

SUMMARY OF LA LETTRE DE CECALAIT, N° 34 (3rd quarter 2000)

(Translation : A. BAPTISTE, Correction : H. LAMPRELL)

Evaluation of the Bentley B 2000 / B

B 2000/B is an automatic MIR (Mid infrared) analyser, developed and marketed by BENTLEY, for analysis of fat, protein and lactose in milk. It can also measure a freezing point equivalent. CECALAIT evaluated its analytical characteristics for 4 months in 1999 (phase I assay, see Lettre de Cecalait, n°33, page 1)

APPARATUS

BENTLEY B2000/B is a dispersive mid infrared spectrophotometer, for the determination of fat, using 2 different filters (filters 1 and 2), protein and lactose in milk. Run by a micro-computer for analyses and calibration, its analysing speed is 450 samples/h.

Mathematically, calibration is done as follows: the instrument measures the energy absorption at specific wavelengths in the mid infrared region. The measure is, first, linearized using a logarithmic algorithm. Then, intercorrection factors are calculated by multiple linear regression (MLR).

TESTS PERFORMED

The following characteristics were evaluated, according to IDF standard 141B:1996 and to the guidebook for infrared analysers issued in France by CNIEL (the milk payment body):

- stability
- carry-over effect
- preservative influence
- linearity
- repeatability
- accuracy

① STABILITY

The stability was evaluated by the duplicate automatic analysis of a set of three milks, corresponding to the usual range of fat and protein, every 20 mn for half a day.

The results show that, for fat (filters 1 and 2) and for protein, the standard relative deviation of reproducibility S_R is always lower than the value inferred by the IDF standard 141B, i.e $S_R < 0.27\text{g/kg or }l$.

② CARRY-OVER EFFECT

The carry-over effect was evaluated by analysing, for fat (filters 1 and 2), protein and lactose, the same individual milk and distilled water, 20 times, in the following sequence: milk – milk – water – water.

The carry-over effect (Tc %) was estimated with following equation :

$$Tc \% = [(S(\text{water } 1) - S(\text{water } 2)) / (S(\text{milk } 2) - S(\text{water } 2))] \times 100$$

Tc values are in the interval of **0.00 to 0.26%**.

These values comply with the maximum limit of 1% usually allowed, for instance in routine methods of determination of milk composition, used for milk payment purposes.

③ INFLUENCE OF THE PRESERVATIVE

The test was performed on 37 individual cow milks, from two different herds and the results obtained in following cases were compared:

- on raw milk.
- on milk + Bronopol 0.02 %, at 4°C.
- on milk + Bronopol 0.02 %, at 20°C.

Samples were bottled after 4 hours without preservative at 10 to 15°C. For each milk, samples with and without preservative were analysed one after the other, to avoid a drifting effect.

The results do not show any significant difference (at the 1 % limit) between preserved or unpreserved milk or due to the storage temperature. However, for fat determination, higher residual standard deviations were observed when studying the linear regression associated with the storage temperatures; especially when using filter 2, the most sensitive to the quality of milk homogenization. Later, it will be better to remain vigilant, due to possible fat disruptions through the mechanical homogenization. With phase II assays analysing more samples in routine, the actual importance of this phenomenon should be better known.

④ LINEARITY

Linearity was evaluated for each channel by manual analysis, in triplicate, without shaking, of a set of 11 milks with :

- ♦ fat ranging from 0 to 85 g/l,
- ♦ protein ranging from 5 to 50 g/kg.

The analysis followed first increasing, then decreasing fat and protein levels. Linearity was estimated on raw data, before applying intercorrection factors.

The results show that the manufacturer's linearity adjustment is satisfactory for the whole range of fat and protein tested. However, it should be optimized for a broader range, for high level milk, for instance Jersey cows' milk at the end of lactation or ewe's milk.

5 REPEATABILITY

Repeatability was evaluated by automatic analysis of 123 individual milk samples and 49 herd milks, preserved with 0.02% bronopol, with fat ranging from 21 to 81 g/l and protein from 24 to 63 g/l.

Each set of 20 samples was analysed in duplicate. The stability of the analyser was checked during the tests.

The values were corrected by linear regression using an orthogonal network of 13 recombined milk samples, following the technique described by O. LERAY in 1989.

The results are given in table 1, page 2, in « La Lettre de CECALAIT » n° 34.

The repeatability values comply with IDF standard 141B specifications, i.e. $S_r = 0.14 \text{ g/kg}$ and $r = 0.4 \text{ g/kg}$.

6 ACCURACY

Accuracy was evaluated, as in 5, by duplicate (not consecutive) automatic analysis of :

- 110 individual milk samples (same range as in 5), preserved with 0.02% bronopol, for milk recording purposes,
- 52 herd milks (same range as in 5), preserved with 0.02% bronopol for milk payment purposes.

The stability of the analyser was checked during the tests. Reference methods used were the official methods for milk payment, ie :

- ♦ the Gerber method for fat.
- ♦ the Amido Black method for protein.

The instrument was calibrated using MLR from a set of 13 recombined milk samples (see 5).

Accuracy was estimated by using :

- the mean bias to the reference values (*moyennes des écarts*).
- the standard deviation of the differences (*écarts types des écarts*).
- the residual standard deviation ($S_{y,x}$).
- the equations of the estimated linear regressions, where Y is the result of the reference method and X the B 2000/B result.

Tables 2 and 3, page 4, in « La Lettre de CECALAIT » n° 34 show the results on individual and herd milks.

↳ For fat, the mean biases are :

- + 0.53 g/l and - 0.04 g/l, for filter 1,
- + 0.14 g/l and - 0.084 g/l, for filter 2,

respectively for individual and herd milks.

The slopes are not significantly different from 1.00. The residual standard deviations are :

- 0.84 g/l and 0.467 g/l, for filter 1,
- 0.46 g/l and 0.336 g/l, for filter 2,

respectively for individual and herd milks. Obviously, using filter 2 reduces the error of the precision of estimation. This is typical of the method.

↳ For protein, the mean biases are :

- + 0.04 g/l and + 0.56 g/l, respectively for individual and herd milks.

The slope is not significantly different from 1.00 for herd milks, but it is for individual milks. The residual standard deviations are :

- 0.552 g/l and 0.274 g/l, respectively for individual and herd milks.

However, the deviations observed for slopes and biases remain very small and still comply with users wishes.

In conclusion, for fat and protein, the analytical characteristics of B 2000/B comply with the limits fixed in IDF standard 141, i.e. residual standard deviation of 1.0 g/kg for individual milks and 0.7 g/kg for herd milks.

The mean biases for herd milks are slightly over +/- 0.15 g/kg for protein. It may come from the delay –about a month– between the preparation of the calibration samples and the sampling of herd milks. These differences are then acceptable.

Moreover comparing the herd milk analyses performed on the B 2000/B and on another instrument, already marketed (MS 4000, with B filter for fat) showed equivalent results for the two instruments, i.e. :

- for fat, respectively for filter 1 and 2 :
 - mean biases of -0.021 and + 0.013 g/l
 - residual standard deviations of 0.375 and 0.169 g/l,
- for protein, a mean bias of -0.065 g/l and a residual standard deviation of 0.118 g/l.

7 general conclusion

The analytical characteristics of BENTLEY B 2000/B : instrumental stability, carry-over effect, linearity, repeatability, accuracy, have all been found satisfactory. They all comply with the requirements of milk payment and milk recording purposes.

The procedure authorizing the use of this new material for milk payment purposes (in France, see La Lettre de CECALAIT, n° 33) is still going on. The instrument is now being tested in routine conditions (phase II) for fat and protein and also for FPD.

For abbreviations and bibliography, please see page 5 in La Lettre de CECALAIT n° 34

AFNOR VALIDATION

AFNOR (French standardisation body) validated recently the following alternative methods :

↳ **EIAFOSS**, manufactured by FOSS, an immunoenzymatic test for the detection of *Salmonella* in human or animal foods

↳ **VIDAS *E. coli* O157 (ECO)** and **VIDAS immunoconcentration *E. coli* O157 (ICE)**, manufactured by BioMérieux, an immunoenzymatic test for the detection of *Escherichia coli* O157, in all human foods.

Questionnaire « web site » : the answers

In our last issue, we informed you about the principal points that were highlighted by the answers to our « microbiology » questionnaire.

Now, the questionnaire about a future web site has been fully analysed. Here are the highlights !

A lot of laboratories replied (121 french laboratories, 45 foreign laboratories, that is to say a response of 27.9%). Thank you very much.

↳ using Internet now and later

Questionnaires had all been sent by mail, but also by e-mail where an e-mail address was available. The latter being the case for about 12% of the questionnaires for French laboratories and 37% of the questionnaires for foreign laboratories. Only 4% of the 121 answers from France whereas 33 % of the 45 foreign answers arrived by e-mail. Foreign labs seem more familiar with this media. Moreover, as postal delays may be very long, it is more interesting to try to reduce them.

Anyhow, there are more connected laboratories abroad : almost all foreign labs who answered are already connected versus only 47% of the French laboratories who answered. Nevertheless, it is true that Cecalait's foreign customers are quite important organizations or laboratories. Moreover, most of the non-connected French laboratories are thinking of being connected in the near future.

For us, it is obvious that we have to build a bilingual site.

↳ What you would like to find on CECALAIT's site

Table 4, page 6 in La Lettre de CECALAIT n°34 gives a summary of what the laboratories, who answered the questionnaire, are expecting from CECALAIT's site.

French and foreign laboratories are generally interested in the same topics, except for consulting our catalogue or a schedule of events.

For more details....

- information about CECALAIT means mostly to know the name of the persons in charge for each type of problem.,
- Something listing IDF and AFNOR methods by analyte or product seems very interesting, but should also include ISO methods.
- faster results from ringtests is interesting in both directions : from laboratories to CECALAIT, but even more from CECALAIT to laboratories.

In conclusion, many thanks for answering the questionnaire. We will now try to meet your wishes. We are hoping to meet you soon on the net.....

INTERESTING NEW STANDARDS

IDF STANDARDS

IDF 155A :1999. MILK AND MILK BASED DRINKS. Determination of alkaline phosphatase activity using a fluorimetric method.

It is a joint IDF/ISO/AOAC, superseding 1992 provisional standard. The corresponding ISO text is ISO 11816-1. It is also issued as an European and French standard NF EN ISO 11816-1.

IDF 128A :1999. MILK. Definition and evaluation of the overall accuracy of indirect methods of milk analysis. Application to calibration procedure and quality control in the dairy laboratory.

It is a joint IDF/ISO/AOAC, superseding 1985 provisional standard. The corresponding ISO text is ISO 8196.

INTERNATIONAL STANDARDS

ISO 11870, March 2000. MILK AND MILK PRODUCTS - Determination of fat content - General guidance on the use of butyrometric methods

ISO 1736, March 2000 DRIED MILK AND DRIED MILK PRODUCTS - Determination of fat content - Gravimetric method (reference method) . It replaces the 1985 version.

ISO 8381, March 2000 MILK-BASED INFANT FOODS - Determination of fat content - Gravimetric method (reference method). It replaces the 1987 version.

ISO/TS 11133-1, June 2000 MICROBIOLOGY OF FOOD AND ANIMAL FEEDING STUFFS - Guidelines on preparation and production of culture media - Part 1 : general guidelines on quality assurance for the preparation of culture media in the laboratory

ISO 11843-2, May 2000, Capability of detection - Part 2 : methodology in the linear calibration case

INTERNATIONAL (and French) STANDARDS

NF EN ISO/CEI 17025 may 2000, General requirements for the competence of testing and calibration laboratories. This text replaces and amends NF EN 45001 issued in december 1989.

EUROPEAN (and French) STANDARDS

NF EN 12469, July 2000 BIOTECHNOLOGY - Performance criteria for microbiological safety cabinets. This replaces NF X44-201, a French standard issued in 1984

↳ Also issued the 6th edition of the inventory of IDF/ISO/AOAC methods.

Bulletin FIL-IDF n° 350, 2000. Inventory of IDF/ISO/AOAC international adopted methods of analysis and sampling for milk and milk products. 6th edition (the former one was issued in 1996).

INTERESTING RECENT EEC REGULATION

Commission Decision 2000/443/EC of 18 May 2000 amending Decision 97/404/EC of 10 June 1997 setting up a Scientific Steering Committee and Decision 97/579/EC of 23 July 1997 setting up **scientific committees in the area of consumer health and food safety** (JOL 179 of 2000/7/18).

Commission Directive 2000/42/EC of 22 June 2000 amending the Annexes to Council Directives.... 86/363/EEC....on the fixing of **maximum levels for pesticide residues** infoodstuffs of animal origin.....(JOL 158 of 2000/6/30).

➤ As usual, **regulation n° 2377/90**, of the Council concerning **maximum residue limits of veterinary drugs in foods** of animal origin, has been amended.

- Annexes I, II and III were amended by **regulation 1286/2000 of 2000/6/19** (JOL 145 of 2000/6/20)

- Annexes II and III were amended by **regulation 1295/2000 of 2000/6/20** (JOL 146 of 2000/6/21)



On July 17th 2000, Commission proposed most radical shake up of food safety hygiene rules. Eventually, 4 new regulations will merge, harmonise and simplify very detailed and complex hygiene requirements previously scattered over 17 existing directives (including directive 92/46).

The first regulation is based upon four key principles :

- application to all food and food operators, from the farm to the table

- primary responsibility for food safety of food operators, right through the food chain. They will have to use programmes for self-checking and modern hazard control techniques.

- traceability of all food and food ingredients,

- responsibility left to Member States for adapting the rules to local situations such as traditional food production or food businesses in remote islands, secluded mountain areas and other geographically isolated regions.

The second regulation will set out specific additional hygiene rules for food of animal origin. The third regulation will regroup the obligations of the veterinary authorities in the Member States. Finally, the fourth regulation will recast, update and improve the transparency of animal health measures.

Sources : [hygiène@egroups.fr](mailto:hygiene@egroups.fr)

<http://www.tageblatt.lu> of 2000/7/18

http://www.terre-net.fr/_phytonet/phyto_actusdetail...

http://europa.eu.int/comm/dgs/health_consumer/

Official Journals of the European Communities of the last 45 days may be consulted on Internet <http://europa.eu.int/eur-lex>

Older texts may be ordered on Internet <http://www.eudor.com>

IDF'S NEW ORGANIZATION

(Abstract of the lecture given by Mr JAMET of CNIEL at CECALAIT's annual meeting, June 2000).

The IDF was created in 1903 and has about 40 member countries, represented by their National Committees. They represent all people involved in the dairy sector, i.e. farmers and their cooperatives, companies, suppliers, government, research institutes, schools and universities. Its mission is « to promote and enhance the image, trade, production and consumption of milk and milk products world wide by :

- collecting and disseminating scientific, technical, economic and legal information,
- providing a platform for meaningful exchange of professional knowledge and discussion» .(in : www.fil-idf.org)

This implies :

- « promoting the study of all aspects of the dairy industry,
- acting as a forum for dairy specialists through regular publications and specialized events,
- representing the dairy industry in decisions affecting standards, hygiene, analysis, etc,
- consulting and advising the dairy industry and international organizations, e.g. FAO or WHO »
(in : www.fil-idf.org)

A new strategic plan has been layed out since 1996 to make IDF more dynamic. Its objectives are :

- attracting other active professionals, especially farmers and companies,
- having a more important role in international discussion and publication,
- giving more responsibility to the general secretary.

The plan was adopted by the end of 1998, under IDF's new president and has been enforced since then.

Before this new plan, IDF scientific, technical and standardizing activities were carried out by 6 Commissions (for instance Commission E, standards for analysis and laboratory techniques). Their work was rather autonomous, not depending on the National Committees or on IDF's secretariat. Moreover, they had more retired than active professionals as members.

The new organization

➔ EXECUTIVE

➤ THE DIRECTOR

IDF's general secretary becomes a general director, responsible for the execution of IDF's activity. He must explain it to the Management Committee (see below) and to the Council, where each National Committee is represented.

➤ THE « MANAGEMENT COMMITTEE »

It is IDF's executive body. It has 6 members : the president, 2 vice-presidents, 2 office members, the president of the Programmes Coordination Committee (see below)

➔ ADMINISTRATION

IDF's secretariat based in Brussels is small : about 10 people. Actually organizing an event is quite totally delegated to the National Committee in charge.

➔ THE SCIENTIFIC, TECHNICAL, STANDARDIZING WORK

➤ PERMANENT COMMITTEES AND TASK FORCES

The former 6 Commissions were disbanded and split into 19 Permanent Committees and 3 « task forces ». The Permanent Committees are composed of Action Teams, each corresponding to a specific and short-term topic.

The Permanent Committees are no longer managed by a group of experts (4 or 5), but by a president and a vice-president with short mandates. Compared to the former Commissions, this is a more flexible organization, where the required knowledge is more specific. It should be more attractive to active professionals wishing to participate in IDF's activity.

However, for work concerning analysis methods, a Method Standard Steering Group, composed of the presidents of the 5 Permanent Committees concerned, has been temporarily set up.

Work is left either to a Permanent Committee, or to a Task Force for topical issues.

Their reports will be consultable on IDF's intranet by the experts registered by the National Committees. The use of this Intranet should make experts' work quicker and easier, as less meetings or travel will be necessary. Thus IDF hopes to solve the long lasting problem of a shortage of experts. It also hopes to attract more professionals, especially farmers and companies.

➤ THE PROGRAMMES COORDINATION COMMITTEE

The Permanent Committees must explain their work to the National Committees (see above) , but also to the Programmes Coordination Committee. This body supervises and coordinates the different work.

Finally, it is responsible for all documents bearing IDF's logo. These must have been approved by the Permanent Committee in charge, but also submitted to the National Committees and to the Programmes Coordination Committee, which may possibly question

Permanent Committees after considering National Committees observations.

At last, the Programmes Coordination Committee arbitrates the possible contentious issues between the Permanent and the National Committees. This function did not exist before, possibly enabling some Commissions to ignore the observations or even the opposition of some National Committees.

The Programmes Coordination Committee is composed of 8 members, all having transversal competences, allowing them to supervise the work of one or several Committees. They also make sure that the work is coordinated and transversal. The president belongs to the Management Committee (see above).

➤ ORGANIZING EVENTS

IDF's new strategic plan also aims to produce a better management of the programme of its specialized events, especially to make them more famous. In particular, the Annual Sessions are from now on replaced by the World Dairy Summit, where interesting conferences,

open to all dairy professionals will be separated from IDF's internal business.

➤ Finally, it was reminded that dairy professionals strongly expect IDF's standardization of analytical techniques. In principle, IDF should focus on physico-chemical aspects and on characterisation of dairy products... Indeed most ISO horizontal microbiological standards should become joint ISO/IDF methods, with new work needed only if the methods are not immediately applicable to dairy products. The Committee «microbiological methods of analysis» will survey this field. Another Committee in charge of the automatic milk analysis methods is also concerned.

It is also hoped that, being able to act more quickly now, IDF may start the examination of alternative methods or of proprietary techniques.

Abbreviations

CNIEL : Centre National Interprofessionnel de l'Economie Laitière

FAO : Food and Agricultural Organization

IDF : International Dairy Federation

WHO : World Health Organization

FORTHCOMING EVENTS

➤ REMINDER

16 – 20 SEPTEMBER 2000
DRESDEN, GERMANY
IDF World Dairy Summit

IDF Secretariat
41, square Vergote
B-1030 BRUSSELS BELGIUM

18 – 20 SEPTEMBER 2000
AVEIRO, PORTUGAL
5th international conference on applications of
magnetic resonance in Food Science

Fundação Joao Jacinto de Magalhaes
Edificio I
Campus Universitario de Santiago
3810-193 AVEIRO, PORTUGAL

20 – 22 SEPTEMBER 2000
PRAGUE, CZECH REP.
Chemical reactions in food IV
European Conference on new knowledge on
chemical reactions during processing and
storage of foods

Pr Dr Jiri DAVIDEK
Dpt Food Chemistry & Analysis
Institute of Chemical Technology
Technicka 1905
166 28 PRAGUE 6, CZECH REP.

10 OCTOBER 2000
BRUSSELS, BELGIUM
CEN seminar on food safety and European
standardization

CEN
Comité Européen de Normalisation
European Standardization Committee

16 – 20 OCTOBER 2000
ANTWERP, BELGIUM
CAC 2000. 7th international conference on
chemometrics in analytical chemistry

CAC 2000 secretariat
University of Antwerp
Dpt Chemistry
Universiteitsplein 1
B-2610 ANTWERP-WILKIJK, BELGIUM

19 – 20 OCTOBER 2000
LONDON, UNITED KINGDOM
Food safety in Europe. Challenge and
opportunities

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http://www.foodsafetyeurope.com/body_home.html

OTHER EVENTS

4 – 5 OCTOBER 2000
EDINBURGH, SCOTLAND
5th EUROLAB symposium
Upon ISO 17025, accreditation, management

Lesley DEAKIN
National Engineering Laboratory
East Kilbride
GLASGOW, G75 0QU, UNITED
KINGDOM

E-mail : eurolab@nel.uk

13 – 16 NOVEMBER 2000
NAGANO, JAPAN
Pacific Congress on milk quality and mastitis
control

Secretariat for PC2000
c/o Philpot and Associates International
PO Box 120
HOMER LA 71040-0120, USA