Fresh

PerkinElmer
For the Better

HUMAN HEALTH | ENVIRONMENTAL HEALTH

Volume 3 March 2009
INDIA ANALYTICAL SCIENCES

Dear Customers

At PerkinElmer, we're taking action to improve the health and safety of people and their environment. Engaged in a proactive fight against illness, contamination and threats to our well-being, PerkinElmer conceives and delivers scientific solutions and components to meet our society's ever-changing needs. From earlier insights and more effective therapies to cleaner water and safer buildings where we work, learn and play; we're taking action to create a better tomorrow.
We are keeping you updated about the scientific solutions and instrumentations available from PerkinElmer for practically all the segments of Industry, Institutions and environment. Any inputs or suggestions are always welcome. Happy reading.
We solicit your queries, enquiries and feedbacks

What's Fresh in this edition…!

- New High Resolution liquid chromatograph (HRLC) from PerkinElmer.
- Confirming the purity of polymorphs by Hyper DSC.
- Dynamic reaction cell in ICP-Mass spectrometer for reducing interference.
- Raman Station 400 for analysis of fats in edible oils.

Can your Fast LC system ….

- Be used for normal HPLC applications with 300mm columns?
- Be used with 4.6mm i.d. columns for ultra fast or high resolution analysis?
- Sustain 10000psi pressure for entire flow rate up to 3000 ul/min
- Has wide variety of injection volumes?
- Has internal needle wash solvent facility?
- Auto sampler with derivatization and dilution facility?

If the answers are NO!!

Then PerkinElmer brings you a new High resolution LC(HRLC) which has all the above facilities in addition to what you have with your Fast LC… to expand your separation capabilities.

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Series 275 HRes UV/Vis LC System

Expand your separation capabilities

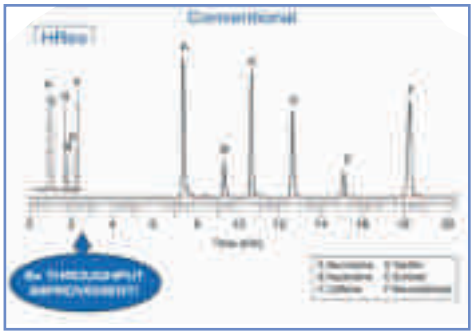
With over 30 years of experience developing liquid chromatography solutions, PerkinElmer is a leading provider of scientific instruments, consumables and services to environmental, biomedical, pharmaceutical, energy, food/agriculture, chemical, forensics and general industrial markets worldwide. Now experience the power of high resolution to liquid chromatography system.

The flexible PerkinElmer® Series 275 HRes LC System leverages small particle-size column technology at ultra-high pressures, giving you greater flexibility to develop methods across a wider operating range. The Series 275 HRes LC System enables today's LC laboratories to achieve higher levels of performance and throughput to meet escalating scientific and business challenges. Precisely designed and configured to minimize system delay volume, optimize operating pressure and flow, and maximize detector response, the Series 275 HRes LC System is compatible with sub-3 µm particle-size high resolution columns for improved performance. Our Brownlee™ HRes LC columns, available in a wide variety of stationary phases and dimensions, can be operated over a wide range of flow rates and operating pressures.

The flexibility of the System allows you to run all your HRes

and your existing conventional methods on a single LC system A wide variety of mixers and detector flow cells are available to address both HRes and conventional applications.

The Key benefits of PerkinElmer Series 257 HRes LC system are



Series 275 HRes UV/Vis LC System reduce retention times

- Greater operating flexibility to optimize method development
- Improved resolution and throughput with higher pressure operating range (500-10,000 psi)
- Outstanding linearity and repeatability even at small injection volumes and high operating pressures
- "Best-in-class" cycle time with exceptionally low carryover drives increased throughput
- Extremely low dispersion for maximum resolving power High-speed data capture assures excellent peak fidelity at high sample throughput
- 21 CFR Part 11 architecture allows operation in a regulated environment.

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Confirming the Polymorphic Purity with Hyper DSC

HyperDSC is a technique where valid DSC measurements are made whilst scanning at rates of up to 500 °C/min. Power-Compensation DSCs, such as the Diamond DSC, are unique in their performance allowing high scan rates to be obtained and rapid measurements to be made at these rates, giving valid measurement of the heat flows occurring in the sample. Two main advantages of this technique are

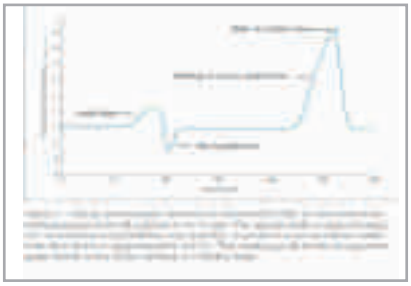
- ability to analyze the sample without changing it
- significant increase in sensitivity.

Whilst the increase in sensitivity is significant for many measurements, Polymorphism Many materials have complex molecular structures that are able to exist in more than one crystalline form, a phenomenon termed polymorphism. Different forms may have different properties and for pharmaceutical use it is important to be able to produce a pure and stable crystalline form of any material to be offered for use as a drug. Using a Differential Scanning Calorimeter (DSC) different forms of such materials may be identified from their melting profiles and differing melting points. Sometimes melting points are so close together that a high resolution analyzer such as the PerkinElmer® Diamond DSC is needed to distinguish them, though on other occasions they may be more distinct.

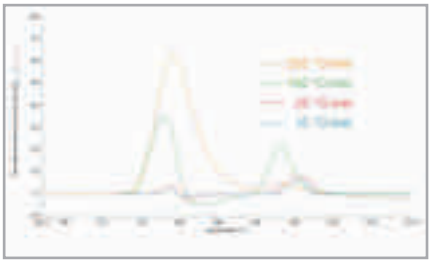
An example is shown above (Figure 1) where one form has melted and then recrystallized into a second form, which has then melted at a higher temperature, a classic picture of polymorphism. However, from a slow scan of Carbamazepine, it is impossible to tell whether just one pure form existed to begin with or not.

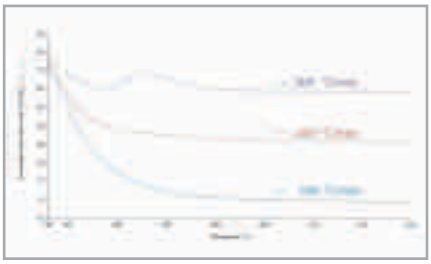
We can see that the sample is recrystallizing, yet we do not know whether all of the higher melting forms resulted from this recrystallization or not, and consequently, whether there was any high melting impurity present in the initial sample.

By scanning very quickly, Hyper DSC™ offers the potential to prevent this recrystallization, enabling us to measure the sample as received, so that the polymorphic purity can be confirmed.



We can find a rate which is fast enough to prevent recrystallization and so enable us to determine how many forms were present initially? Samples of Carbamazepine were heated at increasing scan rates to find out. Different materials will exhibit different kinetics, but the principle shown here is that by making measurements using very high scan rates, true sample properties can be measured without giving the sample time to change. In this case, the polymorphic purity of a pharmaceutical material has been confirmed in a manner not possible using slow scan rates.





Carbamazepine Form III. Thermograms of the tail of the main melting peak at very high heating rates, showing that at 500 ° C/min there is no higher melting form.

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ELAN ICP-MS System with Dynamic Reaction Cell

for reduction of chemical interference using chemical resolution

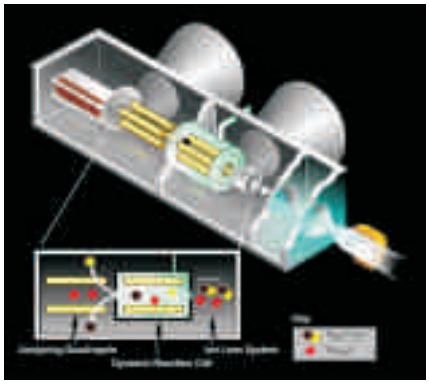
The detection sensitivity of ICP-MS has always been the principle driving force for its rapid adoption in laboratories carrying out ultra-trace element determinations. Since its introduction in 1983, performance in ICP-MS has continually improved, especially in terms of sensitivity. These improvements have resulted in a continual downward trend in detection limits, Sensitivity and background play a key role in determining detection limits. In comparison to other atomic spectroscopic techniques, ICP-MS has relatively few spectral interferences. But those that do occur, can become problematic, especially as sensitivity increases, and the required levels of detection decrease.

Eliminating interferences has been the focus of much research effort in ICP-MS. An obvious approach is to eliminate the interfering species in the sample itself, before it is ever introduced into the spectrometer for analysis. Many successful procedures have been developed using techniques such as flow injection, chromatography, electro-thermal vaporization and evaporative pre-concentration, for example. Each of these techniques has specific advantages, limitations and application.

argon-based interferences, especially for the determination of Fe, Ca and K – all critically important impurity elements in semiconductor manufacturing. The lower energy avoids ionization of the argon and its polyatomics which interfere with Fe, Ca and K isotopes, while ionization in the lower-energy plasma still produces sufficient analyte ions for detection. Though ppt-level detection limits for these three elements can be achieved, the low plasma temperatures limit the ability to ionize all but the most easily-ionizable elements, and the ability to decompose complex sample matrices is compromised.

A far more desirable approach would be to eliminate the interference before the analyte ever entered the mass separation device without compromising the efficiency of the plasma ion source. This can be achieved using a process called "chemical resolution.

Chemical resolution is a process to selectively remove interfering polyatomic or isobaric species from the ICP-MS ion beam using controlled ion-molecule chemistry. A Dynamic Reaction Cell is used to carry out the chemical resolution process. By introducing a reactive gas into an enclosed cell placed in the ion path, the ion beam containing the analyte and the interfering species can be chemically scrubbed of the interference before entering the mass spectrometer for analysis.



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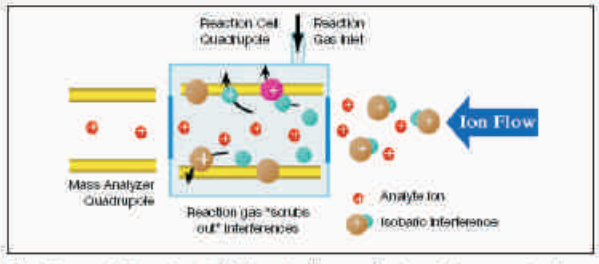
The DRC cell is positioned to communicate between the ion lens and the quadrupole mass separation device of the ICP-MS. The ion beam enters the cell through an aperture, and is efficiently transmitted through the cell by the action of a quadrupole transmission optic element. As the ion beam passes through the cell, isobaric polyatomic interferences selectively react with the fill gas to be transformed into non-isobaric species, so that the analyte is transmitted into the mass spectrometer unencumbered by the interference. Because of the selectivity and efficiency of the interference resolving process, the analyte ion is transmitted with high efficiency, maintaining excellent signal levels.

Advantages of the DRC Approach

1. The DRC device is fundamentally different from the collision cell in several ways.
2. It is operated under thermalized (lower energy) conditions so that ion-molecule reactions are carefully controlled and can be highly specific for the interference being eliminated .
3. A quadrupole is used in the cell device, rather than a hexapole. This is an extremely important distinction because it allows a bandpass filtering function to be applied to the cell, to intercept formation pathways of secondary interferences, which would otherwise interfere with the analyte.
4. While the primary interference may have been eliminated, secondary interferences can be created, once again increasing the background signal at the analyte mass.
5. The DRC exhibits a higher degree of selectivity, resulting in accuracy and detection limits orders of magnitude better than that available from a collision cell using a hexapole. Also high degree of stability as against the collision cell technology and high resolution ICP-MS.
6. Transmission of analyte ions through the DRC, avoids the significant sensitivity losses typical of high-resolution magnetic sector ICP-MS systems.
7. Another major advantage of DRC technology is that a normal, robust plasma with high RF power is always used for the analysis.



The Dynamic Reaction Cell chemically scrubs interfering species from the ion beam, using a reaction gas.

Conclusions

A new breakthrough in interference reduction in ICP-MS has been achieved using the process of chemical resolution in a dynamic reaction cell. Interferences once considered intractable in ICP-MS, degrading the detectability of important elements such as Fe, K, Ca, Se, As and Cr, are found to be greatly reduced or altogether eliminated. Though there is still much to learn about this very new technology, already significant achievements have been reported.

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Raman Station 400 for the analysis of Edible Oils and Fats.

The PerkinElmer RamanStation™ 400 is the ultimate research-grade benchtop Raman spectrometer offering unparalleled versatility and performance. Revolutionary in terms of its performance, ruggedness and flexibility, the RamanStation 400 offers users the most versatile sampling interface, coupled with powerful software, giving users instant access to high performance Raman spectroscopy. Unlike traditional systems, the RamanStation does not require constant user adjustment or alignment, this increases productivity and ensures that experimental results are of a consistently high quality. The ease of use of the RamanStation 400 instrument is complemented by PerkinElmer's tried and tested Spectrum™ data collection and analysis software.

Raman spectroscopy is an ideal method for the analysis of edible fats and oils, which are composed of esters of fatty acids and glycerol. Samples which come from natural sources have a very complex composition, but the key parameters that need to be determined are typically the average chain length of the fatty acids and the extent of saturation. Taken together, these properties determine the melting/softening temperature of the fat sample, which is important in spreading fats such as margarines and butters. In addition, there is a growing interest in the potential health benefits of increasing/decreasing the amounts of various fatty acids in our diet. FAMEs (fatty acid methyl esters) where the spectra of compounds with the same chain length (see Figure 1)

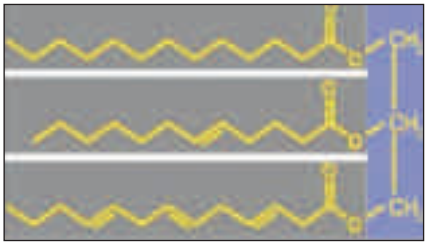

Figure 1. Structure of a Fatty Acid Methly Ester (FAME).

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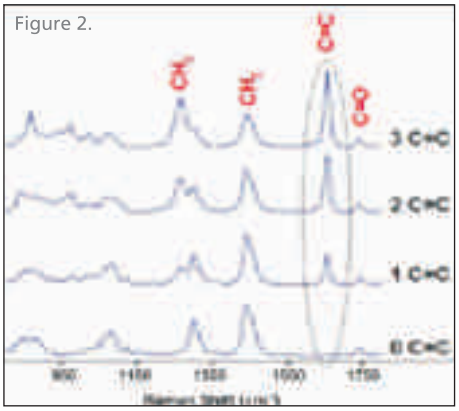

Figure 2. Raman spectra of a series

of FAMEs with unsaturation rangingfrom 0 to 3 C=C and increasing degrees of unsaturation (i.e. double bonds) show a simple monotonic increase in the intensity of the C=C bond at 1655 cm-1 (see Figure 2). This direct link between readily identified spectral feature and the composition of the samples is also found in complex edible oils and fats. Figure 2 shows the spectra of three different plant-derived oils where, again, differences between the oils are reflected in the spectra.

Here the C=O vibration at 1747 cm-1 can be used as an internal standard while different degrees of unsaturation give variations in the relative intensity of the 1655 cm-1 (C=C stretch) absorption band. Similarly, increasing chain length will give increased intensity in the CH2 scissor and twist vibrations which lie at ca. 1442 and 1302 cm-1.

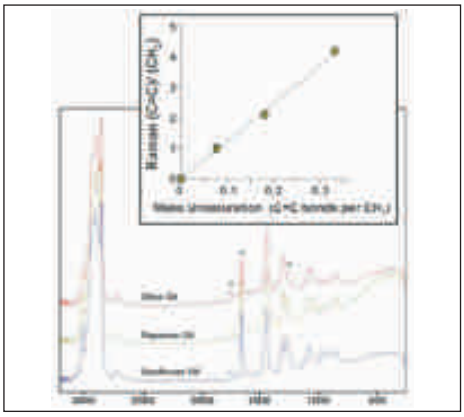

Table1. Wavelengths of peak related to oils and facts.

1276	Symmetric rock in cis (=C-H)
1302	In phase methylene twist
1443	Scissoring mode of methylene (CH2)
1655	cis double bond stretching (C=C)
1747	Ester carbony stretching (C=O)
2850-2980	Symmetric & asymmetric C-H stretching of methyl and methylene grups
3007	Symmetric C-H streetch in = C-H