MA SC
ICAR Sub-Committee on Milk Analysis

Approval procedure for milk analysers in milk recording

Disclaimer : Through this procedure and using these protocols, ICAR recognises and confirms to users that the method evaluated in these conditions and fulfilling the technical requirements is appropriate for the use and purposes of milk recording, so allowing ICAR members to refer to that recognition – so-called ICAR approval – with no more need for complementary evaluations (unless it be locally demanded). This approval for use covers the field of application and the instrument configuration tested during the evaluation and cannot constitute itself an agreement for any use other than milk recording within ICAR.

Foreword

The international ICAR approval for milk analysers was launched in 2002 as applicable as soon as an analyser is successfully evaluated according to ICAR agreed protocols and locally approved in three different countries.

This international approval procedure is here to complement and to take into account the case of an evaluation directly organised by ICAR that allows manufacturers not to go through the preliminary three local or national stages.

Additional new procedures complete the initial protocol for the evaluation of milk analysers. This includes broadening the scope to non-automated milk analysers (i.e. manually served) that can be used as master instrument for calibration purposes, and also to new analysers that do not differ from a former ICAR approved version of a same manufacturer.

1. ICAR approval procedures

ICAR recognizes two ways to achieve the international approval of a milk analyser by ICAR, both being based on the international Standard ISO 8296-3 | IDF 128-3:

1.1 Independent national evaluations

As described in the international standard, the procedure relates to already existing national evaluations and approvals obtained in three different countries. This process allows an instrument to progressively go towards an international validation through successive national evaluations and in the end obtain the ICAR approval as the international recognition of fit-for-purpose.

Advantages lie in spreading evaluation costs over a longer period of time and limiting possible risks of non-compliance to one evaluation.

Details of the procedure are given in Annex A.

1.2 International evaluation

This is based on three independent evaluations in different countries but without going through national evaluations and approvals. It is organised and monitored by an international organisation, i.e. ICAR. Thus the experiments can be organised and performed simultaneously or closely in time. This procedure may result in a direct approval granted by ICAR.

The advantage for manufacturers is a reduction in administration in that only one ICAR application is required. Manufacturers can also rely on the organiser to propose approved laboratories to perform work in suitable countries.

The risk is that any possible instrument modification (e.g. to overcome a possible technical weakness/failure) will need to be applied to each of the instruments under evaluation with the related consequences for delays and costs.

Details of the procedure are given in Annex B.

2. References for the evaluation

2.1 Reference document

The following international standard applies:

ISO 8196-3 | IDF 128-3 Milk - Definition and evaluation of the overall accuracy of alternative methods of milk analysis - Part 3: Protocol for the evaluation and validation of alternative quantitative methods of milk analysis.

This ISO-IDF standard is applicable to any alternative quantitative methods of milk analysis. It confirms and completes the content of the former ICAR protocol "**Protocol for the evaluation of milk analysers for ICAR approval"** from which it derives, thus making it also recognized outside of the ICAR sphere.

2.2 Complement for manual milk analysers

The field of application of ICAR approval also includes non-automated milk analysers (manual) that can be found suitable for use as master instruments for lab monitoring and calibration transfer.

For such devices, the ISO recommendation for the second phase of evaluation on the need for an assessment in two routine laboratories for two months should be replaced by the need for an assessment in one laboratory for two months.

2.3 Complement for new versions of already approved milk analysers

This part refers to the case where the configuration of the instrument is changed (e.g. upgrading for higher testing speed rate) or the analyser submitted for approval is an updated version (more attractive, with more or improved features for users) of a former model where there is no (claim for) significant change either in analytical principle, in main instrument parts, in their functions and in accompanying utensils for the execution of the measurements.

Proof must be brought clearly demonstrating that the new instrument does not actually differ with regard to the analytical performance, therefore verifying that the precision and the accuracy are not significantly modified. This can be verified through adequate comparisons with an instrument of a former ICAR approved version.

The standard protocol of ISO 8196-3 | IDF 128-3 should be applied for all usual checks of the compulsory part provided, but replacing the reference method(s) by a milk analyser of the former ICAR approved version.

Both instruments should be calibrated with the same calibration materials. Compliance should be assessed through the mean difference (not statistically different from zero), the slope (not statistically different from 1,00) and the standard deviation of differences s_d and the standard deviation of repeatability s_r (both not statistically different from the limit of standard deviation of repeatability σ_r).

Details of the protocol and compliance limits are given in Annex C.

A positive conclusion on equivalent performance can result in an extension of ICAR approval to the new analyser version.

Conclusion on poorer performances, i.e. beyond the stated limits, would indicate non equivalent devices hence verification should revert to the standard evaluation according to the ISO-IDF protocol with, as a follow-up, the evaluation and assessment of accuracy against the reference method.

3. Type of evaluation

The choice of evaluation type (between 2.1, 2.2, 2.3) depends on the technical characteristics of the device and prior granted approval.

Both complements 2.2 and 2.3 do not exclude each other and can be used in conjunction, for instance for manual devices deriving from an already approved automated routine device.

The manufacturer may choose for the evaluation method based on technical, strategic and economical criteria. The simplified protocol mentioned in 2.3 can revert to applying a full protocol where the technical characteristics do not fit with the similarity pre-requisite or the evaluation results do not comply with the stated limits.

Therefore, before undertaking any evaluation process - especially through the three independent national evaluation method according to clause 1.1 - the manufacturer is advised to check with ICAR the suitable type of evaluation by submitting instrument characteristics to the ICAR secretariat. The ICAR Secretariat will advise the manufacturer on the adequate protocol(s) after consultation with the MA SC. The form in Annex G (or similar) will be used.

In the case that the ICAR international evaluation is chosen, ICAR will decide on the suitable protocol prior to the organisation of the evaluation.

Annex A

Approval procedure through independent national evaluations / approvals

1. Before the approval request to ICAR

The instrument has been submitted for evaluation in three countries according to the milk analyser evaluation protocol of ICAR and with results meeting requirements as defined in the protocol. Reports are to be collected by the manufacturer or the requesting organisation.

2. Request for approval

The approval request is sent to the General Secretariat by the manufacturer or the requesting organisation together with the (three) evaluation reports and the subsequent national approvals by competent bodies. The forms to be used are appended in Annexes D and E.

The General Secretariat registers the request and transmits it to the examination committee with the appropriate documents (files). The examination committee is composed of at least three experts designated by and who may be members of MA SC.

3. Examination and decision delivery

Reports are examined by the experts and, if needed, discussed on the occasion of a meeting with MA SC. Otherwise, general position (positive or negative) and eventual comments can be made by examiners through a standard template for every point evaluated (Annex F). In case a negative decision is taken, it is fully explained and argued. The period of examination should not exceed two months from ICAR Secretariat dispatch.

The examination committee comes to its conclusion, which is then circulated for agreement to the working group. When not agreed, a further re-examination is required to reach final consensus (within two months), otherwise the chair informs the General Secretariat of the decision of the group :

a- Positive : Endorsement by ICAR Board, addition into the list of instruments approved by ICAR, publication in ICAR Newsletter and on the website of MA SC (list of instruments with date of ICAR approval delivery) ; three reports available on request.

b- Negative : All possible remarks and comments on elements of the instrument/method or the evaluation necessary to be improved must be fixed before a further approval request.

4. Cost of administrative accounting and technical examination

The requesting organisation is charged the administrative costs of the entire process (i.e. registration, examination of technical data, publication). A fixed amount in Euros (exclusive of VAT) is established by Service-ICAR SRL and reviewed every year. It is invoiced to the requester at the opening of each case.

5. ICAR approval delivery

On the basis of a positive conclusion from the Sub-Committee on Milk Analysis, the ICAR Board endorses the ICAR approval which is officially delivered to the manufacturer or the requesting organisation and announced via the usual ICAR communication media after all fees have been paid.

Annex B

Direct international evaluation / approval

1. Request for evaluation and approval

The manufacturer addresses a formal request to the General Secretary of ICAR for evaluation, aiming to obtain ICAR approval of a well defined analyser.

Any technical description and information on the measurement principle and functioning must be included with the request.

2. Process

ICAR General Secretariat registers and transmits the request with the appropriate documents to the Sub-Committee on Milk Analysis which will advise ICAR on technical admissibility (principle, functionality, fit-to-purpose) within one month. The consultation committee is composed of at least three expert members of the MA SC.

ICAR will liaise with the manufacturer in order to agree on the organisation and costs of the evaluation. The decision will be made on the three countries and competent laboratories from a list of accredited laboratories recognised as competent in analyser evaluation by ICAR.

ICAR will establish contact with the evaluating laboratories to make the agreement on the task to undertake according to the ICAR evaluation protocol for milk analysers and agree on financial compensation. through ICAR.

ICAR will make a quotation of all the costs for further invoicing to the manufacturer and will make a contract on the basis defined with the manufacturer.

Involved laboratories carry out evaluations and produce reports according to the ISO-IDF protocol and requirements. They are requested to fill in the summary table of results for their respective parts that will be collated in a single table by the ICAR Secretariat.

ICAR (Service ICAR) will pay laboratories for their services and will invoice the manufacturer for the same amounts, plus the cost of overall organisation by ICAR and technical examination within ICAR.

3. Examination and decision delivery : Idem Annex A

4. **Cost of administrative accounting and technical examination :** Idem Annex A

5. ICAR approval delivery : Idem Annex A

Annex C

Alternative comparison for the evaluation of a milk analyser deriving from an already approved analyser

Since the modification of the new analyser from the former device version includes only minor changes such as software upgrading or changes claimed as of negligible influence on the analytical precision (e.g. performance speed increase), a simplified method can be applied to avoid, where possible, an intensive and costly comparison against reference methods.

A previously approved instrument (e.g. a former version of the instrument tested, that is similar with regard to the principle and hardware), with every technical guarantee that the analytical response is not altered, can be used to verify whether the new instrument shows similar behaviour in term of trueness (mean and standard deviation of differences) and repeatability (ranges between duplicates).

1. Instruments : The former approved device and the new evaluated device must be compared under repeatability conditions i.e. same location and environmental conditions, no or little delays between each device testing, with same samples and same number of replicates (minimum 2 required).

2. Samples : The samples should be of the best physicochemical quality. They should be carefully split in the appropriate number of sub-samples fitting to the number of replicates so as to keep the results of replication series independent of the former testing (e.g. 4 sets of vials required for duplicate series on each of both instruments).

3. Analyses : They must be performed in compliance with ISO 8196-3 with special respect to the sample numbers and replicate numbers stated and after both devices have been calibrated with the same calibration sample set in compliance with ISO 8196-2.

4. Repeatability : Same calculations as in ISO 8196-3 are performed. Both devices must show repeatability values complying with the limit of repeatability of the standard.

5. Trueness : The same type previously approved device is used as the reference method. The same data analysis as in ISO 8196-3 is performed, including detection of possible outliers and covering parameters such as mean of differences and standard deviation of differences. The slope must comply with limits derived from the repeatability error as follows :

	fat	protein	lactose	urea	SCC
Limit s_r	0.014	0.014	0.014	1.4	4%
Limit $\bar{d}$	0.014	0.014	0.014	1.4	4%
Limit b	1±0.03	1±0.03	1±0.03	1±0.03	1±0.03
Limit $s_{y,x}$	0.014	0.014	0.014	1.4	4%

6. Compliance : If compliance with the stated limits is not achieved, then it can be concluded that either one (or both) of the two compared instruments is (are) not optimised. Hence the problematic instrument(s) should be appropriately adjusted and the comparison be redone. When non-compliance persists, it is concluded that the two methods are different and the manufacturer is reverted to a classical evaluation against the reference method.

Note

a- Usual practice is to use distinct sample vial sets per device so as to prevent sample handling and re-heating from being potential sources of error and sample damage. Nevertheless, abnormal residuals, so-called outlier bias to the regression line, can stem from insufficient sample set quality (e.g. sample damage or imperfect sample splitting for distinct sample vial sets per device). Confirmation that vial contents are actually different can be made through re-testing (with other devices) the pair of vials of the outlier sample. If compliance cannot be achieved because of the presence of outliers, calculate and report results with and without outliers.

b- If compliance is achieved for repeatability but not for accuracy and if milk quantity allows, re-analyse the sample set of the evaluated device with the previously approved device in duplicate. Compliance with duplicate testing could indicate an effect of the sample set. Re-evaluate with another sample preparation assuring the lowest standard deviation between samples as measured by the sample homogeneity test, e.g. not exceeding 0.008 % fat.

c- If non compliance for accuracy is confirmed, investigate linearity and milk component interactions (re inter-corrections in MIR analysis).

d- If compliance cannot be reached through optimising linearity and correcting milk component interactions of either of the devices, the conclusion must be that the devices perform different.

Annex D

- REQUEST FORM FOR MILK ANALYSER APPROVAL BY ICAR -

Requesting organisation (name): ……..…..…… **Country:** .....….....

Address: .....…… **Phone:** .....….....

.....…….....…… **Fax:** .....….....

..... **E-mail :** .....….....

represented by (Mr, Mrs) : ……..…….....…….....…… **Function :** .....….....

hereby

makes the request to ICAR to grant ICAR international approval to the milk analyser designated here below for the application in milk recording specified in the following :

Manufacturer (name)	:
Instrument (name)	:
- Type	:
- Configuration (*)	:
- Analytical principle	:

Animal species	Cow	Sheep	Goat	Buffalo	Other
- milk components / criteria tested : ⇨ Fat (F) ⇨ Protein (P) ⇨ Lactose (L) ⇨ Urea (U) ⇨ Somatic cells (SCC) ⇨ ⇨					
- maximum testing rate (nr test/hour)					

(*) e.g. alone / combined

Enclosed documents as proof of the three required national approvals :

Countries (name)			
Evaluation centers/organisations (name)			
Official national approval certificates (doc n°)			
Technical reports (doc n°)			

Date: 20….

Signature :

Return to: **ICAR Secretariat, ServiceICAR, Via g. Tomassetti 3,I- 00161 Rome, Italy**
Tel : +39/ 0644 20 26 39– Fax : +39/ 06 44 26 67 98 – e-mail : icar@icar.org

Annex E

- **Summary form for assessment results of a milk analyser evaluation –**

Resquesting organisation :
Instrument / Type / Manufacturer : / / **Animal species :**

	Evaluation 1			Evaluation 2			Evaluation 3								
Evaluation centre Country															
Reference method															
Evaluation criteria (units)	**ESTIMATED VALUES FOR STATISTICAL PARAMETERS OF THE EVALUATION**														
	FAT (g/100 g)			**PROTEIN (g/100 g)**			**LACTOSE (g/100 g)**			**UREA (mg/100 g)**			**SOMATIC CELLS (1000 cells/ml or %relative)**		
	Eval 1	**Eval 2**	**Eval 3**	**Eval 1**	**Eval 2**	**Eval 3**	**Eval 1**	**Eval 2**	**Eval 3**	**Eval 1**	**Eval 2**	**Eval 3**	**Eval 1**	**Eval 2**	**Eval 3**
Range : min. – max. **Mean of reference values $\bar{y}$** **SD of reference values s_y**															
Carry over ratio															
Linearity Δe/ΔL															
<u>Repeatability</u> **Average SD : s_r** **Relative s_r :** **Average s_r %** **Low level s_r %** **Medium level s_r %** **High level s_r %**															
<u>Within lab reproducibility</u> **Average SD : s_R** **Relative SR :** **Average s_R %** **Low level s_R %** **Medium level s_R %** **High level s_R %**															
<u>Accuracy</u> **Animal samples $s_{y,x}$** **N^{ber} animal samples Na** **N^{ber} herds N_{h1}** **Herd samples : $s_{y,x}$** **N^{ber} herd samples : N_{h2}**															
<u>Calibration</u> **Mean bias : $\pm \bar{d}$** **Slope : $b \pm s_b$**															

Annex F

Examination Committee of Milk Analyser Evaluation Reports

- Reporting form for examiners -

Name of the examiner : **Country :**

Date of the examination :

Instrument / Type / Manufacturer : / /

Animal species :

Milk component(s) :

Specific comments :
1- Daily precision (repeatability and short-term stability) :
2- Carry-over effect :
3- Linearity :
4- Measurement limits (lower and/or upper limits) :
5- Repeatability :
6- Accuracy / Trueness :
7- Ruggedness :
8- Practical convenience :

Advice of the expert : **1- Valid for approval :** Yes / No **2- Invalid for approval :** Yes / No

Comments : (i.e. justification of negative position / advice for manufacturer, …)

Annex G

- REQUEST FORM FOR ICAR ADVICE ON EVALUATION TYPE –

Requesting organisation (name): ……..…… **Country:** .....….....

Address: .....……..... **Phone:** .....….....

.....……..... **Fax:** .....….....

.....…….. **E-mail :** .....….....

represented by (Mr, Mrs) : ……..……..…… **Function :** .....….....

hereby

makes the request to ICAR to advise on the suitable protocol to apply, in the frame of ICAR international approval, for the evaluation of the milk analyser designated below for the application of milk recording with the following specifications and the technical documentation included :

Manufacturer (name)	:
Instrument (name)	:
- Type	:
- Configuration (*)	:
- Analytical principle	:

Animal species	Cow	Sheep	Goat	Buffalo	Other
- milk components / criteria tested : ⇨ Fat (F) ⇨ Protein (P) ⇨ Lactose (L) ⇨ Urea (U) ⇨ Somatic cells (SCC) ⇨ ⇨					
- maximum testing rate (nr test/hour)					

(*) e.g. alone / combined

Date: 20….

Signature :

Return to: **ICAR Secretariat, Villa del Ragno, Via Nomentana 134, I-00162 Rome, Italy**
Tel : +39/ 06 86 32 91 41 – Fax : +39/ 06 86 32 92 63 – e-mail : **icar@eaap.org**

(Part reserved to the ICAR reply)

ICAR recommends to apply the protocol(s) related to ticked in square(s) in the following :

2.1 Routine devices ☐ 2.2 Manual devices ☐ 2.3 Updated devices ☐

Additional comments, recommendations :

……

……

Date: 20….

Signature :