EVALUATION OF THE FT-NIR DAIRYQUANT B4 ANALYSER

The DairyQuant B4 is a near infrared spectrometer (range 3700-14885 cm^{-1}) manufactured by Q-Interline (Denmark) and commercialised in France by Inforlab-Chimie. It is used for the determination of composition components in solid dairy products (butter, cheese…).

This instrument uses a high resolution industrial infrared spectrometer based on Fourier transform (IRTF) incorporating a patented double pivot Michelson interferometer with mirrors. This construction ensures the stability of the optical alignment. A resolution of 32 cm^{-1} is used for the butter application.

Various parameters (fat, protein, dry matter or moisture, chloride…) can be determined with a PLS calibration.
The apparatus is computer controlled with InfraQuant software, which ensures the signal treatment for the routine analyses. A second software (Horizon QI) for the development of calibrations can be supplied.

The tests:

The evaluation tests, realised in reusable Teflon caps, were performed in ACTALIA Cecalait physico-chemistry laboratory (reference and infrared analyses) in September and October 2013. The repeatability and the accuracy were evaluated for moisture and salt in unsalted and salted butter.

The calculation were performed according to ISO 21543/IDF 201 : 2006 standard.

1- EVALUATION OF THE REPEATABILITY

1.1- Samples

The tests were performed on 15 samples of unsalted butter and 30 samples of salted butter from supermarket. Then, butter come from many different sites of production.

1.2- Procedure

The repeatability of the instrument is evaluated using 15 samples of unsalted and salted butter for moisture and 30 samples of salted butter for salt (NaCl). The quantitative analyses were performed in two sets (unsalted butter and salted butter) in consecutive duplicate for each sample. A control milk was analysed before each set to verify the stability of the analyser.

1.3- Results

The following table present the results obtained:

PRODUCT	PARAMETER	n	min	max	M	Sx	Sr	Sr (%)	r
UNSALTED BUTTER	**Moisture (g/100g)**	15 (13)	13.18 (15.35)	16.31 (16.31)	15.562 (15.843)	0.821 (0.303)	0.067 (0.048)	0.43 (0.31)	0.185 (0.134)
SALTED BUTTER	**Moisture (g/100g)**	15 (13)	12.98 (15.12)	16.17 (16.17)	15.354 (15.684)	0.913 (0.278)	0.142 (0.099)	0.93 (0.63)	0.394 (0.273)
	NaCl (g/100g)	30 (29)	1.45 (1.45)	2.51 (2.51)	2.053 (2.047)	0.236 (0.237)	0.046 (0.040)	2.26 (1.94)	0.129 (0.110)

Table 1: Quant repeatability criteria for moisture and NaCl in butter samples

n, min, max: number of results, minimum and maximum value; M and Sx: mean and standard deviation of results; Sr and Sr%: absolute and relative standard deviation of repeatability; r: maximum deviation of repeatability (95% of cases)

The values in brackets correspond to values recalculated after elimination (deviations between duplicate higher than 3Sr and values out of calibration range 15-16.5% for moisture).

1.4- Conclusion

Despite the absence of standard criteria, it can be noted that the relative standard deviations of repeatability vary between 0.31% and 1.94% according to the parameter measured. For moisture, the performances are better for unsalted butter than for salted butter (0.31% against 0.63%).

If we compare to the reference methods used to evaluate the accuracy, the Sr values obtained (0.048 g/100 g and 0.099 g/100 g) are higher (moisture ISO 3727-1, Sr limit equal to 0.036 g/100 g).

2- EVALUATION OF THE ACCURACY

2.1- Procedure

The accuracy of the instrument was evaluated according to the evaluation of the repeatability. The instrumental values were carried out by a calibration of the manufacturer. The following reference methods were used :

- Moisture: drying method according to ISO 3727-1/IDF 80-1: 2001 (test in duplicate for unsalted butter and single test for salted butter),

Salt: chloruremeter method (single test).

The results obtained for moisture in unsalted butter samples by the reference method correspond to the mean of two replicates performed in repeatability condition.
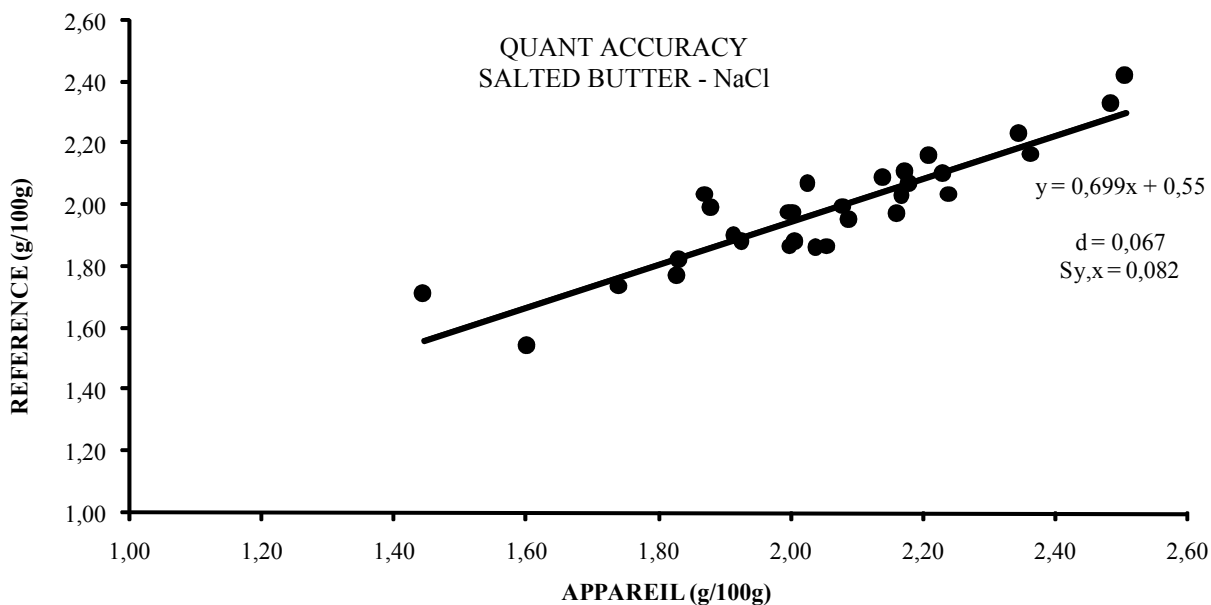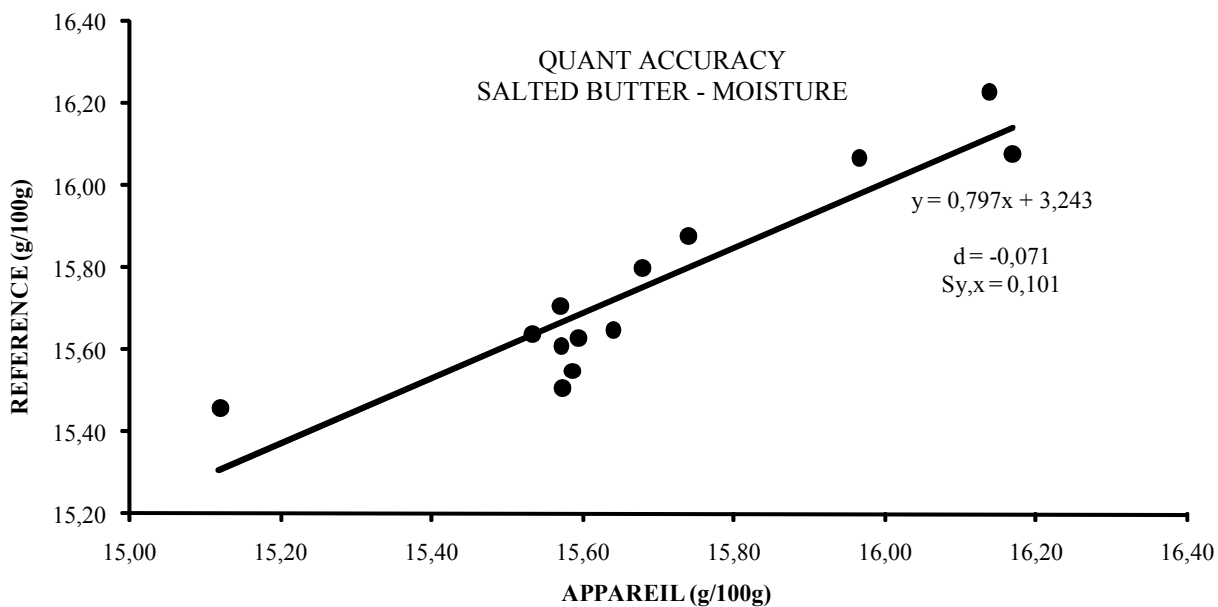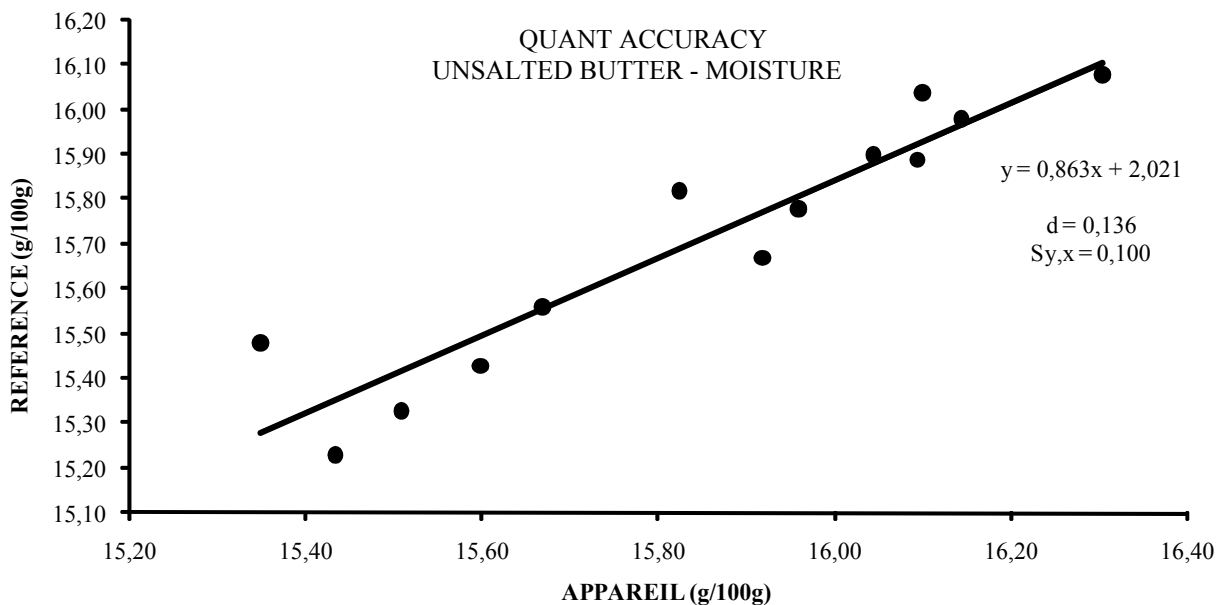
2.2- Results

The following table and figures present the results obtained:

		n	min	Max	Y	Sy	d	Sd	Sy,x	Sy,x (%)	RMSEP
UNSALTED BUTTER	**Moisture (g/100g)**	15 (13)	14.18 (15.23)	16.08 (16.08)	15.587 (15.707)	0.472 (0.279)	-0.026 (0.136)	0.439 (0.104)	0.204 (0.100)	1.31 (0.63)	0.425 (0.169)
SALTED BUTTER	**Moisture (g/100g)**	15 (13)	14.46 (15.46)	16.23 (16.23)	15.595 (15.755)	0.481 (0.242)	-0.241 (-0.071)	0.462 (0.111)	0.122 (0.101)	0.80 (0.64)	0.507 (0.129)
	NaCl (g/100g)	30	1.54	2.42	1.986	0.183	0.067	0.107	0.082	4.00	0.125

Table 2: Quant accuracy criteria for moisture and NaCl in butter samples

n, min, max: number of results, minimum and maximum value; Y: mean results using reference method; Sy: standard deviation of the results from the reference method; d, Sd: mean and standard deviation of deviations; Sy,x, Sy,x (%): residual and relative standard deviation; RMSEP: prediction error.

The values in brackets correspond to values recalculated after elimination of the abnormal values according to Grubbs 5%, and the out of calibration range results (15-16.5% for moisture).

QUANT ACCURACY
UNSALTED BUTTER - MOISTURE

$y = 0,863x + 2,021$

$d = 0,136$
$Sy,x = 0,100$

REFERENCE (g/100g)

APPAREIL (g/100g)

QUANT ACCURACY
SALTED BUTTER - MOISTURE

$y = 0,797x + 3,243$

$d = -0,071$
$Sy,x = 0,101$

REFERENCE (g/100g)

APPAREIL (g/100g)

QUANT ACCURACY
SALTED BUTTER - NaCl

$y = 0,699x + 0,55$

$d = 0,067$
$Sy,x = 0,082$

REFERENCE (g/100g)

APPAREIL (g/100g)

Figures 1,2 and 3: Relation between the reference and instrumental results for moisture and NaCl in butter samples

It can be noted that:

- the RMSEP values for moisture in unsalted and salted butter are respectively equal to 0.169 g/100 g and 0.129 g/100 g. The linear regression slopes (respectively 0.897 and 0.763) are not different from 1 (P = 5%).

- the RMSEP value for salt is equal to 0.125 g/100 g. The slope (0.699) of the linear regression is significantly different from 1 (P = 1%).

2.3- Conclusion

Despite the absence of standard criteria, the performances observed enable RMSEP predictions respectively equal to 0.169 g/100 g and 0.129 g/100 g (1.1% and 0.8%) for moisture in unsalted and salted butter, and 0.,125 g/100 g (6,3%) for salt. The RMSEP values observed are close to the manufacturer specifications (RMSECV limit: 0.1 g/100 g for moisture and 0.05 g/100 g for NaCl). The residual standard deviations of linear regression obtained are in accordance (moisture) or nearest (salt) of these specifications.

CONCLUSION

Because the absence of standard criteria, it is difficult to interpret the results obtained. As the evaluation was performed using manufacturer calibrations optimised for samples from a unique production entity, the RMSEP performances observed for each parameter measured are considered as maximum.
Consequently, according to the regression parameters obtained, the performances can be improved by a specific adjustment of the calibrations (slope and intercept according to the final equation Y = b.X + a) in local samples or by development of calibrations specific to the site.

According to the evaluation report of the FT-NIR DairyQuant B4 analyser – X. QUERVEL and Ph. TROSSAT – October 2013