ANALYZER EVALUATION REPORT

CDR FoodLab®

Figure 1 : CDR FoodLab®

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1. INTRODUCTION

1.1- OBJECTIVES

CDR asked ACTALIA Cecalait to evaluate the performance of the CDR FoodLab® device relating to milk and dairy products. The following parameters and matrix were evaluated:

- Urea in milk
- Lactose in lactose free milk (measurement range 0,01 to 2 g/100g)
- Ammonia in whey

1.2- THE DEVICE

CDR FoodLab® is a versatile photometric analyzer for the determination of a large range of chemical criteria in food products. The device is equipped with LED sources, reading cells and incubation cells thermostatically controlled at 37°C, allowing for 16 determinations to be made in parallel.

The device used for this study had the following characteristics:

CDR FoodLab®
Type : SLB 222
Serial number: B-222003/1112
Year of production: 2019

The unit was installed in a temperature-controlled room (20-23°C – air conditioning), without direct sunlight. The installation procedure was performed by CDR.

1.3- CONSUMABLES

The reagents used in this study were as follows:

- Reagent Kit 300010 for lactose
- Reagent Kit 300054 for ammonia
- Reagent Kit 300004 for urea

Reagents are ready to use and packaged in bags of 10 tests. They have a shelf life of one year.

1.4- TESTS

The evaluation tests were carried out at ACTALIA Cecalait physics and chemistry laboratory in July 2019 : determinations on the CRD FoodLab® analyzer, determinations according to reference method NF EN ISO 14637 for urea and according to NF V 04-217 method for ammonia.

Reference analysis by the HPLC method for lactose were carried out at ACTALIA Control and Quality site of Villers Bocage.

The evaluation tests covered the following points:

- Stability of the device for each parameter
- Method repeatability and accuracy

The raw absorbance data from the CDR FoodLab® were used for this evaluation.

The absorbance was then converted into rates using the reference values obtained within the scope of the accuracy study for each parameter.

Therefore, the accuracy of each parameter can only be assessed on the basis of the residual regression standard deviation Sy,x and of estimation accuracy ± 2.Sy,x. Indeed, because of this approach, the accuracy regression equation obtained on the basis of rates leads to a regression slope of 1.00 and a zero ordinate at the origin.

2. DETERMINATION OF LACTOSE CONTENT IN MILK

2.1- Device stability assessment

Stability was achieved by analyzing 2 milk samples with different lactose contents (table 1). The milks were made from a mixture of 2 UHT drinking milks: milk with a reduced lactose content of 2,7 % and delactosed milk < 0,1 g/100ml, with the addition of a preservative (bronopol 0.02%). The analysis were duplicated every 15 minutes in order to obtain at least 10 measurement cycles.

Table 1 : Lactose level in samples used for stability assessment

	Level 1	Level 2
Lactose (g/100g)	0,80	1,50

The table below shows the results obtained:

Table 2 : CRD FoodLab® lactose stability[1]

	Lactose (Abs)		Lactose (g/100g)	
	Level 1	Level 2	Level 1	Level 2
M	0,6180	1,0712	0,825	1,533
Sr	0,020	0,022	0,032	0,035
Sr (%)	3,29	2,07	3,85	2,27
SR	0,020	0,028	0,031	0,043
SR (%)	3,19	2,60	3,73	2,83
r	0,056	0,062	0,088	0,096
R	0,055	0,077	0,085	0,120

Repeatability standard deviations in the range of 2.3 to 3.9 % and reproducibility standard deviations of approximately 2.8 to 3.7 % can be observed depending on the sample rates.

With regards to standard deviation of reproducibility observed, in the absence of requirements, it can be noted that the latter are very close to the observed repeatability deviations reflecting a generally low « instrumental stability » error.

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

2.2- Device repeatability assessment

Device repeatability was achieved by duplicate analysis of 26 milk samples of 2 UHT milks (see 2.1) with added preservative (bronopol 0,02%). Samples had a lactose content between 0.01 and 2 g/100g.

The results obtained are presented in the following table:

Table 3 : CDR FoodLab® lactose repeatability[2]

	n	min	max	M	Sx	Sr	Sr (%)	r
Lactose (Abs)	26	0,1985	1,1332	0,5354	0,254	0,011	2,03	0,030
Lactose (g/100g)		0,169	1,630	0,696	0,024	0,017	2,44	0,047

Over the measurement range of 0.16 to 1.63 g/100, a repeatability standard deviation of 0.017 g/100g can be observed.

There is no standard method specific to lactose free milk, but it can be compared to existing standard methods for the determination of lactose in milk, i.e. Sr = 0,022 g/100g (Sr% = 0.44) for the HPLC method according to ISO 22662 and Sr = 0.037 g/100g (Sr% = 0.74) for differential pH-metric method according to ISO 26462 standard.

2.3- Device accuracy assessment

The assessment of accuracy against the HPLC method was performed with the same milk samples as those used for the repeatability assessment (see 2.2).

The accuracy was assessed on 26 samples after elimination of samples with outliers (elimination of samples based on regression residues greater than 2 x standard deviation of regression residue deviations: 5%threshold).

The results obtained are presented in the table below:

Table 4 : CDR FoodLab® lactose accuracy criteria[3]

	CDR FoodLab® Lactose (g/100g)
n	26
min	0,179
max	1,621
Y	0,692
X	0,692
Sy	0,405
Sx	0,408
Sd	0,044
Sy,x	0,045

[2] N : number of results ; min and max : minimum and maximum values; M : result average ; Sr (Sr%) : absolute (and relative) standard deviation of repeatability ; r : maximum deviation of repeatability in 95 % of cases.

[3] n, min, max : number of results, minimum and maximum values; Y,X : average of results by reference and instrumental method ; Sy, Sx :standard deviation of results by reference and instrumental method; Sd : standard deviation of deviations ; Sy,x : residual standard deviation.

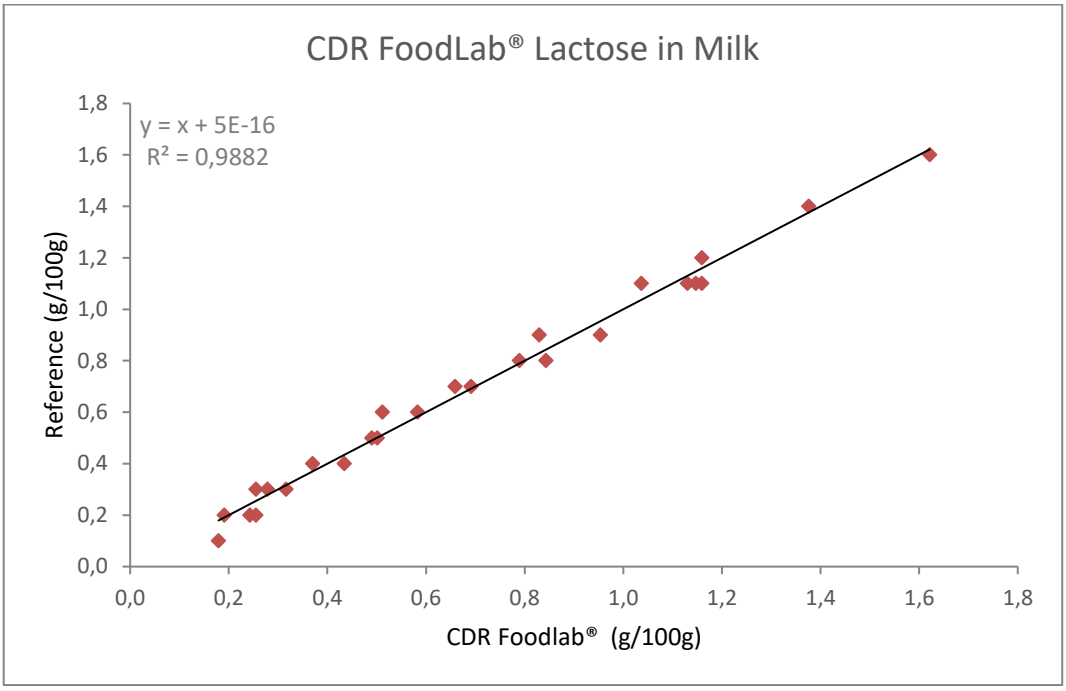

Figure 2 : Relationship between instrumental results and references in g/100g of lactose

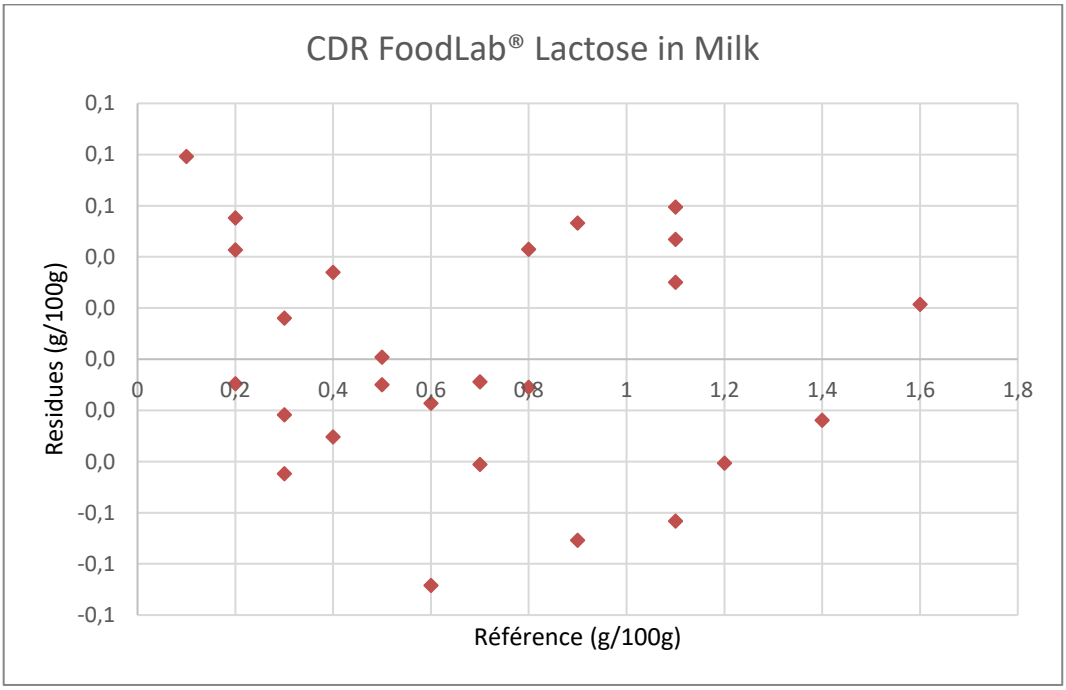

Figure 3 : Residues at regression according to reference values in mg/100g of lactose

With regards to the relationship between the results of CDR FoodLab® method (calculated from the regression equation) and the HPLC reference method, the following can be observed:

- A residual regression standard deviation Sy,x of 0.045 g/100g is observed, resulting in an estimation accuracy of ± 0.09 g/100g.

3. DETERMINATION OF UREA CONTENT IN MILK

3.1- Device stability assessment

Stability was achieved by analyzing 3 samples of whole milk with the addition of a preservative (bronopol 0,02%), with different urea contents, twice every 15 minutes in order to obtain at least 10 measurement cycles. The samples used were ACTALIA Cecalait ETG Urea.

Table 5 : Urea level in samples used for stability assessment

	Level 1	Level 2	Level 3
Urea (mg/l)	180	500	800

In order to assess the stability of the device, repeatability and reproducibility were calculated for each level.

Le table below shows the results obtained:

Table 6 : CRD FoodLab® urea stability[4]

	Urea (Abs)			Urea (mg/l)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
M	0,3979	0,9472	1,4942	181,69	500,95	818,80
Sr	0,011	0,016	0,036	6,204	9,163	20,74
Sr (%)	2,68	1,66	2,39	3,41	1,83	2,53
SR	0,012	0,016	0,033	7,028	9,561	19,057
SR (%)	3,04	1,74	2,19	3,87	1,91	2,33
r	0,030	0,044	0,099	17,186	25,382	57,439
R	0,034	0,046	0,091	19,468	26,485	52,787

Repeatability standard deviation of 1.8 to 3.4 % and reproducibility standard deviations of 1.9 to 3.9 % can be observed depending on the samples rates.

With regards to the standard deviation of reproducibility observed, in the absence of requirements, it can be noted that the latter are close to the observed repeatability deviations reflecting a general low « instrumental stability » error.

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

3.2- Device repeatability assessment

The repeatability of the device was achieved by duplicate analysis of 34 samples with a urea content comprised between 170 and 800 mg/l. The samples were raw milks with the addition of a preservative (bronopol 0,02%): 5 ETG samples of ACTALIA Cecalait urea and 29 tank milks from the Franche-Comté region.

The results obtained are shown in the table below:

Table 7 : CRD FoodLab® urea repeatability[5]

	n	Min	Max	M	Sx	Sr	Sr (%)	r
Urea (Abs)	34	0,3711	1,5231	0,6251	0,223	0,012	1,95	0,034
Urea (mg/l)		166,15	835,58	313,78	129,29	7,072	2,25	19,59

The repeatability r obtained of 19.6 mg/l is slightly higher than that of the reference method NF EN ISO 14637 ($r \leq 15$ mg/l i.e. Sr 5.42 mg/l).

3.3- Device accuracy assessment

The assessment of accuracy in relation to the differential pH-metric method NF EN ISO 14637 was carried out with the same milk samples as those used for the repeatability assessment (see 3.2).

The accuracy was assessed on 34 samples after elimination of samples with outliers (elimination of samples based on regression residues greater than 2 x standard deviation of regression residue deviations: 5% threshold).

The results obtained are shown in the table below:

Table 8 : CDR FoodLab® urea accuracy criterion[6]

	CDR FoodLab® Urée (mg/l)
n	34
min	173,79
max	834,04
Y	313,78
X	313,78
Sy	130,52
Sx	131,26
Sd	13,944
Sy,x	14,160

[5] n : number of results ; min and max : minimum and maximum values; M : result average ; Sr (Sr%) : absolute (and relative) repeatability standard deviation ; r : repeatability maximum deviation in 95 % of cases.

[6] n, min, max : number of results, minimum and maximum values; Y,X : result average by reference and instrumental method ; Sy, Sx : result standard deviation by reference and instrumental methods ; Sd : standard deviation of deviations; Sy,x : residual standard deviation.

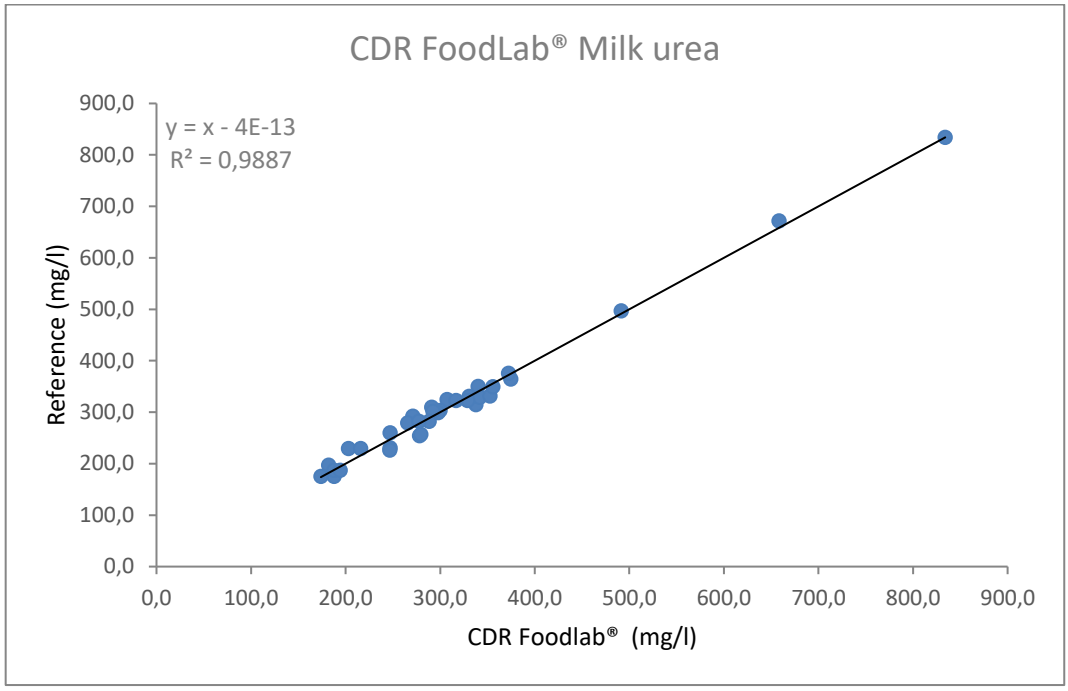

Figure 4 : Relationship between instrumental results and references in mg/l urea

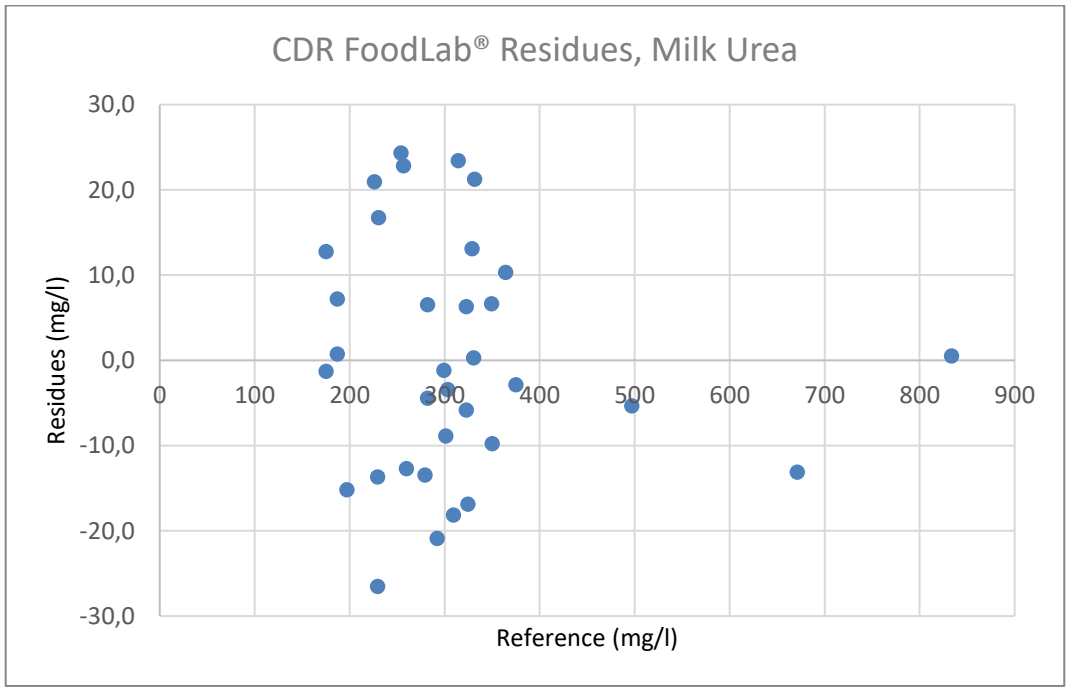

Figure 5 : Residues at regression according to reference values in mg/l of urea

With regards to the relationship between the results of CDR FoodLab® method (calculated from the regression equation) and the differential pH-metric reference method NF EN ISO 14637, the following can be observed :

- A residual standard deviation of regression Sy,x of 14.2 mg/l is observed, resulting in an estimation accuracy of ± 28 mg/l.

4. DETERMINATION OF LACTOSERUM AMMONIA CONTENT

4.1- Device stability assessment

Stability is achieved by analyzing 3 samples of whey with the addition of a preservative (bronopol 0,02%), and different ammonia contents, twice every 15 minutes in order to obtain at least 10 measurement cycles. The samples used are whey samples from the Franche-Comté region.

Table 9 : Ammonia level in samples used for stability assessment

	Level 1	Level 2	Level 3
Ammonia (ppm)	20	30	50

In order to assess device stability, repeatability and reproducibility were calculated for each level.

The table below shows the results obtained :

Table 10 : CDR FoodLab® ammonia stability[7]

	Ammonia (Abs)			Ammonia (ppm)		
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
M	0,3752	0,8086	1,8050	21,83	31,48	53,67
Sr	0,037	0,026	0,033	0,826	0,569	0,735
Sr (%)	9,88	3,16	1,83	3,78	1,81	1,37
SR	0,035	0,025	0,033	0,778	0,564	0,726
SR (%)	9,31	3,13	1,81	3,56	1,79	1,35
r	0,103	0,071	0,091	2,287	1,576	2,035
R	0,097	0,070	0,090	2,154	1,562	2,011

Repeatability standard deviation of 1.4 to 3.8 % and reproducibility standard deviations of 1.4 to 3.6 % according to samples rate.

It can be noted that the standard deviation of reproducibility observed is of the same order as the standard deviation of repeatability, indicating good instrumental stability.

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

4.2- Device repeatability assessment

The repeatability of the device was achieved by duplicate analysis of 33 whey samples containing between 12 and 113 ppm of ammonia. Samples were whey from the Franche-Comté region with added preservative (bronopol 0,02%).

The results obtained are presented in the table below:

Table 11 : CDR FoodLab® ammonia repeatability[8]

	n	Min	Max	M	Sx	Sr	Sr (%)	r
Ammonia (Abs)	33	0,2368	3,1301	0,8649	0,614	0,017	1,92	0,046
Ammonia (ppm)		12,93	112,66	34,58	0,808	0,572	1,66	1,585

Repeatability r obtained using the CDR FoodLab® is of 1.59 ppm compared to an average value of 2.46 ppm in the NF V 04 217 standard over the considered rate range.

4.3- Device accuracy adssessment

Accuracy assessment with respect to the enzymatic method NF V 04-217 was performed with the same whey sample as those used for the repeatability assessment (see 4.2).

Accuracy was assessed on 33 samples after elimination of samples with outliers (elimination of samples based on regression residues greater than 2 x standard deviation of regression residue deviations: 5% threshold).

The results obtained are presented in the table below:

Table 12 : CDR FoodLab® ammonia[9] accuracy criteria

	CDR FoodLab® Ammonia (ppm)
n	33
min	13,03
max	111,12
Y	34,58
X	34,58
Sy	21,38
Sx	22,21
Sd	6,020
Sy,x	6,116

[8] n : number of results ; min and max : minimum and maximum values; M : average of results ; Sr (Sr%) : absolute (and relative) of repeatability ; r : maximum deviation of repeatability in 95 % of cases.

[9] n, min, max : number of results, minimum and maximum value ; Y,X : average of the results by reference and instrumental method ; Sy, Sx : standard deviation of results by reference and instrumental method ; Sd : standard deviation; Sy,x : residual standard deviation

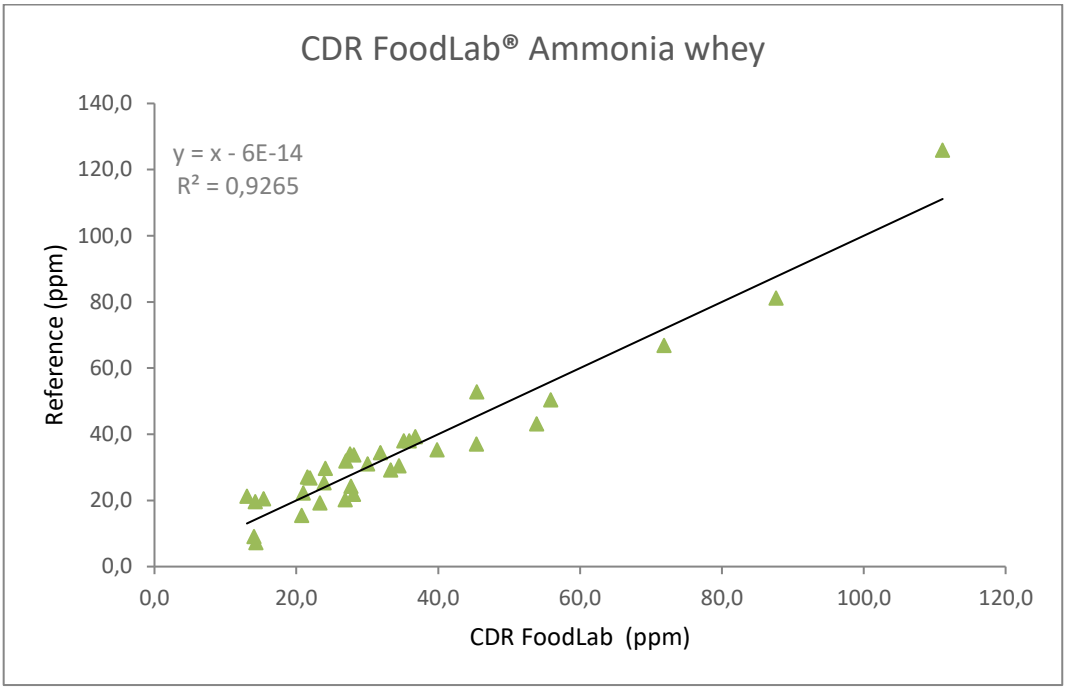

Figure 6 : Relationship between instrumental results and references in ppm of ammonia

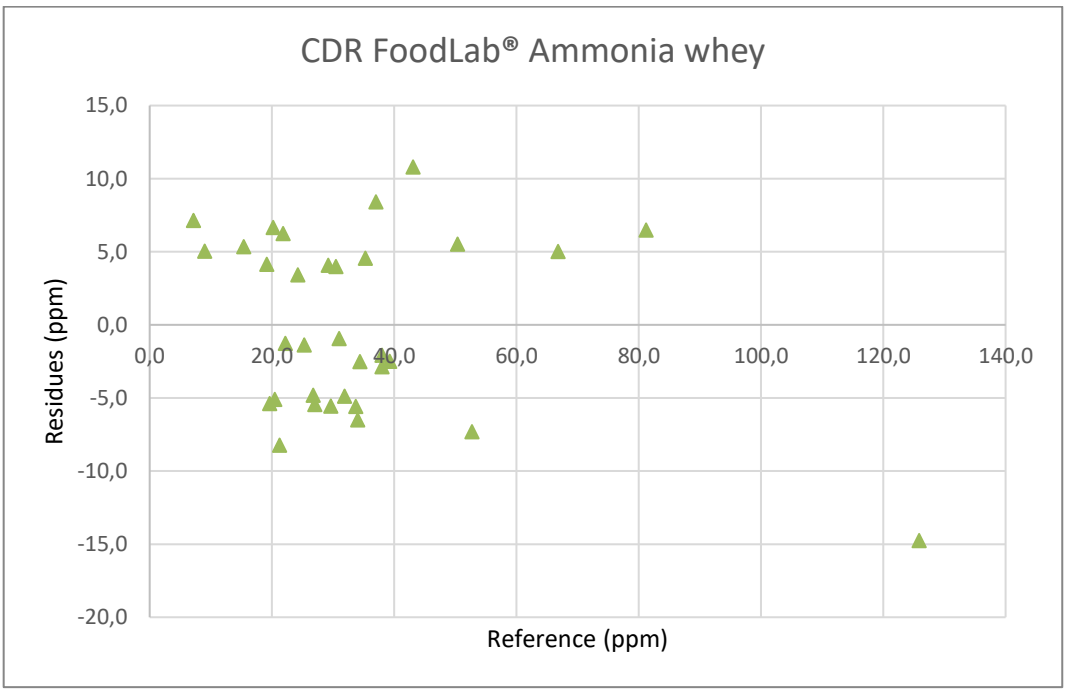

Figure 7 : Residues at regression according to reference values in ppm of ammonia

With regards to the relationship between the results of the CDR FoodLab® method (calculated from the regression equation) and the enzymatic reference method NF V 04-217, the following can be observed:

- A residual standard deviation of Sy,x regression of 4.2 ppm is observed, resulting in an estimation accuracy of about ± 8 ppm.

5. GENERAL CONCLUSION

At the end of the evaluation of milk's lactose (measurement range 0.01 – 2 g/100g), milk urea and whey ammonia models, we can conclude that:

- The CDR FoodLab® device is easy to use thanks to the operating procedures that are integrate with the methods.
- No recurrent problems were found during the tests with the CDR FoodLab® device. However, the importance of sample collection and the addition of reagents that are performed with a pipette must be noted. The pipette must be used with great precision so as not to introduce performance issues.
- In terms of instrument performance, we can note :
    - For the determination of lactose in lactose-free milk, a higher repeatability than standard methods (Sr% = 2.44 vs 0.44 for ISO 22662 method and 0.74 for ISO 26462 method) and a correctness allowing to obtain an estimation accuracy < 0.1 g/100 g (0.09g/100 g)
    - For the determination of urea in milk, repeatability in the same range as that of the reference method and an estimation accuracy of $\pm$ 28 mg/l.
    - For the determination of ammonia in whey, a significantly better repeatability than that of the NF V 04-217 standard method (1.59 ppm vs 2.5 ppm) and an estimation accuracy in the range of $\pm$ 8 ppm on this type of product (for a rate range of about 12 to 113 ppm)

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6. FIGURES

7. TABLES