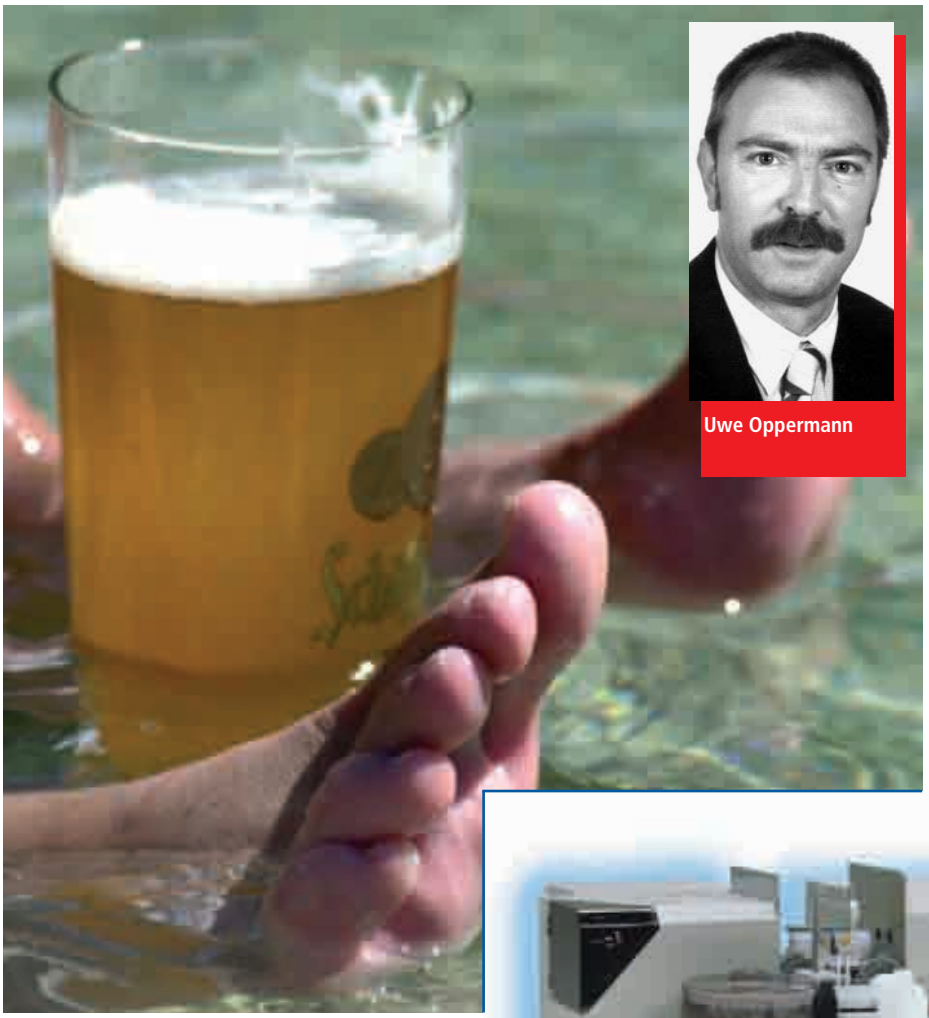
Spectroscopy in Food Analysis



Uwe Oppermann

A wealth of spectroscopic methods exists for residue analysis and quality control. The method and type of instrumentation employed are dictated by the composition and formulation of the samples to be measured. Spectroscopy is the method of choice if quick and simple information about the sample material is required, both qualitatively and quantitatively. The optimum spectroscopic method is determined by the sample characteristics.

The widest application range is covered by the UV-VIS spectrometers, which are ideal for the quantitative and qualitative analysis of samples in absorption, transmission and reflection measurement modes. Sensitive determinations of trace and ultra trace elements in aqueous and organic solutions can be determined using atomic absorption spectrometers.

FTIR Spectrometers, from the near to the far IR region, allow specific identification of substances. They can be used in combination with a range of accessories, including microscope, each accessory being selected according to the sample material and properties. Fluorescence spectrometers that are capable of highly sensitive quantitative analyses complete the range of spectroscopic methods suitable for the analysis of food stuffs.

Trace Elements in Drinking Water

Water for human consumption must meet the criteria laid down in paragraphs 5–7 of the new European drinking water regulations (2003). The concentration levels of the essential and toxic elements are tightly controlled so that a constant water quality can be maintained. Quantitative measurements of elements at the trace and ultra-trace concentration levels are carried out using atomic absorption spectrometers, like the Shimadzu AA-6800 for example. For electrothermal atomisation, the graphite furnace GFA-EX7 with digital control and an automatic sample preparation station is also available (Fig. 1).

The concept of AA-6800 allows the full-automatic changeover from flame to furnace mode and element specific optimisation of the atomizer position. There are two methods of background correction for the determination of trace elements in samples with complex matrices when using flame and graphite furnace:

- Deuterium correction for compensation of spectral interferences through molecular absorption and particulate scattering
- High speed self reversal technique (high current pulse technique) used to compensate for interferences caused by overlapping absorption lines and structured background [1].

Fig. 1: Fully automatic atomic absorption spectrometer AA-6800

Hops in Beer

With an average consumption per head of 131 L per year, the Germans belong to the greatest beer drinkers in Europe. In addition to the control of water for brewing using AAS, the determination of hop content in the beer is the most important quality assurance measurement. The taste is determined in the first instance through the hop content: the more hops, the bitterer and stronger the taste of the beer.

The analytical wavelength used for hops determination is 275 nm. Following dilution with iso-octane, the hops concentration can be determined directly. Using the post-processing capabilities of the UV Probe software, it is possible to

Fig. 2: UV-VIS Spectrophotometer UV-2401PC

Fig. 3: FTIR Spectrometer IRPrestige-21.

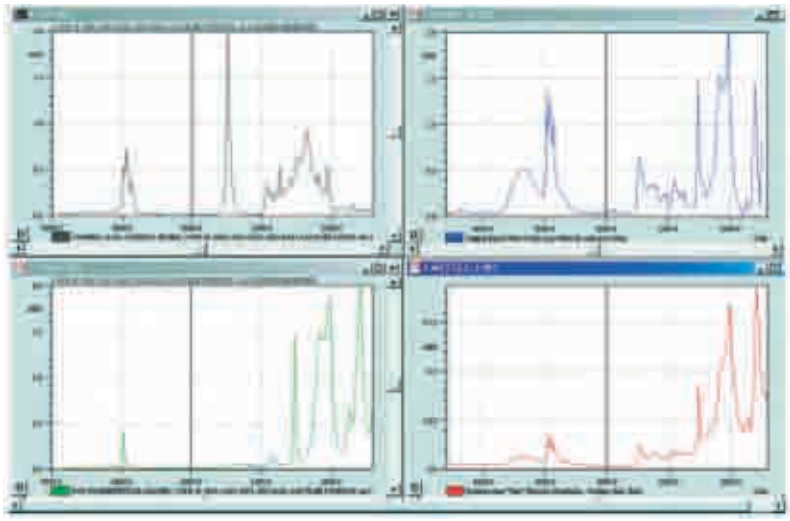


Fig. 4: IR Spectra, top left; Suberine acid diethylester, bottom left; polydimethyl siloxane. Top, right wine cork and underneath, bottom, right; a champagne cork.

differentiate between the following α-bitter acids (Humulon, Cohumulon, Adhumulon) and the β-bitter acids (Lupulon, Colupulon, Adlupulon). The samples require dilution and measurement at the wavelengths 275 nm, 325 nm and 350 nm. The relationship between the α- und β-bitter acids can be directly calculated via the UV Probe software.

Qualitative Analysis of Fruit Juice Drinks

Aqueous products such as fruit juice drinks, lemonade and isotonic fitness drinks are difficult media to measure using FTIR spectroscopy for two reasons. Firstly, the entire Mid-IR region is dominated by the absorption of the water content. Secondly, the materials commonly used to prepare the optical windows are usually hydroscopic or occasionally soluble in water. The use of the ATR technique (attenuated total reflectance) enables the analysis of aqueous solutions. When ATR is used in conjunction with modern FTIR spectrometers, such as the FTIR-8400S or the IRPrestige-21 (Fig. 3), measurements in the mid-IR region are possible.

The ATR accessory used often contains a zinc selenide crystal with high refractive index, which is completely covered by the sample. ATR operates on the principle of total internal reflection of the light beam results due to the differences in refractive index between the crystal and sample. Information about the sample properties is obtained each time the light beam passes from the optically denser crystal material to the optically less dense sample material.

This guarantees the identification of sugar and sugar substitutes like aspartame. FTIR systems can also be used to discover the levels of particular substances in materials like food packaging. The same methods can be applied to the determination of viscous substances like polydimethyl siloxane and suberine on the surfaces of wine and champagne corks. Utilising a single reflection ATR with the FTIR-8400S for surface measurement results in the IR beam penetrating the sample by approximately 2 µm.

How Much Meat is in the Sausage?

The quality of essential food products like meat must be continuously monitored. To verify the meat content of sausage products, the hydroxyproline content (a parameter for collagen metabolism) is determined. The collagen metabolism is a measure of the protein content of connective tissue. The determination of hydroxyproline enables indirect calculation of the meat content. UV-VIS spectrometry is the method of choice for this kind of measurement and the determination is carried out according to paragraph 174 35 of the LMBG (from the German Foodstuffs and Requirements Law).

Hydroxyproline can be quantitatively determined using the UV-2401 and the UV Probe software, since this system configuration allows the optimisation of application oriented methods. The photometric determination of the red solution (due to the presence of 4-dimethylaminobenzaldehyde) is performed at a wavelength of 560 nm. The process can be easily automated by utilizing a sample introduction system (peristaltic sipper) used in combination with a flow through cell. In addition to the hydroxyproline method, this spectrophotometer can be used for the control of other food stuffs such as the determination of nitrite and nitrate content in sausages or the determination of total phosphorus in butcher reports.

Determination of Platinum Metals in Grain Products

In contrast to the toxic elements like lead, cadmium, and arsenic, which are continuously monitored according to the European drinking water regulations and the law for indirect introduction of waste waters, there is no regulation for maximum contaminant levels of platinum metals. Even in the German foodstuffs and requirements law, where the determination of lead and cadmium is strictly controlled according to § 35 LMBG L00.00–19/3, there are no regulations for the platinum metals.

Catalytic converters, nowadays a standard feature in each car, are used to reduce pollution caused by exhaust fumes from petrol and diesel engines and contribute considerably to the prevention of environmental pollution via automobiles. However, new research by Italian and French scientists shows that although on the one hand catalytic converters do reduce pollution on the other hand, they also actively contribute to emissions, specifically through the liberation of very fine particles of precious metals from the platinum group such as platinum, palladium and rhodium [2]. As platinum metals are accumulated in the food chain, it is important to carry out

appropriate analyses. Atomic absorption spectrometry is a suitable method for the reliable routine determination of platinum and rhodium.

In a fully automatic multi element sequence programmed on AA-6800, the calibration is done from a stock standard solution using the sample preparation station ASC-6100 in combination with the diluter ASK-6100. All results were generated from standard solutions, diluted sample solutions as well as a certified reference material (bran RM8433).

Table 1 shows the instrumental parameters using conventional hollow cathode lamps and the recommended currents for low and high current measuring mode using the high speed self reversal method. The concentrations in corn bran, calculated using a linear calibration were 5,48 µg·kg^{-1} for rhodium and 52,31 µg·kg^{-1} for platinum and are in good correlation (1:10) to the ratio of both metals in the catalytic converter. It is obvious that the platinum metals are accumulated in the environment and in the food chain. The platinum concentration of more than 50 µg·kg^{-1} in the reference material for example was five times higher than the certified cadmium concentration. Further research work on this subject is underway and the availability of reference materials including certified concentrations of platinum metals is essential.

Element	Pt	Pd	Rh
Wavelength [nm]	265.9	247.6	343.5
Slitwidth [nm]	0.5	0.5	0.5
Atomisation	Graphite furnace	Graphite furnace	Graphite furnace
Lampcurrent D_2 BGC*[mA]	14	10	12
Lampcurrent SR BGC*[mA]	10/300	10/300	10/300
Atomisation temperature [°C]	2600	2700	2500

Table 1: Instrumental parameters for determination of platinum, palladium and rhodium

Summary

„State of the Art" spectrometers are the correct tools for solving a wide range of application problems in food analysis. Since the list of contaminants and their maximum contaminant level is expected to be even more stringently controlled in future, there is a real need for system configurations with lowest detection limits and highest precision. Shimadzu offers total hardware and software solutions in a wide product range for the accurate determination of those contaminants in addition to the competence and know-how of a market leader in spectroscopy.

References

[1] U. Oppermann, GIT Laborfachzeitschrift, 46, 430–433 (2002)

[2] C. Barbante et al., Environ. Sci. Technol., 35 (5), 835–839 (2001)

Uwe Oppermann
Shimadzu Deutschland, Duisburg
Albert Hahn Str. 6–10
47269 Duisburg, Germany
uo@shimadzu.de

Lorna Moffatt
Shimadzu UK Branch
Milton Keynes