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EXTRACT

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EVALUATION OF THE ORACLE INSTRUMENT

ACTALIA Cecalait was asked to carry out an evaluation of the performance of the fat analyzer ORACLE on dairy products. This instrument, produced by CEM, is based on NMR technology with no method development. It uses a new NMR technology that completely isolates detection of protons on fat molecules from all other NMR signals.

The instrument used in this study was:

Type of instrument: ORACLE MAGNET
Model no: 904800
Serial no: OM3004
Normalization standard (PN: 523020 - SN: NS60007)
Oil standard (P/N 160840)
High precision heater block (44.20 °C ± 0.15 °C)

The instrument was installed in a temperature controlled room (20-23°C – air-conditioning), without direct sunlight. The installation procedure was performed by CEM. Pads, samples and film were necessary for this evaluation.

THE TESTS

Short term reproducibility, repeatability and accuracy were evaluated. Short term reproducibility was performed on 3 cheese samples with different fat levels. Repeatability and accuracy of the instrument were evaluated on 30 samples of dairy products (4 cream samples, 2 sour cream samples, 4 yogurt samples, 6 cheese samples, 4 processed cheese samples, 4 dried milk samples, 2 ice cream samples, 2 milk dessert samples and 2 chocolate mousse samples).
Some samples were Certified Reference Materials (CRM's) sourced from ACTALIA Cecalait, the other samples were bought in a supermarket.

The following standards for reference methods were used for the evaluation of the accuracy of the instrument:

-Fat content in cream	Rose-Gottlieb method according ISO 2450 | IDF 16
-Fat content in sour cream	Weibull-Berntrop method according ISO 8262-3 | IDF 124-3
-Fat content in yogurt	Weibull-Berntrop method according ISO 8262-3 | IDF 124-3
-Fat content in cheese and processed cheese	Schmid-Bondzynski-Ratzlaff method according ISO 1735 | IDF 5
-Fat content in dried milk	Rose-Gottlieb method according ISO 1736 | IDF 9
-Fat content in ice cream	Rose-Gottlieb method according ISO 7328 | IDF 116
-Fat content in milk dessert	Weibull-Berntrop method according ISO 8262-3 | IDF 124-3

The determination of the fat content of cheeses and processed cheeses obtained from the ISO 1735 method are globally equivalent to those which could be obtained from the AOAC 933.05 method. Indeed, we can observe only few technical differences between the two methods: the sample size (1g vs 3g) and the drying temperature (99-101°C vs 102°C±2°C). These differences are not likely to impact significantly the result.
A Rose-Gottlieb principle has been tested on sour cream, yogurt and dessert samples, but unfortunately, a gelification (inside the tube) occurred during the analytical process (likely linked to matrix effect: sugars content and pH of the product) and prevented to obtain results.
Note: The scope of ISO Rose-Gottlieb standardized method doesn't include these products

For the fat determination with the instrument, samples were pre-dried in sample holders overnight (4 hours for yogurt and milk dessert) in oven with film and pads. Then, they were conditioned in the high precision heater block at the magnet temperature for 1 hour. Samples were transferred in to the ORACLE and analyzed within 35 seconds.
Before every set, a reference sample (milk) was analyzed to ensure results.
Long term stabilities were performed before analysis.

EVALUATION OF SHORT TERM REPRODUCIBILITY

The short term reproducibility was evaluated by analyzing 3 cheeses, with different fat contents, in duplicate, every 15 minutes to obtain at least 15 sequences. To evaluate the stability of the instrument, the repeatability and reproducibility were calculated by level.

The following tables present the obtained results:

	Level 1	Level 2	Level 3
Fat (g/100g)	40	21	5

Table 1: Fat content of samples used in short term reproducibility evaluation

(g/100g)	Level 1	Level 2	Level 3
M	42.419	21.371	6.322
Sr	0.090	0.052	0.014
Sr (%)	0.21 %	0.24 %	0.21 %
SR	0.115	0.064	0.016
SR (%)	0.27 %	0.30 %	0.25 %
r	0.253	0.146	0.038
R	0.323	0.180	0.044

Table 2: ORACLE stability[1]

The results indicate that the relative reproducibility (SR %) is in the same scale for the 3 levels. Furthermore, the reproducibility of the instrument is lower than the reproducibility of the reference method (R= 0.40 g/100g).

EVALUATION OF THE ACCURACY

The accuracy of the instrument were evaluated on cream, sour cream, yogurt, cheese, processed cheese, dried milk, ice cream and milk dessert

The following table and figure present the results obtained:

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**

Table 3: ORACLE accuracy criteria in all samples[2]

[1] M: mean; Sr and SR (Sr% and SR%): absolute (and relative) standard deviations of repeatability and reproducibility; r and R: maximum deviation of repeatability and reproducibility in 95 % of cases.

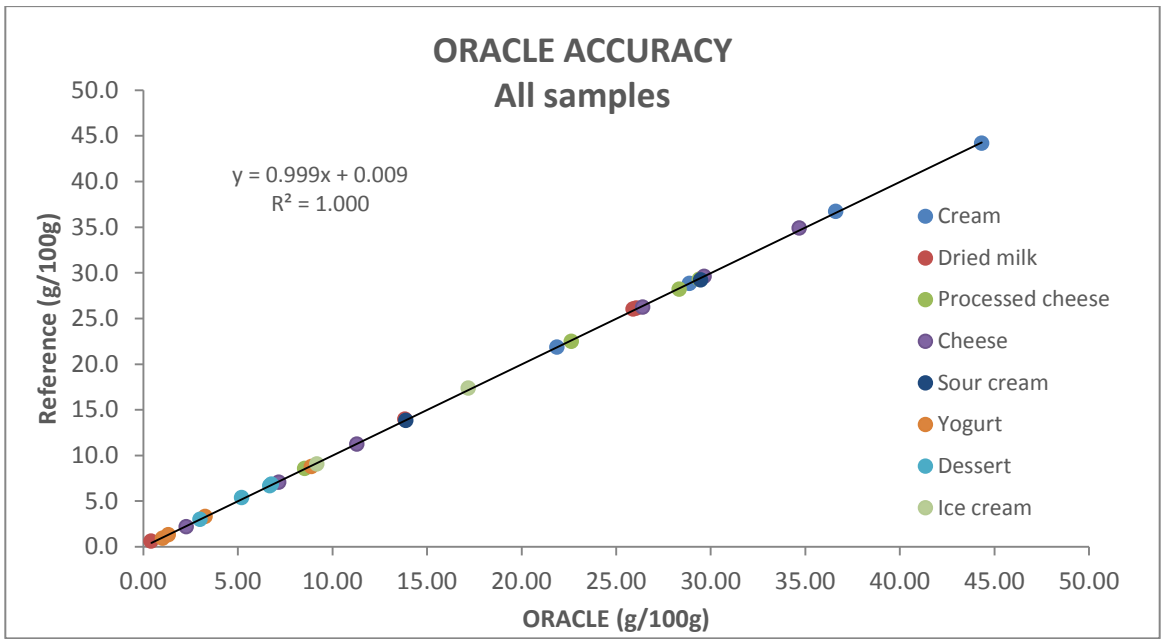

Figure 1 : Relation between ORACLE and reference results in all samples

It can be noted that the mean and the standard deviation of deviations are respectively equal to 0.02 and 0.12 g/100g. The regression slope (0.999) and the intercept (0.009) are not significantly different, respectively from 1.00 and zero (P=5%).

GENERAL CONCLUSION

The ORACLE system is easy to use. Only simple tests have to be performed to check the device operability (as long-term stability for example).

In the absence of standardized limits of repeatability and accuracy for such NMR instrument, we can conclude as follows:

- The ORACLE instrument presents a good performance of repeatability for all the products and below reference method limits.
- For accuracy, we can observe a very good performance (slope and bias closed respectively to 1.00 and 0.00, and mean difference of +0.02 g/100 g). The samples have been analysed on the ORACLE system using the same parameters, bringing to the conclusion, the instrument is very robust.

This study has been focused on fat determination by ORACLE system, but also available from CEM the linkage of the dry matter analyser (SMART 6) and the fat analyser (ORACLE) in order to skip the pre-drying process overnight (or 4 h for yogurt and dessert) and the conditioning step (1 h in the heating block). The total test time for measurement of both dry matter and fat content is announced to be <5 minutes.

[2] n, min, max: number of results, minimum and maximum values; Y: mean results using the reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x, Sy,x%: absolute and relative residual standard deviation.