Analyser B 2000 ® Evaluation on Freezing Point Depression

Analyser B 2000 ® has already been evaluated by CECALAIT for milk composition parameters : fat, protein and lactose. The results were presented in CECALAIT's Newsletter n°34. The actual evaluation is devoted to determinating the freezing point equivalent. Repeatability evaluation indicates that absolute standard deviation of repeatability is smaller than the thermistor cryoscope reference method limit. Repeatability and accuracy performances are in accordance with the manfacturer (BENTLEY) specifications.

APPARATUS

B 2000 ® is an automatic analyser manufactured and marketed by BENTLEY Instruments for analysis of fat, protein, and lactose in milk. CECALAIT evaluated these criteria and presented them in Newsletter n°34. B 2000 ® can also measure a freezing point equivalent.
It works using a filter mono beam infrared system combined with a conductivity cell. The apparatus is coupled with a micro-computer which deals with running and signal processing

Experimental

CECALAIT's physico-chemistry laboratory conducted the reference and infrared analyses for FPD (Freezing Point Depression) from August to October 2002.

The following characteristics were evaluated :
- repeatability
- accuracy

1-Repeatability

Repeatability was evaluated by automatic analysis of 104 herd milks, from Franche-Comté, preserved with 0,02% bronopol. The sampling and analyses was realised over three days with a first set of 54 samples, a second set of 40 samples and a third set of 10 samples. Due to the low amplitude of the results obtained with the second set, the 10 milks from the third set were artificially watered at 4% and 8%.
Measures were analysed automatically and each set of ten milks was duplicated in a non consecutive way.
The evaluation was carried out with samples covering a range of rates from 480 to 534 (m°c x-1).

Results

Repeatability was estimated by using the standard deviation of repeatability.

N	M (m°c x –1)	Sx (m°c x –1)	Sr (m°c)	Sr (%)	r (m°c)
54	518	6.9	0.8	0.15	2.1
40	519	4.7	1.1	0.22	3.1
10	498	11.9	0.9	0.17	2.4

N : number of samples
M : mean results
Sx : standard deviation of the results
Sr et Sr% : relative and absolute standard deviation of repeatability
r : maximal difference between duplicates in 95 % of occurrences

Conclusion

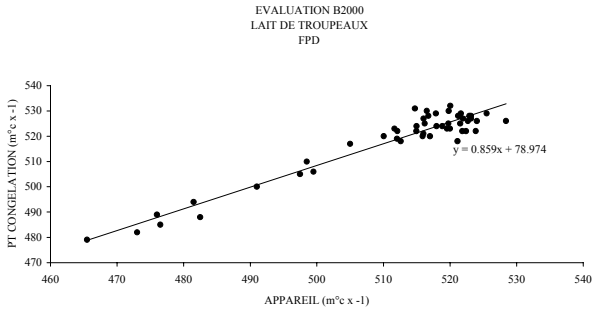
The standard deviation of repeatability Sr is smaller than the limit of the Thermistor Cryoscope Reference Method (ISO 5764:2002), r = 4 m°c and Sr = 1.4 m°C). The results (r mean = 2.5 m°c) comply with the manufacturer BENTLEY Instruments (r < à 2.8 m°c).

2- Accuracy

The apparatus was evaluated with the same milk samples as the ones used for the repeatability evaluation (sets 2 and 3). Immediatly after the instrumental analyses, the samples were cooled to 20 °C, then analysed using the reference method (plateau seeking).

Infrared analyses

Infrared analyses were carried out in duplicate in a non consecutive way. In order to check the stability of the apparatus,



a control milk was inserted every 20 samples. The mean value of the duplicate was calculated and used for determinating results.

The evaluation concerned the values obtained by the calibration performed by the BENTLEY company, which was not adjusted with local milks.

Reference analyses

Determination of freezing point was performed using thermistor cryoscope method following ISO standard 5764 : 2002.

Routine analyses were performed and checked in case of important residual matters.

Statistical analyses

Accuracy is evaluated by using mean results of deviations, standards deviations of differences, residual standard deviations and assessed linear regressions equations, taking the reference method for explained variable Y, and B 2000 for the explicative

variable X.

Compared to set 2, set 3 of 10 milks showed a systematic bias due to the zero adjustement. The results from samples of this set were corrected from the value of this bias (-14 m°c) before calculation of these parameters (except for residual standard deviations). For the calculation of residual standard deviation, each set was submitted to a specific linear regression. Adjusted results according to regression parameters were then gathered and the residual standard deviation calculated.

Results

n	50
min (m°c x –1)	466
max (m°c x –1)	528
Y (m°c x –1)	518.5
X (m°c x –1)	511.7
Sy (m°c)	13.8
d (m°c x –1)	-6.8
Sd (m°c)	4.4
Sy,x (m°c)	3.3
b	0.859
a	79

n, min, max : number of samples, minimum and maximum value
Y,X : mean results with reference and instrumental methods
Sy : standard deviation of results with the reference method
d : mean of the deviations
Sd : standard deviation of the differences
Sy,x : residual standard deviation for the linear regression
b,a : slope and biais of the linear regression

The table and figure above give the results and the linear regression obtained.

The slope and bias of the linear regression (b= 0.859 a=79) are significantly different from 1 and 0 (P=1%). The residual standard deviation of the regression obtained is 3.3 m°c.

Conclusion

The standard deviation being 3.3 m°C allows for an accuracy in estimation of +/- 6.6 m°c. The results comply with BENTLEY specifications (Sy,x< 4 m°c).

CONCLUSION

Results of repeatability and accuracy comply with BENTLEY's specifications. The mean deviations observed between instrumental and reference values may be explained by the fact that evaluation concerned values obtained from the calibration performed by the manufacturer.

An adjustment based upon analysis of samples from the collection area is necessary, as required by IDF standard 128. It is also advisable to make more precise blanking in order to avoid any gaps.

Abbreviation

FPD : Freezing Point Depression

Bibliography

ISO 5764:2002 - Milk -- Determination of freezing point -- Thermistor cryoscope method (Reference method)

QUERVEL, X. et TROSSAT, P. **Rapport d'évaluation de l'analyseur B 2000, critère détection du point de congélation**, CECALAIT, 2003, Poligny, 3 p.
