

Two techniques, one system

Nexera-i MT — HPLC plus UHPLC

Vanillin - natural or synthetic?

Analyzed using IRMS and GCMS

Anniversary: 60 years of GC and IR

Shimadzu as pioneer of the first hour















#### **APPLICATION**

Vanillin – natural or synthetic?
GCMS and IRMS acquire the
isotopic fingerprint of vanillin
Safe, clean and free of harmful
substances? Analytical methods
for plastic packaging of foods

New – Application Handbook Liquid Chromatography

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#### **PRODUCTS**

Rivals in femtogram-level analysis — The new GCMS-TQ8050 rivals HRMS's sensitivity

Two techniques, one system – Nexera-i MT – Method transfer made easy

#### LATEST NEWS

GC pioneers of the first hour 60 years of gas chromatography

Technological driver and market leader Milestones in 60 years of IR history 1

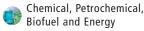
Long-time partners – Shimadzu testing equipment at Vladivostok University's Engineering School

# Rivals in femtogram-level analysis

The new GCMS-TQ8050 rivals HRMS's sensitivity



#### MARKETS

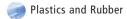












Automotive

since Shimadzu launched the triple quadrupole gas chromatograph mass spectrometers GCMS-TQ8030 and GCMS-TQ8040 in 2012 and 2014, the systems have become wellestablished in environmental and food safety (multicomponent residual pesticide analysis), foren-

sic toxicology and metabolomics (metabolites analysis).

As regulatory bodies are increasingly opening the market of ultra-trace analysis not only to HRMS (high-resolution MS) but also to triple quadrupole systems, it is necessary to develop systems

that cover HRMS's sensitivity with robustness and ease of use.

## Instrument detection limit on femtogram level

In this scope, Shimadzu has collaborated with referenced laboratories in the field of POPs (Persistent Organic Pollutants) analysis to develop adequate solutions.

The new GCMS-TQ8050 is the brand-new system of the GC-MS/MS product range. It achieves an instrument detection limit (IDL) approximately ten times more precise than the previous systems and is capable of femtogram level (parts per quadrillion) analysis.

## New high-efficiency detector

The Shimadzu's GC-MS/MS range of instruments is already well-known for its UFMS (Ultra-Fast Mass Spectrometry) features. Customers improve throughput as well as quality of data.

In order to leverage the UF-transmission off-axis ion optical system to the utmost, a new high efficiency detector with heightened amplification performance has been adopted. A new shield to further reduce noise pushes the limit of detection and improves robustness of the system for daily analysis.

Combining off-axis ion optical, overdrive lens system and the new detector features ensures that noise is minimized during trace analysis, where high amplification levels are required.

I.D.	Compound name	Egg fat		Pig fat		Salmon		Oliveoil	
		GC-MS/MS	GCHRMS	GC-MS/MS	GCHRMS	GC-MS/MS	GCHRMS	GC-MS/MS	GCHRMS
1	2,3,7,8-TeCDD	ND	ND	ND	ND	ND	ND	ND	ND
2	1,2,3,7,8-PeCDD	ND	ND	ND	ND	ND	ND	ND	ND
3	1,2,3,4,7,8-HxCDD	ND	ND	ND	ND	ND	ND	ND	ND
4	1,2,3,6,7,8-HxCDD	ND	ND	ND	ND	ND	ND	ND	ND
5	1,2,3,7,8,9-HxCDD	ND	(0.3)	ND	ND	ND	ND	ND	ND
6	1,2,3,4,6,7,8-HpCDD	(0.6)	(0.5)	ND	ND	ND	(0.2)	ND	ND
7	OCDD	(0.3)	3.6	(1.0)	(1.1)	ND	(0.5)	ND	ND
8	2,3,7,8-TeCDF	(0.2)	(0.3)	ND	ND	(0.3)	(0.4)	ND	ND
9	1,2,3,7,8-PeCDF	ND	(0.2)	ND	ND	ND	ND	ND	ND
10	2,3,4,7,8-PeCDF	(0.1)	(0.2)	ND	ND	(0.2)	(0.3)	ND	ND
11	1,2,3,4,7,8-HxCDF	ND	ND	ND	ND	ND	ND	ND	ND
12	1,2,3,6,7,8-HxCDF	ND	ND	ND	ND	ND	ND	ND	ND
13	1,2,3,7,8,9-HxCDF	ND	ND	ND	ND	ND	ND	ND	ND
14	2,3,4,6,7,8-HxCDF	ND	ND	ND	ND	ND	ND	ND	ND
15	1,2,3,4,6,7,8-HpCDF	ND	ND	ND	ND	ND	ND	ND	ND
16	1,2,3,4,7,8,9-HpCDF	ND	ND	ND	ND	ND	ND	ND	ND
17	OCDF	ND	ND	ND	ND	ND	ND	ND	ND

Table 1: Comparison table GCMS-TQ8050 vs. HRMS on a real sample. Quantitative value of DXNs contained in real sample (pg/g).

In addition, the new detector has a long service life, at least five times more than previous systems, enabling high sensitivity analysis with longer stability.

#### Result

The new GCMS-TQ8050 perfectly fits for trace analysis applications of extremely small quantities of dioxin in food & feed and the environment, POPs components, endocrine disrupting chemicals, impurities in pharmaceuticals and banned drugs in hair samples.

Table 1 shows a real sample comparison between the GCMS-TQ8050 and a JEOL HRMS system.

Combined with the MIURA GO-HT series clean-up system and BUCHI Speedextractor PSE (see dioxin S3 offer), it provides a powerful tool for dioxins analysis in food & feed to meet the EU regulation 589/2014 since June 2014.

#### Literature

Commission Regulations (EC) No 152/2009 (Feed), (EU) No 589/2014 (Food)



Further information on this article
• Flyer: Dioxin S3



Figure 2: Off-axis ion optics













## Vanillin – natural or synthetic?

### GCMS and IRMS acquire the isotopic fingerprint of vanillin

anillin is the most popular flavor worldwide. It is traditionally produced from vanilla pods (Vanilla planifolia or other species) by maceration. However, natural vanilla production covers only 1 % of global demand. 99 % of vanillin flavor is produced synthetically (petrochemical origin) or biotechnologically (e.g. from ferulic acid, eugenol).

Between natural and synthetic vanillin is a cost factor of 100:1. It's no wonder that vanillin is one of the most imitated additives in food products worldwide. How can consumers be protected from false declaration and fraud?

Natural and synthetic vanillin is chemically identical, but an isotopic signature is left by the raw material and the production process. Consequently, the "isotopic fingerprint" can be used for differentiation.

## GCMS and IRMS reveal compound-specific isotope signature

A common elemental analyzer, coupled to a high resolution sector field mass spectrometer (Isotope Ratio Mass Spectrometer, IRMS) can be used for the determination of the isotope signature from pure material with high precision and accuracy. However, the vanillin (target compound) in a vanilla extract from a complex matrix ranges in concentration from 2-300 µg mL<sup>-1</sup>.

The substances should therefore first be separated from each other, after which isotopic ratios of the target compounds as well as of accompanying compounds when needed can be determined (Compound Specific Isotope Analysis, CSIA).



Natural vanilla. The name comes from the Spanish »vainilla«, small pod.

This can be accomplished by coupling of GCMS and IRMS. The Austrian-based company Imprint Analytics uses a Shimadzu GCMS (GCMS-QP2010 Ultra) for this coupling technique as a worldwide first.

The CSIA technique was first used commercially for the determination of the <sup>13</sup>C/<sup>12</sup>C ratio in vanillin (Hoffman und Salb, 1979 [1]). However, the determination of only <sup>13</sup>C cannot differentiate between synthetic (petrochemical) and biotechnological (e.g. ferulic acid, eugenol) origin of vanillin. Furthermore, the <sup>13</sup>C signature can be manipulated by enriched raw material so as to counterfeit a natural product. More differentiation characteristics should be included.

## Determination of the isotopic ratios of hydrogen

For the above reason, more differentiating parameters should be determined. Additional determination of the isotopic ratios of hydrogen has been proposed (Greule et al., 2010 [2]). However, little data is available for

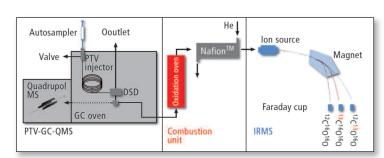


Figure 1: Method for measuring the carbon isotopic ratio in vanillin (schematic drawing). The sample is cold injected and the solvent is blown out from the PTV (vent). The Detector Switching Device (DSD) from Shimadzu is located after the separation column, where the matrix substances are separated. The gas flow is split after the DSD: the smaller part is transferred to the quadrupole mass spectrometer, where the substances are identified; the larger part is flashed in the oxidation oven. The CO<sub>2</sub> is transferred into the IRMS after removal of water and the isotopic ratio is measured.

this today. Imprint Analytics has built up a large database in the past years which can be used to determine the origin and authenticity of such samples.

Which methods does Imprint Analytics use to verify whether vanillin is from natural, natureidentical or synthetic origin in extracts and end-products?

#### **Materials and methods**

Vanillin is extracted from an essence (or from the end-product). The solvent is 2-Methoxy-2-methylpropane (MTBE). 200 µL of the sample are used on the auto sampler (Shimadzu, AOC-5000).

Two separate analyses are performed to determine the isotopic signatures of carbon and hydrogen.

The samples are cold injected with a 10  $\mu$ L syringe into the Optic-4 Multimode Inlet (GL Sciences, Tokyo, Japan) at 35 °C. The solvent is removed for some seconds by blowing it out and the injector is then heated up to 285 °C. The sample is transferred onto the column by splitless injection mode. Separation is done using a ZB-5MSi column (60 m x 0.25 mm 1.4  $\mu$ m, Phenomenex, Torrance, USA). The GC program is:

Temperature program: 35 °C, 1 min.

Heating rate: 20 °C min<sup>-1</sup> until 300 °C,

Pressure program: He carrier: 300 kPa, flow control: linear velocity, APC1 (pressure after Detector Switching Device, DSD) 100 kPa;

Splitless time: 0.7 min.

The DSD from Shimadzu is located after the separation column. It enables total separation of the solvent and transports other substances into the heart-cut purge. Vanillin and other substances with similar retention times are transported by a T-piece into an inert silica glass capillary with two directions:

 separate substances which are identified in the Shimadzu GCMS-QP2010 Ultra

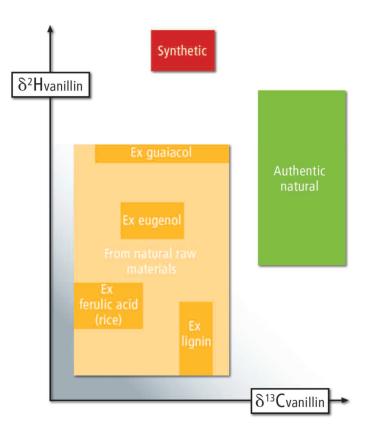


Figure 2: Isotopic signature of vanilla flavour. Possible isotopic signature ranges from natural, biotechnological and synthetic vanillin are shown (data from Imprint Analytics).

 organic substances which are oxidized in the oven into H<sub>2</sub>O und CO<sub>2</sub> (Hekatech, Wegberg, Germany).

Water is removed with a Nafion<sup>TM</sup> membrane from the He flow (Hekatech, Wegberg, Germany) and CO<sub>2</sub> gas is forwarded to the IRMS (Nu Instruments, Wrexham, UK) where the isotopic ratio <sup>13</sup>C/<sup>12</sup>C is determined (figure 1).

The determination of hydrogen isotopes is similar, but more sample material is required. Injection volume and blow-out time in the Optic-4 Multimode Inlet are therefore increased. Vanillin is carried into the pyrolysis oven instead of an oxidation oven. In the pyrolysis oven, vanillin is reduced to H<sub>2</sub> (and CO). H<sub>2</sub> is transferred to the IRMS to determine the isotopic ratio of <sup>2</sup>H/<sup>1</sup>H.

Measured values are normalized in two steps to ensure high measurement accuracy: each run with a reference gas, and each sample row with a reference material (in case of hydrogen two reference materials) with known isotopic ratio. The normalization process is verified by quality control samples. More replicate measurements are carried out for both elements to ensure a high precision

Measured values are compared with a reference database (figure 2). In this way, it is determined whether vanillin is from natural, nature-identical, or synthetic origin. The composition of the isotopes can also be used to prove authenticity and the determination of the geographical origin of the natural vanillin. The declaration of bourbon vanilla can be verified by this procedure, e.g. whether the vanilla really originates from Madagascar.

#### Outlook

All vanillin sources can be determined using the technology and method described. But new possibilities to produce vanillin in the future by other ways will occur. For this reason, this method should be developed further. Oxygen isotopes should be considered in the future to ensure robust results on the authenticity of vanillin. A third measurement is required, where the oxygen of

vanillin reacts to CO in the platinum oven.

A large number of flavor substances are falsified (e.g. benzaldehyde from bitter almond, or cinnamaldehyde in cinnamon), just to mention some. Imprint Analytics will develop more method applications for this field.

#### **Authors**

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#### Literature

- [1] Hoffman, P.G., Salb, M. (1979) Isolation and stable isotope ratio analysis of vanillin. J. Agric. Food Chem., 1979, 27 (2), pp 352-355
- [2] Greule, M., Tumino, L.D., Kronewald, T., Hener, U., Schleucher, J., Mosandl, A., & Keppler, F. (2010) Improved rapid authentication of vanillin using δ13C and δ2H values. European Food Research and Technology, 231: 933-41

Further information on this article

• Website: Imprint

Analytics















## Safe, clean and free of harmful substances?

### Analytical methods for plastic packaging of foods



he influence of cups, plastic bottles and film packaging on human health and the environment is a challenge. Supermarket shelves are full of products that are, in part, packaged in highly complex plastics.

These types of packaging materials are lightweight and also highly stable, and protect expensive as well as easily perishable products against environmental influences. At the same time, plastic packag-

ing carries colorful printed advertising messages and fact-oriented product information.

But plastic packaging materials are increasingly the subject of criticism, because undesirable and potentially hazardous substances can migrate from the packaging into the foods when in direct contact. More plastic also means more waste. Plastics not only end up in recycling yellow bags and on landfills but also in rivers and

seas – and in the stomachs of fish and seabirds.

Just to quantify, a brief look at Germany as one of the largest European countries. In 2013, around 17.1 million tons of plastic packaging wastes were generated. The increase to this highest level is attributable mainly to changes in consumption patterns. Nearly 72 percent, i.e. 12 million tons of the packaging wastes went into recycling [1].

#### EU Ordinance on the Avoidance and Recovery of Packaging Wastes

As early as 1998, the European Union adopted the Ordinance on the Avoidance and Recovery of Packaging Wastes (VerpackV) and amended it in the most recent version of 2014 [2].

The objective of VerpackV is to avoid or reduce the environmental impacts of packaging waste. In addition, the share of beverages filled in reusable drinks packaging and environmental-

friendly one-way drinks packaging should increase to at least 80 %.

According to a report of the European Environmental Agency (EEA) in 2013, Europeans recycled more waste from 2001 to 2010: the recycling share increased from 23 percent to 35 percent – in absolute numbers this was 63 million tons of household waste which found a new destination [3].

## VerpackV and harmful substances

VerpackV also regulates the concentration of harmful substances such as heavy metals that may be present in packaging materials. Accordingly, packaging or its components may only enter circulation if the cumulative concentration of lead, cadmium, mercury and chromium VI does not exceed 100 mg/kg.

In addition to the REACH Ordinance Annex XVII, there is a ban on bringing certain cadmium-

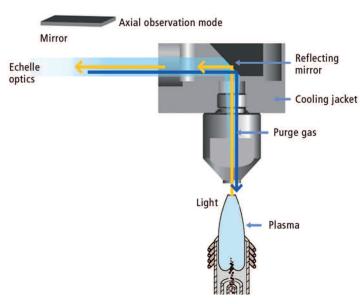
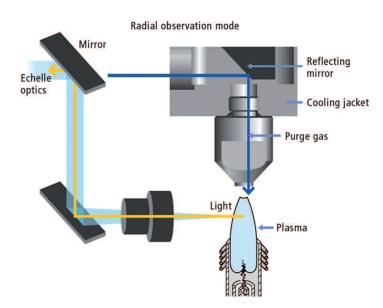


Figure 2: ICPE-9820 ICP-OES spectrometer with 'dual view' optics

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Sample	BCR680			BCR681					
Element	Dry Method	Wet Method	Microwave M.	Certified V.	Dry Method	Wet Method	Microwave M.	Certified V.	*Detection Limit
	[mg/L]	[mg/L]	[mg/L]	[mg/L]	[mg/L]	[mg/L]	[mg/L]	[mg/L]	[mg/L]
Cd	141	140	140	140.8	21.0	21.4	21.7	21.7	0.02
Pb	105	< 0.2	108	107.6	13.1	< 0.2	13.5	13.8	0.2
Cr	105	112	112	114.6	16.2	17.2	17.5	17.7	0.03
Hg	< 0.2	24.0	25.6	25.3	< 0.2	4.3	4.6	4.5	0.2
As	28	31	30	30.9	4	4	5	3.93	0.5

Table 1: Distribution of the elements Pb, Cd, Hg and Cr in polyethylene (reference material BCR 681), \*Detection limit at sample preparation with delution of 0.2 g/20 mL

containing compounds into circulation if the cadmium content exceeds 100 mg/kg (0.01 % by mass). These restrictions are most important for plastic products as they are produced and distributed in high quantities.

In this case, a distinction is made between cadmium used for coloring certain substances and mixtures (e.g. cadmium sulfide or cadmium selenide) and cadmium used as a stabilizing agent (e.g. cadmium stearate) for certain mixtures or products of vinyl chloride polymers or copolymers. The addition of cadmium used as a dye is restricted in nearly all types of plastics, while the admixture of cadmium as a stabilizer pertains to 13 vinyl chloride (co)polymer product groups (i.e. PVC), for example packaging materials.

## Recycled plastics for food packaging?

Particularly, recycled plastics as materials for food packaging are in focus, for instance PET bottles. The plastics experts group at the Federal Institute for Consumer Health Protection and Veterinary Medicine has explained this as follows:

The legal regulations for consumer goods are also applicable to recycled PET plastics. However, due to possible improper use and the generally non-homogeneous input material, additional quality assurance measures are required. The input material for the mechanical recycling process should consist of at least 99 % original 'food grade' PET. The recycling process applied must be able to remove migration-relevant substances from the polymer matrix that may be present due to prior use. The required cleaning efficiency is to be verified once in

terms of a 'worst-case' scenario using model contaminants (surrogates) and using a migration test or evaluation. An adequate quality assurance analysis method is essential for monitoring of the production process [4].

Using spectroscopy, chromatography, mass spectrometry and materials testing technology, Shimadzu, as one of the world's leading manufacturers of analytical instrumentation, offers the

in the sample compartment without any complex sample preparation.

The sample to be examined is irradiated from below with high-energy X-rays. When these hit an atom, an electron from one of the lowest energy levels of the K and L shells is excited energetically to such an extent that it is ejected from its orbital position. The resulting 'hole' is immediately occupied by an electron falling

ICP-OES spectrometers such as the ICPE-9820 (figure 3), which are characterized by high sensitivity, a wide dynamic measuring range and high sample throughput. The ICPE-9820 with CCD (charge coupled device) detector is equipped with vacuum optics, setting new standards with respect to performance, speed and operating costs.

After applying a suitable digestion method the plastic samples are injected to the ICPE-9820. At argon plasma temperatures of appr. 10,000 K the atoms and ions are excited and subsequently moving back to ground state. At this step energy (light) is emitted in UV-VIS range.



Figure 3: ICP-OES Spectrometer ICPE-9820

complete hardware and software range for reliable determination of hazardous substances and identification of the materials used.

## Qualitative and quantitative determination of heavy metals

For rapid qualitative and quantitative determination of heavy metals in plastics (such as cadmium in the desired concentration range) used in food packaging, energy-dispersive X-ray fluorescence spectroscopic analysis is the method of choice. Shimadzu's EDX series (EDX-7000P/8000P) enables the analysis of food packaging like plastic films, PET bottles, polystyrene yoghurt containers and much more, in the low-ppm range (figure 1). The sample can be positioned directly

from a higher shell. This process releases energy known as secondary energy or X-ray fluorescence energy.

In the present example, this element-specific fluorescence radiation corresponds to the energy difference between the K or L shell and the shell of the higher energy levels from which the 'falling' electron originates. By knowing the energy ratios of the individual elements, reliable quantitative analysis of an unknown sample is possible: the detected X-ray fluorescence is element-specific.

## Simultaneous determination of heavy metals

Simultaneous determination of heavy metals is carried out using

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The radiation emitted is processed in the optical system and is measured by the CCD detector. The emission spectrum (detected in the range of 167 to 800 nm) contains information of over 70 elements, which can be used for quantification. This quantitative determination of elements is carried out using calibration curves from multi-element standards. Figure 2 shows the optical system of the ICPE-9820 in 'dual view' for axial and radial plasma observation.

## Various sample preparation methods

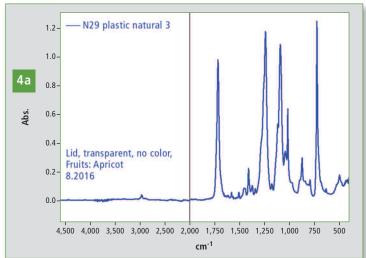
Various methods are available for sample preparation. A widely used method is microwave digestion, carried out in microwavepermeable pressure vessels in a microwave oven. An example is the treatment of samples using a mixture of concentrated nitric acid with addition of a small amount of hydrogen peroxide at elevated pressure-temperature conditions. In addition, wet-ashing or dry-ashing, in which the organic constituents of a solid sample are digested via combustion in a crucible, are also possi-

The ICP-OES system configuration described here provides a current state of the art overview of the determination of heavy metals in plastic packaging according to the European Packaging Ordinance. The actual concentrations of the individual substances in the homogeneous sample material can vary considerably and can be analyzed easily and rapidly using the ICPE-9820 following sample preparation and dilution.

## Identification of plastics, including main components and additives

FTIR spectroscopy using the IRAffinity-1S spectrophotometer in combination with ATR (attenuated total reflectance) technology offers an ideal system configuration for the identification of plastics. The determination of polymers takes place via diamond-ATR and enables unequivocal determination of the main





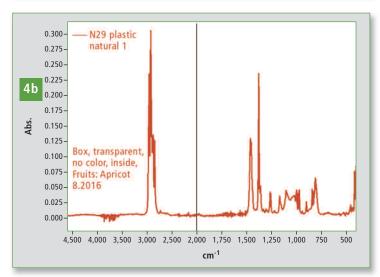


Figure 4: Shown here is the packaging for apricots – a plastic box with lid and a label 'Recycling 5 for polypropylene' under the box. The box is made of polypropylene (IR spectrum figure 4b), while the lid is made of PET (IR spectrum figure 4a).

components as well as the identification of additives, for instance plasticizers, as a function of the concentration.

PVC for instance is a very hard and brittle plastic in its initial

form needing a plasticizer to make it more flexible. Soft PVC is used, for example, in the production of packaging films applied as outer packaging of PET beverage bottles. PVC films are also still used at deli counters. The use of the particularly harmful, endocrine disruptive plasticizer DEHP (a phthalate) has, however, been banned in packaging of fat-containing foods. Plasticizers used in food packaging materials pose a risk for migration of undesirable substances into the food. The food industry therefore uses other plastics such as polyethylene (PE), polypropylene (PP), polycarbonate (PC) and polyethylene terephthalate (PET).

## The use of infrared spectroscopy

PE, PP, PC and PET were investigated via infrared spectroscopy in about 30 food packagings (table 1) obtained from supermarkets in the Netherlands and Germany. The packagings (transparent, colorless) were examined from the inside and outside. Where the packaging consisted of several components such as lids, bottle caps or film layers, for instance as tamponage for liquids, these components were included in the measurements. Consumer information on the label informing of the polymer used was also determined.

It is always astonishing that a polymer may be listed on the outer packaging label as PP or PET – and found in the measurements – while the inner packaging actually consists of a PE. Will this not be a real challenge for the recycling industry?

Sample measurement using the FTIR spectrometer takes place non-destructively and without much time and effort. In particular, comparison of the IR spectrum of the actual measured sample with comprehensive spectral libraries allows the identification of the material. In addition, it is possible to obtain a clear indication of whether it is a pure substance, a product mixture or even a recyclate. In this investigation, only surfaces that could interact with an infrared penetration depth of up to 2 µm were considered.

Looking at the polymers identified in Table 2, those listed on the label and the packaged food, it

Sample	Location of the label	Recycling symbol		Identification via FTIR	
No: Type of food			Container, cup, tray	Lid, cover	Film, adhesive, tamponade
N02: meat	Container, external surface	1 PET	PE, PET	none	PP, PE
N03: meat	Container, external surface	1 PET	PE, PET	none	PP, PE
N04: chicken meat	Container, external surface	1 PET	PE, PET	none	PP, PE
N05: Egg carton	Container	1 PET	PET	none	kein
N06: chicken meat	_	_	PS	none	PET plus
N07: Strawberries	Container, external surface	1 PET	PET	PET	PE, PE+ resin
N08: Salmon/egg salad	Container, external surface	1 A-PET	PET	PET	PET plus
N09: Grapes	Container, external surface	1 PET	PET	PET	none
N10: Sausages	_	_	PE, PET	none	PE, PET, starch
N11: Nuts	Container, external surface	1 PET	PET	PET	PET
N12: Wine cup, disposable	_	-	PS	none	PP
N13: Sliced cheese	_	_	PET	none	PET
N14: Small cake	_	_	PS	PET	PP
N15: Mini waffles	Container, external surface	1 PET	PET	none	PP
N16: Flavored water	_	_	PET	PE, PE plus	kein
N17: Cookies	Container, external surface	05 PP	PP	none	none
N18: Cup, disposable	Cup, external surface	05 PP	PP	none	none
N19: Cup, disposable	Cup, external surface	06 PS	PS	none	none
N21: inner Sausage	_	_	none	none	Cellulose
N22: Organic cling film	_	_	none	none	PE, PET
N23: Sweets, wine gums	Film	Symbol	none	none	PP plus
N24: Apples	Container, black	Symbol	PS	none	PVC
N25: Sweets, wine gums – sour	Film	5 PP	PP	PP, PP plus water?	
N26: Organic bread	_	_	none	none	PP
N27: Bread	_	_	none	none	PP
N28: Tomatoes	_	_	PET	PET	
N29 - Apricots	Container, external surface	5 PP	PP	PET	
N30: Sweets, liquorice	Film	05 PP	none	none	PP
N31: Fruits, self-service	-	-	none	none	PE
N32: Red currants	Container, external surface	PET	PET	PET	none

Table 2: Transparent, colorless packaging material and the infrared-identified main polymers found in the packaging

can be concluded that each polymer is used ubiquitously. No trend could be found. In these types of packaging, the visual appearance, the value and ultimately the price determines the so-called 'make-up.' Among the polymers found, no PVC film could be identified.

#### **Summary**

The determination of plastic food packaging requires careful quality assurance analysis using EDX, ICP-OES and FTIR spectrometry. Investigation of transparent packaging materials using FTIR-ATR technology, however, reveals

that the plastics indicated on the recycling symbol are not always unequivocal. In some PET and PP samples, other plastics have been identified. Such mixtures place high demands on the recycling industry. Further investigations using complementary analysis methods on this topic should provide more information and lead to a complete solution – including colorless and dyed materials up to black plastics.

[4] Gesundheitliche Beurteilung von Kunststoffen im Lebensmittelverkehr: Stellungnahme der Kunststoffkommission des Bundesinstituts für gesundheitlichen Verbraucherschutz und Veterinärmedizin zur Verwendung von Kunststofferzeugnissen für Mehrweganwendungen und von Kunststoff-Recyclaten für die Herstellung von Lebensmittelbedarfsgegenständen, Bundesgesundheitsblatt, 1995, 38, 73



FTIR spectrometer IRAffinty-1S

#### Literature

- [1] Umweltbundesamt, Abfall- und Kreislaufwirtschaft – Verpackungsabfälle 2/2016
- [2] Verordnung über die Vermeidung und Verwertung von Verpackungsabfällen (Verpackungsverordnung – VerpackV), 2014
- [3] EEA Report, Managing municipal solid waste, ISSN 1725-9177, 2/2013

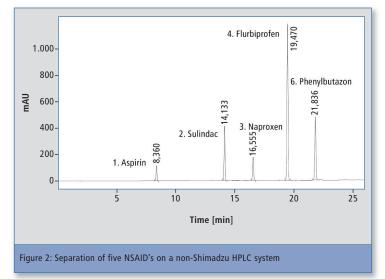
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faster and better analytical methods that improve efficiency and throughput especially in R&D environments.

However, many of the analytical methods used in quality control laboratories, including those listed in the pharmacopeia, are conventional HPLC methods. Transferring these HPLC methThe Nexera-i MT incorporates two analytical flow lines with different system volumes into a single compact integrated LC. By automatic switching between these flow lines, Nexera-i MT seamlessly performs both HPLC and UHPLC analyses, preserving the relative separation pattern by compensating automatically for differences in system volume.

Figure 1: Nexera-i MT system



| Nomal | Advanced | End Time : 36.00 min | Method 20 min Gradient\_HPLC\_Volume adjust 500ul.lcm | End Time : 36.00 min | Method 20 min Gradient\_HPLC\_Volume adjust 500ul.lcm | End Time : 36.00 min | Method 20 min Gradient\_HPLC\_Volume adjust 500ul.lcm | End Time : 36.00 min | Method 20 min Gradient\_HPLC\_Volume adjust 500ul.lcm | End Time : 36.00 min | End Time : 36.0

Consequently, Nexera-i MT achieves exceptional analytical reproducibility when switching from a system with large volume to a system with smaller volume, or vice-versa.

The same technology also allows Nexera-i MT to match any existing HPLC or UHPLC method run on competitive instrument platforms, eliminating the need to consider and carefully match plumbing to achieve identical system volumes between instruments.

The instrument offers enhanced functionality to support the transfer of existing HPLC methods to faster UHPLC analyses while assuring high cross-compatibility between the former and new method conditions. It can also be used for quick method development in UHPLC mode and seamlessly convert into HPLC methods for broader applicability by using a simple conversion program.

## Two independent flow lines

Nexera-i MT features two independent and dedicated flow lines, one for UHPLC and the other for HPLC analyses. Newly developed Analytical Conditions Transfer and Optimization (ACTO) technology minimizes

the effect of system volume differences between different systems on analytical results.

In addition to drastically improving the efficiency and quality of method development and transfer efforts in quality control departments, Nexera-i MT's dual flow lines also maximize operational efficiency by enabling a single instrument to run both HPLC and UHPLC analyses, as opposed to separate dedicated instruments for each method.

As an example application figure 2 shows the analysis of five non-steroidal anti-inflammatory drugs on a conventional HPLC system from another vendor.

#### **Method conditions:**

#### Column:

ACE Excel 3 SuperC18 150 x 4.6 mm (ACT)

#### Mobile phase:

A: 0.1 % formic acid in H<sub>2</sub>O B: 0.1 % formic acid in MeCN

Gradient program					
Time	% B				
0	20				
20	70				
25	70				

Followed by 11 min reequilibration time (20 % B) 36 min run time

Flow rate: 1 mL/min Temperature: 40 °C Detection at: 254 nm Sample: 0.6 mg Aspirin, 0.1 mg Sulindac and 0.2 mg of Naproxen, Flurbiprofen, Phenylbutazone in 1 mL Acetonitrile / Water (50:50 v/v)

## Method transfer to a different system

lethod Editor (Instrument Parameters)

If the HPLC system that was used to develop and run this method needs replacement but the same system is no longer available, the user will face the problem of having to transfer the method to another system, with different performance parameters and system volume. This may result in a totally different method that will render future results not comparable to previous data. On the other hand, when purchasing an older instrument to match the method conditions, the chance can be missed to get the latest equipment that could still be used when a more efficient, faster (UHPLC) method is required.

In this case, the Nexera-i MT system is the ideal solution. The customer acquires state-of-the-art equipment offering all the latest technology, including pressure resistance up to 660 bar, auto-validation function and touch panel control, and can accurately

reproduce HPLC analysis from previously used methods.

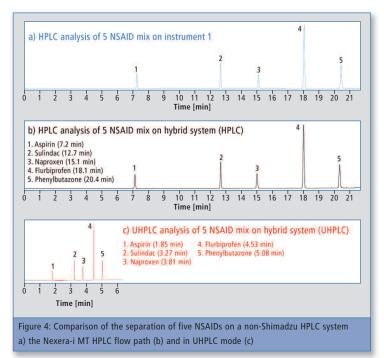
To illustrate these features, the analysis shown in figure 2 was transferred to the Nexera-i MT system. Method conditions remained the same, while a small difference in system volume between the HPLC flow path and the other instrument was accounted for in the software (figure 3).

Using this feature and exactly the same analysis conditions including column and sample, the chromatogram could be reproduced almost identically (figure 4a, b).

## Method transfer tool increases efficiency with a mouse-click

Furthermore, by using the method transfer tool in the LabSolutions Software the method could be switched easily to the UHPLC flow path and run time was decreased by 19 min to an overall run time of 7 min (figure 4c, page 12).

And it doesn't take an expert chromatographer to perform this kind of increase in efficiency. The ACTO function included in LabSolutions is an easy-to-use interface offering all necessary steps at the click of a mouse.



The user only has to open the chosen HPLC method and click the method transfer button in the user interface (figure 5). Original and new method conditions including column dimensions and flow rate can be entered, and the software automatically calculates and incorporates

the new gradient profile and method parameters to a new UHPLC method, which runs through the UHPLC flow path of the system.

The combination of the Nexera-i MT system with LabSolutions and the systems' unified graphical

Configuration Column Inner Diamete Inner Diamete Particle Size Particle Size 3.0 System Flow line 2 (Mixer Volume 300uL) Flow line 1 (Mixer Volume 40uL) 36.00 Total Flow Total Flow 1.0000 mL/mir 0.40 mL/min 80 25 Response Response Injection Volume Injection Volume: 8.0 1.7 uL Before inj. (308uL) Gradient start: Gradient program Time A.Conc B.Conc C.Conc D.Conc A.Conc B.Conc C.Conc D.Conc 20.0 1 INIT 80.0 20.0 0.0 2 20.00 30.0 70.0 2 6.95 1000 00 100.0 0.0 Figure 5: Method transfer calculator

user interface and software allow intuitive operation independent of the user's experience level. In addition, where another CDS is used, available software drivers allow the Nexera-i MT's special features to be used with most other vendor software packages.

Further information on this article www.shimadzu.eu/ nexera-i-mt



APPLICATION















## New - Application Handbook Liquid Chromatography

PLC, UHPLC as well as SFC systems are able to quantitatively analyze substances in mixtures containing multiple ingredients by separating and detecting target substances.

They are used to separate, quantify, qualify or purify single components from a sample containing various analytes in different matrices.

Shimadzu offers a wide variety of application-specific systems, such as automated sample pretreat-

ment systems for amino acid analysis or on-line sample trapping for the quantification of residual pesticides in food or soil.

Further information on this article Download: Application Handbook Liquid Chromatography



















## GC pioneers of the first hour

### 60 years of gas chromatography



ith its 120 kg, the GC-1A could have been named a 'Sumo wrestler' among GCs. Everything, from sample introduction, separation, detection up to chromatogram evaluation, was included within a single structural framework.

The GC-1A was Japan's first gas chromatograph; it was developed by Shimadzu in 1956, only four years after the British scientist A.J. Martin was awarded the Nobel Prize for his groundbreaking research on gas chromatography, which he utilized together with his colleague A.T. James.

With the GC-1A, Shimadzu pushed forward the legacy of its founder family. Genzo Shimadzu Junior, the second-generation company leader, was one of the 10 most important inventors of Japan and was honored by the emperor at that time. In 2016, Shimadzu celebrates its 60th GC anniversary. Within the 141 years of Shimadzu's company history, chromatography paved the way and has set numerous milestones that today have become technical standards and serve consumer protection, environmental protection and product safety.

#### **Major analytical** technology

Since its commercial introduction, gas chromatography has evolved into a major analytical technology. Today, tens of thousands of GC systems are in use in all areas of industrial and pharmaceutical research and development, in basic research, environmental research, and in quality control. The separation of complex mixtures as well as the identification and quantification of the individual components is still considered to be one of the most important tasks in instrumental analysis.

"Contributing to society through science and technology" is Shimadzu's core philosophy. This also includes the continuous development of existing technologies, exploring possibilities and pushing existing technical boundaries. This has made Shimadzu a market leader and an established name in science and industry.

#### GC milestones: hardware and software, central systems, detectors and accessories

Much of what has been technically established in GC today has often been achieved through hardware and software milestones. Between the first GC-1A and the current ultra-modern, universal Tracera system with its novel BID (barrier ionization discharge) detector that can detect all substances except for helium and neon, there have been numerous world premieres, advances in performance and innovations that have set technical and economic trends and standards.

• With the GC-3A series, Shimadzu introduced the first mini GC, as a development result of downsizing instru-

Year launched	Model name
1956	GC-1A
1966	GC-4A
1981	GC-8A
1988	GC-14B
1992	GC-17A
2000	GC-2010
2004	GC-2014
2009	GC-2010 Plus
2010	GC-2025
2013	Tracera

Table 1: History of Shimadzu gas chromatography systems



- The GC-4A marks the step from isothermal to temperature-programmed chromatography, which allows the separation of complex mixtures of substances in significantly less time. The GC-4A, for the first time, implements the patented dual-flow operation of two packed columns.
- Automated chromatogram evaluation starts with the C-1A integrator.
- The GC-8A is the first asbestos-free GC system. In combination with the AOC-8A autosampler, automated measurement of sample sequences is possible.
- The GC-9A and -14B mark the triumph of capillary versus packed column chromatography. Now, it is possible to achieve separations of complex mixtures with improved resolution and within a much shorter
- The GC-9A is the first gas chromatograph coupled to a mass spectrometer. In addition to quantitative evaluation, the mass spectrometer allows for the identification of unknown sample components and leads to new scientific findings in environmental, pharmaceutical and materials research.



- With the GC-17A, gas pressures and flows are now electronically adjustable and can be modified via flow and pressure programs.
- The GC-2010 has set a longterm standard with respect to detection limits. Its outstanding reproducibility remains undefeated until today.
- The LabSolutions software with self-explanatory operation of the GC controls and novel

diagnostics functions, make gas chromatography accessible to all users.

- The GC-2014 and GC-2025
  models for routine analyses
  are reliable and robust as well
  as extremely cost-effective.
  The GC-2025 is the first system
  manufactured from RoHscompatible components and is
  fully recyclable.
- The most recent development is the Tracera with its novel BID

detector – the innovative technology for the helium plasma generation combines sensitivity with so far unrivalled robustness and long-term stability, opening up entirely new possibilities in trace analysis.

#### Wide range of GC systems

Today, routine as well as high-end systems determine Shimadzu's product range – from small and versatile through ultrasensitive and highly productive systems up to instruments that break new technological grounds, for example the multiple heart-cut technology.

Numerous detectors complete the picture, including new and advanced technologies such as the BID helium ionization detector.

System solutions, e.g. detectorswitching or splitting, backflushing or autosamplers offer users customized as well as flexible application possibilities.

Shimadzu's 60 years of GC development stands for the company's ambition to continuously broaden the horizon for GC analysis with innovative and novel developments.

Further information on this article www.shimadzu.eu/ 60-years-gc

















## Technological driver and market leader

#### Milestones in 60 years of IR history

ince the middle of the 20<sup>th</sup> century, spectroscopy has been at the core of Shimadzu's analytical business unit and a main driving force in the company's growth and reputation. Knowhow, technological skills and innovative power have brought Shimadzu to a leading position in spectroscopy.

The promotion of the 60<sup>th</sup> IR anniversary kicked off in September at the JASIS conference in Tokyo, Japan.

The documented history of spectroscopy already began in the 17<sup>th</sup> century. Sir Isaac Newton first applied the word spectrum to describe the rainbow of colors that combine to form white light. During the early 1800s, Joseph

von Fraunhofer made experimental advances with dispersive spectrometers that enabled spectroscopy to become a more precise and quantitative scientific technique.

Chemical IR spectroscopy emerged as a science in 1800 through the work of Sir William Herschel. Early IR instrumentation was based on prisms or grating monochromators. Albert Michelson invented the interferometer in 1881. This working principle is still used in modern IR spectrophotometers today.

First double beam self-recording infrared spectrophotometer

In the first half of the 20th century, Shimadzu grew significantly under the leadership of Genzo Shimadzu Jr. He showed rare talent as a remarkable inventor, and under his guidance Shimadzu



progressed hand-in-hand with science, building itself into a leader in technology. In this spirit, the company developed its first double beam self-recording infrared spectrophotometer: the AR-275. The instrument became commercially available in 1956 and was robust enough even for export overseas.

Several technical issues were encountered in this development process: the manufacturing of a large high quality artificial rock salt crystal & the sensitivity and responsiveness of the thermocouple used as a detector. One of the lessons learned at this early stage was to develop and use an own thermocouple, as the originally applied component imported from overseas had a compromised vacuum.

In 1959 the IR-27 platform was launched. This second generation of double beam self-recording spectrophotometers was considerably smaller than the standalone AR-275 and was designed as a table top instrument. The final model of this range was the IR-27G, launched in 1965. Weighing 130 kg, it was a heavy and bulky instrument requiring a very solid table.

## A brief overview of technological milestones

The AR-275 marked the start of an outstanding line of infrared products spanning six decades of innovation. Some of the most important Shimadzu milestones of the past 60 years include:

- 1965: IR-27G First dispersive table top instrument
- 1981: IR-435 First instrument with data processor. On the verge of the computer era, the IR-435 paved the way from analog to digital
- 1984: FTIR-4000 First Fourier Transform Infrared Spectrophotometer, made possible by the rapidly developing computer technology. FTIR lowered the threshold for use of the IR spectrophotometer, and measurements could be done in seconds. New applications became



AIM-9000 - Automatic Failure Analysis System featuring a unique concept in the field of micro sample analysis

possible, including foreign substance analysis, verification testing, functional group identification and quantitative analysis.

• 1990: FTIR-8100 First FTIR with dynamic alignment stan-

Year launched	Model name		
1956	AR-275		
1959	IR-27		
1964	IR-27C		
1965	IR-27G		
1970	IR-400, IR-450		
1974	IR-430		
1977	IR-410		
1978	IR-408, IR-420, IR-440		
1981	IR-435		
1984	IR-4000		
1986	IR-460, FTIR-4100		
1987	FTIR-4100, FTIR-4300		
1988	IR-470		
1990	IR-8100, FTIR-8100M		
1991	FTIR-8500		
1992	IRG-8000		
1993	FTIR-8200, FTIR-8200D FTIR-8600		
1994	FTIR-8100A, FTIR-8200A FTIR-8200PC, FTIR-8600PC, µIR-8000		
1997	FTIR-8300, FTIR-8700		
2000	FTIR-8400, FTIR-8900		
2002	FTIR-8400S,		
	IRPrestige-21		
2008	IRAffinity-1		
2013	IRTracer-100,		
	IRAffinity-1S		

Table 1: History of infrared spectrophotometers from Shimadzu dard. The following generations featured the new Shimadzu "IRSolutions" software running on PCs

- 2002: IRPrestige-21 First FTIR with mid-near-far infrared measurement range and the flexibility to add the far infrared measurement range (MCT detector) and the near infrared measurement range (InGaAs detector)
- 2008: IRAffinity-1 New benchmark for entry level instruments with best in class performance
- 2013: IRTracer-100 First FTIR with rapid scan function and LabSolution SW platform

#### Towards the future

Finally, in this IR celebration year, Shimadzu has launched the AIM-9000 FTIR microscope and failure analysis system as successor to the AIM-8800.

The AIM-9000 is intended as an FTIR microscope for all users. All important steps for micro sample analysis, observation, measurement and analysis are highly automated, making this system also suitable for less experienced operators. Combined with the IRTracer-100, the AIM-9000 offers a staggering

S/N ratio of 30.000/1 and has a wide range of accessories for many different application types.

Shimadzu's history in IR spans many technological eras. Today, the Shimadzu spectroscopy portfolio includes software and hardware solutions such as UV-VIS spectrophotometers, FTIR spectrophotometers & microscope systems, fluorescence spectrophotometers, energy dispersive X-ray fluorescence spectrometers, Atomic Absorption Systems (AAS) and ICP-OES systems as well as ICP-MS instruments.

A complete overview of model names and release dates of infrared spectrometers sold by Shimadzu is shown in table 1. In the past six decades the total number of Shimadzu infrared spectrophotometers sold throughout the world has reached more than 20.000 units, underlining Shimadzu's position as a leader in spectroscopy.

Further information on this article www.shimadzu.eu/ 60-years-ir



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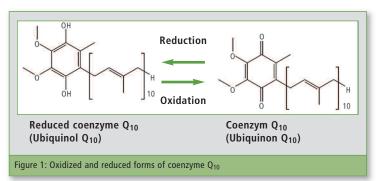






## Coenzym Q<sub>10</sub> — analysis of a sensitive substance

#### Analysis of unstable compounds using online SFE-SFC



ree radicals are short-lived molecular fragments of oxygen and are suspected of being responsible for aging processes but also for damage to cells or enzymes. Cells, however, have their own defense mechanisms to counter free radicals. Such antioxidants, like ascorbic acid or coenzyme Q<sub>10</sub>, react with free radicals and are provided by dietary intake.

UV-radiation, pollutants in the air or chemicals lead to the formation of highly reactive oxygen species, so-called free radicals in the human body.

If they accumulate, they can cause cell damage and other signs of aging, for instance the formation of wrinkles. Free radicals are even associated with a number of diseases – including cardiovascular disease and cancer.

In addition to the body's own antioxidants mentioned above, substances provided by dietary intake can exhibit similar effects. The best-known examples are vitamins or their precursors. This also includes coenzyme Q<sub>10</sub> and its reduced form, which is structurally related to vitamin E and K.

Additionally to its antioxidant properties, coenzyme Q<sub>10</sub> is also involved in important biochemical processes such as cellular respiration due to its easy oxidizability. So, how can such a sensitive and easily converted substance be carefully analyzed?

## Supercritical fluids for gentle extraction

For substances that are sensitive to oxidation, analysis using supercritical carbon dioxide (CO<sub>2</sub>) offers a gentle alternative to conventional solvent extraction. Supercritical fluids combine the properties of gases and liquids: they are low in viscosity and exhibit high diffusivities similar to gases, but they are also readily soluble like liquids.

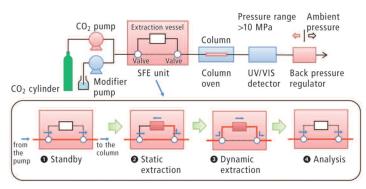


Figure 2: Flow diagram of the automated online SFE-SFC analysis

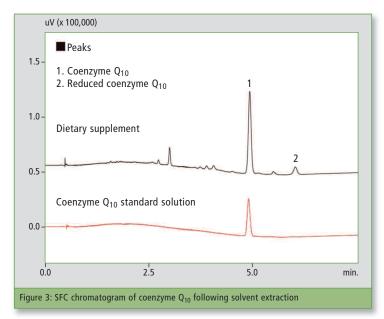
CO<sub>2</sub> (at or above its critical temperature and critical pressure) is the most commonly used supercritical fluid for chromatographic purposes. In addition to its suitable physico-chemical properties and availability, it is highly inert, non-toxic and inexpensive.

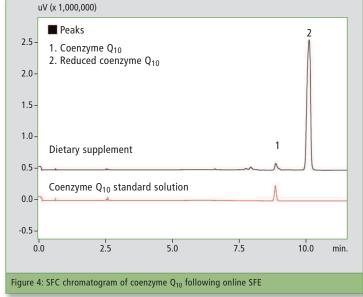
So far however, techniques like SFC (supercritical fluid chromatography) and SFE (supercritical fluid extraction) could only be used as individual steps in an analysis. The newest generation of instruments now combines the entire analysis, integrating sample pretreatment, chromatographic separation and detection into a single system. With the online coupling of SFE and SFC, manual extraction and transfer steps are now replaced by a fully automated process. This does not only reduce time and personnel expenditure but also eliminates manual errors that can occur, e.g. during decanting or pipetting.

An additional advantage of the closed online SFE-SFC system is the gentle sample handling. After being placed in the instrument, the samples can be analyzed virtually without decomposition, as the entire analytical process takes place without exposure to light or air and free from humidity. Therefore, the system is ideal for the analysis of sensitive samples, for instance light-sensitive, easily oxidized or readily hydrolyzed substances.

## Online SFE-SFC analysis of reduced coenzyme Q<sub>10</sub>

Online SFE-SFC analysis is also a gentle alternative to conventional solvent extraction for the sensitive coenzyme Q<sub>10</sub> (figure 1).





To obtain a direct comparison of the two techniques, the proportion of reduced and oxidized coenzyme  $Q_{10}$  in a dietary supplement was determined using both techniques.

The analyses were carried out using Shimadzu's Nexera UC (Kyoto, Japan), a modular allround SFE-SFC system for highperformance, high-speed applications. For solvent extraction, the content of a capsule of the dietary supplement was suspended in ethanol and the coenzyme Q<sub>10</sub> was extracted in an ultrasonic bath. After filtration, the sample was analyzed using SFC under the conditions listed in table 1.

For the online SFE-SFE analysis, about 5  $\mu$ L of the content of the capsule was dripped onto a filter paper and placed directly into an extraction vessel. The automated analysis is subsequently carried out under the conditions listed in table 2. The static extraction step is followed by dynamic extraction in order to transfer the sample to the column, where the actual chromatographic separation takes place. A flow diagram of the process is shown in figure 2.

#### **Summary**

Both chromatograms, each including a standard solution of

the oxidized form for comparison, are shown in figures 3 and 4.

It can clearly be seen that following solvent extraction almost all of the coenzyme  $Q_{10}$  is present in its oxidized form, whereas in the online SFE-SFC analysis only a very small proportion of the analyte oxidized and is largely still present in its original reduced form

Online SFE-SFC is therefore very well suited for the analysis of sensitive compounds such as substances that are sensitive to oxidation. Additionally, the Nexera UC avoids errors due to its high degree of automation and simplifies sample handling.

System	Nexera UC SFC-UV system		
Column	Shimpack UC-RP (150 mm L. x 4.6 mm I.D., 3 µm)		
Column temperature	40 °C		
Mobile phase	A: CO <sub>2</sub>		
	B: MeOH		
Flow rate	3 mL/min		
Time program	5 % B (0 min) → 50 % B (5 - 8 min)		
Back pressure	10 MPa		
Detector	UV/VIS @ 220 nm		
Injection volume	1 μL		

Table 1: Analytical conditions for solvent extraction-SFC

System	Nexera	uC SFC-UV system		
SFE				
Extraction volume	0.2 mL			
Static extraction	Time:	0 - 2 min		
	Conc. B:	5 %		
	Back pressure:	10 MPa		
	Flow rate:	3 mL/min		
Dynamic extraction	Time:	2 - 4 min		
	Conc. B:	5 %		
	Back pressure:	10 MPa		
	Flow rate:	3 mL/min		
SFC				
Column	Shimpack UC-RP (150 mm L. x 4.6 mm I.D., 3 μm)			
Column temperature	40 °C			
Mobile phase	A: CO <sub>2</sub>			
	B: MeOH			
Flow rate	3 mL/min			
Time program	5 % B (0 min) → 50 % B (9 - 13 min)			
Back pressure	10 MPa			
Detector	UV/VIS @ 220 nm			

Table 2: Analytical conditions for online SFE-SFC analysis

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View from the Zolotoy Bridge in Vladivostok. The cable-stayed bridge was opened in July 2012 on the occasion of the asia-pacific economic summit (APEC).

## Long-time partners

## Shimadzu testing equipment at Vladivostok university's Engineering School

wo years ago, the Far Eastern Federal University (Vladivostok, Russia) opened the unique Center of Mechanical Tests and Material Structural Analysis on the basis of Welding Department.

Shimadzu's entire testing equipment product range is present: systems for static and dynamic tests of metals and composites,

polymers, elastomers and plastics, construction materials, ceramic products, food, products from pulp and paper, textile and pharmaceutical industries. Today, bachelors, masters, PhD students or scientific researchers can conduct investigations in this new highly sophisticated laboratory.

The Center of Mechanical Tests and Material Structural Analysis



Figure 1: Rubber tensile test with AG-X

was established as part of the university's Engineering School. It consists of three departments: the Mechanical Tests Laboratory, Material Structural Analysis

Center activity: "We take part in many interesting projects together with the largest Far Eastern enterprises. For example, during the building of bridges across the for rebuilding of ship diesel engine shaft parts using laser deposition. This technology avoids the need to replace worn-out parts of ship machinery, while

carried out for many scientific projects. In addition, many Baccalaureate, Masters and PhD theses were written based on work carried out in the Center.

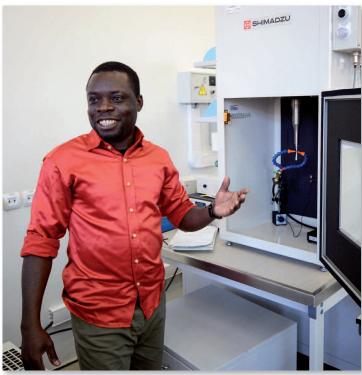


Figure 2: Introduction to USF-2000

Laboratory and Sample Preparation Laboratory. The Center became a basis for scientific research as well as for special investigations required by different factories, plants etc. Highly qualified specialists from the university work in close cooperation with the representatives of many industries.

#### Dozens of projects currently running

Currently, specialists from the Center of Mechanical Tests and Material Structural Analysis take part in more than 30 different projects. The field of research is quite wide with analysis of metals and composites as well as other construction materials, testing of polymers and ceramics, and special investigations for food, textile and pharmaceutical industries.

The head of the Center is Dr. Ekaterina Gridasova, Associate Professor of the Welding Department. She described to Shimadzu News the main directions of the

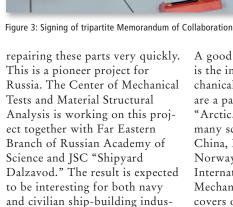
Eastern Bosphorus Strait and the Golden Horn Bay (Zolotov Rog), both in Vladivostok, Russia, many tests of high-strength bolts, fittings, cable-stayed connections etc. were made using Shimadzu instruments such as the UH-1000kNI hydraulic universal machine.

#### **New laser deposition** technology

Another important project is the development of a new technology



Figure 4: Dr. Ekaterina Gridasova, head of the Center



The laboratory doesn't only work with metals and alloys. Some time ago, a test run of silent block materials was conducted. These components are used for stabilizer bars in automotive applications. This work was done for "Repair Technology Center", the manufacturer of automobile industry spare parts.

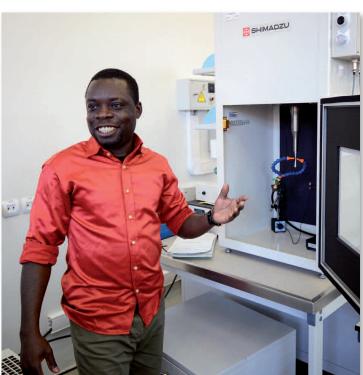
tries.

#### The international "Arctic" project investigating ice cores

An important sphere of the Center's activity is the support of University scientific researches. Different investigations are being A good example of such research is the investigation of ice mechanical properties. These studies are a part of a large project called "Arctic." In the second year, many scientists from Russia, China, India, South Korea and Norway are taking part in the International Winter School "Ice Mechanics." They investigate ice covers of Russky Island water areas at the places of sampling as well as in the laboratory. For compression tests of ice cores Shimadzu's AG-100kNX universal test machine has been applied.

#### **Cooperation between** Vladivostok, Osaka and **Dortmund universities**

An important event for the Center was the award of international status. In the beginning of 2015, Far Eastern Federal University (Russia), Material Test Engineering of Dortmund Technical University (Germany) and Welding Research Institute of Osaka University (Japan) signed a tripartite Memorandum. The relationship between these large |



universities from three different countries is interesting not only because of common scientific research opportunities, but also because it provides new, interesting educational programs for students.

Today, in the framework of the laboratory, the project of friction welding of ship metal plates has been realized. Far Eastern Federal University receives carbon steel samples from Osaka University and conducts static tests of these samples, while colleagues from Dortmund University carry out dynamic tests.

#### Long-time partnership

The Center of Mechanical Tests and Material Structural Analysis and Shimadzu have been good partners for a long time. Shimadzu's staff gives its clients full-scale assistance and support. Shimadzu organized several ioint workshops and seminars on the basis of Mechanical Tests and Material Structural Analysis Center. Visitors from different laboratories and plants could see how Shimadzu instruments work, while getting much new information about applications of the equipment in food, pharmaceutical, polymers and electronic industries. Such events increase Shimadzu's customer network and enable Shimadzu customers to exchange their experiences.

#### 2017 Fatigue Tests International Symposium

Of course, this relationship will grow continuously. For next year, Far Eastern Federal University is planning to organize a Fatigue Tests International Symposium. Shimadzu will support this event.



Figure 5: The staff of the Center

The relationship between Far Eastern Federal University and Shimadzu is a good example of the fruitful teamwork of a world-leading manufacturer of analytical instrumentation and one of the leading Federal universities of Russia.

**Author**Dr. Ekaterina Gridasova,
Far East State Technical University



### Shimadzu live

#### Car Symposium

Bochum, Germany February 1-2, 2017 www.car-symposium.de

#### Anakon

Tübingen, Germany April 3 - 6, 2017 www.gdch.de/anakon2017

#### **EWCPS**

St. Anton, Austria February 19 - 24, 2017 www.ewcps2017.at

#### **SETAC**

Brussels, Belgium May 7 - 11, 2017 https://brussels.setac.org



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