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An evidence-based trial with the LCMS-8040 Triple Quadrupol







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The ravages of time

IR spectroscopy investigates aging of a polymer bag



or 15 years, it modestly wrapped objects in the household. It had a place in the sun near a window with a view. But then, all of a sudden, the plastic bag disintegrated into dust. At first into small parts, which upon further touching fell apart into even smaller bits. The bag had acquired a 'fractal' life.

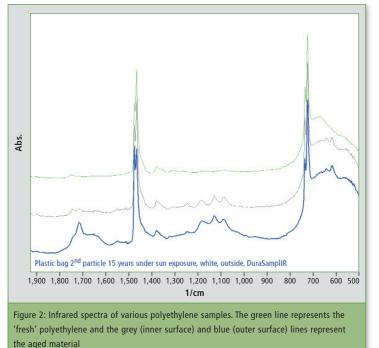
What has happened?

Solar radiation triggered oxidation of the polymer, as has been de-

scribed frequently in the literature. To verify this assertion, fragments of the aged plastic bag were investigated non-destructively using infrared spectroscopy (IR) and the ATR measuring technique (Figure 1). In the present case, a diamond ATR unit was selected. As oxidation is a surface phenomenon, the ATR method is highly suitable. Depending on wavenumber, optical element and window, the penetration depth is about $2 \mu m$ (at 1,000 cm⁻¹) into the surface.



Figure 1: The pressure rod of the DuraSamplIR pushes a disintegrated piece of the plastic bag against the diamond prism



Theory

During polyethylene oxidation, hydroxyl, carbonyl and vinyl groups are formed from the longchain -[-CH₂-CH₂-]n- The polymer chain shortens and becomes more brittle, leading to disintegration of the material after sufficient conversion or progressing of oxidation.

Polyethylene inherently evokes an infrared spectrum that has very few bands. Three significant vibrational ranges leave room for IR windows, one of which is located in the so-called 'fingerprint' region. This is why polyethylene is also used as carrier material for other highly viscous substances (e.g. oils) in order to measure their infrared spectra.

As already mentioned, polyethylene exhibits a simple IR spectrum. The polyethylene spectrum becomes more 'interesting' when aggregants are intended to influence its target characteristics. In this case, vibrational bands enhance the absorptions in the IR window. During oxidation of polyethylene, carboxylic acid derivatives are formed, with CO vibrations which are strongly infrared active. Small amounts already generate strong absorptions in the infrared spectrum at 1,700 - 1,750 cm⁻¹.

Measurements

To generate a reference spectrum, a commercial polyethylene (PE) white plastic bag without print was measured. This bag serves as 'fresh' polyethylene at the defined time 'zero.' The spectrum is free from CO vibrations originating from the group of carboxylic acids.

Inner as well as outer surfaces of aged plastic bag samples were examined (Figure 2). The samples originate from the white parts of the bag which have been exposed to the sun. In this way, it was possible to compare the aged foil to the fresh foil without any distortion of the results due to colored prints and polymer layers containing other substances.

Figure 2 shows the infrared spectra for comparison between fresh and aged foil. In the fingerprint region (1,000 - 1,400 cm⁻¹) the foils differ fundamentally from one another according to different admixtures or fillers. The infrared vibrations of polycarbonate and acrylate do not correspond to the triplet structure in the range of 1,200 - 1,050 cm⁻¹.

Detailed clarification of which of the vibrations in the fingerprint region are due to the aging process would be necessary. The car-

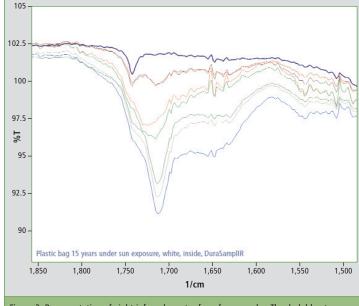


Figure 3: Representation of eight infrared spectra from four samples. The dark blue to bright red spectra represent the inner surface and the green to blue spectra represent the outer surface.

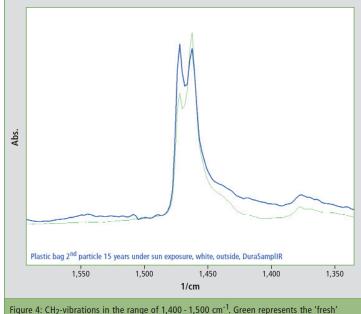


Figure 4: CH₂-vibrations in the range of 1,400 - 1,500 cm⁻¹. Green represents the 'fresh foil while blue represents the aged (outer surface) foil.

boxylic acid vibrations represent a simple range to detect aging. In addition, the aged foil – inner and outer surface – can now be compared. The signal at 1,710 cm⁻¹ is clearly recognizable, arising from the carboxylic acid group. The presence of this vibration points to the aging process.

Trace analysis with plasma

World premiere – The new detector concept: barrier ionizatio

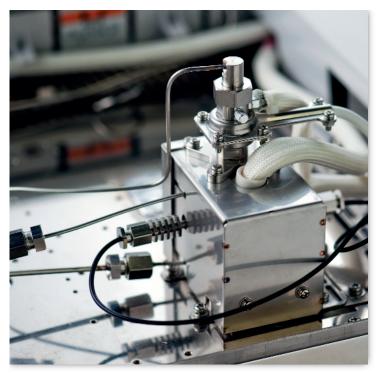


Figure 1: Barrier Ionization Discharge Detector (BID)

he need for a robust, easy to operate but sensitive universal detector in gas chromatography has existed for a long time already. The flame ionization detector (FID) is popular due to its outstanding combination of longterm stability and sensitivity. Unfortunately, its detection spectrum lacks permanent gases such as nitrogen, carbon dioxide etc. The thermal conductivity detector (TCD) is a universal, robust detector and can detect all components. It is not, however, sensitive enough for today's detection requirements. The family of helium ionization detectors (HID) offers a solution. These detectors are highly sensitive, although they

require a certain finesse in handling and some compromises also had to be made with regard to their long-term stability.

The new BID detector concept

With the barrier ionization discharge (BID) detector, Shimadzu launches a new concept in helium ionization technology (Figure 1). As with most HID detectors, a helium plasma is generated by electrical excitation of helium atoms (Figure 2). The discharge gas (ultrapure helium) used for this purpose is introduced at the head of the detector. The electrodes arranged in the upper half



Figure 2: Plasma technology of the BID

(see Figure 3) bring helium to the excited state and create a cool helium plasma.

A thin tube made of quartz glass serves as

dielectric barrier between the electrodes and the helium plasma. In this way, the electrodes are not in direct contact with the helium plasma and are therefore not contaminated or affected by the plasma itself. This is one reason for the long-term stability of the barrier ionization discharge detector. Tests have shown that the relative sensitivity hardly changes, even over more than 3,000 hours of operation. Only during the first weeks of operation does the detector sensitivity increase somewhat (see Figure 4).

In principle, the BID can be divided into two zones (Figure 3). The plasma is generated in the upper zone, ionization and detection of the chromatographically separated components takes place in the lower zone. Ionization of the components does not take place through collision with excited helium atoms, but rather via light emitted from the helium plasma. The ionization energy of the light is 17.7 eV, whereby the BID is able to detect all organic and inorganic compounds at lower ionization energy. Only helium itself and neon cannot be detected.

Figure 3: Cross-sectional drawing of a BID detector. Introduction of the discharge gas and helium plasma generation takes place in the upper half of the detector, while detection of the components eluting from the capillary column takes place in the lower half.

technology

n discharge detector

Improved detection limits

Special 'sapphire cobalt alloy' electrodes have been developed for compound detection. The BID reaches detection limits that are about 50 - 100 times better than the TCD and, depending on the compound 1.5 – five times better than the FID. With five orders of magnitude, the dynamic range of the BID is equal to that of the TCD and only slightly smaller than the dynamic range of the FID.

Despite its high sensitivity, the BID appears to be highly tolerant to high concentrations. In this regard the BID, just like the TCD, can be used for the analysis of liquid samples. The signal arising from the solvent used for the sample does not have to be faded out via 'heart-cut' techniques. Although the detector is overloaded by the solvent, it does not need much more time than a FID to return to baseline level. This is another strong feature of the new plasma technology.

BID bridges an application gap

By combining long-term stability with high sensitivity, the BID bridges a gap between the robust but less sensitive TCD and the highly sensitive, but in terms of operation, quite challenging pulsed discharge helium ionization detector (PDHID).

High sensitivity combined with good tolerance for detector overload brings the BID into play as a possible substitute for a FID detector. Especially for organic compounds containing heteroatoms (e.g. short-chain alcohols, aldehydes and ketones), the much more sensitive BID scores well in comparison with the FID. As a result, the barrier ionization discharge detector not only bridges a gap, but also offers new perspectives for many applications currently requiring multiple detectors (e.g. greenhouse gas analyzers).



relative BID sensitivity

Top athlete

IRTracer-100 for high sensitivity, high resolution and high speed applications

he new IRTracer-100 is a middle-range FTIR system offering high core performance, including the highest sensitivity levels in its class (S/N ratio of 60,000:1), high resolution (0.25 cm⁻¹), and high measurement speed (20 spectra per second in Rapid Scan mode). High core performance stands for sophisticated FTIR analysis, such as highly sensitive measurement of micro contaminants, tracking measurements of rapid chemical reactions, or precise measuring of semiconductor or other materials.

To guarantee high performance with maximum stability, it also features an interferometer with advanced dynamic alignment and built-in dehumidifier and an instrument monitoring function.



Figure 1: IRTRacer-100 Fourier Transform Infrared Spectrophotometer

Customized system configuration

The IRTracer-100 system is extremely flexible with a wide range of accessories and easy-to-use software options meeting the needs of each specific application. Measurements are possible from the near to the far infrared range. The large sample compartment allows an easy setup of accessories including auto recognition.

Specifications are top-level for both hardware and software. Using the IRTracer-100 in combination with the new LabSolutions IR software family which was developed jointly with the IRTracer-100, makes it simple to fully utilize the high performance capabilities and enter sophisticated application areas.

The LabSolutions IR software incorporates data processing functions such as advanced ATR correction and Kubelka-Munk conversion, quantitation functions such as the multi-point calibration curve method and the CLS method, as well as the spectral searching function as standard features. Optional add-on software is also available to further increase the application range with time course, PLS-quantitation, mapping, high level macro programming and much more. For accurate identification of unknown samples, the LabSolutions IR software includes a library of 12,000 spectra.

The gardener did it ...

A legal trial using the LCMS-8040 triple quadrupole



any valuable Mediterranean plants hibernate during the cold season in greenhouses, where they are watered and fertilized regularly in order to reveal their full splendor again in the following summer. But a greenhouse in St. Gallen, Switzerland led to the plants deadly fate. They had been poisoned with an unknown substance. The Canton's police force was called in.

A suspect was quickly found. But how could it be proven that the plants had really been poisoned? This became the responsibility of the forensic science services of the St. Gallen Cantonal Police.

Wherever possible, two independent methods of analysis are used to provide physical evidence. The staff of the forensic science service now had to figure out which methods would stand up to legal scrutiny in court.

LCMS/MS provides clues

One option was Shimadzu's newly installed LCMS-8040 triple quadrupole mass spectrometer. The use of liquid chromatography coupled to tandem mass spectrometry (LCMS/MS) offered an excellent possibility. This technique is superior to other methods in terms of selectivity, due to the underlying 'multi-reaction monitoring' (MRM). The desired parent compound is selected via the first quadrupole (Q1) and fragmented in the collision cell (Q2), while one or more specific fragments are selected by means of the third quadrupole (Q3). It is possible to determine a wide range of analytes with only one analytical method, even without complex sample preparation. Figure 1 shows a schematic representation of the LCMS-8040 triple quadrupole.

Development of the detection method was promptly incorporated into the current user training for the LCMS-8040 triple quadrupole MS and the accompanying LabSolution software.

The fully automated MRM optimization was performed with a dilution of the substance recovered from the suspect pesticide container.

An HPLC method was quickly added, but optimization of the

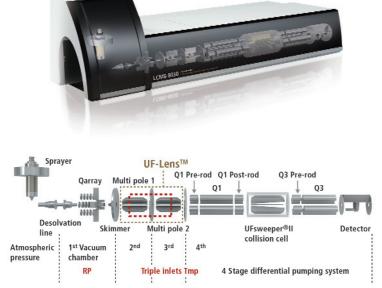


Figure 1: Schematic representation of the Triple Quadrupole LCMS-8040

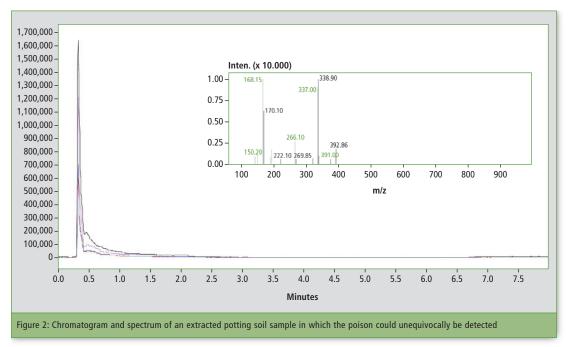
chromatographic conditions was largely omitted due to time pressure. The exciting moment of detection of a plant poison in the potting soil had soon arrived.

The approach

25 g of the contaminated soil was slurried with 50 mL of ultrapure

water and subsequently filtered. In addition, an uncontaminated soil sample was extracted to use as a blank control. Would this fast and straightforward method already suffice to convict the perpetrator? Yes!

In all five potting soil samples investigated, the plant poison



Talking about meats

TOC determination in phosphoric acid in the food industry

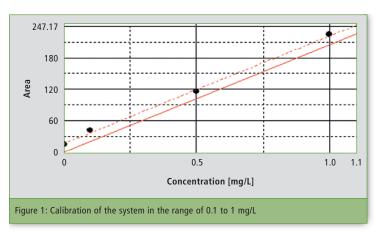
could be detected unequivocally (Figure 2 – chromatogram and spectrum of an extracted potting soil). 1 µL of the filtered solution was injected into the LCMS-8040 which was coupled to the Nexera X2 UHPLC system. In addition to optimized mass transitions (MRMs), the method included a full scan of a defined mass range in the positive as well as in the negative ionization mode. This is possible without loss of sensitivity due to the excellent speed parameters of the LCMS-8040. With a polarity switching time of only 15 ms, this is one of the fastest systems on the market and offers a scan rate of 15.000 u/s and dwell times of 1 msec.

The second detection method

The DART-TOF-MS (Direct Analysis in Real Time - Time of Flight) of a different supplier was selected as the second detection method. DART is an ion source that instantaneously ionizes gases. liquids and solids under ambient pressure. DART does not require any sample preparation, meaning that solids and liquids can be analyzed by mass spectrometry in their native state. Ionization takes place directly on the sample surface. The process involves an interaction between the analyte molecule and electronically excited atoms or vibronically excited molecules.

A DART ionization source can be connected to Shimadzu's single quad LCMS-2020 mass spectrometer as well as to its triple quad LCMS-8030 and LCMS-8040 mass spectrometers.

Science proved ... it was the gardener who killed the plants.



Phosphoric acid is one of the most frequently used inorganic acids in industrial applications. It is applied as starting material for the manufacture of phosphate-containing fertilizers as well as for the production of water-softening agents such as detergent additives.

Phosphoric acid is also used in the food industry - as acidification agent and preservative in beverages or as antioxidant in meats and meat products. Particularly for these types of applications, it is important to apply acids that are pure and free from foreign substances. Manufacturers and processors of phosphoric acids are increasingly using the TOC (Total Organic Carbon) sum parameter for quality control. This parameter is a measure of the contamination of phosphoric acids by organic components.

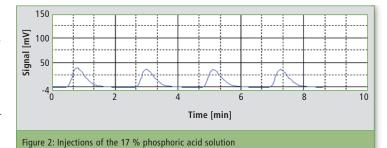
Wet-chemical UV oxidation at 80 °C

The determination of the TOC content in phosphoric acid is carried out via wet-chemical UV oxidation using Shimadzu's TOC- V_{WP} . Its core technology is the powerful oxidation applying a combination of sodium persulfate and UV oxidation at 80 °C. This ensures that all dissolved carbon compounds are converted to CO₂.

In the presence of persulfate ions and UV illumination, OH-radicals are formed which have a strong oxidative effect and convert organic compounds to carbon dioxide. A carrier gas transports the carbon dioxide formed to the NDIR detector where they are detected. Automated reagent preparation eliminates any contamination of the reagent solutions and minimizes the blank value of the instrument.

Calibration of the system was carried out in two different ranges. Calibration is shown here for the range of 0.1 to 1 mg/L. TOC determination in phosphoric acid was carried out using the NPOC (Non-Purgeable Organic Carbon) method. Prior to TOC determination, neutral or alkaline samples are acidified in order to decompose all inorganic carbonates and bicarbonates. This step could be omitted for phosphoric acid.

Concentration of the phosphoric acid does not play a significant role in the determination; it is only necessary to ensure that the acid is not too viscous. The 85 % phosphoric acid solution was therefore diluted 1:5 with water. The resulting 17 % acid solution was transferred into the instrument using an OCT-1 autosampler. For each analysis, a 3,000 µL aliquot of the acid was injected. The 17 % phosphoric acid solution measured in this way resulted in a TOC concentration of 0.61 mg/L. The relative standard deviation over three injections was 1.8 %.



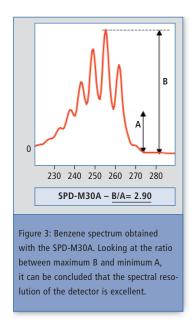
Crucial steps ahead

World premiere – The new HPLC SPD-M30A photodiode array



Figure 1: The 80's reloaded: First generation SPD-M1A HPLC photodiode-array detector

ith the introduction of the SPD-M30A detector, the Nexera UHPLC series adds a new member to the family. Just like Shimadzu's photodiode detectors of the past (Figure 1), the new detector (Figure 2) is also one step ahead of the everincreasing demands of modern



high-performance liquid chromatography.

In the era of UHPLC where peak widths of less than one second are quite common, detectors must



Figure 2: The new SPD-M30A

provide very fast data acquisition rates. At the same time, ultrafast separations should not suffer from loss of sensitivity.

In order to obtain informative data, the instruments must provide high spectral resolution. Combined with high sensitivity, high spectral resolution represents the optimum, and it is one of the strengths of the new SPD-M30A. The sophisticated optical unit, the state-ofthe-art electronics and the flow cell are crucial components as well.

Optical path length and cell volume

To meet today's demands in UHPLC, the detector cells must have the smallest possible cell volume in combination with the longest possible optical path length (typically 1 cm for standard HPLC cells). The flow line and

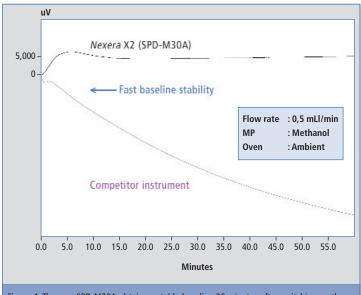


Figure 4: The new SPD-M30A obtains a stable baseline 20 minutes after switching on the instrument, whereas a competitor instrument still exhibits a noticeable baseline drift even after one hour

detector

the shape of the cell must support the fastest possible fluid exchange in order to minimize dispersion. The combination of optical unit and flow cell should be designed in such a way that refractive index effects become negligible.

The benefits of performance

The optical unit was optimized for the use of capillary flow cells. This involves the use of a 1,024element CMOS photodiode-array. This combination allows high spectral resolution of 1 nm (Figure 3). In addition, the entire optical unit is thermostatted. In this way, the instrument is ready for operation faster and is significantly less sensitive to ambient temperature fluctuations (see figures 4 and 5).

To reduce noise even further, a special high-performance Deuterium lamp is used.

The key role of the capillary flow cell

The new capillary flow cell (Figure 6) has an optical path length of 1 cm, which is standard for most analytical cells. Exceptional however, is the cell volume of only 1 µL. Stray light and refractive index effects are reduced to a

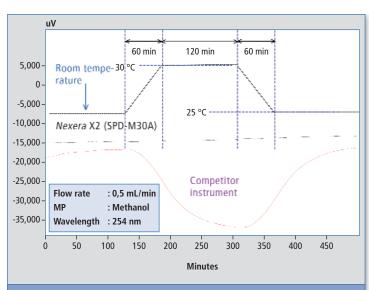


Figure 5: An increase in ambient temperature of + 5 °C (- 5 °C) does not influence the baseline of the SPD-M30A. The baseline of a competitor instrument is clearly influenced by ambient temperature fluctuations.

minimum due to the extremely well reflecting capillary and the use of advanced fiber optics technology.

A cell with this geometry is the optimal solution for use in the semi-micro range in UHPLC as well as in conventional HPLC. Figure 7 shows a chromatogram overlay of the predecessor model, SPD-M20A with the 2.5 µL UFLC cell and the SPD-M30A with the standard cell. There is a clear difference between the signal intensities due to the different optical path lengths. It is obvious as well, that higher plate numbers and improved resolution can be achieved through the low dispersion and the smaller cell volume in the SPD-M30A.

For highly sensitive measurements, the optional HS cell with a path length of 85 mm and a volume of only 8.5 μ L is available. Figure 8 shows a comparison between the standard cell of the new SPD-M30A and the optional HS cell.

To meet the requirements in a regulated environment (for instance GLP and GMP), the cells of the SPD-M30A feature an ID function which ensures traceability to the cell employed based on the data files.

Future-proof design

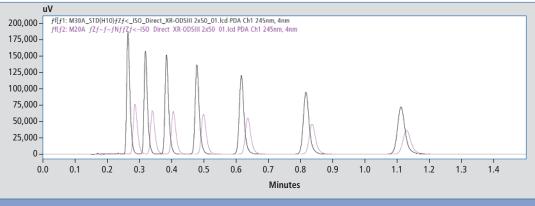
It is known that high data acquisition rates are required for ultrafast separations. In theory, 20 data

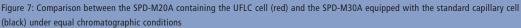


Figure 6: Standard capillary cell of the SPD-M30A

points are needed per peak for proper peak recording. Accordingly, the data acquisition rate should be higher than 20 Hz for peak widths of one second.

In accordance with requirements, the new SPD-M30A allows selection of various data acquisition rates of up to 200 Hz, which yields a distinct peak profile with peak widths of 0.1 sec.





PRODUCTS

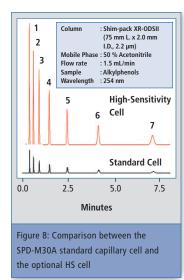


Figure 9 shows a peak which has been recorded with a base width of approx. two seconds at different data acquisition rates. It can clearly be seen that a data acquisition rate even of 100 Hz, in comparison with 40 Hz, allows a more accurate peak representation.

It is also evident that at a peak width at baseline of approx. two seconds, the 200 Hz data acquisition rate does not offer any significant improvement.

Software extends field of use

Additional features in the new LabSolution software extend the

field of use in photodiode-array detection:

- With the 'intelligent Peak Deconvolution Analysis' function (i-PDeA), it is possible to easily quantify overlapping peaks or peaks containing impurities and different spectra, using the first derivative of the spectrum
- •The 'intelligent Dynamic Range extension Calculator' (i-DReC) function enables quantification of highly concentrated samples whose signals exceed the linear detection range by many times. For quantification without extrapolation, the standard calibration curve created within the linear range of the detector is used. In practice, the linear range of the detector is extended over several orders of magnitude without additional hardware.

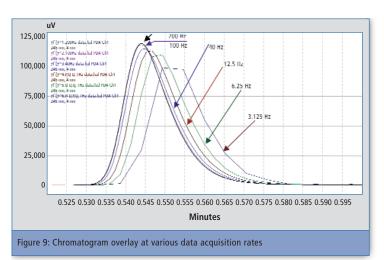
Both functions are highly userfriendly and, in addition to allowing manual parameter setting, automatic parameter selection is also possible.

Conclusion

The new SPD-M30A records highresolution data with the highest quality. In terms of sensitivity and speed the new i-PDeA and i-DReC software features significantly extend the field of use.

Column	Shim-pack XR-ODSII (75 mm L x 2.0 mm I.D., 2.2 m)
Mobile Phase	50 % acetonitrile
Flow rate	1.0 mL/min
Sample	Alkyl phenones
Wavelength	245 nm

Table 1: Analytical conditions of figure 9



Long winter perio wastewaters Efficient



The long frosty period during last winter had an impact on airports. In extreme cases, heavy snowfall resulted in grounding of all air traffic when plowing services were not able to clear the runways. The frigid temperatures, however, left further 'traces.'

Ice and snow on the wings of airplanes increase their total weight and have a negative impact on their aerodynamics. For reasons of safety, airplanes need to be de-iced just before they are ready for takeoff. The de-icing agent that is most frequently used is a mixture of water, glycol and additives. The exact composition depends, among other things, on the outside temperature.

TOC monitors the organic load of wastewater

After spraying the airplanes, the de-icing agent enters the sewage system where it leads to a significant increase of the organic load. Even when the agent used is biodegradable, the effluents must be subjected to a controlled treatment



Figure 1: TOC-4200

d pollutes airport

monitoring by online-TOC prevents environmental damage



process. For the airport operator, it is therefore important to know the organic load of the airport's wastewater. The TOC sum parameter has been well established as an assessment parameter for wastewater analysis.

TOC (Total Organic Carbon) is a measure of the concentration of organically bound carbon and therefore reflects the pollution level of organic substances in wastewater. Depending on the use of the de-icing agent, the TOC values can fluctuate significantly.

Continuous online NPOC monitoring

Of course, one rarely finds an airport that includes a laboratory staffed with laboratory technicians, so an analysis system is needed that can monitor the organic carbon pollution level continuously while operating under maintenance-free and relatively autonomous conditions.

Important for TOC determination is the differentiation between organic and inorganic carbon. Carbonates and hydrogen carbonates are, after all, present in all natural waters. The most widely used method for TOC determination is the so-called NPOC method. In this method, the sample is acidified to convert the carbonates and hydrogen carbonates present in the sample to CO_2 . The resulting carbon dioxide is subsequently purged using a gas stream passed through the sample.

Tried and tested and powerful – the TOC-4200

The TOC-4200 process analyzer is predestined for this application. The TOC-4200 is a powerful analyzer that uses catalytic combustion at 680 °C. After the analyzer has automatically removed the inorganic carbon, a sub-quantity is injected onto a hot (680 °C) platinum catalyst. Here, all organic components present are oxidized to carbon dioxide. The resulting CO_2 is transported by a carrier gas stream to a highly sensitive and CO_2 selective NDIR detector, where it is measured. The TOC concentration is calculated using an external calibration.

The integrated dilution function enables TOC analyses up to 20,000 mg/L as well as automatic sample dilution when the measuring range is exceeded.

The measured value is transmitted directly to a control room that initiates suitable measures when a threshold value is exceeded. The analyzer can also take direct action. For example, a slider can be closed automatically to prevent the discharge of contaminated water into watercourses or stagnant water bodies.

Multiple sample streams in one instrument

An airport may have several separate wastewater collection systems. In this case, multiple sample streams must be measured using one instrument, requiring the use of a multi-stream sampler (see figure 2). The sample is passed through a strainer and reaches the sample chamber where it is homogenized using a rotating knife before being transferred to the instrument for further analysis. In this way, even samples containing large amounts of particulate matter can be measured without any problems. After sampling, the chamber and strainer are cleaned with rinsing water. The rinsing water prevents carry-over effects when switching sample streams. The measuring parameter can be selected individually for each sample stream. In addition, the user can freely select the sequence by which the sample streams are to be measured.

The automatic dilution function, the self-calibration check and the

optimized sampling process allow virtually independent operation of the instrument at the airport. Numerous alarm and status signals simplify detection of exceeded threshold values or indicate maintenance requirements. In addition to the conventional possibilities, Modbus communication is available. An optional web browser enables access to the instrument from any networked computer. This makes the TOC-4200 the ideal instrument for continuous TOC determination of wastewater streams at airports, especially during the winter period.

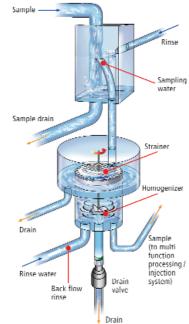
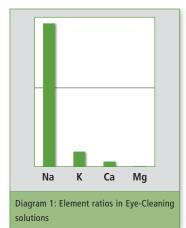


Figure 2: Multi-stream sampler

he eyes are one of the most important sensory organs. But they are very sensitive to accidents, necessitating precautions in case of emergency. An example of an eye accident could be ingress of particles or chemicals (particularly in laboratories). In this case, the eyes need to be rinsed with a special Eye-Cleaning solution.

The quality assurance of such a solution is very important to ensure that the rinsing helps rather than causing even more problems, e.g. through introduction of microorganisms which can cause serious infection of the eyes. To prevent the solution from containing microorganisms, the filling has to be aseptic. It is an efficient way to fill a plastic bottle directly after



extruding and moulding it into the common bottle shape. Filling the bottle at this time with the temperature-stable solution results in fast cooling of the bottle material. The productivity of this manufacturing process known as blow fill seal (bfs) is much higher than when working with air as cooling medium or flushing the bottle with rinse solution, quite apart from the possibility of higher contamination risks. These are some of the reasons that Holopack® (near Stuttgart, Germany) offers this technique (The ready bottled sample is shown in figure 1).

A different aspect in quality assurance is the screening of the contents. Normally, the amounts of salts included in an Eye-Cleaning solution are well known by the manufacturers. But in order to exclude weighing errors or to con-

How to keep a clear view

Quality assurance applying spectroscopy for Eye-Cleaning solutions



trol the stability and real concentration of the elements in the final solution, an additional control step is indispensable for increased safety. Therefore, it is necessary to determine the element concentrations of sodium (Na), potassium (K), magnesium (Mg) and calcium (Ca) in a fast and reliable way.

AAS or ICP-OES?

For the analysis of Na, K, Mg and Ca it is desirable to use both techniques, Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Atomic Absorption Spectroscopy (AAS). With the ICPE-9000 system and the AA-7000 dual atomizer instrument, Shimadzu meets the demands of quality assurance aspects.

The AA-7000 is the system of choice for economic analysis of up to 4-5 elements. When six or more elements should be determined quickly, the ICPE-9000 system is more applicable (compare figure 2). Additional criterions for decisions can be present, e.g. the different detection limits of the analytical systems. But in case of the Eye-Cleaning solution this is not a point because all four analytes are high concentrated to imitate the human tear fluid to avoid eye-irritations.

Analytical Task

The analytical challenge of this sample is the highly varying levels of the elements Na, K, Mg and Ca. For example, sodium is present in a high ppm range whereas the concentration of magnesium is 100 times lower (Diagram 1). Based on the fact that sample preparation nowadays has to be highly automated with the lowest amount of work, one dilution only of the sample should be sufficient to determine all elements, instead of several dilutions to match each analytical working range.

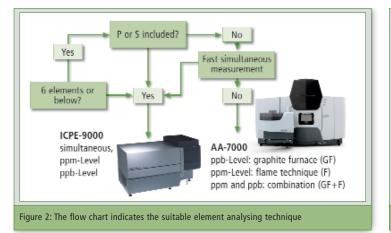


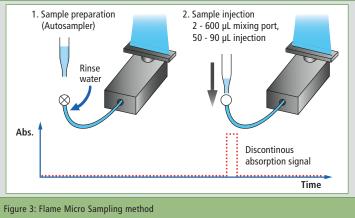
Figure 1: Analyzed product: Sterile Eye-Cleaning solution bottled by bfs-technique A 100-times dilution was selected for working with the Eye-Cleaning solution. In this way, it is possible to determine magnesium (now 0.36 ppm), calcium and potassium with flame atomic absorption technique using the AA-7000F. For determination of sodium, the observed absorption wavelength has to be changed to the less-sensitive 330.3 nm-line, which is of factor 200 less sensitive in comparison to the most common 589.0 nm-line.

Micro Sampling

To have more scope in method development and individualization in the final steps of the method optimization process, Shimadzu's Micro Sampling is perfectly suited to measure the Eye-Cleaning solution. For example, when applying ionisation buffers as prescribed for determination of sodium in drinking water (in accordance with German Drinking Water Directive).

The AA-7000F in combination with the ASC-7000 sample preparation station allows the automated flame Micro Sampling method (Figure 3). In this method, flame





atomic absorption analysis is conducted with small sample volumes (50 - 90 μ L), whereas in the conventional flame method (hereafter "flame continuous method"), the sample is continuously aspirated with a flowrate of approximately 8 mL/min, and larger sample volumes are needed for aspiration.

In the first step (Figure 3) the sample can be diluted and water, ionization buffers and/or standard solutions (standard addition method) can be added. The total mixing volume is 600 µL. The resulting solution is injected in step two.

The flame Micro Sampling method has several advantages compared to the flame continuous method. Analysis is possible with a small amount of sample, and when the autosampler is used, automatic further dilution of the sample and automatic addition of buffer solutions are possible in order to compensate for interferences. Moreover, since only a small amount of sample is introduced, the flame Micro Sampling method is effective for analysis of high matrix samples which may cause clogging of the burner in the flame continuous method, such as the Eye-Cleaning solution. when measuring high matrix samples with common continuous flame method.

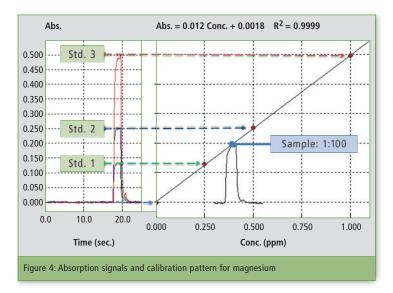
To illustrate measurement results, the calibration plus the absorption signal of magnesium are shown in Figure 4. The recovery was in a

Instrument	AA 7000 with autosampler ASC 7000 incl. Micro Sampling Kit			
Measurement element	Na	К	Ca	Mg
Wavelength	330.3 nm	766.5 nm	422.7 nm	285.2 nm
Slit width	0.2 nm	0.7 nm	0.7 nm	0.7 nm
Flame type	Air-C ₂ H ₂	Air-C ₂ H ₂	Air-C ₂ H ₂	Air-C ₂ H ₂
Gas flow rate	1.8 L/min	2.0 L/min	2.0 L/min	1.8 L/min
Sampling time	20 sec	20 sec	20 sec	20 sec
Interference buffer	CsCl-solution		La ₂ O ₃ -solution	
Working range [mg/L]	10.0 - 40.0	1.25 - 5.00	0.50 - 2.00	0.25 - 1.00

Table 1: Final method parameters for quality assurance of highly concentrated elements in Eye-Cleaning solutions

Final method and results

Method parameters providing the best measured results are summarized in table 1. Using these parameters, it was possible to measure over hours with no clogging of the burner head, as can happen



Analyte	Expected (weighted)	Result (measured)	Recovery
Na	158 mmol/L	157 mmol/L	99.4 %
К	10.0 mmol/L	9.81 mmol/L	98.1 %
Ca	3.24 mmol/L	3.28 mmol/L	101.2 %
Mg	1.48 mmol/L	1.48 mmol/L	100.0 %

Table 2: Measurement results for the Eye-Cleaning solution

good range for all elements included in this research (Table 2). The AA-7000 in combination with Micro Sampling is a "state of the art" atomic absorption spectrophotometer for high precision measurements of element concentrations in Eye-Cleaning solutions as well as in mineral waters (see Shimadzu News 01/2011). In general, this method is an interesting tool for clinical or pharmaceutical applications as it complies with FDA CFR 21 Part 11, and the possibility to optimize all parameters including individual sample dilution even in a multi-elemental measurement sequence emphasises the Micro Sampling technique.

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Moldy off-odors in fruits and vegetables?

Determination of trichloroanisole and tribromoanisole using SPME coupled to Comprehensive GCxGC and mass selective detection (HS-SPME-GCxGC-qMS-NCI)

Erich Leitner

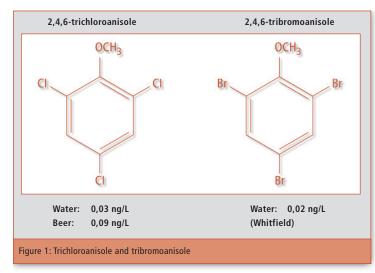
Institute for Analytical Chemistry and Food Chemistry, Technical University of Graz, Austria

he compound 2,4,6,-trichloroanisole (TCA) is often mentioned in the literature, particularly since H.R. Buser's publication identified TCA as a key component of the "cork taste" in wine.¹ A number of other reports have been published on the sensoric effects on food by TCA and also 2,4,6-tribromoanisole (TBA). In 1990, J.C. Spadone found that 20 % of raw coffee from Brazil was contaminated with TCA.² Most recently, TBA was in the news regarding product recalls from the pharmaceutical companies Pfizer and Johnson & Johnson who had to withdraw products from the market as a result of contaminated packaging. A robust and sensitive method to determine TBA and TCA in a range of fruits and vegetables exhibiting unwanted moldy offodours has been developed.

Sensoric Properties and Sources of TBA and TCA

TCA and TBA have an unpleasant moldy off-odour which is registered by humans – even at very low concentrations. These compounds can be perceived at different levels by humans in different food matrices.³ Figure 1 shows the chemical structures of the two compounds.

It should be noted that none of these substances were added to the food products intentionally, but both substances are formed



from halophenols which are used as bactericides (PCP) during the anti-poisoning process of microorganisms. The formation mechanism could be identified in the production of wine stoppers made of natural cork. A number of microorganisms could be detected both in – and on – the natural cork as a result of the production process. Formation occurs either by direct methylation of trichlorophenol and/or tribromophenol or a methylation reaction after a dechlorination of halide phenols with more than three chlorine or bromine atoms. Of the large number of halide anisoles, TCA and TBA have by far the lowest odour reception level.

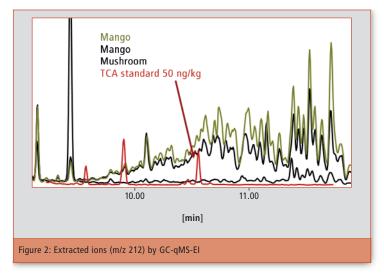
Analytical Methods

Due to the high volatility of the substances, high-resolution gas chromatography (GC) is the method of choice for analysis. An electron capture detector (ECD) or a mass spectrometer is recommended for use in selective and sensitive detection of halide compounds. Initial measurements on different fruits and vegetables with a low resolution quadrupole MS in selected ion monitoring (SIM) mode showed, however, that such a method is not useful in the analysis of real world samples because of massive spectral interferences (see figure 2).

To solve this problem, a combination of multidimensional chromatography (comprehensive GCxGC) with negative chemical ionization (NCI) was used. In comprehensive GCxGC two columns of different polarities are coupled in series. The first column in this set-up usually has conventional dimensions, while the second column is substantially shorter (0.5 - 3 m).

All compounds eluting from the first column are cryo-focused at the 2^{nd} column head by a jet of cold nitrogen gas and subsequently heated up in a constant interval time (modulation frequency, usually 2 - 8 s) by a jet of hot nitrogen gas to be injected into the second column. In this way, peaks from the first dimension are fractionated and additional separation on the second column takes place.

For a better overview of separation, the resulting data are usually shown in so-called contour plots rather than in chromatograms. In a contour plot the retention time on the first column is plotted against the retention time on the



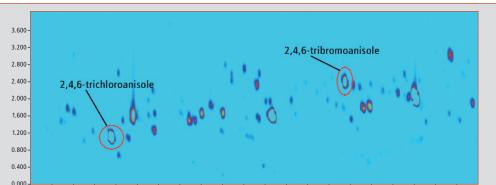
second column, with the maximum length of the y-axis being the modulation frequency. Compounds that are shown parallel to the y-axis are not separated in a one-dimensional chromatographic system. In addition, the modulation of the peaks leads to a better sensitivity as the peak width of normally five to six seconds is reduced in a comprehensive system to 200 - 400 ms.

Using Negative Chemical Ionization

The signal-to-noise ratio (S/N) for 10 ng/kg TCA was increased from 18:1 (electron ionization (EI) in SIM mode) to 72:1 (NCI in SIM mode). If NCI is used in combination with comprehensive GCxGC, a further increase of the S/N to 300:1 is observed, resulting in a quantitation limit of 0.1 ng/kg. The only drawback of this method is the formation of fragments corresponding to the halide atoms (m/z = 35 and 37 for)chloroanisoles and m/z = 79 and 81 for bromoanisoles), making the use of deuterated standards impossible. The figure below shows the contour plot of a standard sample with a concentration of 10 ng/kg of each of different haloanisols. Figure 3 shows the contour plots of halide anisols in a real world sample of mango. Sample preparation was performed by automated headspace solid-phase microextraction (HS-SPME) using a 2 cm 50/30 µm divinylbenzene/carboxene/ polydimethylsiloxane fibre at 80 °C for 60 min. Sample amount was 1 g of homogenized material. Calibration range was 1 - 100 ng/kg.

Conclusion

Comprehensive two-dimensional gas chromatography coupled to automated SPME using NCI was shown to be a selective and sensitive method for the determination of the odour active compounds 2,4,6-trichloroanisole and 2,4,6tribromoanisole. The method is also robust and simple. Determination of the target compounds without any interference from the matrix is possible with this combined method. First results from real world samples (mango, garlic and different types of mushrooms) obtained from a grocery



16.000 16.667 17.333 18.000 18.667 19.333 20.000 20.667 21.333 22.000 22.667 23.333 24.000 24.667 25.333 26.000 26.667 27.333 28.000 28.667 29.333

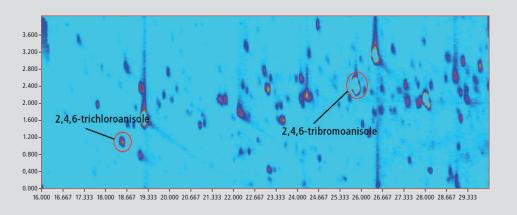


Figure 3: (a) Mango 1 / (b) Mango 2

store showed alarmingly high contamination levels. A thorough investigation of different fruits and vegetables is planned.

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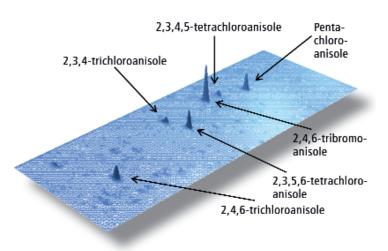
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Erich Leitner

studied technical chemistry at the Technical University of Graz, Austria, focusing specifically on analytical chemistry. He graduated in 1992 and received a PhD in 2005 in food chemistry.

He has over 25 years of experience in organic trace analysis using gas chromatographybased methods. He focuses specifically on the identification of odour active substances using analytical and human sensory methods. 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 411





Standard 10/ng/kg each, haloanisoles

The UV-VIS market has been divided into sections created by the limitations of the component quality of diffraction gratings, detectors and source lamps. The 'normal' banding consists of instruments which are able to read in the order of 2-4 absorbance (abs). 'Research' machines extend

High absorbance readings in UV analysis of Tertiary-Butyl-Catechol (TBC) without colorimetric derivatization





Figure 1: The Shimadzu UV-2700 UV-Vis-NIR spectrophotometer

V-VIS spectroscopy has been a fundamental tool for most laboratories since it was first used routinely nearly 70 years ago. The technique is widely applied in research, quality control and assurance laboratories in the chemical and pharmaceutical industries.

APPLICATION

this range to 4.5 - 5 abs through the use of pre-monochromators to further reduce stray light. Beyond this is a further niche of 'Reference' grade instruments using double monochromators and advanced wavelength drives to reach as high as 7 - 8 abs.

Smaller, lighter and more powerful

With the launch of the new Lo-Ray-Ligh™ grating, Shimadzu has completely redefined the conventional spectrophotometer niches. This new grating offers dramatic reduction of stray light enabling a single monochromator (as in the UV-2600) to deliver > 5 abs performance, and a double monochromator system (UV-2700) to reach > 8 abs – in an instrument which is physically smaller and lighter than most conventional double-beam systems. The grating also offers a wider wavelength range than any earlier generation grating, permitting the UV-2600 to read up to 1,400 nm with the ISR-2600 Plus integrating sphere option which contains an addi-

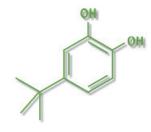


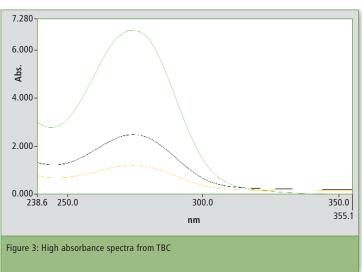
Figure 2: Chemical structure of TBC (Tertiary-Butyl-Catechol)

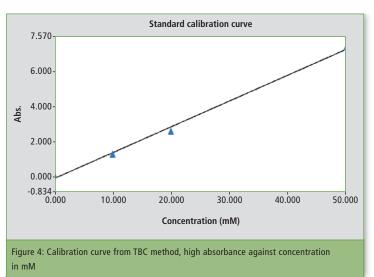
tional InGaAs detector (Indium Gallium Arsenide).

This new development gives spectroscopists a new choice. It is no longer necessary to invest in a very high specification double grating, double monochromator system to cover this absorbance range. The UV-2700 is much more compact, just a third of the size of conventional reference instruments and with a fraction of the costs.

Application example

The following example shows how to reduce costs not only from the instrument but also from the application side. A chemical company has been using a method for determination of TBC, an inhibitor of polymerization reactions applied to increase the handling time for bonding agents, which involves a reaction with NaOH to produce a red color. The color has been used for quantitation as it exhibits lower absorbance reducing the need for dilution. Direct measurement of TBC





at 275 nm has not been possible using conventional 0 - 4 abs UV-VIS instruments as the highest concentrations of TBC at 50 mM have an absorbance of nearly seven Abs.

Using the UV-2700, it is possible to directly measure the absorbance peak of TBC at 275 nm. The following figure shows the absorbance traces of 10, 20 and 50 mM TBC (axis scale 0 - 8 abs).

The figure shows that it is possible to scan a sample which has a maximum absorbance of 6.87 at 275 nm. The ability to quantitate this product directly is demonstrated by a plot of the concentration vs. absorbance from the standard quantitation software in Shimadzu UVProbe.

The relationship of absorbance to concentration remains linear through to very high absorbance. In order to read at very high absorbances, the Shimadzu UV-2700 offers a 'High Absorbance' scan mode which can be used in conjunction with rear-beam attenuation to allow measurement of light intensities less than one ten millionth of normal open beam energy.

Summary

The Shimadzu UV-2700 is an affordable and compact instrument which dramatically increases the concentration range of highly absorbing species which can be directly observed in UV spectroscopy. It offers a performance previously available only in reference grade, double monochromator instruments which were too large and too expensive for most laboratories.

Instrumentation UV-2700, UVProbe software, Attenuator, standard 1 cm square cell holder, 1 cm square quartz cell

New tool for healthcare and cosmetic products

SPFCalculator software for Shimadzu UV-VIS instrumentation



new UV-VIS instrumentation software for the healthcare and cosmetic segments outperforms the new regulations to be applied from July 2013. These adjustments will be fixed by the European Community, relating to the production and marketing of cosmetics [1]. Other regulations are the Colipa [2], FDA [3], Boots Star Rating [4] and JCIA (Joint Commission International Accreditation - Health Organisation). All of them are part of the SPFCalculator Software which even helps to calculate some physical properties. This product has been designed in cooperation with the Aqualis software house in Milano, Italy.

The SPFCalculator presents 17 parameters requested for the qualification of a sun protection product. The program includes more information on the sun screen product than is required by the guidelines. The calculator is easy to handle and just needs a simple training for users. The software can be updated based on changes in the regulations. The seventeen parameters which can be calculated are shown in table 1.

Users can easily test the sun protection product and change its formulation where necessary, before carrying out the very expensive in vivo measurements.

Shimadzu can provide a complete easy to handle solution: a package containing the UV-2600 instrument, the ISR-2600 integrating sphere in combination with UVProbe and the SPFCalculator. It is a total solution in accordance with European Cosmetics Associations requirements. The strength of the package is that other parameters such as color, appearance, quality of packaging etc. can be determined using the same combination.

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According to the Boots Star Ratio System (2008 Revision), Boots, Nottingham, UK, 2008

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UVAPF0	UVAPF	Ratio (UVAPF/SPFlabel)	UVA dose
Exposure energy	Conversion factor	Exposure time	Critical lambda (pre-irradiation)
Critical-lambda (after-irradiation)	UVA-I/UV FDA	Critical lambda FDA	Star rating FDA
UVA/UVB pre- irradiation Boots	UVA/UVB after- irradiation Boots	Critical lambda boots	Boots star rating
Japan PA rating			

Table 1: Parameter selection for the SPF report

Shock waves

World premiere – The new high speed video camera Hyper Vision HPV-X



Figure 1: HPV-X system configuration consisting of camera head and power supply unit

U ltra-high speed recordings with video cameras such as the Shimadzu Hyper Vision HPV-1 or HPV-2 become a standard tool for characterizing and quantifying high speed events in order to make invisible phenomena visible to the human eye.

Shimadzu's HPV-1 and HPV-2 enable recordings at up to one million frames per second (fps) while maintaining excellent spatial resolution at any recording speed.

The need to capture high-speed phenomena is demanded by academic institutions and research institutes and also various manufacturing industries. Applications include fuel injection in combustion engines, dispersion of ink drops by inkjet printers, studies on hydrodynamics, destruction processes, shock wave development, visualization of machining processes as well as fast chemical reactions.

Unique features

The latest development in this application field is the new Hyper Vision HPV-X High-Speed Video Camera equipped with a proprietary high-speed CMOS image sensor, enabling ultra-high-speed continuous recordings at 10 million frames per second while ensuring no blooming effects. This is a unique feature and a key differentiator in comparison to other high speed camera systems.



external memory. Due to limitations of the output amplifier speed, the effective recording speed will be in the tens of thousands of frames per second, but the pixels are sub-sampled to further increase the speed. This results in an extremely poor resolution in the high speed region. With the high speed CMOS sensor, the photo diode and memory are connected 1 to 1, and with the signal from each frame, all pixels are written to memory simultaneously. Since no sequential readout occurs there is no output amplifier limitation, so high-resolution recording even at ultra-high speeds is enabled.

High resolution even at high speeds

The new HPV-X is able to record 128 frames, 20 % more than the HPV-2. In the HP-mode (50,000 pixels), a double-memory function enables recordings of 256 consecutive frames for recording even longer periods. This offers the users the choice of prioritizing either resolution or recording time.

Other high speed camera systems need to lower the resolution as recording speed increases, but the HPV-X maintains the same high resolution even if recording speed is increased. That means that ultra-high-speed phenomena can be analyzed in far more detail than when using a regular high speed video camera. This proprietary feature is included in all Shimadzu Hyper Vision high speed video cameras, but is not available from any other manufacturer. The HPV-X is able to record five million 100,000 pixel images per second or ten million 50,000 pixel images per second.

Easy handling

In contrast to any other high speed camera systems, the HPV-X is very compact and easy to use. Just like the previous models of the Hyper Vision series, the simple system configuration, consisting of the HPV-X camera head connected by cable to a laptop computer, offers a compact and highly portable design that makes on-site setup especially easy. It also retains the same HPV software that has been so popular with current users all over the world, featuring intuitive and easy-to-understand setting screens making it easy to capture ultrahigh-speed videos.

From soft to hard

The new HMV-G microhardness tester

hen thinking of microhardness testing, the determination of structural constituents of hard metallic or ceramic samples usually comes to mind. Studies on crystal segregation, hardness distribution of nitrited layers and welded joints as well as systematic hardness testing of new materials form the majority of areas of application. However, the number of measurements requiring

urement. In this way, it is possible to examine very hard materials as well as thin layers such as metal foils used in the manufacture of solar cells.

Twelve load ranges

Up to twelve load ranges from HV0.001 to HV2 are supported. Thanks to the force-compensated design, up to four additional load ranges can be freely de-

fined, which can be very useful in the measurement of borderline cases. In addition, a motor-driven turret enables easy switching to another objective lens without the need to repeat testing if a too high or too low load range was selected.

> Overall, emphasis has been placed on flexible and convenient



Figure 3: Big alphanumeric LCD touch panel

operation during the development of the new HMG-V. The G-shaped frame increases the sample compartment considerably, enabling straightforward measurement of large-volume samples or components. The measurement turret is available in two versions, with the possibility to mount up to two indenters as well as four objective lenses simultaneously.

Three series for various applications

Three series are available depending on the application need:

- the manual version, suitable for training purposes as well as for high-precision measurement for small measurement tasks in research
- the camera and PC-controlled version for more complex and intensive measurement activities measuring indentation automatically
- the fully automated version for large measurement series and regular hardness distribution, with autofocus feature and an automated stage.

The fully automated HMV-G-FA hardness tester with its multitude of functions offers maximum convenience and performance with minimal work complexity.

Dedicated support functions

Using the integrated 'stage viewer' function, the entire stage is available for sample observation. An 'edge detector' recognizes sample geometries and generates simplified representations on the monitor. With the optimal imaging, even real samples with scratches or light or dark backgrounds can be determined automatically without the need for complex sample preparation such as polishing. The measurement series are then carried out by simply clicking the start and end points or by selecting individual points.

These support functions together with the excellent measuring and precision properties of the new HMV-G series also enable less experienced users to obtain fast measurement results. True to the motto 'Everybody can become an expert'.

Figure 1: Micro Vickers hardness tester HMV-G

particularly small test forces increases rapidly with fast advances in lightweight construction.

The completely redesigned HMVseries hardness tester uniting all required features in one instrument takes this development into account.

The HMV-G models feature a vast test force range from the conventional microhardness range of 0.098 N up to 1.96 N down to the small load hardness range to 19.61 N. In addition, the HMV-G can push forward into the ultra-microhardness range and can apply forces of merely 9.81 milli-Newton to 49.04 milli-Newton for meas-



Figure 2: Multi turret function with up to two indenters and four abjective lenses

Laboratory opening ceremony with hammer stroke

"Excellence in Science" on 1,500 m²: World and European premieres



Traditional Kagamiwari ceremony with the Managing Director of the Analytical Division, Teruhisa Ueda, Shimadzu's CEO Akira Nakamoto, Consul General Kiyoshi Koinuma as well as the European Managing Directors Yasuo Miura and Jürgen Kwass (left to right).

t could not have been planned better: 150 guests from all over Europe were greeted by bright sunshine and luminous cherry blossoms at the opening of Shimadzu's new Laboratory World. According to Japanese culture, flowering cherries symbolize an awakening – the theme for this day that included an opening ceremony with representatives from business and politics, a Japanese Kagamiwari ceremony, a guided tour through the laboratories as well as world and European premieres of new products.

After short speeches from Akira Nakamoto, CEO of Shimadzu Corporation, the Japanese Consul General Kiyoshi Koinuma, Duisburg's Mayor Sören Link as well as Yasuo Miura, President of Shimadzu Europa, they cut the ribbon together and officially opened the new Laboratory World. Subsequent guided tours through the laboratory and seminar facilities were accompanied by live demonstrations of selected applications and technologies at seven different stations. Four product premieres also received center stage.

Kagamiwari ceremony with hammer stroke and rice wine

The inauguration of the Laboratory World was concluded with a Japanese Kagamiwari ceremony. The lid of a keg of Sake was smashed with wooden hammers, resulting in a delightful spray of the rice wine. A mutual 'Kanpai' (toast) in wooden sake bowls with all guests completed the ceremony. Newspaper and television journalists were present and have reported on this event.

In the following days, from 15 -19 April, customers of Shimadzu's European branches were introduced to the 'Laboratory World' featuring individual product segments.

On over 1,500 m², testing facilities were created for Shimadzu's entire product range – from chromatographs, spectrophotometers, TOC analyzers, mass spectrometers and balances up to material testing machines. Mass spectrometry is presented in its own area. Shimadzu has significantly advanced this technology in recent years. In addition, laboratory areas for customer applications and seminar facilities have been expanded. Shimadzu has invested a total of 3,2 million Euros in the expansion of its laboratory and training facilities.

"During the entire week, we have welcomed more than 200 customers", explained Jürgen Kwass, Managing Director of Shimadzu Europa. "The facilities at our Laboratory World are designed in such a way that there are dedicated areas for discussion and exchange of ideas in addition to the spacious product and laboratory stations. Shimadzu anticipates that due to the extended consumer and product safety regulations, the testing and analysis needs - and consequently the need for training - will continue to increase in the future.

One world and three european premieres

In addition to the new 'Laboratory World', Shimadzu has introduced four new products:

- the high-speed Hyper Vision HPV-X camera. Thanks to the world's fastest sensor with 10 million frames/sec (Mfps), the camera records phenomena during shock waves or in machining processes (see article on page 18).
- the HS-20 headspace sampler for gas chromatography for accurate analysis of volatile and semi-volatile compounds in a wide boiling point range
- the *Nexera* X2 UHPLC system with the world's most sensitive photodiode-array detector for



routine analyses and applications in highly regulated environments (see article on page 8)

the highly sensitive Tracera gas chromatograph, which can detect virtually all traces of organic and inorganic compounds due to novel plasma technology – about 100 times more sensitive than the universal TCD and twice as sensitive as the FID (see article on page 4).