Measuring the fat content – quickly and universally
Practical advantages thanks to the latest NMR technology

The moisture, solid matter and fat content levels are important control parameters in the quality monitoring of ongoing food production operations as well as for the incoming goods control of raw materials. However, the time requirement for the analysis can cause problems, as the results are often only available hours after commencement of the analysis, which prevents fast intervention in the ongoing production process. The ORACLE Fat Analyzer (Fig. 1) as a combined microwave and NMR device provides fast, solvent-free and calibration-free technology here and delivers precise results. The ORACLE can be used to determine the fat in ice cream, dairy products, cream, cheese, meat and sausage products, fish, animal feed, dressings, mayonnaise, butter, margarine, sour cream, yoghurt, ketchup, biscuits, crackers, snacks etc. All these foods share one thing in common – they consist of extremely high quantities of water (frequently up to 70 % water), which has hitherto made fat measuring difficult.

Fig. 1: The ORACLE Fat Analyser

Requirements made of modern process analysis

Modern production methods are characterized by larger volumes and faster production, continuous process operations, automation and standardized product quality. This makes new demands of the accompanying and supervisory analysis processes, such as e.g. active occupational safety, speed, integration into information systems, shifting of the measuring work from the analytical laboratory to the production location, robust apparatus (“glove-compatible”) and simple handling by sometimes only semi-skilled staff. The aspect of costs naturally also plays a key role in the time needed for a measuring device to pay for itself. That is why indirect measuring methods are often used that generate spectras or signals which then require product-specific calibrations, generating personnel costs over several months. With the ORACLE, CEM as a specialist in QA and process analytics has developed a fat analyzer for universal

applications, examining a very wide range of samples. Here it is not necessary to carry out any extensive, product-specific calibrations for a vast range of different formulations. This latest NMR technology measures all sample types directly in the unit without any prior calibration. The ORACLE is ready for routine work directly after installation! There has never before been a comparable system in the world that analyses the fat content of the above types of food samples so quickly and precisely in such a versatile, calibration-free, solvent-free manner.

How does the work proceed inside the ORACLE?

The work process consists of just three steps:

1. Drying of the sample in the Smart 6 microwave dryer in order to expel all the water within 2 – 3 minutes.
2. Transfer of the dried sample to the NMR nuclear resonance spectrometer (Trac-Module), (Fig. 2).
3. Analysis of the fat is then carried out in the ORACLE module within 30 seconds.

Fig. 2: Transfer of the dried sample to the NMR nuclear resonance spectrometer (Trac-Module)

The microwave dryer and the ORACLE module are operated completely via a touchscreen. The software has been designed in such a way that it provides menu-guided work instructions by analogy with the use of smartphones. The software evaluates complicated spectra such as e.g. the fat signals directly and the result is displayed for the user. A PC has been integrated into the ORACLE system in such a way that semi-skilled personnel can also carry out the analyses independently without any long training, in other words after at most 15 minutes!

How does the combination of the moisture and solids analysis with subsequent fat analysis function?

Fat analysis in foods by means of NMR (nuclear magnetic resonance) spectroscopy is a reliable technology that

- can be used universally for a large number of samples,
- has long since become established for dry samples,
- works without toxic solvents,
- does not require any product-specific calibration,
- provides results very quickly in less than in a minute, and
- is very easy to operate.

The use of NMR technology for measuring fats is not new and has already been used for many decades on dry samples such as nuts, chocolate or cereals. However, early fat analysis trials on very moist products such as meat and sausage products, dairy products (ice cream, quark, yoghurt, cheese …), delicatessen products, ketchup, mayonnaise and many others failed. The reason for these failures was the disturbing influence of the water on the fat signal. Consequently, prior to determining the fat content the water must be expelled from the sample. However, as drying operations in the drying cabinet last for several hours, this idea was doomed to failure from the start. Accordingly, for CEM as pioneer and manufacturer of the microwave dryer, it was a logical consequence to install the world's fastest dryer, the Smart 6, up front for fast drying of samples within 2 minutes of the fat analysis operation. Microwave drying as the fastest direct drying method is fast enough for the process monitoring and can be applied for a wide range of products and varieties directly at the point of production without any calibration input.

As a microwave-moisture/solids analysis system, the Smart 6 has been in use in a range of production branches for decades now. The sample product is placed on a special sample carrier material (glass fibre carrier) and set down on the scales installed in the microwave unit. Here the water molecules of the sample are heated in the set microwave field and expelled, without the sample surface becoming crusty and thus preventing further expulsion of water. Controlled warming of the sample material is carried out via the integrated temperature sensor, so that the risk of sample decomposition (e.g. caramelisation of carbohydrates) is minimized. To determine the moisture content exactly it is necessary to develop the specific microwave field uniformly and to regulate it continuously. During the drying process the integrated analysis scales record the sample weight continuously and ensure that the unit is switched off when a constant weight is achieved – often after just two minutes of measuring. Thanks to its speed and precision (precision of + 0.1 % dry matter), this method is particularly suitable for at-line process monitoring of substances with high water contents (up to 99.9 %).

The sample dried precisely in this way is now transferred to the ORACLE module, the NMR spectrometer. The fat molecules give off a characteristic signal which the ORACLE software converts directly into the fat content level and displays this to the user. This fat analysis is not falsified by accompanying substances such as sugar, salt, flavoring substances, flavor enhancers, emulsifiers, conservation agents … etc. Nor do any differences in color between the samples exert any interfering influence! Consequently this method can be applied universally.

In this connection the accredited laboratory Actalia Cecalait [1] examined the ORACLE in an international evaluation study with a wide range of different sample types. All the sample types

were analyzed in the ORACLE for their water and fat content and the results were compared with reference contents based on reference methods. The results of unknown samples are thus comparable with the results of the standard methods. A number of different dairy/milk sample types were examined in the study – cream, powdered milk, various cheese types, sour cream, yoghurt, desserts and ice cream, in a bandwidth of 0.5 to 45.0 % fat.

Tables 1 and 2 as well as Figure 3 show the comparability of the ORACLE fat results with the standard reference methods for the different foods.

Samples	ORACLE 1	ORACLE 2	ORACLE mean	Reference results mean	Difference	Residue
Cream 1	44.34	44.32	44.33	44.19	0.14	0.09
Cream 2	21.88	21.86	21.87	21.87	0.00	-0.02
Cream 3	36.65	36.58	36.62	36.71	-0.10	-0.14
Cream 4	28.90	28.86	28.88	28.83	0.05	0.02
Dried milk 1	25.98	25.84	25.91	26.05	-0.14	-0.17
Dried milk 2	26.09	26.06	26.08	26.14	-0.07	-0.09
Dried milk 3	13.83	13.84	13.84	13.98	-0.15	-0.15
Dried milk 4	0.41	0.43	0.42	0.62	-0.20	-0.19
Processed cheese 1	28.39	28.31	28.35	28.22	0.13	0.10
Processed cheese 2	29.42	29.39	29.41	29.30	0.11	0.07
Processed cheese 3	22.63	22.66	22.65	22.48	0.16	0.14
Processed cheese 4	8.54	8.56	8.55	8.56	-0.01	-0.01
Hard cheese 1	34.77	34.60	34.69	34.90	-0.21	-0.25
Hard cheese 2	26.50	26.33	26.42	26.25	0.16	0.14
"Fromage Frais" 1	2.26	2.30	2.28	2.21	0.07	0.08
"Fromage Frais" 2	7.13	7.20	7.17	7.07	0.09	0.09
Soft cheese 1	29.64	29.69	29.67	29.62	0.04	0.01
Soft cheese 2	11.31	11.30	11.31	11.25	0.05	0.05
Sour cream 1	13.86	13.93	13.90	13.85	0.045	0.04
Sour cream 2	29.45	29.49	29.47	29.22	0.250	0.22
Yogurt 1	8.9	8.91	8.91	8.77	0.135	0.13
Yogurt 2	3.31	3.26	3.29	3.30	-0.015	-0.01
Yogurt 3	1.01	1.06	1.04	0.94	0.095	0.10
Yogurt 4	1.27	1.38	1.33	1.32	0.005	0.01
Dessert 1	6.68	6.71	6.70	6.67	0.025	0.02
Dessert 2	3.02	3.01	3.02	3.00	0.015	0.02
Dessert 3	5.19	5.22	5.21	5.37	-0.165	-0.16
Dessert 4	6.8	6.78	6.79	6.86	-0.070	-0.07
Ice cream 1	9.21	9.15	9.18	9.07	0.110	0.11
Ice cream 2	17.18	17.21	17.20	17.36	-0.165	-0.18

Samples highlighted in blue correspond to Cecalait's CRM.

Tab. 1: Comparability of the ORACLE fat results with those of the standard reference methods for different foods

g/100g	Cream	Sour cream	Yogurt	Cheese	Processed cheese	Dried milk	Ice cream	Milk dessert	All Samples
n	4	2	4	6	4	4	2	4	30
min	21.87	13.90	1.04	2.28	8.55	0.42	9.18	3.02	0.42
max	44.33	29.47	8.91	34.69	29.41	26.08	17.20	6.79	44.33
Y	32.90	21.54	3.58	18.55	22.14	16.70	13.18	5.48	16.80
S_y	9.66	10.87	3.61	13.43	9.54	12.14	5.81	1.78	12.53
d	0.02	0.15	0.06	0.04	0.10	-0.14	0.01	-0.05	0.02
S_d	0.10	0.14	0.07	0.13	0.08	0.06	0.14	0.09	0.12
$S_{y,x}$									0.122
$S_{y,x\,\%}$									0.72
Slope									0.999
Bias									0.009

Tab. 2: ORACLE accuracy criteria in all samples

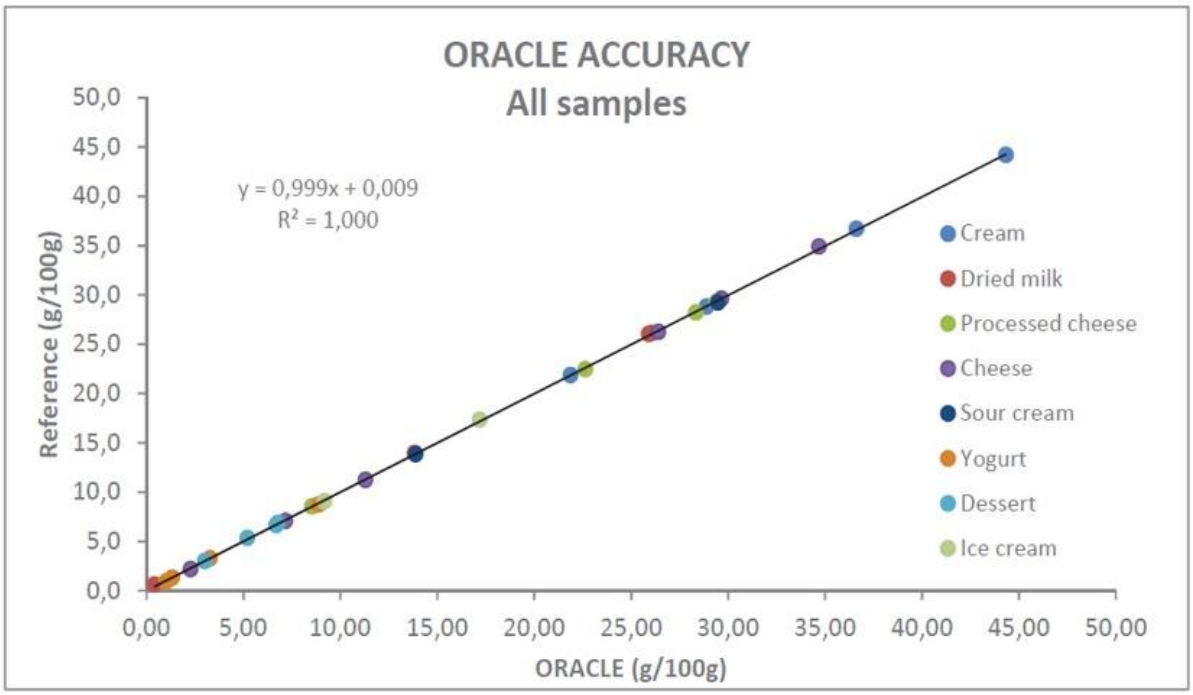

Fig. 3: Relationship between ORACLE and reference results in all samples

Actalia ascertained that the ORACLE fat analyzer was able to analyses all the above dairy samples with the same accuracy as and better precision than the wet-chemical extraction technologies according to Röse-Gottlieb, Weibull-Berntrop and Schmidt-Bonzynski-Ratzlaff. The measurements take only a few minutes and do not require any method development or calibration. In particular the comparison between ORACLE and wet chemistry results produced a perfect linear determination coefficient (R2) of 1,000. Actalia also concluded that the repeatability of the ORACLE was better for all samples than the reference chemistry. Actalia, based in Poligny, France, is a COFRAC-accredited laboratory with expertise in dairy analysis specializing in the provision of technical and scientific findings for validating and unifying analysis methods. Furthermore, Actalia both organizes ring trials and serves as global supplier of SRMs (secondary reference materials) for dairy products.

Can the moisture and fat analysis also be carried out for “dry” samples such as e.g.?

- animal feed,
- feedstuffs,
- snacks,
- crackers and biscuits,
- bakery products and baking ingredients,
- cereals, grain and muesli,
- chocolate,
- sweets,
- raw cocoa and cocoa beans,
- nuts and marzipan,
- whole milk powder,
- starch and baby food,
- egg yolk powder,
- oils and fat etc.

The answer to this is quite simple – there is no need for drying in the Smart 6 microwave dryer. The fat content level of these dry samples can be examined directly in the ORACLE.

Summary and Prospects

In many branches of industry the scope of process monitoring tasks has changed distinctly in recent years, not least due also to changes in legislation. Now there is greater demand for analysis systems that can be used on site or directly in the production process (at-line). In particular in this field of applications a special degree of safety and operating comfort must also be taken into account. The ORACLE analysis system is ideally suited for this, especially for such moisture and fat content determination. No skilled staff is needed to operate the unit. Fat analysis is carried out quickly, independently of a matrix, and the unit is ready for operation immediately after installation. By contrast with other measuring methods, no comprehensive, product-specific calibrations need to be carried out over months. As regards safety at work, the ORACLE sets a new standard by doing without acids or solvents. A maximum of precision has been checked and confirmed by many users. Accordingly, food formulations can be integrated directly at the limit level, resulting in correspondingly higher earnings.

Literature

[1] Actalia Cecalait ORACLE Evaluation report, A. Qudotte, M. Esteves, JR Bondier, P. Trossat, December 2017

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Further information: https://www.youtube.com/watch?v=9_RpF8LwB8k