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Curiosity has landed – Interference filters in current practice

Just a few weeks ago, the Curiosity robot successfully landed on Mars and has already delighted many, including scientists.

Nibble, nibble, little mouse ...

Texture analysis of ginger bread – To protect consumers, modern food technology laboratories increasingly use texture analyzers.

From coal dust to steel

Particle size measurement – The versatile application possibilities of the new SALD 2300.

3D-illustration, NASA/JPL-Caltech

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The "High Absorba of an interference


Curiosity robot (3D-illustration, NASA/JPL-Caltech)

Curiosity has landed – Interference filters in current practice

Just a few weeks ago, the Curiosity robot successfully landed on Mars and has already delighted many, including scientists. Curiosity has been equipped with ten scientific instruments to fulfill its mission. The MastCam is the main camera of the rover.

Both of the camera's optical systems have a filter that enables imaging of the structures to be examined in different visible wavelength ranges. In this way, the surface of Mars can be rendered in true as well as in so-called false colors. In particular via these false color images, it will be possible to highlight differences in mineralogical composition of the Martian soil.

UV-VIS spectroscopy is ideally suited to analysis of interference filters. These optical filters – the same like the ones used for the Curiosity robot – only allow certain wavelength ranges of the visible spectrum to pass through. Depending on the target

Classification	Transmittance range [nm]	Absorption range [nm] (Minimal)	Value [Abs]
Filter 1	270 - 380	270 - 380	0.1
		660 - 770, 700	6.5
		820	4.7
		854	5.3
Filter 2	472 - 490	472 - 490	0.10
		< 330	6.2
		386	6.5
		620	5.87
		637	6.5
		831	6.4
Filter 3	510 - 560	510 - 560	0.11
		< 318	5.88
		808	5.1

Table 1: Interference filter with the approximate transmittance range in wavelengths of the transmission spectra (see Figure 1) and comparison with the "high-absorption" spectra of the filter and the corresponding absorption values (see figures 2 - 4)

nce” method for qualification filter UV-VIS spectroscopy

application, the filters transmit within narrow, broad or wide wavelength ranges. In most cases, an interference filter consists of a multi-layer system designed to enable absorption of a wide range of radiation. This absorption results from reflectance at one or more interfering filter layers. The part of transmitted radiation is determinated by the interference between incident and reflected wavelengths. The shape of the absorption edges, is important for the quality of the interference filter. These edges should indrease sharply enabling a rectangular shaped signal output.

Comparison between three filters

In this application, three interference filters were tested using UV-VIS spectroscopy. The size of the filters of diameter 1 cm requires the use of a suitable mounting bracket, in this case the “rotating film holder“ supporting a small measuring spot via masks. The spectra resulting from this measurement are shown in figure 1. According to the transmission spectra measured, the interference filters are characterized as being more or less narrow-banded (Table 1). The effective range lies within the ultraviolet and the visible range. Compared to filter 2 and filter 3, filter 1 is characterized as being a broadband filter.

Transmission and absorption measurement

Transmission as well as absorption measurements were carried out. Absorption testing is in fact somewhat unconventional. Of interest, however, is the ability of the interference filter to filter out unwanted wavelengths.

Looking at the transmission spectra, one can see that zero-percent transmittance has been graphically achieved. This becomes evident from the formation of a zero-line in figure 1 over the wavelength ranges that should not pass through the filter. The absorption measurement is compliant with the Lambert-Beer law describing absorption in terms of the logarithmic dependence of the transmission: Abs = log (To/T). To is the energy of the transmission before passing through the sample, T after passing through the sample. Abs represents extinction or absorption.

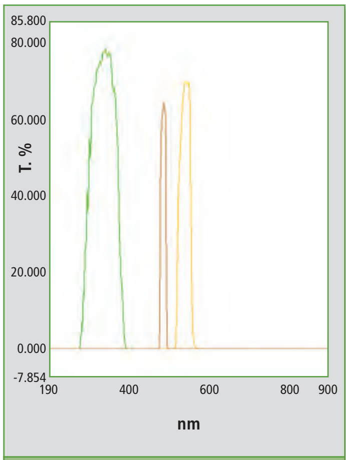

Figure 1: UV-VIS transmission spectra of three interference filters with different wavelength transmittance, the green line representing filter 1, the light-brown line filter 2 and the orange line filter 3. Compare this with the filters used in Curiosity (http://msl-scicorner.jpl.nasa.gov/Instruments/Mastcam/).

Absorption measurements and transmittance

“High absorbance“ measurements, i.e. the percentage of light remaining in the interference filter, exhibit a different transmittance pattern (absorption minima). Raw data of the measurements is presented in figures 2 - 4.

These measurements were carried out using Shimadzu’s UV-2700 and the “high-absorption” method. Using this dual-beam spectrophotometer, it is possible to visualize up to eight absorption units of a material. Looking at the measurements using this method, additional transmittances or more weakly absorbing areas in the interference filter are apparent. The absorption minima indicating transmittances are presented in table 1.

All three filters exhibit individual absorption spectra. Depending on the type of material used for the generation of interferences, specific absorption patterns are created. When the detector is sensitive enough for the interference filters used, these small deviations can be reason for nonspecific background noise.

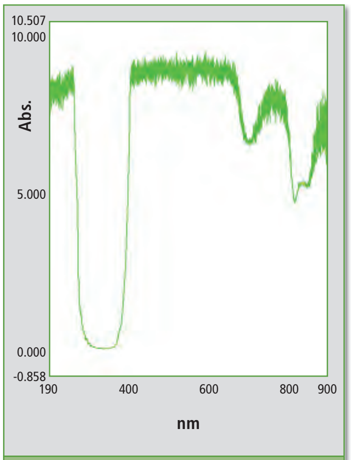

Figure 2: Absorption spectrum of interference filter 1 in the wavelength range from 190 to 900 nm. The spectrum was acquired using a data interval of 0.01 nm at 1 nm slit width and exhibits a transmittance at 270 - 380 nm.

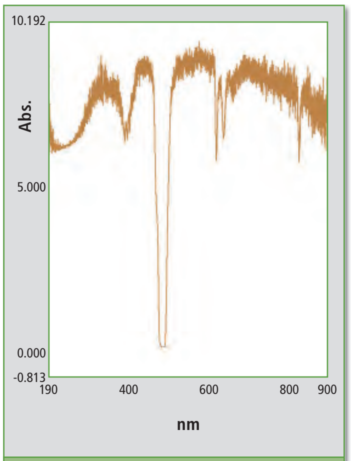

Figure 3: Absorption spectrum of interference filter 2 with a transmittance of 472 - 490 nm

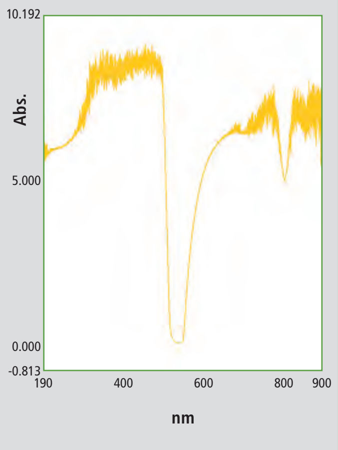

Figure 4: Absorption spectrum of interference filter 3 with a transmittance of 510 - 560 nm

Destruction in the name of

TU Dortmund University and Shimadzu – Universal testing m

Figure 1: Shimadzu in the lecture hall … … Materials engineering lecture 3 at the TU Dortmund University

To teach the fundamentals of materials engineering to large numbers of students, the Department of Materials Test Engineering (WPT) of the TU Dortmund University (Germany) has developed an interactive lecture concept that replaces the former small group laboratory experiments while retaining all active elements (Figure 1). In terms of content, the focus is on visual presentation of practice-oriented knowledge and features mobile small-load testing machines used in advanced research.

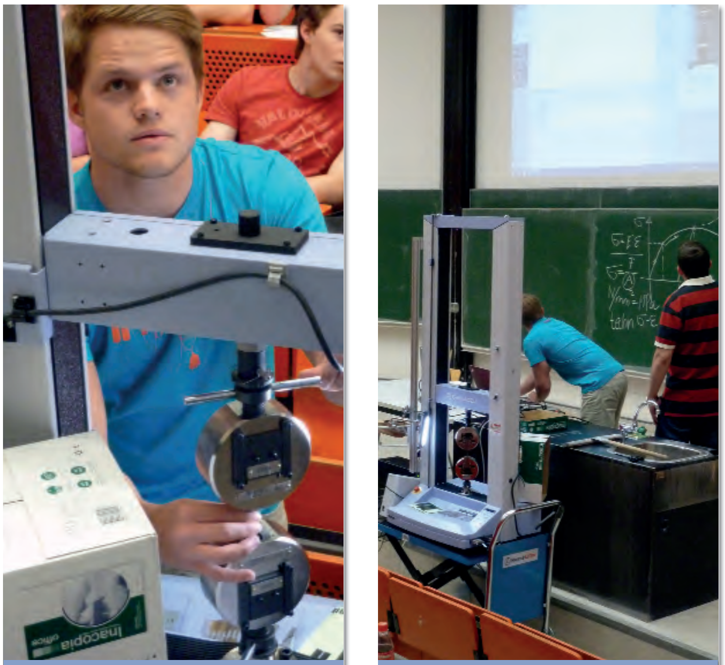

Figure 2: Carrying out a tensile test … … in the lecture hall – TU Dortmund University

Under the guidance and supervision of the staff, students operate measuring and testing systems during the hands-on experiments. The results are subsequently presented and discussed in the auditorium to enhance communication and interaction between instructors and students. As well as increasing motivation and understanding, practical demonstrations of difficult-to-grasp theoretical concepts can significantly reduce failure and dropout rates of students.

The tensile test is a basic experiment used in destructive testing of materials and components. This test was introduced to the young scientists using Shimadzu's AGS-X 5 kN universal testing machine. The instrument is equipped with a TRViewX video extensometer. The student's task

science

achines in education and research

was to prepare and to clamp samples as well as to align the video extensometer (Figure 2). The success of the new lecture concept is evident from the active participation of the students and the highly positive course evaluation.

Accurate measuring and testing for materials assessment

Prof. Dr. Frank Walther is the Head of the Department of Materials Test Engineering (WPT) at the TU Dortmund University in Germany. Basic concepts as well as application-oriented topics in destructive and non-destructive materials and component testing are addressed in research and teaching. The department focuses on the application of various measuring and testing methods to characterize structure-property relationships, assess damage propagation and calculate the (remaining) lifetime of structural materials. Microstructure- and mechanism-based characterization of the fatigue behavior from Low-Cycle-Fatigue (LCF) to Very-High-Cycle-Fatigue (VHCF) as well as corrosion and corrosion prevention are the main focuses. Key qualifications and core research fields are shown in figure 3.

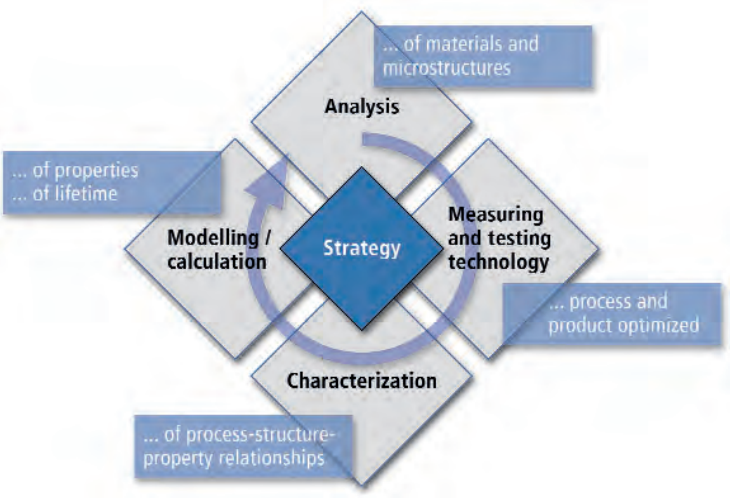

Figure 4: Research strategy

As core elements of the research strategy (Figure 4), the analysis of materials and microstructures is closely linked to process- and production-optimized measuring and testing methods in order to characterize materials and components, model properties and calculate lifetime. Precise description of the influence of manufacturing and operating conditions on fundamental materials parameters serves to optimize the property profile and to increase the functional safety of structural components.

Topics range from fundamental scientific research to application-specific (bilateral) research and development services for industrial companies.

We would like to thank Shimadzu for their excellent cooperation and support of our teaching activities!

Materials and components testing	Fatigue	Corrosion and corrosion prevention
Materialography	Low- and High-Cycle-Fatigue (LCF/HCF)	Electrochemical corrosion testing
Microscopy	Thermo-Mechanical-Fatigue (TMF)	Immersion tests
Destruction testing	Very-High-Cycle-Fatigue (VHCF)	Sol-gel-based coatings
Non-destructive testing	Deformation-induced phase transformations	
Calculation of damage propagation and (remaining) lifetime		
Structure-property relationships of construction materials		
Influence of manufacturing, service loading and corrosion on component properties		

Figure 3: Key qualifications and core research fields

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Holstein and Slovak spotted cattle breeds

Detection of DGAT1 gene polymorphism using microchip electrophoresis

The aim of this study was to detect K232A polymorphism in acyl-CoA:diacylglycerol acyltranferase-1 gene (DGAT1) in the Holstein and Slovak spotted cattle breeds in Slovakia using improved primers and high-throughput microchip electrophoresis (MCE).

Samples of 87 animals of Holstein breed and 42 animals of Slovak spotted breed were genotyped for DGAT1 K232A polymorphism (A and K alleles) using the PCR-RFLP technique.

Clear resolution of three different genotypes was observed following fast, automated analysis of PCR products by microchip electrophoresis. The Holstein breed showed frequencies of 0.8621 for the A allele and 0.1379 for the K allele.

Most animals were AA homozygotes (frequency 0.7356) or AK heterozygotes (frequency 0.2529). Only one KK homozygote (0.0115) was found in the tested sample. The observed frequency of the allele K in tested herds of Slovak Holstein is lower than reported for this breed in other countries. Genotyping data for K232A polymorphism in the Slovak spotted breed showed even lower frequency of the allele K (0.0476) than in the Holstein breed. The results could be used to guide further association studies between this locus and milk traits in these breeds.

Introduction

DGAT1 encodes the enzyme acyl-CoA:diacylglycerol-acyltransferase that plays a fundamental role in the metabolism of cellular diacylglycerol in physiological processes such as intestinal fat absorption, lipoprotein assembly, adipose tissue formation and lactation involved in the metabolism of triacylglycerol in higher eukaryotes [1]. Additional studies have demonstrated the effect of K232A mutation (substitution of lysine by alanine at position 232 of DGAT1 protein) on the function of the resulting enzyme [2]. Other studies have shown that the 2 alleles at the K232A polymorphism segregate in several breeds from different countries [3].

Nevertheless, the estimated frequency of alleles varies greatly according to the population considered, which may reflect different breeding objectives regarding milk composition in different countries and breeds. A significant decrease in protein and milk yields, and increase in fat yield was associated with the lysine substitution (K allele). The alanine variant (A allele) was associated with an increase in protein and milk yields and decrease in fat yield [4; 5].

Microchip electrophoresis has recently attracted much attention in DNA analysis due to its high efficiency, high throughput, time-saving ability, easy operation and low consumption of samples and reagents [6]. In microchip electrophoresis (MCE), nucleic acid fragments are separated at high voltage by capillary electrophoresis in a chip with microfabricated channels, with automated detection as well as on-line data evaluation. MCE has therefore emerged as an effective tool for the electrophoretic separation of a variety of different DNA inputs while providing a viable alternative to conventional agarose or PAGE slab gel electrophoretic applications.

Materials and methods

DNA from hair roots and blood was isolated by the Maxwell 16 Magnetic Particle Processor and corresponding tissue or blood Maxwell purification kit (Promega, USA) following the manufacturer's instructions. Genomic DNA was genotyped by an PCR-RFLP assay for the locus responsible for the DGAT1 K232A substitution. Briefly, PCR reactions were performed in a total volume of 20 µL using 10 - 50 ng of genomic DNA as template, 1X PCR buffer, 2.5 mM $MgCl_2$, 0.2 mM of each dNTP, 5 % DMSO, 0.8 U AmpliTaq Gold DNA polymerase (Applied Biosystems, USA) and 0.4 µM of each primer. The PCR profile included an initial denaturation step at 95 °C for 5 min, 35 cycles of 94 °C (60 s), 63 °C (60 s) and a final extension step of 10 min at 72 °C. Modified primers for the amplification of 352 bp fragment of bovine DGAT1 gene (sequence acc. No. AY065621.1; dgat1 forward: 5'-catcctcttcctcaagctgttct-3'; dgat1 reverse: 5'-gggcgaagaggaagtagtagaga-3') were designed using Primer3 software (frodo.wi.mit.edu/primer3/input.htm).

Restriction endonuclease CfrI (Fermentas, Germany) was used to digest a 352 bp PCR product. The uncut fragment represents the lysine variant, whereas the CfrI fragments of 199 and 153 bp represent the alanine variant.

PCR products were analysed using the automated MCE®-2020 MultiNA microchip electrophoresis system (Shimadzu, Japan) with a DNA-500 kit according the manufacturer's protocol. An SYBR Gold fluorescent dye for DNA staining (Invitrogen, USA) and a 25 bp DNA Ladder (Invitrogen, USA) was used to determine the size of the PCR products.

A software PowerMarker v.3.25 [7] was used to calculate allelic and genotypic frequencies and to estimate the deviation from Hardy-Weinberg equilibrium for a given locus.

Results and discussion

87 animals of Holstein breed and 42 animals of Slovak spotted breed have been tested for K232A polymorphism in DGAT1 gene using novel primers for PCR-RFLP genotyping. New primers have been designed generating the 352 bp PCR product. The uncut fragment represents the lysine variant (K allele), whereas the CfrI fragments of 199 bp and 153 bp represent the alanine variant (A allele). The digestion products could be analyzed effectively by fast, automated and high-throughput microchip electrophoresis showing a clear separation of three different genotypes (Figure 1).

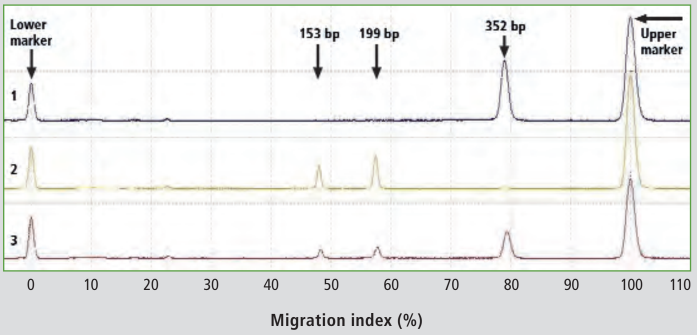

Figure 1: Representative results of PCR-RFLP genotyping of K232A polymorphism of DGAT1 gene in the Holstein breed using the microchip electrophoresis (MCE). Lane 1: genotype KK; Lane 2: genotype AA; Lane 3: genotype KA.

The allele and genotype frequencies as well as the estimation of Hardy-Weinberg equilibrium were calculated by PowerMarker v.3.25 and are summarized in Table 1. The observed frequency of the allele K in the Slovak Holstein cattle herd tested is lower than reported for this breed in the other countries. In German Holstein allele frequencies reported for the lysine variant K range from 0.35 to 0.548 depending on the sample [4], while French Holstein showed a frequency of K allele of 0.37 [9].

The data regarding K232A polymorphism of DGAT1 gene in the Slovak spotted breed showed even lower frequency of the allele K (0.0476) than in the Holstein breed. 38 AA homozygotes (frequency 0.9048) were found, but only four AK heterozygotes (0.0952) in 42 tested animals. Such a low frequency was also reported in the spotted cattle breeds [4; 8]. The chi-square test found a deviation from the Hardy-Weinberg equilibrium of the DGAT1 K232A genotypic frequencies for Holstein and Slovak spotted breeds. This disequilibrium could be related to the sampling of the genotyped animals or can be a result of indirect selection for this locus from the selection for milk production.

The results could be used to guide further association studies between this locus and milk traits, e.g. milk production, fat and protein content in the milk. Well-established diagnostics of DGAT1 variants in individual animals might be extremely helpful to further investigate biochemical pathways involved in the expression of milk production traits or to study possible interactions with other causal genes.

Acknowledgements

This article was written during realization of the "LAGEZ No. 26220120051" and "MARKERY No. 26220220033" projects supported by the Operational Programme Research and Development, funded through the European Regional Development Fund.

Breed	N	Allele frequency		Genotype frequency			HWE	
		K	A	KK	KA	AA	χ^2	p-value
Holstein	87	0.1379	0.8621	0.0115	0.2528	0.7356	0.3490	0.5547
Slovak Spotted	42	0.0476	0.9524	0	0.0952	0.9048	0.1050	0.7459

Table 1: The allele and genotype frequencies and Hardy-Weinberg equilibrium of DGAT1 K232A polymorphism in the tested population of Holstein and Slovak spotted breeds

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Determination of Rare Earth Elements in electronic waste

ICP-OES spectrometry

Figure 1: ICPE-9000 simultaneous ICP-OES spectrometer with dual view

According to IUPAC definition, Rare Earth Elements (REE) are a selection of seventeen elements from the periodic table of elements. In contrast to their name, the REE are present in the Earth's crust much more than expected. Due to their geochemical properties Rare Earth Elements are typically dispersed, and an economic treatment of rare earth minerals and ores is difficult. Nowadays, the biggest ore deposits of REE are located in China (Mongolia) which controls the global market.

As REE are required in the electro- and electronics industry the demand for special metals is booming. In European countries, it may be worthwhile to recycle electronic waste in order to be independent of expensive imports [1].

The Shimadzu ICPE-9000 (Fig. 1) has been optimized for the determination of Rare Earth Elements. This simultaneous ICP-OES spectrometer was used in experiments on electronic waste material such as mobile phones, notebooks, electronic boards and more. Samples were prepared applying crushers and mills, homogenized and finally digested in a microwave digestion procedure.

System configuration for REE analysis optimized

Quantitative determination of the Rare Earth Elements has been carried out using the ICPE-9000 spectrometer and ICPESolution software version 1.31. The simultaneous spectrometer with vacuum optics is especially suitable for fully automatic multi-element analysis of large sample sequences and is characterized by its high flexibility. For instance, the determination of REE must be possible in presence of major elements from the sample matrix such as copper, iron, tin and zinc, in a single method through the application of axial and/or radial plasma observation. Various parameter sets can be executed within one method, which allows the optimization of the system configuration for different sets of samples with high precision and low limits of determination.

The user wavelength registration function is just one important feature of ICPESolution software version 1.31. It allows that analytical wavelengths which are not included in the analysis database can be added by the operator. This function is very useful in view of a huge number of hundreds of analytical wavelengths available for REE in the literature. The key of success for the REE analysis is the proper selection of the optimum lines. A selection of a few analytical wavelengths which have been used for calibration is shown in figure 2.

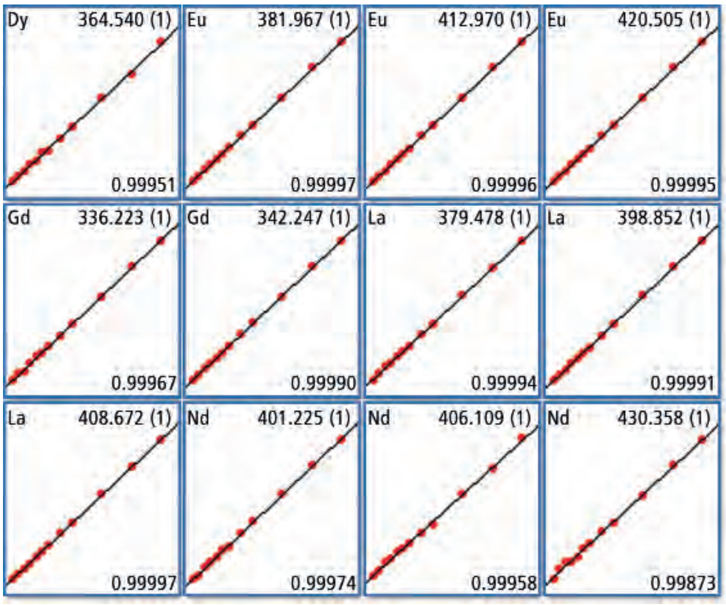

Figure 2: Calibration curves of selected Rare Earth Elements

In a qualitative and quantitative analysis experimental data on standard solutions and sample solutions have been obtained using the instrumental parameters in table 1.

Instrument	ICPE-9000
RF generator power	1.2 KW
Plasma gas	10 L/min
Auxiliary gas	0.6 L/min
Carrier gas	0.7 L/min
Plasma torch	Minitorch
System configuration	axial / (radial)

Table 1: Instrumental parameters for determination of REE

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Golden Delicious, please proceed to radiology!

Energy dispersive X-ray fluorescence spectroscopy of agricultural products

Apple in Wonderland

»What is an apple like you doing here at the radiologist?«
»I am not just an apple, I am a Golden Delicious.«
»So, why are you here, Golden Delicious?«
»For prevention.«
»Prevention? Against peel cancer perhaps?«
»Although it is none of your business, against bitter pit.«
»And what is that?«
»Brown spots, on the peel and in the flesh underneath.«
»Oh, is that serious?«
»Well, I don't really care, but human eyes and taste buds do.«
»Why?«
»It is not necessarily recognized as a beauty mark! Also, it makes me taste corky and bitter. You probably have never eaten an apple, have you?«
»And how will radiology be of any help?«
»Scientists have discovered that the tendency to develop bitter pit is related to the calcium concentration. When this is higher than 30 mg/kg everything is fine. When it is lower than 20 mg/kg I am ill. Between 20 and 30 mg/kg, much depends on magnesium and potassium. And this varies from apple to apple.«
»Oh really ... and what can the radiologist do for you?«
»He can place me in an analytical instrument and irradiate me weakly while my mineral content is being measured. It's that simple.«
»And afterwards?«
»Then I can go.«
»Where to?«
»Where to?" Onto the retail shelf of course. Because I'm fine ...«

Sample	A	B	C	D	E
Ca (ppm)	225	139	246	661	172
K (ppm)	2808	2016	1777	1929	2538
Ratio	12.5	14.5	7.2	2.9	14.8

Table 1: FP-measured results of various apples. EDX-720 measuring instrument in standard configuration. All data in mg/kg (resp. ppm). To increase the shelf life of apples during cold storage, they are often sprayed with $CaCl_2$. This explains the high calcium concentration.

Apple in real life

Apples count among the healthiest fast foods. Although they do not have any record-breaking nutritional value, they do contain a balanced combination of nutrients. Polyphenols strengthen the immune system and can prevent the development of cardiovascular diseases and cancer.

Stored apples in particular, have a tendency to develop bitter pit. These spots on the peel also spread to the underlying flesh. It turns brown and tastes corky.

The tendency to develop bitter pit is related to the calcium concentration. When higher than 30 mg/kg (30 ppm), everything is fine. However, at calcium concentrations lower than 20 mg/kg (20 ppm) the occurrence of bitter pit is very likely. At a value between 20 - 30 ppm, much depends on elements such as magnesium and potassium that are present in apples in concentrations of 25 - 50 mg/kg (magnesium) and 500 - 1500 mg/kg (potassium).

The EDX-720 energy dispersive X-ray fluorescence system is a versatile analytical instrument for agricultural science. It can be used for fast and reliable mineral concentration determination of calcium (Ca) and potassium (K) in apples. The measuring range of the instrument ranges from sodium (Na) to uranium (U) and concentrations from just a few ppm up to 100 %.

For the results presented in table 1, standardless FP measurements of the samples in figure 1 were carried out. Measuring time per sample was 200 seconds. The advantage of this measuring method is that no sample pretreatment whatsoever was necessary.

The entire apple was placed in the instrument and measured. After measurement, the apple was intact and could still be sold to consumers. The large sample compartment of 150 x 300 mm also allows measurement of larger fruits.

In addition to fruits, the EDX-720 can be used for testing of soil or fertilizer compositions.

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Which TOC technique is the best?

Cleaning Validation

TOC-L with ASI-L autosampler

To ensure quality control and safety in manufacturing facilities within the pharmaceutical industry, it is important to apply cleaning validation. This technique is to be conducted after cleaning of production-related equipment in order to ensure that the quantity of residual substances collected from the equipments' surfaces is within permissible limits. Three methods are available for cleaning validation using a TOC analyzer:

1: Rinse sampling
2: Swab sampling – aqueous extraction
3: Swab sampling – direct combustion carbon measurement method

The methods are described here in detail using the $\text{TOC-L}_{\text{CPH}}$ total organic carbon analyzer (Shimadzu Corporation, Kyoto, Japan) for measurement of residual pharmaceutical products and their constituent substances.

Preparation of Residue Sample

To evaluate the cleaning validation sampling methods, residue measurement samples were created by applying various types of pharmaceutical products and their constituents to stainless steel pots. The aqueous and non-aqueous substances used are listed in table 1.

The aqueous and non-aqueous substances were dissolved in water and ethanol or acetone respectively, and the solution concentrations were adjusted to 2,000 mg C/L (= carbon concentration of 2,000 mg/L). Each residue substance measurement sample consisted of a $5\ \text{cm}^2$ area on the surface of a stainless steel pot to which a volume of 100 µL of each solution was applied and dried. Carbon amount in the sample at each application site was therefore 200 µg. Among these, Gentashin ointment (aminoglycoside antibiotic) and Rinderon ointment (corticosteroid) were prepared based on determination of their carbon concentrations using the TOC analyzer equipped with solid sampling module.

Substance	Solubility in water	Solvent used in solution preparation
Tranexamic acid	soluble	Water
Anhydrous caffeine	soluble	Water
Isopropylantipyrine	insoluble	Ethanol
Nifedipine	insoluble	Acetone
Gentashin ointment	insoluble	Ethanol
Rinderon ointment	insoluble	Acetone

Table 1: Sample types

Substance	TOC concentration	Recovery rate [TOC conc. – Blank / Theoretical Conc.]
Blank	0.030	– –
Tranexamic acid	2.14	105 %
Anhydrous caffeine	2.19	108 %
Isopropylantipyrine	2.20	109 %
Nifedipine	2.17	107 %
Gentashin ointment	0.117	4.35 %
Rinderon ointment	0.333	15.2 %

Table 2: Measurement results for Rinse Sampling – TOC Measurement Method

1: Rinse Sampling

The Rinse Sampling method uses TOC measurement of the final rinse water used in the cleaning of a production equipment unit. This method is suitable for systems that cannot easily be disassembled, such as CIP (clean-in-place) equipment and narrow tubing. Sampling is considered to be difficult if the residues are not soluble in water.

To evaluate the recovery of the various substances when using the rinse sampling method, 100 mL of pure water was transferred to the stainless steel pot with its patch of dried sample, and after stirring for 15 minutes to prepare the rinse solution, TOC measurement was conducted.

Substance	TOC concentration	Recovery rate [TOC conc. – Blank / Theoretical conc.]
Blank	0.059	– –
Tranexamic acid	2.19	107 %
Anhydrous caffeine	2.23	109 %
Isopropylantipyrine	1.90	92.2 %
Nifedipine	1.86	89.9 %
Gentashin ointment	0.093	1.70 %
Rinderon ointment	0.208	7.45 %

Table 3: Measurement results for Swab Sampling – Water Extraction TOC Measurement Method

Substance	TOC concentration	Recovery rate [TC conc. – Blank / Theoretical conc.]
Blank	0.00	– –
Tranexamic acid	202	101 %
Anhydrous caffeine	201	100 %
Isopropylantipyrine	210	105 %
Nifedipine	212	106 %
Gentashin ointment	200	100 %
Rinderon ointment	209	104 %

Table 4: Measurement results for Swab Sampling Direct Combustion Method

Measurement Conditions

Analyzer: Shimadzu Total Organic Analyzer TOC-L$_{CPH}$
Catalyst: High sensitivity catalyst
Measurement item: TOC (= TOC by acidification sparge processing)
Calibration curve: 2-point calibration curve using 0 - 3 mg C/L potassium hydrogen phthalate aqueous solution
Injection volume: 500 µL

Since carbon content in each of the residue measurement samples was 200 µg, TOC concentration should be 2 mg C/L if all of the sample were to dissolve in the water.

For the blank, measurement was conducted in the same way using water transferred to a stainless steel pot with no dried sample applied to its surface. The blank concentration measured was subtracted from each TOC concentration and then compared with the theoretical value of 2 mg C/L to determine the rate of recovery. The results are shown in table 2.

Water-soluble tranexamic acid and water-insoluble anhydrous caffeine had high recovery rates as expected. Water-insoluble isopropylantipyrine and nifedipine also had high recovery rates.

However, recovery rates of Gentashin ointment and Rinderon ointment were both low at less than 20 %. Based on these results, it is clear that evaluation of the rinse water using this method is unreliable due to the variation of recovery of substances which are not readily soluble in water.

2: Swab Sampling – Water Extraction

The Swab Sampling Water Extraction method consists of wiping the inside surface of the production apparatus with a fibrous swab material, extracting the adhering material with water, and conducting TOC measurement of the extract solution. Since the residue is physically wiped from a fixed area of the surface of the apparatus using the swab material and then analyzed, sampling efficiency is high. Since however water is used for extraction of the residue, residues that are insoluble in water are difficult to extract. Accordingly, cleaning evaluation with respect to these residues may be difficult for the same reasons as the Rinse Sampling method.

To evaluate recovery of the various substances when using the Swab Sampling Water Extraction method, the sample, which was applied to a stainless steel pot was wiped off with a 5 cm^2 piece of fibrous swab material, which was then placed in a glass jar containing 100 mL of pure water. The residue was then extracted by stirring for one hour, after which TOC measurement was conducted. Since the fibrous swab material used (Alpha 10 obtained from Texwipe Co.) consists of polyester, very little organic material was extracted from the swab itself.

Measurement Conditions

Analyzer: Shimadzu Total Organic Analyzer TOC-L$_{CPH}$
Catalyst: High sensitivity catalyst
Measurement item: TOC (= TOC by acidification sparge processing)
Calibration curve: 2-point calibration curve using 0 - 3 mg C/L potassium hydrogen phthalate aqueous solution
Injection volume: 500 µL
Swab material: 5 cm^2 piece of Texwipe Alpha 10 swab material washed in pure water and dried

Since the carbon content in each of the residue measurement samples was 200 µg, TOC concentration in the extraction solution would be 2 mg C/L if all of the sample were wiped off.

For the blank, measurement was conducted in the same way by wiping the stainless pot, which had no sample applied before conducting extraction. The measured blank concentration was subtracted from each TOC concentration and then compared with the theoretical value of 2 mg C/L to determine the rate of recovery. Results are shown in table 3.

As expected, water-soluble tranexamic acid and water-insoluble anhydrous caffeine had high recovery rates while water-insoluble isopropylantipyrine and nifedipine had high recovery rates of about 90 %. However, recovery rates of Gentashin ointment and Rinderon ointment were both low at less than 10 %. Based on these results it is clear that evaluation of the rinse water using this method is unreliable due to the variation of recovery of substances which are not readily soluble in water.

3: Swab Sampling Direct Combustion Method

The Swab Sampling Direct Combustion method consists of wiping the inside surface of the production apparatus with a piece of inorganic quartz filter paper swab material, and then conducting measurement using a direct combustion carbon measurement system. The swab material with adhering residue is simply placed in the sample boat and the carbon content is measured directly by the TOC analyzer with a connected Solid Sample Combustion Unit (SSM-5000A, Shimadzu Corporation, Kyoto, Japan). Using this method, water-insoluble residues that are difficult to extract in water can also be collected, and measurement can be conducted quickly and easily without the need for any pretreatment such as sample extraction or similar.

To evaluate the rate of recovery of the different types of substances using the Swab Sampling – ➧

Direct Combustion method, quartz glass filter paper swab material was used to wipe the sample adhering to the stainless steel pot and placed in the SSM-5000A sample boat. TC measurement was then conducted.

Substance	Solubility in water	Recovery rate		
		Rinse sampling – TOC measurement method	Swab sampling – water extraction – TOC measurement method	Swab sampling – direct combustion method
Tranexamic acid	soluble	105 %	107 %	101 %
Anhydrous caffeine	soluble	108 %	109 %	100 %
Isopropylantipyrine	insoluble	109 %	92.2 %	105 %
Nifedipine	insoluble	107 %	89.9 %	106 %
Gentashin ointment	insoluble	4.35 %	1.70 %	100 %
Rinderon ointment	insoluble	15.2 %	7.45 %	104 %

Table 5: Summary of measurement results

Measurement Conditions

Analyzer: Shimadzu Total Organic Analyzer TOC-L$_{CPH}$ + SSM-5000A Solid Sample Combustion Unit (IC circuit bypass using system with cell switching valve set)
Cell length: Short cell
SSM carrier gas: 400 mL/min oxygen gas
Measurement item: TC Calibration curve: 1-point calibration curve using 1 % C glucose aqueous solution
Swab material: Advantec quartz glass paper QR-100 (diameter 45 mm) heat-treated at 600 °C for 15 minutes

Since carbon content in each of the residue measurement samples was 200 µg, the TC value would be 200 µg if all of the sample were wiped off. For the blank, measurement was conducted in the same way by wiping a stainless pot with no sample applied. The measured blank value was subtracted from each TC value and then compared with the theoretical value of 200 µg to determine the rate of recovery. Results are shown in table 4. A high recovery rate of about 100 % was obtained for all substances regardless of whether they were water-soluble or water-insoluble.

Conclusion

The methods used here and their respective recovery rates are summarized in table 5. When using the Rinse Sampling and the Swab Sampling Water Extraction methods, substances that do not dissolve easily in water were found to include those havingboth high and low recovery rates. It is thought that this may be due to differences in the strength with which the substances adhere to the stainless steel pot. Accordingly, it is probable that residue evaluation using these methods would be difficult for substances with low recovery rates.

In contrast, high recovery rates were obtained for all substances when using the Swab Sampling Direct Combustion method, regardless of whether the substances were water-soluble or water-insoluble and thereby permitting residue evaluation. This method is therefore considered to be the most effective measurement method for conducting cleaning validation, especially when multiple compounds are being manufactured in the same vat.

LATEST NEWS

NEW: Comprehensive application handbook

Online and laboratory TOCs

As a leader in TOC technology, Shimadzu summarizes its experience in an application hand-book containing the following chapters:

1. Environmental analysis
TOC analysis is carried out on a wide variety of matrices – from groundwater to seawater, from drinking water to wastewater, from soils to sewage sludge. This chapter discusses the diversity in environmental applications and their many different challenges.

2. Pharmaceutical industry
TOC determination is described in the Pharmacopoeia – not just the method, but also tests to verify the suitability of a TOC analyzer for the analysis.

3. Chemical industry
Incoming goods control plays an important role in the chemical industry. In addition to the targeted analysis of known compounds, sum parameters can help to assess the raw chemicals for impurities.

4. TOC special applications
Due to its significance, the TOC sum parameter is widely applicable. The user's scientific curiosity and ingenuity often solves an analytical problem, and TOC is key to the solution.

5. TOC in daily practice
This application range applies to the individual modules, kits, options and support functions of Shimadzu's TOC analyzers. But fundamental concepts and methods are also described.

6. TOC process analysis
Especially during process control, it is important to obtain fast, continuous and informative data on organic pollution levels of waters. In TOC process analysis, the sample is fed continuously to the measuring instrument for subsequent measurement. In this way, the control room can react promptly to any possible process changes. The analyzer can be tailored to the specific measuring task.

We will gladly send you a copy. Please enter the corresponding number on the reply card or order via Shimadzu's News App or News WebApp. **Info 407**

From coal dust to steel

Particle size measurement – The versatile application possibilities of the new SALD-2300

Coal and steel, mines and ironworks – what once made the Ruhr district the industrial center of Germany has now evolved into a knowledge region through structural transformation and high-tech innovation, with world-class research facilities and characterized by new industries and a strong service sector.

The attraction of the Ruhr district is still based on its central location within Europe, associated with an excellent infrastructure of highly trained professionals including, for instance, the world's largest inland port.

While the city of Essen with its coalmines was the leading mining city in the 19th century, Duisburg – today the home of Shimadzu Europa – became one of the most significant iron and steel centers. To this day, almost one third of all raw iron produced in Germany still comes from blast furnaces in Duisburg, while steel production and metalworking is focusing increasingly on high-tech products.

Traces of heavy industry still dominate the Duisburg skyline. But the former production facilities have long since been transformed into leisure facilities and cultural sites. Innovative start-up companies have settled on the former factory sites. New history is being written at historic locations. Since the 1960's, the Ruhr district has successfully implemented its structural transformation.

SALD-2300

Shimadzu has also been involved in this process.

History and new technologies

As successor to the successful SALD-2201 standard model, the measuring range of the SALD-2300 has been extended to 17 - 2500 µm. This enables measurement of both dry and liquid dispersions up to a maximum size of 2500 µm. Conversion of the instrument from dry to wet measurement mode is revolutionary and can be achieved within seconds.

The unique design of the optical path enables automated measurements within a 1-second time interval. In this way, time-dependent processes can be recorded with a temporal resolution that was previously unattainable.

Proven functions and accessories have been retained and further refined. Also the SALD-2300 is available with a high-concentration cell. A calibration of the instrument is still not necessary. Evaluation according to the Mie solution as well as the Fraunhofer approximation is possible. When the refractive index is unknown, a software assistant helps to search for an appropriate refractive index.

Using a more powerful laser (similar to the SALD-7101H), it is even possible to measure strongly absorbing substances such as carbon black. In comparison to the predecessor model, samples with a 10-fold lower concentration can be measured. The stirrer of the MS-23 sampler enables pumping of particles with densities of up to 7.8 g/cm^3. Since this value corresponds to the density of steel, even steel balls with diameters of 2.0 mm can be measured.

Coal and steel – two materials that can be measured using Shimadzu's new particle measurement instrument. The SALD-2300 combines history with innovative technology.

Circuit board failure analysis

Infrared microscopy

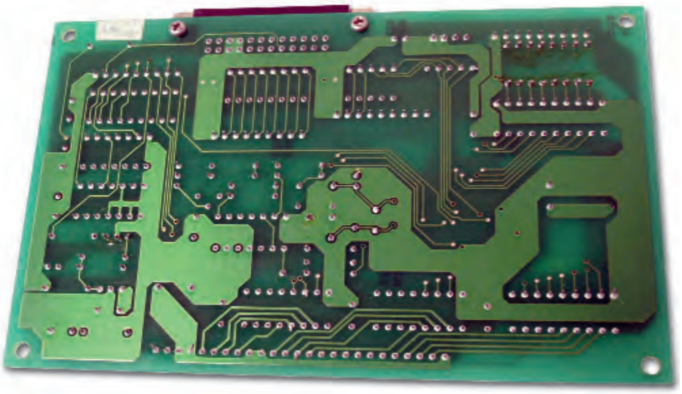

Figure 1: View of a typical circuit board with faultless circuit

Non-destructive materials testing can be carried out using ATR ('Attenuated Total Reflection') microscopy. ATR measurement is used to identify not only the material but also its physical condition. What causes imperfections such as caverns, bubbles or failures in circuit boards? The manufacturer or user of the board needs to know. How can these imperfections be identified? Infrared spectroscopy provides the answer.

For this case, the IRPrestige-21 FTIR spectrophotometer was used in combination with the AIM-8800 infrared microscope and the ATR-8800M objective.

The ATR objective is equipped with a Germanium crystal. The objective head is placed on the sample surface, an infrared beam is passed through the sample at a 30° angle and is reflected off the sample. The beam penetrates approximately 1 µm into the sample surface and records spectral information of the material. Contact surface diameter of the objective is about 50 µm.

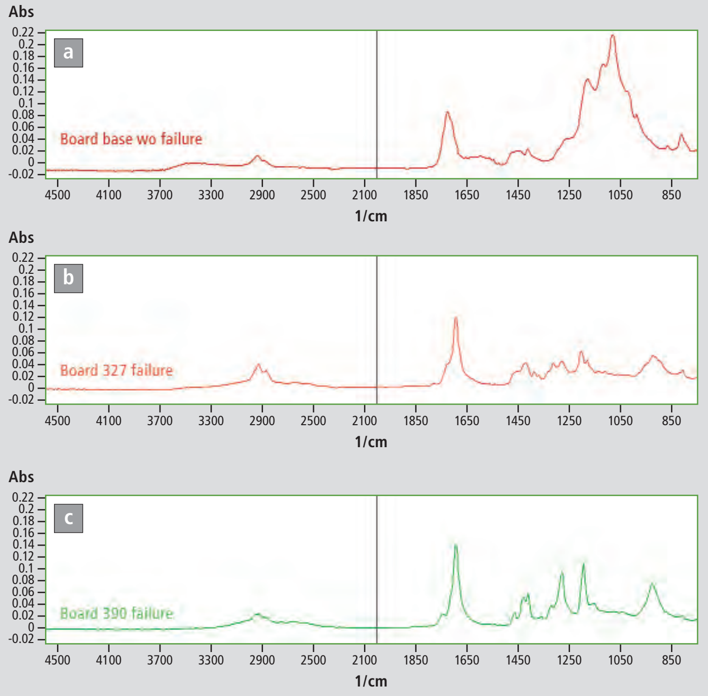

Figure 2: Three infrared spectra resulting from micro-ATR measurement of a circuit board: (a) dark-red line: spectrum of circuit board surface, (b) red line: spectrum of a failed surface, (c) green line: spectrum of another failure

In the diagram above, all three infrared spectra are superimposed and the section at 1700 cm^{-1} has been expanded. The different band positions of the CO signals can be clearly distinguished. The signal at 1720 cm^{-1} originates from the circuit board backing material and the signals at 1690 cm^{-1} arise from the failures.

Cause of failure identified

Boards are manufactured – depending on quality – from fiberglass mats in a network of resins or from paper impregnated with resins or phenols. The infrared spectrum (a) in figure 3 shows typical features of silicone (broad bands at 1200 - 950 cm^{-1}) and resins (~ 1720 cm^{-1}, -C = O, for instance acrylic resins band). In individual measurements for the determination of failures, additional spectra were detected. These can be observed in spectra (b) and (c). In all failure spectra, the dominant signal of an acid at 1690 cm^{-1} ([-C = O]-OH acidic group) is evident.

Spectrum (b) was derived from one failure and closely matches succinic acid (amber). Amber is a natural resin. Library search for spectrum (c) closely matches adipic acid.

The range of failure

With this knowledge, larger areas can also be analyzed using the 'mapping' function. Figures 6 and 7 show a map of the failure on the circuit board.

The view in figure 6 of the circuit board was obtained from images as 400 x 300 µm frames. Measuring area corresponds to an area of 1.25 to 1.65 cm. 800 spectra were measured with an interval of 500 µm.

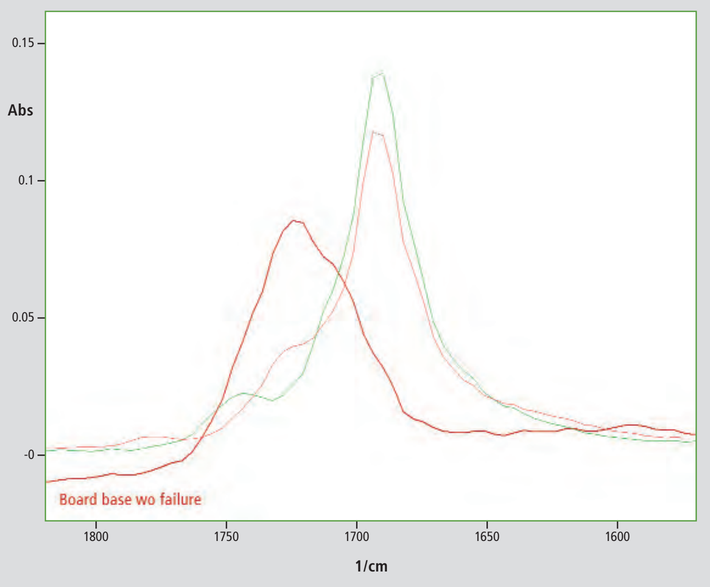

Figure 3: Section of the spectrum of figure 2, representation of the different band positions of the carbonyl groups

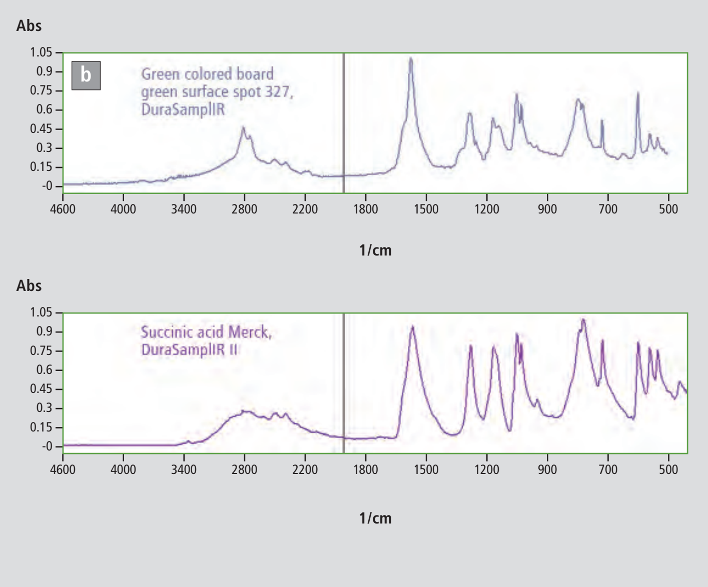

Figure 4: Spectral library search for spectrum (b) closely matches the spectrum of succinic acid

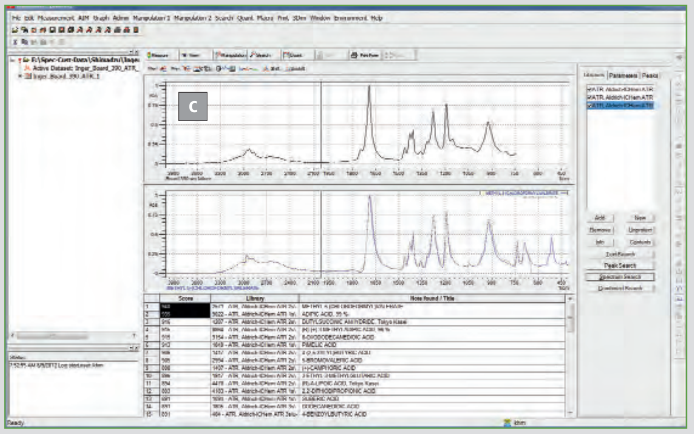

Figure 5: Spectrum (c) and search result of the Aldrich-Ichem library of STJapan-Europe. Adipic acid was a hit.

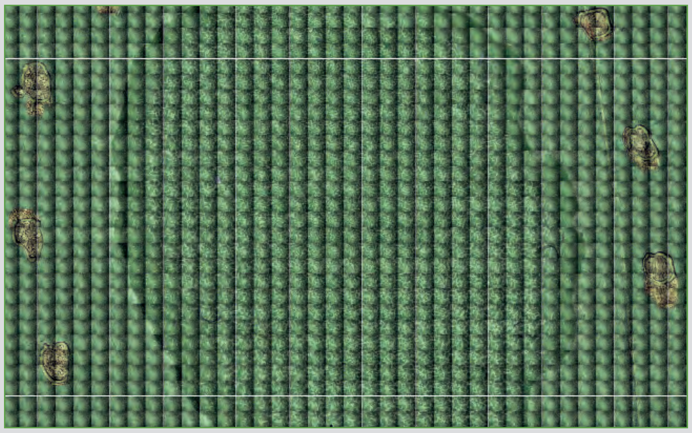

Figure 6: Representation of the mapping in observation mode on a selected visible area of the sample. The view was created from 400 x 300 µm frames.

The 3-D map of the analysis of a circuit board is shown in figure 7, where area has been mapped against intensity of a selected wavelength. The maxima in the figure point to changes on the surface. All the maxima correspond to the spectrum of the coating with a signal of the CO bands at 1690 cm^{-1}. Spectrum (a) of the backing material from figure 1 is found in the flat areas. This view is indicative of an unevenly distributed layer and various layer thicknesses. Where only spectrum (c) was found, the layer thickness was at least 1 µm, while the other positions represent layer thicknesses of less than 1 µm.

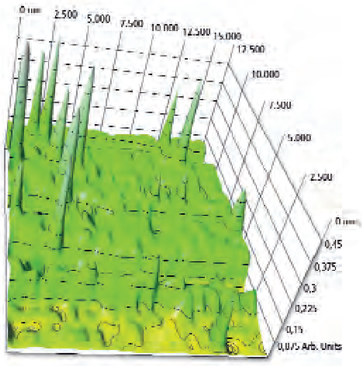

Figure 7: 3-D view of analysis of 800 infrared spectra, with area mapped against intensity at an analytical wavelength of approximately 1700 cm^{-1}

Nibble, nibble, little mouse ...

New texture analyzer EZ-Test-X available

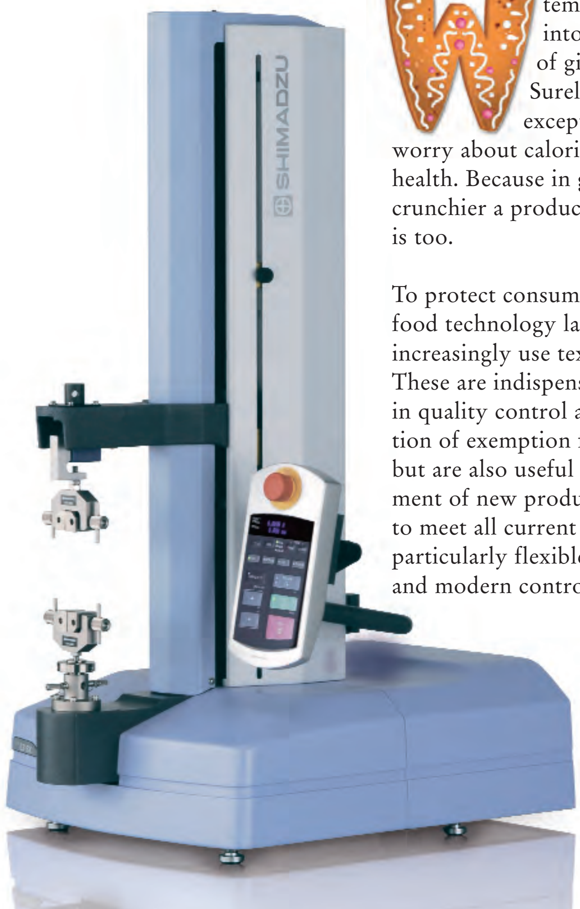

Figure 1: EZ-Test SX with compact screw-type grips

Who can resist the temptation to bite into a tasty piece of ginger bread? Surely not many, except those who worry about calories and dental health. Because in general, the crunchier a product, the harder it is too.

To protect consumers, modern food technology laboratories increasingly use texture analyzers. These are indispensable not only in quality control and in verification of exemption from liability, but are also useful in the development of new products. To be able to meet all current requirements, particularly flexible instruments and modern controls are required.

This is where the newly developed EZ-Test-X texture analyzer comes in. Thanks to the new controller, all load cells are now optionally available in class 1 or even 0.5 accuracy according to ISO 7500 and EN 10002-2. Each load cell covers a vast measuring range from 1/1 up to 1/500 of the nominal capacity selected, in a continuous manner. In this way, measurements of up to 5,000 N but particularly in the most critical lower range of only two millinewtons, can be carried out with excellent precision.

Test speed and positioning accuracy have also been further optimized to attain test speeds of up to 2,000 mm/min with an accuracy of 0.1 %. A high-performance controller that can send up to 1,000 data per second to the PC guarantees this performance.

Not only for the food industry

The EZ-Test-X is not only suitable for the food industry but can also be used wherever very small forces or compact samples must be reliably and reproducibly tested. Typical application areas include, for instance, the measurement of filaments, adhesive tests, shear tests of sensitive electronic components or friction tests of plastic films. The comprehensive Trapezium-X software supports all common standards and regulations, allowing independent optimizing and parameterizing of the tests.

Classic standard methods to determine the characteristics of plastics, metals, ceramics and other materials or products can also be carried out in this way.

To do so, it is possible to connect additional highly sensitive measuring sensors such as clip-on extensometers as well as the new TRViewX video extensometer with the unique accuracy class 0.5 according to EN 9513.

A wide range of accessories ensures a secure hold as well as an exact positioning of the test specimen. The broad arm of the crosshead enables measurement of large-size samples such as circuit boards, but entire loaves or even ginger bread can be tested.

Looking forward to Christmas ...

Figure 2: Modern non-shift wedge type grips in combination with a high-precision load cell

DYMAT 2012 – Looking back

Testing machines and high-speed cameras for materials research

The location of the event – concert hall Fribourg

The dynamic behavior of materials protects human life, preserves instruments and increases efficiency. Improved crash resistance of automobiles, trains, ships and aircraft makes travel as well as transport of goods safer. Today, space satellites are better shielded from impact by tiny particles of space debris, and turbine blades are more resilient than ever. What are the effects on buildings of shockwaves caused by explosions? How do metals behave at high speed? In all these cases, the dynamic behavior of materials plays a crucial role.

Within the DYMAT association, engineers and materials scientists from all over the world exchange research results and new insights on the dynamic behavior of materials.

HPV-2 high-speed camera

As a company with a large material science division, Shimadzu has supported the 10th International DYMAT Conference in Freiburg, Germany, from 2 - 7 September 2012 and has presented a selection of its testing machines. In particular, the HPV-2 high-speed camera records the fastest crack propagation in materials. In ballistic research, the high resolution of 312 x 260 pixels at a speed of one million frames per second (fps) documents the moment immediately following a ballistic impact test in the highest detail.

As up to four cameras can be synchronized, it is possible to record 3D images or images from four different viewing directions. The specific configuration of the camera enables easy implementation of applications such as DIC (Digital Image Correlation).

Dynamic universal testing machines such as the MMT also received much attention. These systems are used for vibration testing of artificial joints, bones, teeth and tissue engineering, and also for fatigue testing of complex products. The MMT table model enables dynamic tests at frequencies of up to 100 Hz, test loads up to 500 N and strokes up to 20 mm. A wide range of accessories, for instance strain measurement systems and air chambers, allows for even more flexible operation while enabling additional testing procedures.

The week-long conference covered various topics related to materials such as steel, concrete, polymers and energy-absorbing materials. Composites and biomechanics were also discussed and there were numerous lectures on experimental techniques. During the breaks, scientists networked and exchanged information. In numerous conversations with users from around the world, Shimadzu received many inspiring impulses.

Joyful and healthy science

Nexera UHPLC – Fast analysis of resveratrol in red wine

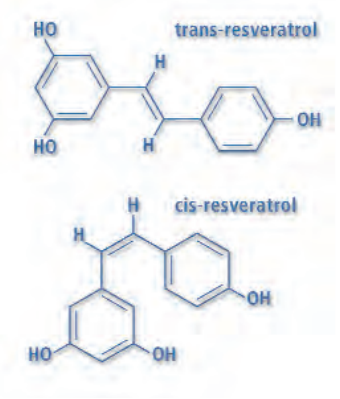

Figure 1: Structural formulae of trans-resveratrol and cis-resveratrol

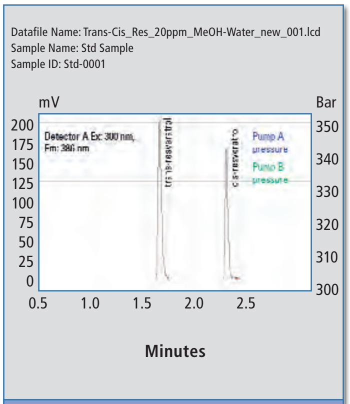
Datafile Name: Trans-Cis_Res_20ppm_MeOH-Water_new_001.lcd
Sample Name: Std Sample
Sample ID: Std-0001

Figure 2a: Chromatogram of a 20 ppm standard mixture of trans- and cis-resveratrol (fluorescence detection)

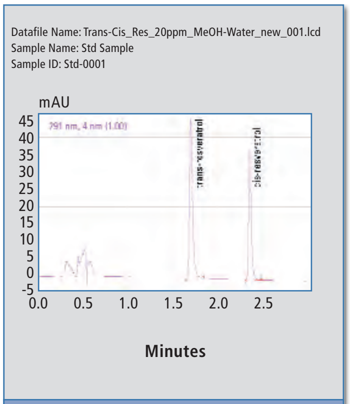
Datafile Name: Trans-Cis_Res_20ppm_MeOH-Water_new_001.lcd
Sample Name: Std Sample
Sample ID: Std-0001

Figure 2b: Chromatogram of a 20-ppm standard mixture of trans- and cis-resveratrol (UV absorption SPD-M20A)

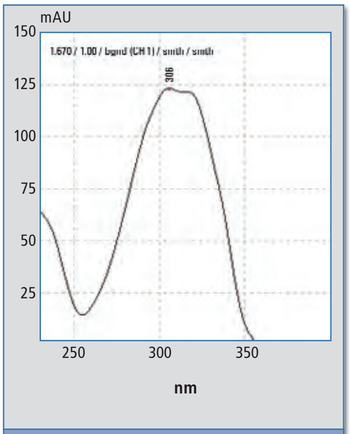

Figure 3a: UV absorption spectrum of trans-resveratrol

Figure 4: The RF-20 AXS fluorescence detector with thermostated flow cell

The substance resveratrol is a component of a plant's innate immune system, which is produced under stress due to fungal attacks, bacteria, virus infections or harmful environmental influences. In chemical terms, resveratrol is a phytoalexin and belongs to the group of polyphenols. In addition to the anti-oxidative effect, it has more health benefits such as anti-inflammatory and chemo-preventive effects and is therefore the focus of current research.

Resveratrol occurs naturally as trans-resveratrol and as cis-resveratrol (see figure 1, structural formulae of resveratrol) and is present mainly within grape skins and – in lower amounts – in grape seeds, stems, vines and roots of the Grapewine. High concentrations are found mainly in vines whose immune systems are challenged by climatic fluctuations. To put it simply, German wines will contain larger amounts of resveratrol than French wines. Resveratrol is generally more pronounced in red wines than in white wines, as the mash remains in the pressed juices for longer.

The research

In this application, a fast and high-resolution analysis of resveratrol using Shimadzu's *Nexera* UHPLC system is presented. Figures 2a and 2b show chromatograms of a 20-ppm standard mixture of trans- and cis-resveratrol. The analytical conditions are listed in table 1. Figures 3a and 3b show the UV absorption spectra of trans- and cis-resveratrol.

Separation column:	Shim-pack XR-ODS III (150 mm x 2 mm, 2.2 µm)	
Mobile phase:	A: 0.2 % formic acid – water (MilliQ) B: 0.2 % formic acid – acetonitrile (HPLC Grade)	
Gradient program		
Time	**Function**	**Value**
0.0 min	BCONC	23 %
1.00 min	BCONC	23 %
2.5 min	BCONC	40 %
2.51 min	BCONC	100 %
4.00 min	BCONC	100 %
4.01 min	BCONC	23 %
Flow rate:	0.7 mL/min	
Column temperature:	60 °C	
Detection (fluorescence):	RF-20 AXS EX: 300 nm, EM: 386 nm measuring cell temperature 20°C	
Detection (UV, photodiode array):	SPD-M20A D2 lamp, 291 nm	

Table 1: Analytical conditions

Due to the photosensitivity of resveratrol (a major amount of trans-resveratrol is converted to cis-resveratrol during UV radiation), connection of a fluorescence detector in series with the photodiode-array detector is recommended to prevent decomposition by the strong excitation energy of the fluorescence detector.

In addition to the extremely high sensitivity (signal-to-noise ratio of

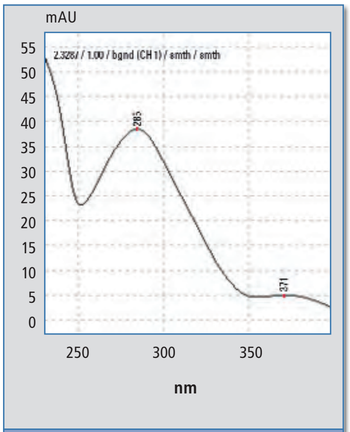

Figure 3b: UV absorption spectrum of cis-resveratrol

the Raman scattering of water > 2,000), the RF-20 AXS (Figure 4) also offers the possibility to thermostat the flow cell. By lowering the cell temperature, the temperature-quenching effects can be reduced and the sensitivity can thereby be increased even further. Figures 5a and 5b show an overlay of trans- and cis-resveratrol at various cell temperatures (20 °C, 30 °C and 40 °C). For both forms it can be seen that when comparing the absorption at 20 °C versus 40 °C, a sensitivity increase of more than 10 % becomes apparent.

The sample put to the test

Shimadzu's anniversary wine released at the occasion of the 60th birthday of UV spectroscopy and the 55th birthday of IR spectroscopy was analyzed for its resveratrol content. Wine sample preparation was carried out using liquid-liquid extraction according to the protocol in figure 6.

The fluorescence chromatogram for an injection volume of 1 µL is shown in figure 6.

Quantification resulted in concentrations of 35.7 mg/L trans-resveratrol as well as 40.4 mg/L cis-resveratrol, which correspond to a real concentration in wine of 7.1 mg/L trans-resveratrol and 8.1 mg/L cis-resveratrol. These concentrations are within the expected range for German red wines.

This experiment, therefore, contributes to the scientific knowledge that red wine, when consumed in moderation, also has beneficial health effects.

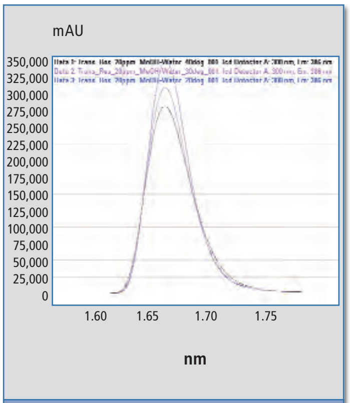

Figure 5a: Overlay of trans-resveratrol at 20 °C, 30 °C and 40 °C cell temperature

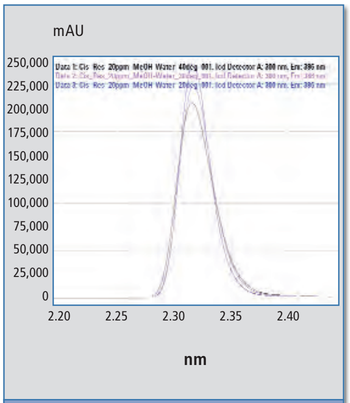

Figure 5b: Overlay of cis-resveratrol at 20 °C, 30 °C and 40 °C cell temperature

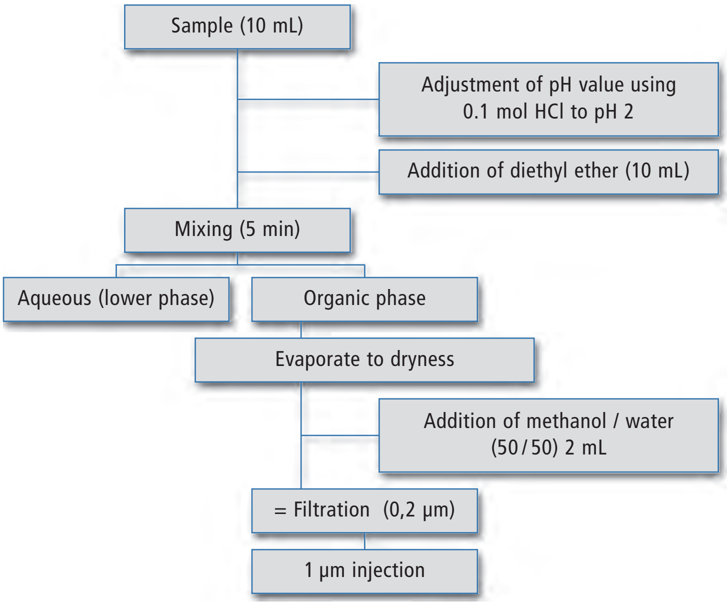

Figure 6: Sample preparation

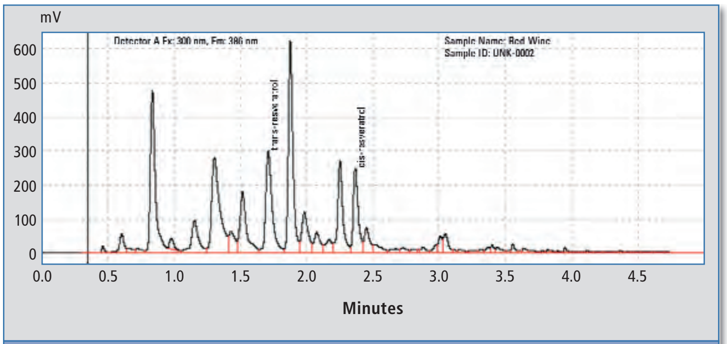

Figure 7: Fluorescence chromatogram: analysis of Shimadzu's anniversary wine

Attractive differences ...

TD-20 thermal desorber – Comparative emission analysis of d

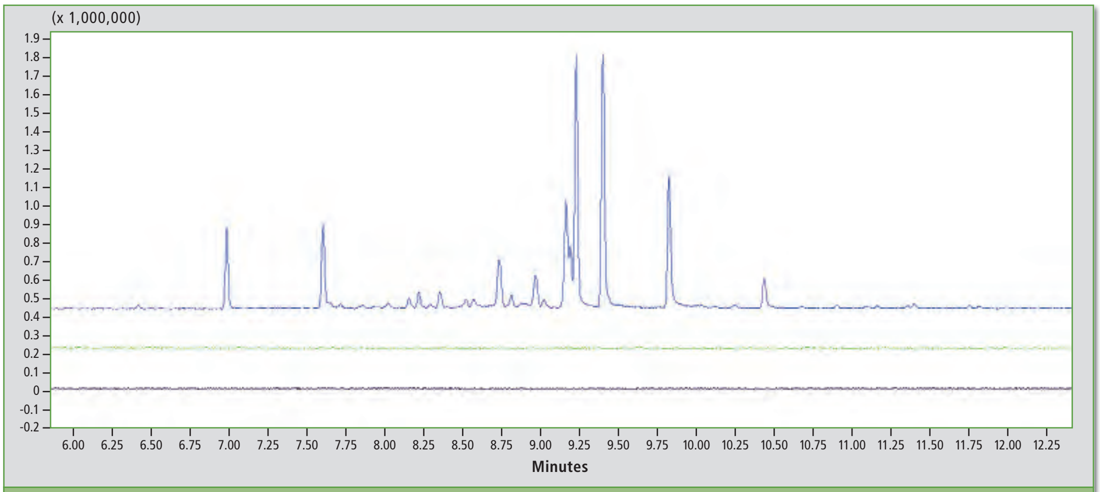

Figure 1: Comparison of low molecular weight emissions from a smelling PVC sample (blue chromatogram) and a reference PVC sample (green chromatogram). The samples were compared with a blank measurement e.g. lab air in a glass tube (black chromatogram). TD-20 conditions: 90 °C, 30 minutes.

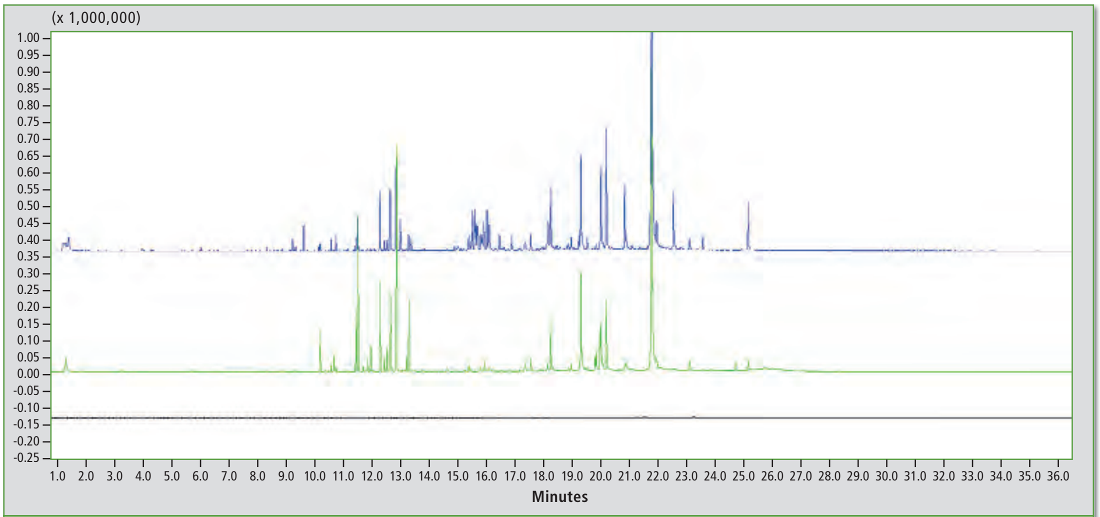

Figure 2: Comparison of emissions from a rubber batch with good physical performance (green chromatogram) and poor physical performance (blue chromatogram). The black chromatogram represents a blank measurement. TD-20 conditions: 120 °C, 60 minutes.

By Franky Puype and Jiri Samsonek
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Polymers are all around us and are indispensable in daily life. Although many polymer manufacturers produce high quality products, it may happen that a defect appears. The source is usually not known and a chemical analysis needs to be done. Defects such as smell, discoloring, fracture, fogging, blooming and contamination are easy to detect if a reference sample is available. This so-called comparative method is generally accepted by the scientific community.

The objective of defect analysis is not to find “bad guys” and “good guys” but to predict unexpected factors and lead to new approaches and prevention. In terms of economics, defect analysis can prevent much trouble for producers and help their customers during a warranty reclaim period.

TD-20 as an excellent tool for polymer analysis

From an analytical point of view, screening of the non-bounded molecules in a polymer matrix usually suffices to find the origin of the defect. Direct thermal extraction of volatiles and semi-volatiles from the polymer by use of the TD-20 has the great benefit that it is quick and requires almost no sample preparation. A piece of polymer is placed in a glass tube and plugged on both sides with glass wool.

Cooling – Sampling – Desorbing – Go!

The TD-20 thermal desorbing system includes a programmable 6-port high temperature valve connected to a cold trap containing TENAX TA trapping material. This trap is cooled before and during sampling to 50 °C below ambient temperature in order to trap a wide range of analytes.

Figure 3: TD-20: Rear, Thermodesorption tubes in the sampler. Front, Heater for desorption step.

efects in polymers

During sampling the polymer in the glass tube is heated, and emissions evolve in a temperature range from ambient to 280 °C according to the needs of the operator. This wide temperature range is useful in evaluation of defects such as smell or high molecular weight contaminants.

After sampling, the trap with the TENAX TA heats quickly to its maximum temperature and the analytes of interest are redesorbed and swept into the analytical column. The connected GC-MS gets its start signal from the TD-20 and off we go!

Figure 4: TD-20 with GCMS-QP2010 Ultra

Smell evaluation

In the following example the TD-20 proves great performance in the profiling of volatiles from PVC samples with complaints of smell (Figure 1). After comparative analysis, a mixture of free aliphatic alcohols was detected in the sample with smell. These aliphatic alcohols should esterify to the plasticizer diisononyl phthalate. This non-esterified fraction causes a typical smell due to its volatility.

Rubber analysis

The TD-20 is a dynamic instrument, the software is easy to handle and its performance guarantees sufficient sensitivity for many applications and a very good reproducibility even for difficult matrixes. By performing an emission fingerprint, much new information can be obtained relating to differences between batches.

Figure 2 shows chromatograms of a defective rubber with poor physical performance and a reference rubber with good performance. Both chromatograms indicate emissions from hydrocarbons and typical rubber additives. A quick comparison of peaks shows that the defective rubber contains nonylphenol which was not present in the reference sample. Nonylphenol is an alkylated phenolic plasticizer and has many isomers. The presence of nonylphenol, in this case as a contaminant, has a high impact on the physical properties of rubber material.

Sample tube (trap tube)	
No. of samples	48
Tube size	1/4" (6,35 mm) x 90 mm
Heating temperature	80 - 400 °C (set in 1 °C units)
Control flow rate range	21 - 150 mL/min (set in 1 mL)
Transfer line	
Plumbing material	Silcosteel©
Maintained temperature	80 - 350 °C (set in 1 °C units)
Valve unit	Valve
Valve	Valco 6-port high temperature valve (1/16")
Plumbing material	Silcosteel©
Maintained temperature	80 - 300 °C (set in 1 °C units)
Cold trap unit	
Trap tube	3.2 outer diameter (2 mL inner diameter) x 100 mm, Silcosteel© tube
Adsorbent	Tenax TA (50 - 60 mg)
Cooling temperature	from 50 °C below RT to 0 °C (set in 1 °C units)
Heating temperature	80 - 350 °C (set in 1 °C units)
Heating time	0 - 30 min (set in 0.01 min units)
Interface unit	
Maintained temperature	80 - 350 °C (set in 1 °C units)
Gases	
Carrier gas	Helium (99.9999 % purity), 0.5 - 0.9 MPa
Purge gas	Air (dehumidified), 0.2 - 0.3 MPa
Purge gas Consumption	3 L/cycle (max.)
Entire system	
Specified environment	Temperature: 18 - 28 °C; 50 % - 70 % Humidity
Storage environment	Temperature: 0 - 40 °C
Power Supply	AC 220 V - 240 V (with down transformer)
Dimensions	323 (W) x 500 (H) x 500 (D) mm + 190 (B) x 206 (W) x 235 (D) mm (down transformer)
Weight	43 kg + 20 kg (down transformer)

Table1: TD-20 unit specification

Need for Speed!

New UFMS systems

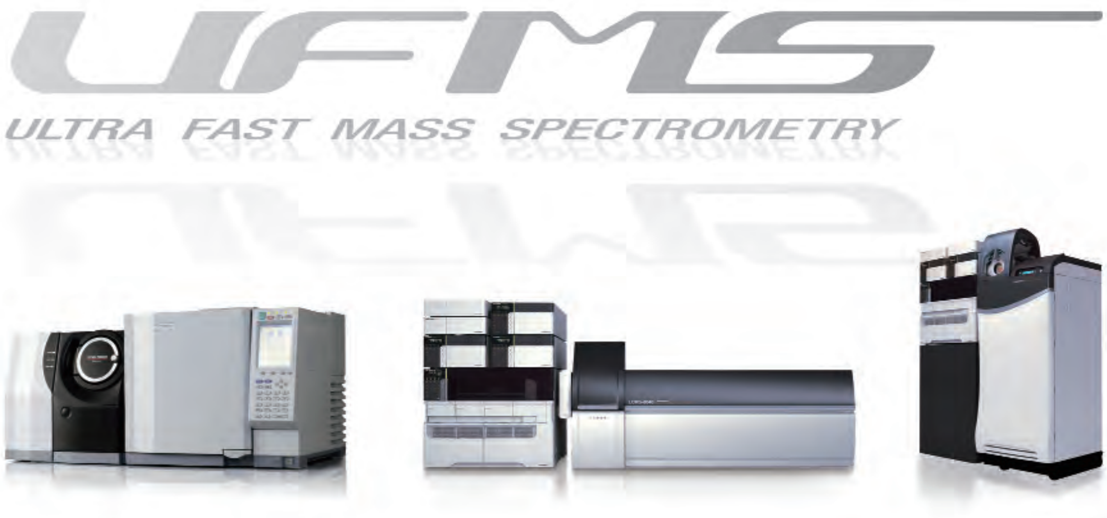

Figure 1: GCMS-TQ8030, LCMS-8040 and LCMS-8080

The ever-increasing number of samples in high-throughput analysis, especially in liquid chromatography (LC), sets the trend towards accelerating separations in order to meet the increasing need to handle more and more sample material in the shortest time possible. Fast chromatographic separations can currently generate peak widths of just a few seconds. Detectors coupled to such LC systems must therefore also meet the requirements for fast and valid signal acquisition and transmission.

For mass spectrometric analysis speed does not only mean high throughput but also the generation of as much information as possible over a short period of time. Modern and fast GCMS and LCMS analysis systems attain much higher data quality and more data points per peak compared to the systems used to date. At the same time, they provide significantly improved spectral quality as well as improved and reliable quantification reproducibility.

Shimadzu has introduced three new fast UFMS systems that combine outstanding data quality with excellent speed parameters.

The new LCMS-8040, LCMS-8080 and GCMS-TQ8030 triple quadrupole mass spectrometers offer a maximum of functionality for various application areas such as food quality control and drug testing. Together with the LCMS-8030, the new systems form a technological platform meeting any individual requirement.

LCMS series

The most important requirement for fast triple quadrupole LCMS analysis in a regulated environment includes, in addition to system sensitivity, fast data acquisition rates in terms of scan speed and the number of MRM transitions (Multiple Reaction Monitoring) that can be measured per second. For more complex analyses, fast polarity switching times also play an important role.

In addition to MRM acquisition, fast triple quadrupole MS systems offer the possibility even for fast LC analyses to customize the gain in information of a measurement without compromising individual problem-solving needs of the user. This is achieved by combining various experimental approaches (production scans, neutral loss scans, precursor ion scans etc.)

In addition, the LabSolution software combined with fast LCMS systems enables system optimization for a particular analyte within a few minutes. Methodically optimized compounds can be measured subsequently via the single methods, or combined and measured via an individually customizable complete method.

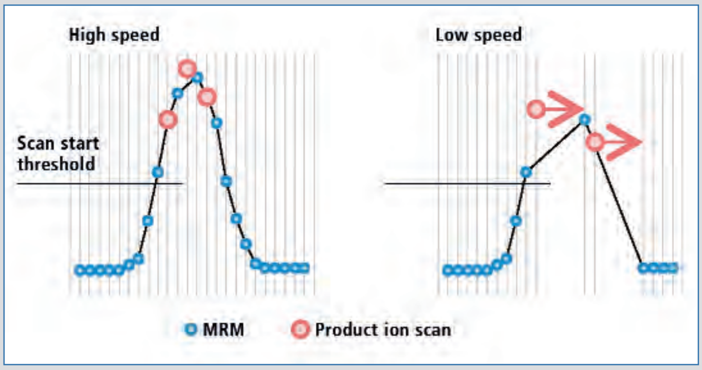

Figure 2: Triggered production analysis during MRM acquisition

Stability and easy maintenance support the 'time is money' aspect. The high sensitivity of the LCMS-8040 and LCMS-8080 extends the individual application ranges considerably.

LCMS-8040 – the stable and powerful work horse

With its improved ion optics and advanced collision cell technology, the LCMS-8040 provides significantly higher MRM sensitivity. Due to the improved ion focusing and higher collision cell efficiency, a five-fold increase in sensitivity (measurement of the standard compound reserpine) of the LCMS-8040 could be achieved in comparison to its smaller brother, the LCMS-8030.

The LCMS-8030 and the LCMS-8040 are unbeaten in terms of their scan speed of 15,000 u/s (in 0.1 u scan steps). This high speed pays off right from the start, as Shimadzu's MRM optimization technique is based on fast automated flow-injection analyses taking just a few minutes. Complex substance mixtures can be optimized in an unattended workflow without any additional syringe injections. MRM parameters optimized for the LCMS-8030 as well as methods developed using these parameters can be transferred to LCMS-8040 systems. Short dwell and pause times ensure acquisition of a high number of data points per peak and reliable quantification, also during rapid and complex analysis. In addition, the high scan speed and voltage adjustment times allow different experiments to be combined in one analysis, for instance a combination of

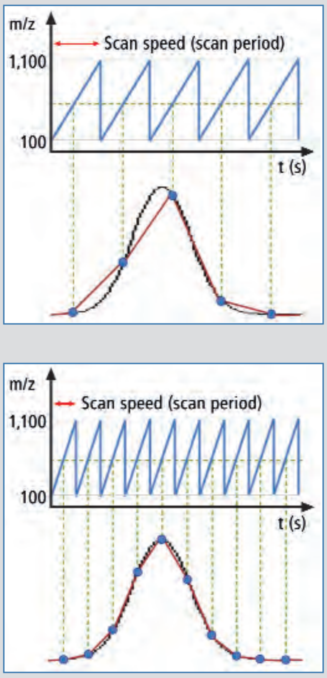

Figure 3: Influence of data points on signal shape

MRM's and triggered product ion scans for quantification and additional qualitative confirmation of the results (see figure 2, left).

Certain patented quadrupole technologies combine high sensitivity of the system with fast data acquisition rates. Fast polarity switching times of 15 ms enable the simultaneous measurement of positively and negatively ionized compounds within one run. The fast data acquisition rates generate more data points and therefore have a direct influence on the signal shape and the quantitative result (Figure 3).

The LCMS-8040's new UFsweeper™ II technology enables ultrafast acquisition of up to 555 MRMs per second with minimal dwell and pause times of approximately 1 ms. Even at such high MRM processing frequencies, the generated fragments are removed quickly and efficiently from the collision cell. This suppresses crosstalk while ensuring unequivocal identification and quantification. To further increase the number of data points for a multicomponent analysis, the LabSolution software enables time scheduling of the MRM acquisition based on elution times of the different analytes (MRM synchronization).

Cleaning and maintenance of the LCMS-8040 is fast and easy. The possibility to exchange the desolvation line without breaking the vacuum ensures stable operation without long downtimes. The LabSolution software combined with several method packages (including database and MRM acquisition methods for pesticides, veterinary drugs, drugs of abuse or water quality analysis) enables a fast and simple analysis startup.

LCMS-8080 – 'Best in Class' in terms of sensitivity and quantification

Transfer of as many ions as possible to the detector is essential in order to attain highest sensitivities. Complex technologies such as coaxial hot gas provide an efficient ionization by combining and heating the gas stream of the ion source which enables the LCMS-8040 to achieve this goal easily. Hot-Source Induced Desolvation (HSID) efficiently removes neutral contaminations. In addition, the laminar flow technique enables easy transfer of large amounts of ions to the detector with high transmission efficiency at high speeds. These unique technologies are advantageous in many applications where a high degree of sensitivity is essential.

However, based on its design, the LCMS-8080 does not achieve the speed advantages of the 8030/8040 series. But the extremely high sensitivity still enables fast high-end analyis of even traces in very complex matrices. The LCMS-8080 completes this series of LCMS systems.

The small footprint of the complete system also saves laboratory space. In addition, the system also runs under the well-established user-friendly LabSolution software.

GCMS series – unique sensitivity and speed

The new GCMS-TQ8030 offers the outstanding performance and ease of use of the GCMS-QP2010 single-quadrupole while integrating the UFMS technology of the LCMS-8030.

The GCMS-TQ8030 offers a sensitivity and speed that no other triple quadrupole GCMS system can match. This is based on Shimadzu's vendor proprietary technologies such as the high-efficiency ion source, noise elimination technology, high-speed scanning control (ASSP) and UFsweeper technology (Table 1).

Description	Patent	Incorporated in MS system	Advantages for users
High-Sensitivity Ion Source	US7939810	GCMS-TQ8030 GCMS-QP2010 Ultra	Ultra high sensitivity
High-Speed Scanning Control (Advanced Scanning Speed Protocol, ASSP™)	US6610979	GCMS-TQ8030 GCMS-QP2010 Ultra GCMS-QP2010 SE	Ultra high scan speed with superior mass spectra quality (no skewing) and no intensity drop
Overdrive Lenses (Noise Elimination Technology)	US6737644	GCMS-TQ8030 GCMS-QP2010 Ultra GCMS-QP2010 SE	Improved signal-to-noise ratios due to noise elimination
High-Performance Quadrupole Mass Filter	US5227629	GCMS-TQ8030 GCMS-QP2010 Ultra GCMS-QP2010 SE	Adjustable and highly precise mass resolution
High-Efficiency Collision Cell UFsweeper®	patent pending	GCMS-TQ8030	Ultra high MRM speed analysis without cross-talk

Table 1: Shimadzu's manufacturer-proprietary technologies

The UFSweeper with its unique design forms a pseudo-surface potential which accelerates ions out of the collision cell. Together with the cleaning mechanism after each transition, this achieves analysis without crosstalk even during ultrafast measurement (600 transitions/s). The unique ability of the GCMS-TQ8030 to carry out high-speed data acquisition with the ASSP functionality, MRM and full-scan (Q3) in a single run (even in fast GC or comprehensive GC applications) yields previously unmatched results of highest quality. Based on its sensitivity and selectivity, the MRM data can be used for quantitative target analysis. Q3 full-scan data with complete spectra and LRI-information (linear retention index) are used for accurate identification in order to avoid false-positive or false-negative results, while also providing information on non-target compounds. This methodology offers the most efficient tool for routine analysis, e.g. a QuEChERS prepared matrix for pesticide screening.

Comprehensive GC is an application area in which the detector speed of the mass spectrometer is extremely important. This analytical application drastically increases the separation performance of the system, whereby two columns are connected in series and a modulator placed between the two columns controls the injection onto the second column. ➧

Figure 4: Twin-line configuration of the GCMS-TQ8030

The second column is generally a short capillary column (2.6 meter) with a reduced internal diameter (ID = 0.1 - 0.15 mm) and a stationary phase orthogonal to the stationary phase of the first dimension column. Typical peak base widths that can be attained in comprehensive GCxGC applications are between 200 - 600 ms. The system must therefore be able to detect one data point for the required information with a sampling frequency of 50 - 20 Hz (one data point every 0.02 - 0.06 sec with complete information over a mass range of at least 350 amu). Only then is it possible to provide reliable quantitative information. Shimadzu offers both GCMS series (single and triple quadrupole) for comprehensive applications (Figure 5).

In addition to the ultra-high scan speed of the single and triple quadrupole, the patented ASSP function represents a key feature. Using this unique technology, a breakthrough in terms of detection sensitivity in high-speed analysis has been achieved. In addition, the spectral quality is unchanged, even at a scan speed of 20,000 u/s of the quadrupole system.

Flexibility is the key characteristic of Shimadzu's GCMS series. The high-performance turbo molecular pump with dual inlet and the differential vacuum construction with optimized surfaces enable adjustment of column flow rates up to 15 mL/min (GCMS-QP2010 Ultra) or 10 mL/min (GCMS-TQ8030). This enables the use of a twin-line MS system in which two columns are installed simultaneously in the mass analyzer without any loss of sensitivity. A twin-line system can be used for smooth acquisition of data sets on different columns without the need for column handling or ventilation of the MS. This drastically increases productivity (Figure 4).

GCMS-Software

The GCMSsolution software, well-known from the GCMS-QP2010 series, offers fast MS/MS method development training. The AART function guarantees automatic adjustment of retention times and can be used, for instance, for method transfer from a conventional to a fast column. The COAST function plays an important role in MRM method development and enables straightforward development of various methods for an MS/MS detection mode.

GCMS-QP2010 Ultra and GCMS-TQ8030

- High-performance quadrupole systems offer highest sensitivity at ultra-high speeds (20,000 µ/s, 100 Hz)
- Provide the most flexible systems, extendable with multidimensional techniques and a vast selection of peripheral equipment for future needs

GCMS-QP2010 SE

- Routine instrument with outstanding price-performance ratio. Meets all requirements of numerous application areas
- User-friendly, easy functionality for straightforward maintenance (Easy sTop) of the inlet system

Summary

Users benefit from Shimadzu's UFMS systems through further improved data quality and higher speeds never before been. Unique technologies set a new global standard in mass spectrometry by providing deeper insights and results, thereby leading to better product, customer and patient safety.

We will gladly send you additional information. Please enter the corresponding number on the reply card or order via Shimadzu's News App or News WebApp. **Info 408**

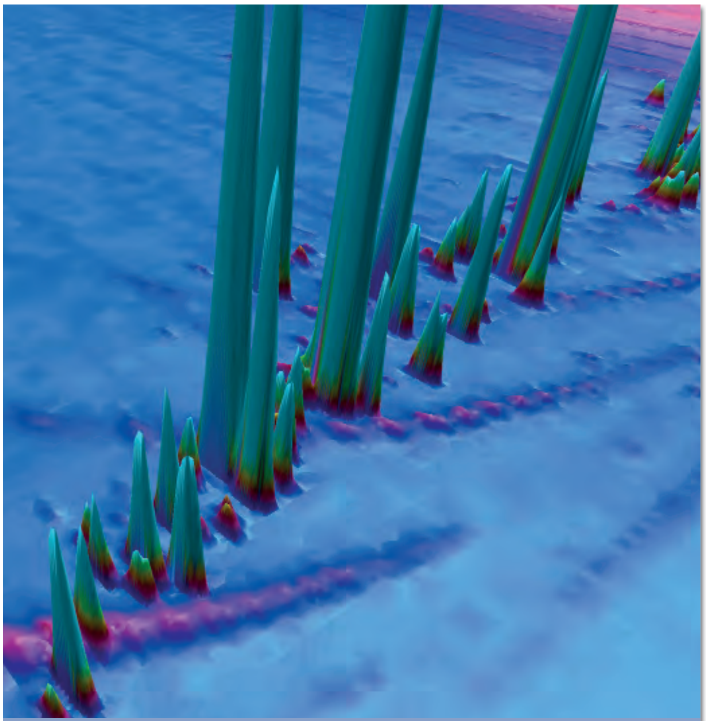

Figure 5: Three-dimensional information obtained by GCxGC application