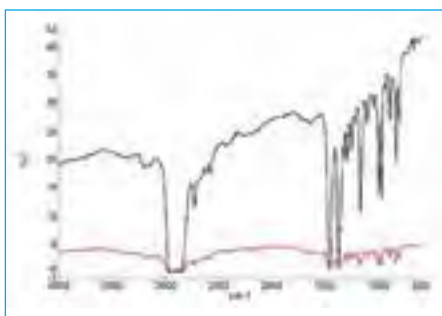


Figure 2: Spectra from a microplastic particle in Product 1. Transmission spectrum (black) and reflectance spectrum (red).

The transmission spectrum has a much higher signal than the reflectance spectrum and gives better sensitivity for this measurement. In addition, the bands in the reflectance spectrum are more intense due to the fact that the IR beam is effectively passing twice through the sample, known as transmittance. For smaller particles this does not cause any problems; but for larger particles the path length may be too large leading to totally absorbing bands, thus making identification more difficult. However, in this case, it would be possible to identify the material from either the transmission or reflectance spectrum. The mesh may interfere with the transmission measurement, slightly decreasing the amount of energy reaching the detector. This explains the baseline slope observed in the spectrum, but it does not significantly impact the overall measurement. To obtain the best quality spectrum of the material, the sample can be transferred onto an IR-transmitting window material, such as potassium bromide

(KBr). A KBr window was placed onto the mesh containing the sample and the mesh inverted thereby transferring the microplastic particles directly onto the KBr window.

A “Visible Image Survey” was collected over the area containing the majority of the particles in Product 1. Selecting the “Analyze Image” function in the Spectrum 10 software invokes the intelligent automated routine for detecting particles within this Visible Image Survey, which is displayed as “analyze image result” shown in Figure 3.



Figure 3: The Analyze Image software routine detects the particles in Product 1.

This routine will automatically detect any particles present in the visible image and mark them as regions of interest. It will then calculate the maximum rectangular aperture size that can fit wholly inside each of the particles, thus maximizing signal-to-noise when the data is scanned. In the past, manual selection of the regions of interest and setting of apertures took a considerable amount of time. Clicking “Scan Markers” initiates the collection of transmission spectra (using equivalent apertures for the background) for each particle, displaying ratioed sample spectra in real time as they are collected. Automatic processing of the spectra, using software routines such as Search, Compare, or Verify, can be performed during data collection. In this case, the analysis of the microplastics, a spectral search was performed against a library of polymer spectra to give the identity of each of the particles as shown in the results screen in Figure 4.

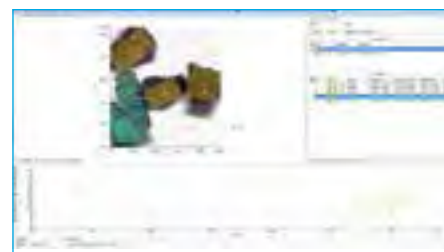


Figure 4. Results screen for the detection and identification of particles

The results show that Product 1 has two different types of polymers present, polypropylene and polyethylene. Product 2 contains only particles of polyethylene. Representative spectra are shown in Figure 5. Small differences are observable in the spectra of the polyethylene between the two different products, most likely due to additives present.

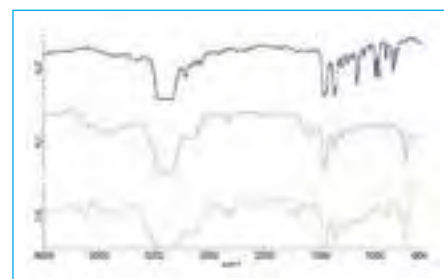


Figure 5: Top – spectrum of polypropylene in Product 1. Middle – spectrum of polyethylene in Product 1. Bottom – spectrum of polyethylene in Product 2.

Summary

Microplastics are a major concern regarding their impact on the environment and as such their use in consumer products is increasingly being prohibited. An automated IR microscopy system has been shown to be an invaluable method for the detection and identification of a source of microplastics in cosmetic formulations. The work presented here will be extended to analyze samples of microplastics collected from European river systems to illustrate how widespread this pollution problem is within marine environments.

References

1. Labo magazine – Oktober 2010 Wasserverschmutzung durch Mikroplastikpartikel, www.labo.de

Polymers, paints and mining

Analysis of Automobile Paint Chips Using an Automated IR Microscope

Author:

Ian Robertson
PerkinElmer, Inc.
Seer Green, UK**Introduction**

The information obtained from paint chips involved in road traffic accidents is extremely important for piecing together evidence in criminal cases. Traces of paint can be transferred from a vehicle onto other surfaces or materials, such as victims clothing, and these can be matched to the paint type of the vehicle. This is achievable since the paint chips are multi-layered materials consisting of several coats of paint. The layer combinations are unique for an individual manufacturer, model, color, and year of a particular vehicle. Infrared (IR) spectroscopy is a standard technique used for the measurement of paint samples with ASTM method E2937 - 13 acting as a standard guide for using infrared spectroscopy in forensic paint examinations. Infrared microscopes are routinely used for measuring extremely small paint samples down to a few micrometers in size allowing spectra to be recorded for each of the layers.

This application note describes the use of the different sampling modes and automation features of the Spotlight™ 200i IR microscope system applied to an automobile paint chip sample retrieved from the roadside at the scene of a road traffic accident.

There are three main sampling techniques for infrared spectroscopy of solid samples: transmission, (specular) reflectance, and Attenuated Total Reflectance (ATR). All of these sampling techniques can be applied to standard (macro) IR accessories as well as IR microscopes for microsamples. Each of these techniques has been applied to this sample on the IR microscope and the relative advantages and disadvantages of each are described.

Reflectance

Specular reflectance measurements are obtained from direct reflection from the cross-section surface of the sample. Reflectance sampling mode requires minimal sample preparation as the sample is simply secured in a small clamp device and placed on the stage of the IR microscope. The visible image from the sample in reflectance in Figure 1 shows

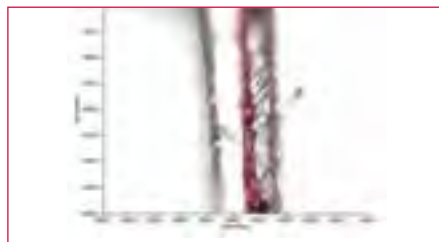


Figure 1: Visible image of cross section of a paint chip sample on IR microscope.

that there are multiple layers present. Sample preparation and spectral data are easy using the reflectance technique. However, it is not the optimal technique, since there are distortions in reflectance spectra due to refractive index changes and peak shifts. Therefore, reflectance spectra are generally not suitable for comparison against reference spectra that have, in the vast majority, been recorded in transmission. Reflectance spectra from the layers in this sample are typical of a reflectance spectrum from a paint sample and are shown as Figure 2. The background spectrum was recorded using a gold mirror reference.

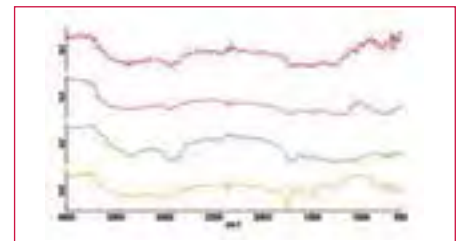


Figure 2: Specular reflectance spectra of paint layers.

Attenuated Total Reflectance (ATR)

The technique of ATR is also a surface technique with the IR radiation only penetrating 1-2 micrometers into the sample. Therefore, the sample to be measured can be a thick sample

requiring less sample preparation; a simple cut of the sample to generate a cross section of the layers is sufficient. The sample for ATR can be supported in a clamp device for the microscope or embedded in a resin block and polished. Embedding provides better support and avoids distortion of the sample when pressure is applied using the ATR crystal, also aiding to preserve it, allowing repeat measurements if required. A section of the paint chip was embedded in a resin for these ATR measurements, as shown in Figure 3.

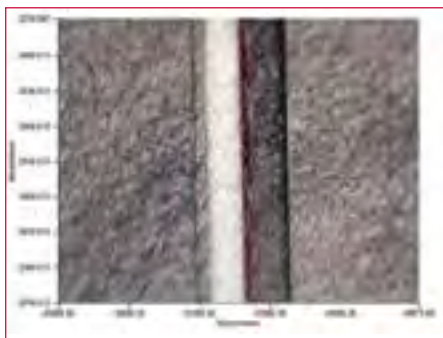


Figure 3: Visible image of a cross section of a paint chip embedded in resin for ATR measurements.

The Spotlight 200i system can be fitted with an automated drop-down ATR crystal (100 micrometer tip size) that allows measurements to be performed at discrete points on the sample, or allows linescans or maps to be collected. An alternative is to use the macro ATR accessory for the IR microscope. The crystal on this accessory is clamped across the entire sample allowing direct measurements anywhere on the sample without any crystal movement. This gives a significant improvement in the spatial resolution that can be achieved, allowing thinner layers to be measured. A linescan measurement was performed across the paint chip using the Macro ATR accessory with the data shown in Figure 4.

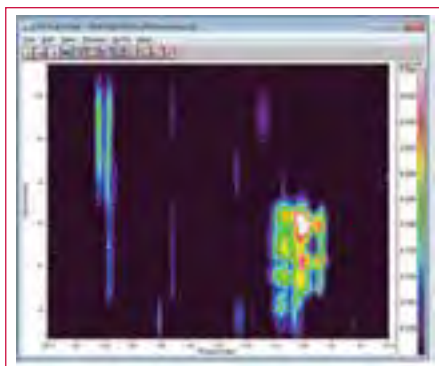


Figure 4: ATR linescan data for paint chip.

The spectra obtained for the different layers are shown in Figure 5.

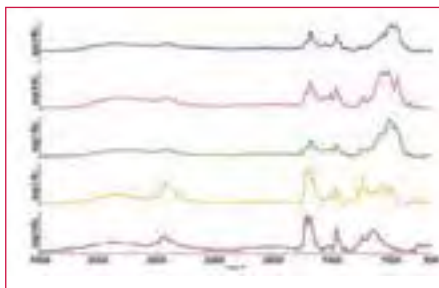


Figure 5: Spectra obtained for the different layers in the ATR experiment.

The advantage of ATR spectra over specular reflectance spectra is that ATR spectra closely resemble those of the equivalent transmission spectrum. ATR correction processing routines will correct for the wavelength-dependent intensity differences between ATR and transmission spectra, allowing ATR spectra to be compared against the extensive library databases of transmission spectra. Spectra are shown in Figure 6 comparing the ATR spectrum with the transmission sample for the same paint layer demonstrating the equivalence of the spectra using the two techniques.

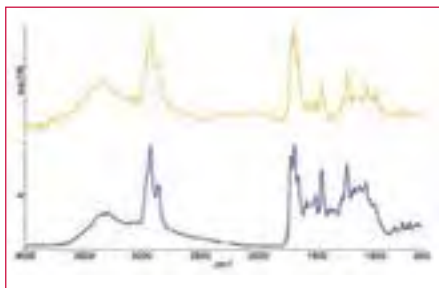


Figure 6: ATR-corrected spectrum (top) and transmission spectrum (bottom) for one of the layers in the paint chip sample.

Transmission

IR transmission measurements generally require the sample to be in the region of 10 to 20 micrometers thick in order to avoid totally absorbing bands. For paint chip samples, this requires the sample to be microtomed to an acceptable thickness prior to measurement. The paint chip sample was microtomed and placed onto the surface of a potassium bromide (KBr) window, shown in Figure 7.

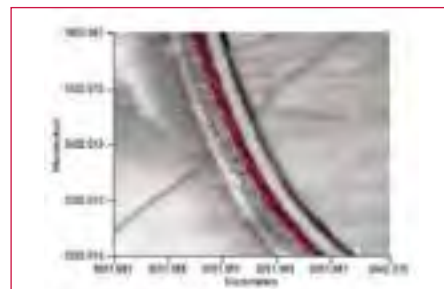


Figure 7: Microtomed paint chip sample is shown on KBr window for transmission measurement.

In order to obtain spectral information from all the layers in the sample a linescan was set up to measure spectra at five-micrometer intervals across the entire width of the paint chip. The linescan data is shown as Figure 8.

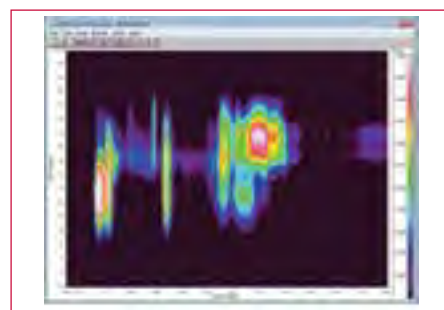


Figure 8: Transmission linescan data for paint chip sample.

The linescan data shows different spectral features in different areas of the sample, which represent the different layers. Spectra for each of the layers were extracted from this linescan data and are shown in Figure 9.

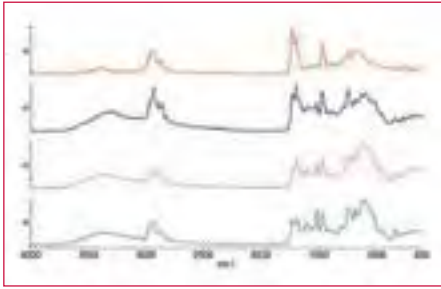


Figure 9: Shown here are transmission spectra of the layers in the paint chip.

The transmission spectra can be compared against standard libraries of paints and additives in order to identify the layers present.

Summary

IR microscopy is an invaluable technique for the measurement of the multiple layers of paint chips. The choice of sampling technique will be dependent

on the sample preparation techniques available to the user and the information required from the spectrum.

Specular reflectance offers a no-sample preparation solution and will be able to show spectral differences between layers, but the spectra will contain distortions and cannot be directly identified or compared to transmission spectra libraries.

ATR also offers a no-sample preparation solution if the sample is held in a clamp device on the microscope sample stage. However, embedding the sample is advantageous. The ATR spectra are directly comparable to transmission spectra and libraries once ATR correction has been applied to adjust the relative intensities of the spectral bands. The use of the macro ATR accessory for the microscope

will generate better spatial resolution, allowing thinner layers to be measured.

Transmission is the preferred method for measuring paint samples, since transmission spectra are directly comparable to existing spectral databases and reference spectra. In addition, transmission measurements do not exhibit distortions or require the mathematical corrections required for reflectance and ATR spectra. However, this measurement requires the sample to be no thicker than 15 to 20 micrometers, thus requiring some level of sample preparation of the samples to be microtomed.

References

1. PerkinElmer Technical Note 007641A-03, Spatial Resolution in ATR Imaging

Hyphenation Technology

Technologies typically classed as 'Hyphenation'

- Modification of the sample environment
- Evolved Gas Analysis (EGA)
- Simultaneous Analysis

- TG-FTIR
- STA-FTIR
- DSC-RAMAN SPECTROMETER
- PHOTO DSC (DSC-UV)
- TGA-MS
- TG-GCMS
- Photo DMA & controlled humidity DMA



Polymers, paints and mining

Characterizing polymer laminates using IR microscopy

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Introduction

Multilayer polymer films, or laminates, are used in a wide variety of industries. A major use of these materials is for packaging of foods and consumer products. The composition of multilayer films can often be quite complex as they may have to satisfy a variety of requirements to preserve the contents. A package must collate and contain the product, requiring strength and the ability to seal the packaging. It must be machineable at a reasonable cost. In the case of food products, it must be able to preserve the contents and protect it from external influences that would affect the product quality or safety, ultimately leading to increased shelf-life. Each of the layers in the laminate will perform a different barrier function, protecting the product from different external factors, such as moisture, light, oxygen, microbial materials and other chemicals or flavors.

Generally, traditional polymer materials, such as polyethylene terephthalate (PET), polyethylene (PE), polystyrene (PS), and polypropylene (PP), have been used for packaging materials. These packaging materials account for a significant proportion of materials ending up at

landfill sites or recycling plants. Some of these materials biodegrade slowly or do not biodegrade at all and are environmentally unfriendly. Consequently, there is increasing focus on the use of biodegradable or compostable polymers that can be used as packaging materials. Bio-based materials are partly or entirely made of renewable raw materials, such as cellulose, starch or polylactic acid (PLA). These bio-based plastics can be biodegradable, but are not always. Compostable plastics can be completely biodegraded by microorganisms leaving only water, carbon dioxide, and biomass. These materials are more environmentally friendly and are expected to be used increasingly in the future.

Infrared microscopy has long been the most important technique for characterizing multilayer polymer films. Infrared spectroscopy has the ability to identify materials and the addition of an infrared microscope allows for small samples (down to <10 microns) to be analyzed, including the determination of the identities of the different layers of laminates. This Application Note describes the use of infrared microscopy applied to “traditional” multilayer polymer films as well as the newer compostable materials.

Infrared Microscopy of Multilayer Polymers

Infrared microscopy of polymer films can be performed using transmission or Attenuated Total Reflectance (ATR) techniques. Infrared transmission measurements require the sample to be optically thin, generally not thicker than 20 to 30 microns. This requires the sample to be prepared as a thin film by the use of a microtome. The sample can then be placed on an infrared transmitting window material, such as potassium bromide (KBr), for measurement of transmission spectra. ATR measurements can be performed on optically thick materials as ATR is a surface technique. The sample needs to be physically supported, either in an embedding resin or in a sample clamp specially designed for use in infrared microscopes. ATR measurements have the additional benefit of generating spectra at a significantly better spatial resolution than transmission measurements.¹

Transmission of Laminate

A polymer laminate sample was cut to a thickness of 25 microns using a microtome and taped flat onto a 7 mm diameter KBr window. This sample was then placed in a standard microscope

sample holder on the microscope stage of the PerkinElmer Spotlight™ 200i. A visible image of the sample is shown as Figure 1. The laminate is approximately 350 micrometers across (top to bottom).

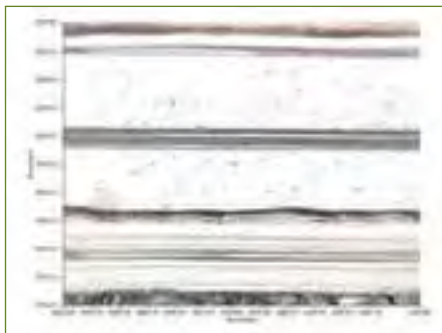


Figure 1: Visible image of polymer laminate measured in transmission.

If detailed information is required about all of the layers in the laminate then it is possible to setup a linescan, collecting spectra at very small intervals across the laminate. Such an experiment was set up to collect spectra at 3 micrometer steps across the laminate, using an aperture size of 5 micrometers with a total of 140 spectra collected. The linescan data is shown as Figure 2.

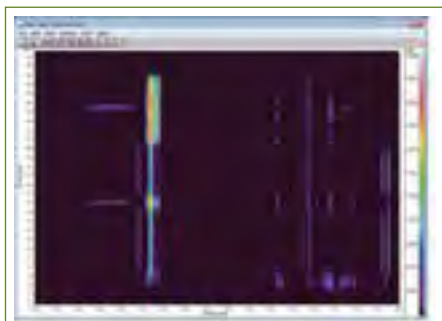


Figure 2: Linescan data for polymer laminate transmission measurements.

The results indicated that several different polymer types were present in the sample as shown in Figure 3. These were identified using Search libraries as; PET, modified PS, PE, ethylene-vinyl acetate (EVA), and ethylene-vinyl alcohol (EVOH).

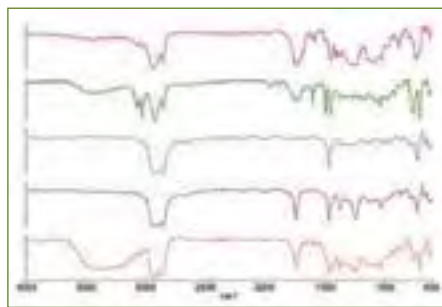


Figure 3: Spectra of the polymers present in the different laminate layers.

Profiles can be generated to show the distribution of the different polymer types throughout the laminate giving significant structural information. The profiles for polystyrene (1600 cm^{-1}), polyethylene (1450 cm^{-1}), ethylene-vinyl acetate copolymer (1746 cm^{-1}), and ethylene-vinyl alcohol copolymer (3334 cm^{-1}) are shown as Figure 4.

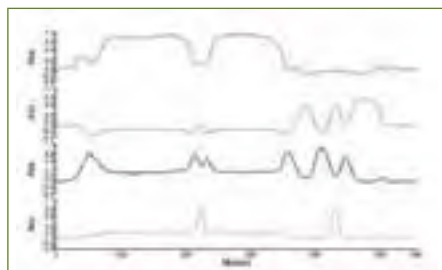


Figure 4: Distribution profiles for polymer types in laminate. From top to bottom: polystyrene, polyethylene, ethylene-vinyl acetate co-polymer and ethylene-vinyl alcohol co-polymer.

If the only requirement for the analysis is to detect and identify the layers in the laminate, then the Analyze Image function within the Spectrum 10 software can be used. This function will analyze the visible image of the sample, detect the layers present, and maximize the measurement area for each layer, all completed automatically. In the case of a multilayer sample, it will collect a single spectrum for each layer, giving the maximum signal-to-noise and significantly reduce the analysis time compared to mapping or measuring a linescan on the same sample. Figure 5 shows an example of a five-layer laminate.

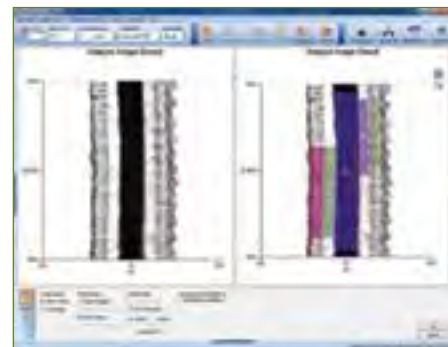


Figure 5: Automatic detection of layers in a laminate shows five layers.

After detection of the laminate layers, spectra were automatically recorded at the marker positions, shown in Figure 6. An automatic library search identified each of the layers as polyethylene terephthalate (layers 1 and 5), ethylene-vinyl acetate copolymer (layers 2 and 4), and silica-loaded polyethylene (layer 3).

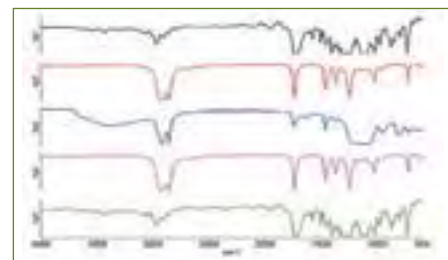


Figure 6: Spectra of layers 1 are shown (top) to 5 (bottom) in polymer laminate.

ATR measurements of polymer laminates

ATR provides a fast and easy way of measuring an infrared spectrum of a material. ATR on an infrared microscope is capable of measuring spectra of very small materials down to just a few micrometers in size. A macro ATR crystal/accessory for the microscope has been utilized to collect data on food packaging laminates. This ATR accessory can generate spectra at a significantly better spatial resolution than transmission measurements¹. For the ATR measurements the samples were embedded in a resin and polished to give a flat, clean surface for the ATR measurement. Embedding the sample generates a stronger multilayer surface than simply clamping the sample and prevents deformation or compression of the sample under ATR pressure.

A sample of a multilayer food package manufactured using “traditional” polymers was prepared for ATR measurement in the Spotlight 200i. The visible image of this sample was measured and is shown as Figure 7. The width of the laminate is seen to be approximately 200 micrometers and consists of several polymer layers.

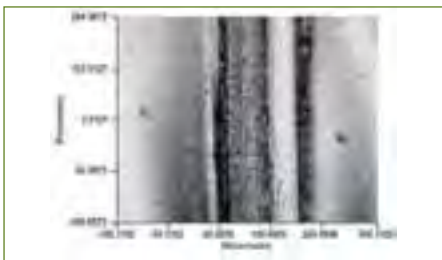


Figure 7: Visible image for multilayer food packaging material.

The macro ATR crystal for the IR microscope was placed in contact across the entire width of the sample. Spectra were collected across the laminate with an effective aperture size of 5 x 5 micrometers at a step size of 5 micrometers. The linescan data collected is shown as Figure 8.

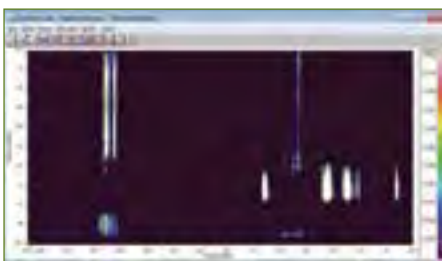


Figure 8: Linescan data for food packaging material.

Several different polymer types seem to be present in the sample. The spectra obtained from the major layers are shown in Figure 9. A search against polymer databases identifies the layers as polypropylene, polyethylene terephthalate, polyethylene, and modified polyethylene.

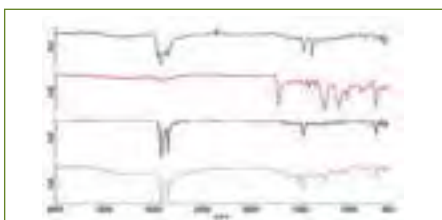


Figure 9: Spectra of major layers are identified as PP, PET, PE and modified PE.

In addition, several other minor layers were detected and their infrared spectra shown as Figure 10. A region of the data (around 160 micrometers in the display) gave no spectral details at all, as it was a thin foil layer.

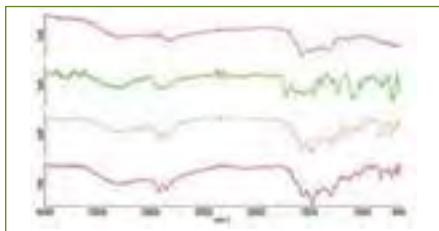


Figure 10: Spectra of minor layers in multilayer food packaging material.

A new generation of biodegradable polymer materials has been developed as a replacement for the “traditional” polymer packaging material. A compostable food packaging material has been analyzed on the Spotlight 200i. The sample was prepared for IR-ATR microscopy in the same way as the “traditional” packaging material that was shown previously.

The visible image of the embedded sample appears as Figure 11. The laminate is seen to be approximately 80 micrometers wide, consisting of a small number of visible layers.

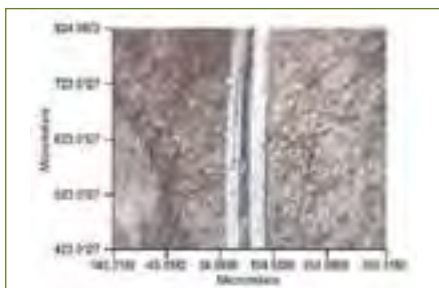


Figure 11: Visible image of a compostable food packaging laminate.

The infrared data collected on the sample is shown as Figure 12. The sample consists of three major layers each of approximately 25 micrometers width.

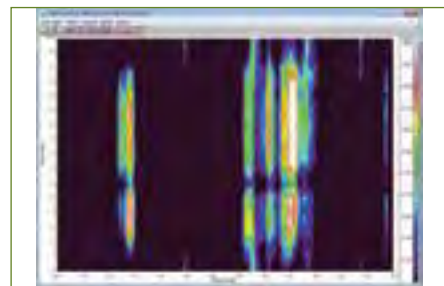


Figure 12: Linescan data for compostable laminate sample.

The spectra are shown as Figure 13. The spectra of the layers all look similar, however, they exhibit spectral differences in the C=O region between 1700-1760 cm^{-1} . The materials are known to be polylactic acid (PLA)-based copolymers. The region at approximately 60 micrometers in the display does not exhibit spectral features, as there is a thin layer of foil present in the sample.

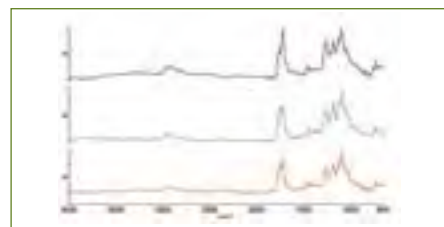


Figure 13: Shown here are spectra of the three different layers in the compostable polymer laminate.

Summary

Packaging materials, especially food packaging, are complex materials in order to satisfy the numerous requirements for the product contained within. Multilayer laminates are a means of fulfilling these requirements. However, disposal of food packaging materials is a significant environmental problem. Biodegradable packaging materials are a possible solution.

IR microscopy has been shown to be an excellent technique for the characterisation of these “traditional” and newer multilayer materials. Transmission or ATR measurements can easily be deployed depending on the sample preparation that is available.

Reference

1. PerkinElmer Technical Note 07641A_03, Spatial Resolution in ATR Imaging



Polymers, paints and mining

The analysis of precious metals in mining with the PinAAcle 500 atomic absorption spectrometer

Authors:
Ken Neubauer
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Introduction

When mining for precious metals, ores are extracted from the ground and subjected to various sample preparation procedures in order to remove the metals of interest. A commonly used procedure to isolate metals from the ore is fire-assay, which leaves a matrix-free “button” of the metal. This button is then dissolved in the appropriate acids and analyzed. By knowing the amount of sample used in the sample preparation and the analytical results, the concentration of the metals in the ground can be determined. These analyses are typically done with flame atomic absorption (AA) spectrometry due to its low cost, analytical speed, simplicity, and robustness. This work will focus on the analysis of precious metals in simulated digested precious metal buttons, with an added emphasis on

assessing the lowest limits which can be accurately measured.

Experimental

All analyses were performed on the PerkinElmer PinAAcle™ 500 AA spectrometer operating with 10 cm burner head, according to the conditions in Table 1. All samples and standards were introduced manually using self-aspiration through the high-sensitivity nebulizer with the spacer removed. The gas flow rates were optimized to give the highest sensitivity and a steady signal. For all analyses, a three-second integration time and three replicates were used. Standards were prepared in either 2% HNO₃ (copper, silver) or 15% aqua regia (gold, palladium, platinum) to simulate the dissolution of a button sample after fire-assay sample preparation.

Results and Discussion

The ability to accurately measure low concentrations was assessed by establishing low-level calibration curves with regression values > 0.999. The lowest standards were those whose absorptions were greater than the blank and gave relative standard deviations (RSDs) < 5%. For every element, lower concentrations could be measured, but the RSDs were greater than 5%, a result of statistics when considering small numbers. Table 2 shows the calibration standards, with the resulting calibration curves appearing in Figure 1. To assess the accuracy of the measurements, a standard at the mid-point of each calibration curve was measured, with typical results shown in Table 3.

Table 1. PinAAcle 500 AA spectrometer instrumental conditions.

Parameter	Gold (Au)	Palladium (Pd)	Platinum (Pt)	Copper (Cu)	Silver (Ag)
Wavelength (nm)	242.80	244.79	265.94	324.75	328.07
Slit (nm)	0.7	0.2	0.7	0.7	0.7
Lamp	HCL	HCL	HCL	HCL	HCL
Air Flow (L/min)	4.40	4.40	4.40	4.40	7.80
Acetylene Flow (L/min)	2.02	2.02	1.86	2.02	1.58

Table 2. Low-level calibrations.

Element Calibration	Standards (µg/L)	Calibration Type
Au	50, 60, 70, 80	Linear Through Zero
Pd	50, 60, 70, 80	Linear Through Zero
Pt	500, 750, 1000, 1250	Linear Through Zero
Cu	10, 20, 30, 40	Linear Through Zero
Ag	5, 10, 20, 30	Linear Through Zero

Table 3. Mid-level standard quantitative read back.

Element	Standard (µg/L)	Read-Back (µg/L)	% Recovery
Au	65	67.7	104
Pd	65	69.2	106
Pt	850	836	98
Cu	25	24.1	96
Ag	15	14.5	97

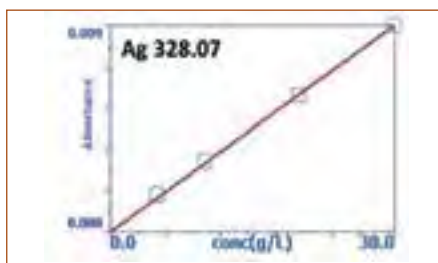
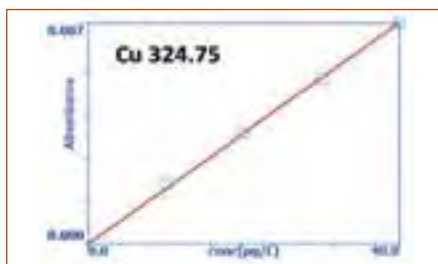
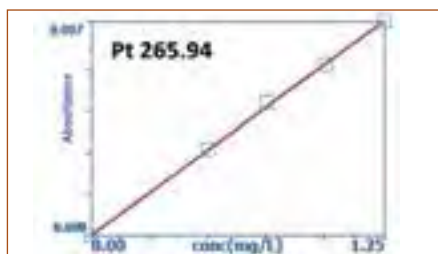
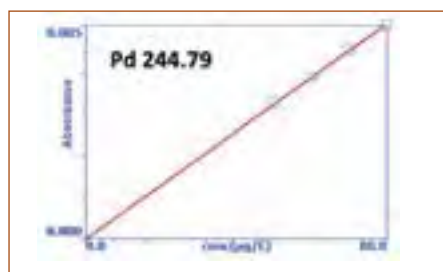


Figure 1. Low-level calibration curves for gold, palladium, platinum, copper, and silver.

Because of the importance of measuring precious metals at low Table 4. Detection limits. levels, detection limits were determined under the same instrumental conditions and timings as the calibration and quantitation studies (Table 4) using the following formula:

$$\text{Detection Limit} = \frac{(3 \cdot \text{SD} \cdot \text{CC})}{0.0044}$$

SD = Standard deviation
CC = Characteristic concentration

The characteristic concentration is determined by running a standard, recording the absorbance, and using the following

formula: Characteristic Concentration = $(\text{Conc} \cdot 0.0044) / \text{Abs}$

Conc = Concentration of the standard
Abs = Absorbance

An explanation of the “0.0044” constant can be found elsewhere¹.

Conclusion

This work has demonstrated the ability of the PinAAcle 500 AA spectrometer to accurately measure low-level gold, palladium, platinum, copper, and silver in matrices which result from the fireassay preparation of ore samples. The Syngistix Touch™ software operated from the PinAAcle’s large touchscreen display allows for simple operation when analyzing samples. If more flexibility is desired, Syngistix™ for AA software can run from PinAAcle’s on-board computer – this full-featured software provides flexibility for method development, allows off-line post-analysis data reprocessing, and enhanced reporting capabilities, among other features.

In addition to enhanced software capabilities, the PinAAcle 500 has also been optimized for use in corrosive environments with samples prepared in highly acidic matrices. Examples include acid-resistant coatings on the flame shield and instrument boards.

With its high corrosion resistance, flexible software options, and enhanced analytical capabilities, the PinAAcle 500 is an excellent instrument for measuring precious metals in a mining environment.

References

1. “Sensitivity Versus Detection Limit”, Technical note, PerkinElmer, Inc.

Table 4. Detection limits.

Element	Matrix	Detection Limit (µg/L)
Au	15% aqua regia	10.5
	1% HNO ₃	8.2
Pd	15% aqua regia	13.7
	1% HNO ₃	14.2
Pt	15% aqua regia	56.2
	1% HNO ₃	56.5
Cu	1% HNO ₃	2.1
Ag	1% HNO ₃	1.4

Consumables Used

Component	Part Number
Au Hollow Cathode Lamp	N3050107
Pd Hollow Cathode Lamp	N3050158
Pt Hollow Cathode Lamp	N3050162
Cu Hollow Cathode Lamp	N3050121
Ag Hollow Cathode Lamp	N3050102
Nebulizer Capillary Tubing	09908265
High-Sensitivity Nebulizer with Tantalum Capillary	N3160152
Au 1000 mg/L Standard	N9303759 (125 mL), N9300121 (500 mL)
Pd 1000 mg/L Standard	N9303789 (125 mL), N9300138 (500 mL)
Pt 1000 mg/L Standard	N9303791 (125 mL), N9300140 (500 mL)
Cu 1000 mg/L Standard	N9300183 (125 mL), N9300114 (500 mL)
Ag 1000 mg/L Standard	N9300171 (125 mL), N9300151 (500 mL)
Autosampler Tubes	B0193233 (15 mL), B0193234 (50 mL)

TurboMatrix™ Headspace Samplers with built-in trap

A PerkinElmer exclusive, this technology allows samples to be introduced into the column without using a gas syringe or multiport valves. Instead, carrier gas pressures are precisely regulated to manage transfer, eliminating many of the sources of variability and contamination found in other systems.



Headspace Gas Chromatography – for applications involving the solvent-free extraction of volatile compounds, it's an unsurpassed technique, eliminating the time-consuming steps and risk of human error associated with other GC sample-preparation methods.

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The heated needle area is constantly flushed with carrier gas to remove contamination. Because the column or transfer line is inserted all the way to the needle, maximum inertness and minimal dead volume are maintained.

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A solenoid valve interrupts the carrier gas flow and the vial acts as a reservoir of carrier gas. During injection, as the pressure decays, sample volume is transferred to the column. This prevents carrier gas from diluting the sample and avoids expansion of the sample before injection.

– including forensics, food and beverage, pharmaceuticals and environmental – TurboMatrix™ Headspace (HS) and Headspace Trap samplers are the clear choice for laboratories seeking outstanding throughput and precision.

Critical applications with exceptional performance

- Rapid blood-alcohol analysis

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- Assess fermentation characteristics of samples such as beer and wine

News and Event Update

NexION® 350 wins select science award 2015

We are excited to announce that PerkinElmer's NexION® 350 ICP-MS was awarded the SelectScience 2015 Scientists' Choice Award for Best Spectroscopy Product at Pittcon Conference!

SelectScience, an independent, expert-led scientific review resource for the worldwide scientific community, began the Scientists' Choice Awards in 2007 to enable scientists to voice their opinions on the best laboratory products. Once a year, SelectScience invites members to nominate their favorite products of the year in each category. Select Science has announced the winners of the 2015 Sci-

entists' Choice Awards and PerkinElmer bagged the same for ICPMS as Best Spectroscopy Product of the year 2014. This year's awards, nominated and voted by scientists around the world, celebrate new products that have significantly contributed towards laboratory efforts in 2014.

Congratulations and thank to all involved in development and commercialisation of this award winning product, continuing PerkinElmer's long history of excellence and leadership in ICP MS and atomic spectroscopy.



Company of the year award 2014 instrument business outlook

PerkinElmer, Inc. (NYSE:PKI), a global leader focused on improving the health and safety of people and the environment, today announced that it has been named 2014 "Company of the Year" by Instrument Business Outlook (IBO), a newsletter published by Strategic Directions International, Inc., an industry analyst firm focused on life science and analytical instruments, equipment and related products.

IBO described PerkinElmer as an "industry pioneer" and "prodigious developer

of advanced-technology instrumentation," selected for its "strong financial performance, market leadership, innovative product introductions and key strategic investments." The award recognizes how the Company has "led the way in adapting to the challenges and opportunities of the industry."

"PerkinElmer is truly honored to receive this distinction for our many technological, strategic and financial achievements in 2014," said Robert Friel, Chairman and CEO, PerkinElmer. "We are commit-

ted to delivering advanced solutions and services to help customers gain critical insights, make breakthrough discoveries, and positively impact human and environmental health."

IBO cited PerkinElmer's strengths in several markets, including diagnostics (newborn and infectious disease screening in emerging markets), food testing, and environmental analysis, as key differentiators in its selection for the award.

News and Event Update

Pharma Expo

“Asia Pharma Expo is an international trade fair for the South Asian pharmaceutical industry and takes place in Dhaka. The Asia Pharma Expo in Dhaka took place from Thursday, 08. January to Saturday, 10. January 2015

At the fair, national and international exhibitors were present their latest products and innovations to an expert audience.”

PerkinElmer stall was neatly decorated and illuminated with display of instruments and prominent back drop. PerkinElmer had focused on “Residual solvent analysis (USP 467) and Elemental Impurity Analysis (USP 232/233)” theme. Solutions to new regulations implemented were widely talked. Visitors had shown interest in solutions and services. The eastern team with channel partner were receiving the customers to explain the GCMSHS, FTIR, DSC, Services and consumables. The trade show was very successful for organizers and exhibitors.



Dhaka Textile and Garment machinery exhibition

12th Dhaka Int’l Textile and Garment Machinery Exhibition designed to be the trendsetter for the industry player to showcase new technology, state-of-the-art equipments, materials and services, as well as an excellent avenue for international suppliers and visitors to expand business to the lucrative market and accelerate Bangladeshi technological advances that will impart effective

quality, high speed and competitive cost to gain that all important edge in textile and garments industry. PerkinElmer first time participated as exhibitor to explore and expand its food print in Bangladesh textile analysis. In fact we were only company at exhibition as analytical instrument provide for textile. We promotes UV-Vis and FTIR, ICP, AAS and GCMS.



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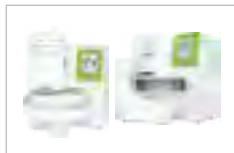
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- DMA
- CHNS/O & N₂ Analyzers



CHNSO analyzer



TGA



DMA



DSC & STA

Hyphenated techniques

- TG-IR
- TG-MS
- TG-GC/MS
- UV-DSC/UV-DMA
- GCMS



TG-IR-GCMS



TGA-MS



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