EVALUATION OF THE CDR FOODLAB® INSTRUMENT

ACTALIA Cecalait was asked to carry out an evaluation of the performances of the <u>CDR Foodlab[®]</u> analyser on milk and some liquid dairy products. This instrument, produced by CDR, is a polyvalent photometric analyser for the determination of a large panel of chemical criteria in food products. The instrument is equipped with LED sources, reading and thermostated incubation at 37 °C cells, enabling the realisation of 16 determinations in parallel.

The characteristics of the instrument used for this study were:

- CDR Foodlab[®]
- Type: SLB 222
- Serial No: B-222003/1112
- Production year: 2019.





The instrument was installed in a temperature controlled room (2-23 °C – air-conditioning), without direct sunlight. The installation procedure was performed by CDR.

Lactose in lactose-reduced milk, urea in milk and ammonia in whey have been evaluated using respectively the following ready-to-use reagents kits: 300004, 300010 and 300054, which were packed in bag of 10 tests with a one year shelf-life. The analyses were carried out without prior preparation of the samples.

THE TESTS

The evaluation tests (instrumental and reference analyses according to ISO 14637 for urea and NF V 04-217 for ammonia) were carried out in ACTALIAT Cecalait physico-chemistry laboratory in July 2019. The reference analyses for lactose by HPLC were performed in ACTALIA Contrôle et Qualité at Villers Bocage.

The repeatability and the accuracy of the method and he stability of the instrument for each parameter, were evaluated.

As only the absorbance raw data of the CDR FoodLab[®] were available, they were transformed in rates using the reference values obtained within the context of the stability and accuracy evaluation for each parameter.

So, the evaluation of the accuracy of each parameter focused only on the residual standard deviation of regression Sy, x and the estimation accuracy $\pm 2.Sy,x$. Indeed, because of this approach, the accuracy regression equation obtained on the basis of the transformed absorbances leads to a regression slope at 1.00 and an intercept at zero.

1. EVALUATION OF THE STABILITY (INTRA-LABORATORY REPRODUCIBILITY)

The stability of the instrument was evaluated by analysing:

• For determination of lactose content in milk:

2 milk samples with different lactose contents: 0.80 g/100 g (level 1) and 1.50 g/100 g (level 2). The milk samples are a mix of 2 UHT milks: one at lactose-reduced content 2.7 % and one without lactose < 0.1 g/100ml.

• For determination of urea content in milk:

3 whole milk samples with different urea contents: 180 mg/l (level 1), 500 mg/l (level 2) and 800 mg/l (level 3). The samples used were SRMs produced by ACTALIA Cecalait.

• For determination of ammonia content in whey:

3 whey samples with different ammonia contents: 20 ppm (level 1), 30 ppm (level 2) and 50 ppm (level 3). The samples used were whey samples from Franche-Comté region

Bronopol was added to the samples to give a final concentration at 0.02 % for each determination. The analyses were performed in duplicate, every 15 minutes to obtain at least 10 measurement cycles.

To evaluate the stability of the instrument, the repeatability and reproducibility were calculated for each level.

The results obtained are presented in the following tables:

Table 1: CRD FoodLab[®] stability lactose¹

	Lactos	e (Abs)	Lactose (g/100g)			
	Level 1	Level 2	Level 1	Level 2		
М	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		

Table 2: CRD FoodLab[®] stability urea²

		Urea (Abs)		Urea (mg/l)				
	Level 1	Level 1 Level 2 Level 3		Level 1	Level 2	Level 3		
М	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	166	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		

Table 3: CDR FoodLab[®] stability ammonia³

	A	mmonia (Ab	s)	Ammonia (ppm)				
	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3		
М	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		

It can be noted that:

• For the determination of lactose content in milk:

The standard deviation of deviations are in the range of 2.3 to 3.9 % and the standard deviations of reproducibility are about 2.8 to 3.7 % according to the samples rates.

Concerning the standard deviation of reproducibility observed, with no standard criteria, it can be noted that they are very close to the observed repeatability deviations, reflecting a generally weak "instrumental stability" error.

^{1 2 3} M: mean; Sr and SR (Sr% and SR%): absolute standard deviation of repeatability and reproducibility (and relative); r and R: maximal deviation of repeatability and reproducibility in 95 % of cases.

• For the determination of urea content in milk:

The standard deviations of repeatability are in the range of 1.8 to 3.4 % and the standard deviations of reproducibility are of 1.9 to 3.9 % according to the samples rates.

Concerning the standard deviation of reproducibility observed, with no standard criteria, it can be noted that they are very close to the observed repeatability deviations, reflecting a generally weak "instrumental stability" error.

• For the determination of ammonia content in whey:

The standard deviations of repeatability are in the range of 1.4 to 3.8 % and the standard deviations of reproducibility are of 1.4 to 3.6 % according to the samples rates.

The standard deviation of reproducibility observed is in the same range of the standard deviation of repeatability, indicating a good instrumental stability.

2. EVALUATION OF THE REPEATABILITY

The repeatability of the instrument was evaluated by analysing:

• For the determination of lactose in milk:

26 samples of milk (mix of 2 UHT milks) with lactose contents between 0.01 and 2 g/100 g.

• For the determination of urea in milk:

34 samples of raw milk with urea contents between 170 and 800 g/l: 5 samples of urea SRMs produced by ACTALIA Cecalait and 29 producer's milk from Franche-Comté region.

• For the determination of ammonia in whey:

33 samples of whey from Franche-Comté region with ammonia contents between 12 and 113 ppm.

The samples were analysed in duplicate and Bronopol was added to the samples to give a final concentration at 0.02 %.

The results obtained are presented in the following tables:

Table 4: CDR FoodLab[®] repeatability lactose urea and ammonia⁴

	n	min	max	М	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
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
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

It can be noted:

• For the determination of lactose in milk: a standard deviation of repeatability of 0.017 g/100 g on the measurement range of 0.16 to 1.63 g/100 g.

No standard criteria exist for lactose-reduced milk, but it can be compared to the existing standardised methods for the determination of lactose in milk: 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 the differential pH-metric method according to ISO 26462.

• For the determination of urea in milk: a repeatability r obtained (19.6 mg/l) slightly higher than the reference method (ISO 14637): r = 15 mg/l; Sr = 5.42 mg/l.

• <u>For the determination of ammonia in whey</u>: on the range of the considered rates, a repeatability r obtained using CDR FoodLab[®] equal to 1.59 ppm against 2.46 ppm using the reference method (NF V 04-217).

3. EVALUATION OF THE ACCURACY

The accuracy was evaluated by analysing 26 samples for the determination of lactose content in milk, 34 samples for the determination of urea in milk and 33 samples for the determination of ammonia in whey. The samples were the same samples analysed for the repeatability evaluation.

Samples with aberrant reference values were eliminated on the basis of the regression residuals greater than 2 x standard deviation of regression residuals: 5% threshold.

⁴ N: number of results; min and max: minimum and maximum values; M: mean of the results; Sr (Sr%): absolute (and relative) standard deviation of repeatability; r: maximum deviation of repeatability in 95 % of cases.

The results obtained are presented in the following table and figures:

	n	Min	Max	Y	Х	Sy	Sx	Sd	Sy,x
Lactose (g/100g)	26	0.179	1.621	0.692	0.692	0.405	0.408	0.044	0.045
Urea (mg/l)	34	173.79	834.04	313.78	313.78	130.52	131.26	13.944	14.160
Ammonia (ppm)	33	13.03	111.12	34.58	34.58	21.38	22.21	6.020	6.116

Table 5: CDR FoodLab[®] accuracy criteria lactose, urea and ammonia⁵



Figure 1: Relation between instrumental and reference results in g/100g of lactose



Figure 2: Relation between instrumental and reference results in mg/l of urea



Figure 3: Relation between instrumental and reference results in ppm of ammonia

⁵ n, min, max: number of results, minimum and maximum value; Y,X: mean of the results using the reference and instrumental method; Sy, Sx: standard deviation of the results from the reference and instrumental method; Sd: standard deviation of deviations; Sy,x: residual standard deviation

Concerning the relation between the results obtained using CDR FoodLab[®] method (calculated with the regression equation) and the reference method, it can be noted:

• For the determination of lactose in milk, a residual regression standard deviation (Sy,x) of 0.045 g/100 g, so a precision of estimation of \pm 0,09 g/100g.

• For the determination of urea in milk, a residual regression standard deviation (Sy,x) of 14.2 mg/l, so a precision of estimation of \pm 28 mg/l.

• For the determination of ammonia in whey, a residual regression standard deviation (Sy,x) of 4.2 ppm, so a precision of estimation of about \pm 8 ppm.

4. CONCLUSION

The evaluation of the lactose content in milk (range of 0.01 - 2 g/100 g), the urea content in milk and the ammonia content in whey allows the following conclusions:

- The CDR FoodLab[®] instrument is easy to use thanks to the use procedures incorporated in the methods.
- No recurring problems were noted during tests with the CDR FoodLab[®] instrument. It can nevertheless be noted the importance of sampling as well as the addition of the reagents made with a pipette. The pipette must be used with precision so as not to introduce performance problems.
- Concerning the performance of the instrument, it can be noted that:
 - For the determination of the lactose content in lactose-reduced milk, the repeatability is superior to the standard methods (Sr% = 2.44 vs 0.44 for ISO 22662 and 0.74 for ISO 26462) and the accuracy enables to obtain a precision of estimation < 0.1 g/100 g (0.09 g/100 g).
 - \circ For the determination of the urea content in milk; the repeatability is close to the reference method and the precision of estimation is of ± 28 mg/l.
 - \circ For the determination of the ammonia in whey, the repeatability is significantly better than the NF V04-217 reference method (1.59 ppm vs 2.5 ppm) and the precision of estimation is about \pm 8 ppm for this type of product (for a range of about 12 to 113 ppm).

According to the evaluation report of the CDR Foodlab[®] analyser – A. OUDOTTE and P. TROSSAT – July-August 2019