FOSS

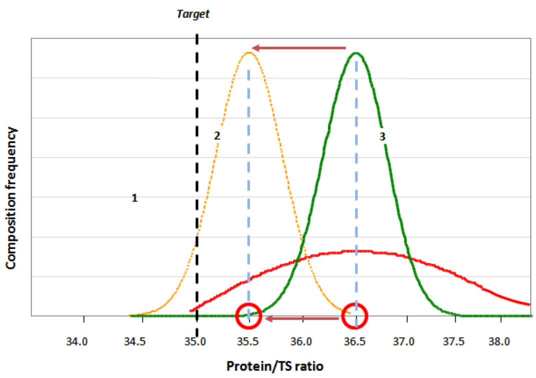
Application Note

AN 0040

Rev. 3

ProFoss™

In-line WPC/MPC Calibration Model



Continuous in-line process monitoring in Whey Protein Concentrate (WPC) and Milk Protein Concentrate (MPC) processing is becoming increasingly important. Consistent and high-quality standardized products manufactured at low cost are a key goal for competitive success in a global market.

The ProFoss helps you achieve this goal by reducing protein and Total Solids (TS) variation and streamlining your manufacturing process. The solution provides you with fast, accurate, and continuous ´real-time` results for critical process parameters like Protein, TS, Protein/TS ratio and Fat content.

Continuous in-line analysis of the Protein and TS content in the WPC in the outlet of the ultrafiltration process will give you the possibility to control the final product protein concentration closer to the specification.

You can use the results either for manual adjusting the production process or for automatic control by interfacing the ProFoss with your automatic process regulation system.

Using ProFoss the operator or the automatic process regulation system can immediately react to changes in the raw material or filtration process.

With the ProFoss system, you can run your process with a protein/TS ratio production target much closer to the product specification giving you an increased raw material utilization and with a consistent product quality over time.

With the ProFoss solution you can:

- Track exactly how your process is performing instead of waiting hours for results from standard wet chemistry analysis in the laboratory.
- Control the manufacturing process to a precision limited only by your control system
- Detect not only the actual process situation but also follow trends and predict possible future out-of-specification situations and react before they actually happen
- Identify what you have in the process line at any time – product type, quality, etc. thanks to the unique ProFoss qualification software. In-line process qualification is a 'lifeguard' to the production and for preventing unreliable results to be used by your process regulation system.

In-line Performance Evaluation

This application note describes the results that can be expected when using the ProFoss solution for in-line analysis of WPC or MPC in the outlet of the filtration unit.

For an in-line analysis solution it is important to measure the product composition level as fast and precise as possible. But equally (or perhaps more important) is the ability of the system to detect process variations (trends) fast and reliably. The smaller the variation that can be detected (Process Variation Detection Limit - PVDL), the better process regulation can be applied and the production target can be moved closer to the product specification.

As measurements on the ProFoss are made at such short time intervals, corrections to production can be made much faster than with process monitoring using manual sampling and laboratory analysis where a time lag of several minutes or even hours is common. Adjustment of a process is seldom based on a single sample manually collected - re-testing is required and this further increases the time lag in the regulation system.

In-line measurements also minimize possible sampling errors, and the effect of short term production variations no longer have significant influence on the overall performance.

Using ProFoss the true product concentrations and trends are obtained instantaneously.

The lowest concentration level change that a process regulation system can react on is the process detection limit of the monitoring system. We can define the process variation detection limit as the repeatability of the monitoring system. As a traditional repeatability cannot be calculated on an in-line process instrument (different samples all the time) the best measure of repeatability is the standard deviation of differences between adjacent results – this is an estimate of the smallest process changes that can be detected by the analyser. This figure is often 5 – 10 times smaller than the figure for the calibration model accuracy calculated as Standard Error of Prediction (SEP).

The PVDL has been estimated based on the variance between 2 adjacent moving average measurements for a period of several hours where the production process is stable.

In the table below, a calculated PVDL in terms of standard deviation of differences is given for the parameters including in the calibration models.

Process Variation Detection Limits

Component	PVDL
Protein	0.034%
TS	0.064%
Fat	0.013%

Table 1 Process variation detection limit

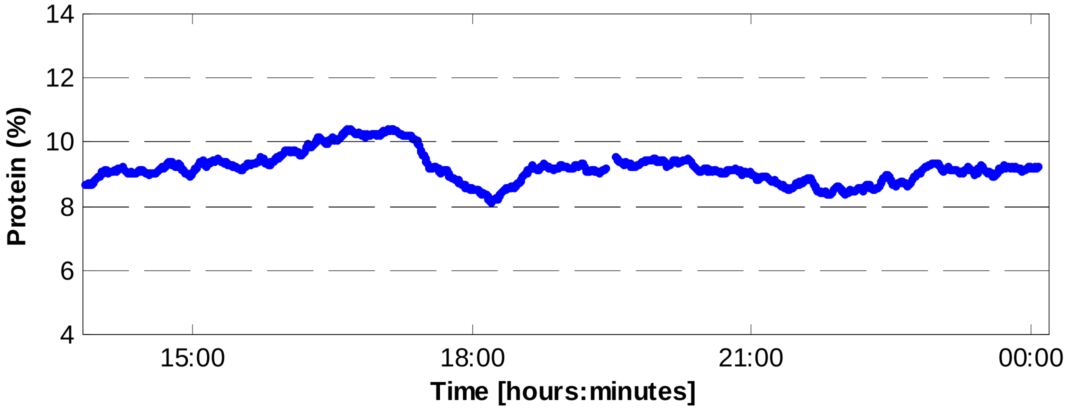


Fig. 1 Typical protein measurement over a 10 hour period

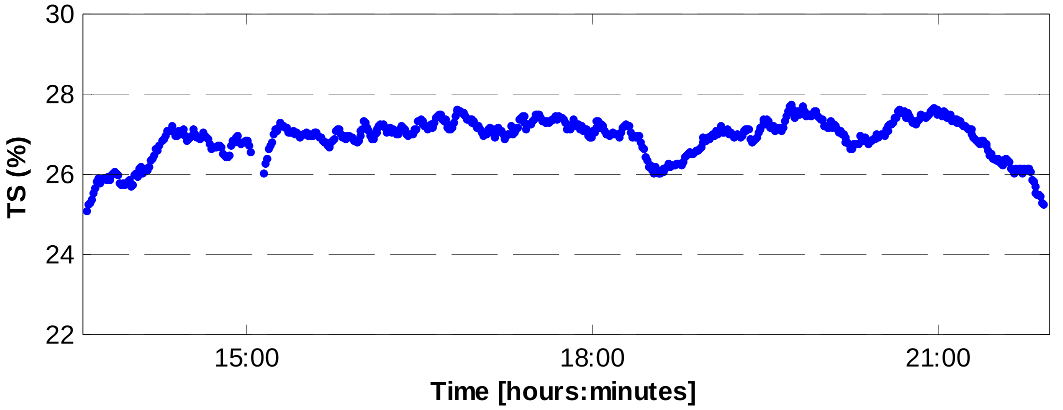


Fig. 2 Typical TS measurement over a 8 hour period

Samples Used in the Calibration

The calibration is based on ProFoss WPC and MPC data collected in-line in the outlet of the filtration unit. The calibration can be used for WPC and MPC and contains calibration for Protein, TS and Fat. The concentration range included in this calibration can be seen in the table below.

Component	N	Min %	Max %
Protein	8285	1.11	35.7
TS	8205	6.50	57.3
Fat	2834	0.02	2.77

N: Number of samples in the calibration set.
Min %: Minimum concentration in the calibration set.
Max %: Maximum concentration in the calibration set.

Table 2 Calibration sets

Performance

The calibrations for Protein, TS and Fat were developed using an ANN modelling.

The performance was evaluated using independent validation sets and the results are presented in the table below.

Calibration version: ProFoss WPC/MPC vers 3000.

Component	Model	N	Acc. %	Min %	Max %	RSQ
Protein	ANN	579	0.47	1.11	35.7	0.99
TS	ANN	544	0.68	6.47	57.3	0.99
Fat	ANN	240	0.17	0.02	1.99	0.81

Model: Artificial Neural Network (ANN).
N: Number of independent samples in the validation set.
Acc.: Independent test set accuracy expressed as Standard Error of Prediction (SEP(C)) corrected for bias (1 SD absolute) [1].
Min.: Minimum concentration in the validation set.
Max.: Maximum concentration in the validation set.
RSQ: Linear correlation between ProFoss result and reference result.

Table 3 Calibration performance

[1] Accuracy of individual sets will depend on sampling, sample handling, reference method used and range. The performance example outlined in this note should only be regarded as a guideline for the expected performance of new installations. The performance of new installations will always depend on the uniformity of the flow and homogeneity of the product, as well as the reproducibility of the reference method used to verify the performance. An indication of the obtainable performance can be found as approximately 2 times square root of the square sum of the sampling error and the intra laboratory reproducibility of the reference method.

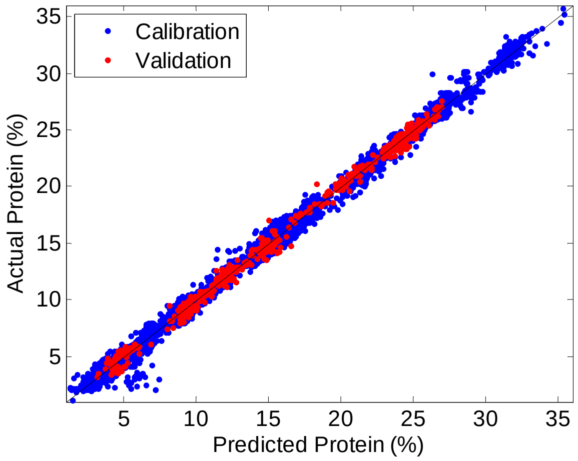
General

The performance of the calibration has to be validated with your samples (minimum 25 samples with reference values) following the International Standard IDF 201/ISO 21543 – "Milk products – Guidelines for application of near infrared spectrometry" or the FOSS ProFoss Operation and Performance Qualification documents.

If the samples you are measuring exceed the stated calibration validation ranges, or have non-common variations of other components, this might influence the performance of the calibrations.

The graphs below show predicted results versus the reference ("actual") values for the independent validation sets. The histograms to the right show the individual performance of each sample in the independent validation sets, expressed by its residual (reference results – predicted results).

Protein



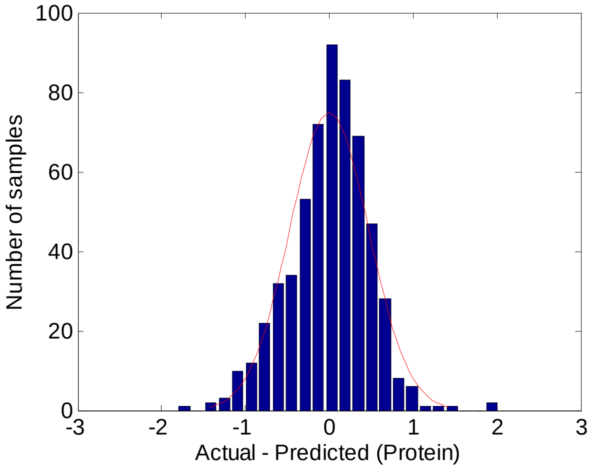
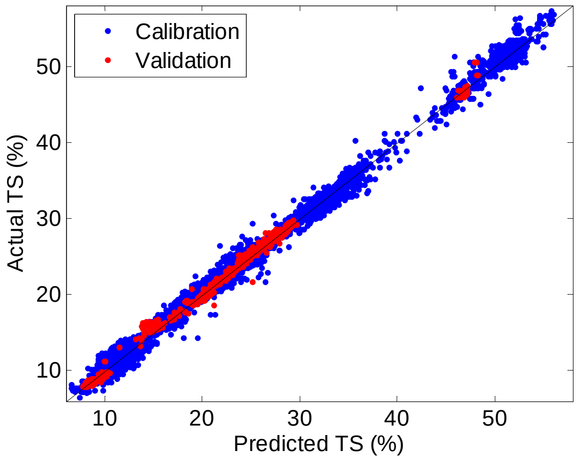


Fig. 3 Protein calibration and residual plot

Total Solids



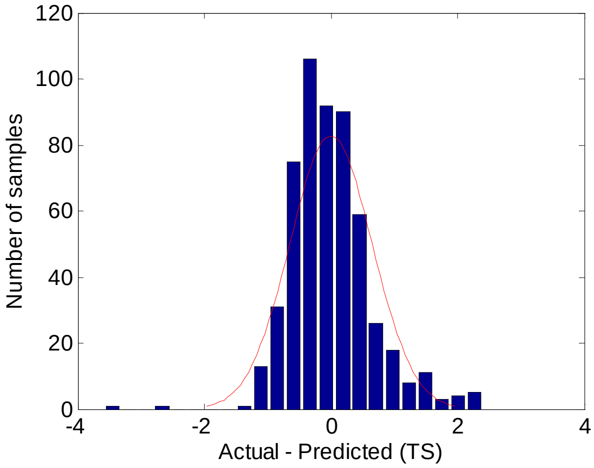


Fig. 4 Total Solids calibration and residual plot

Fat

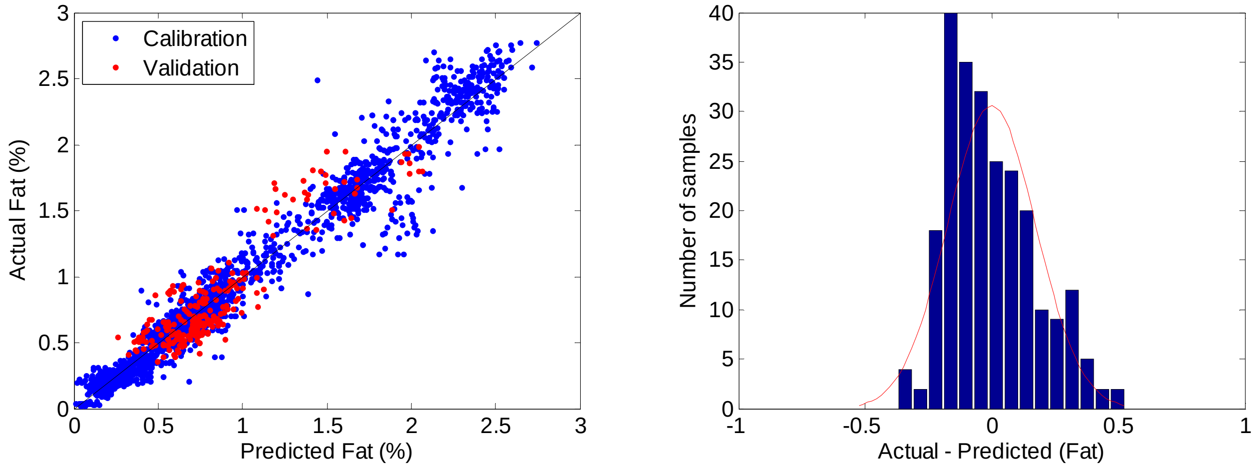


Fig. 5 Fat calibration and residual plot

The graphs below show in-line predicted results (blue line) compared to laboratory results (red).

Protein

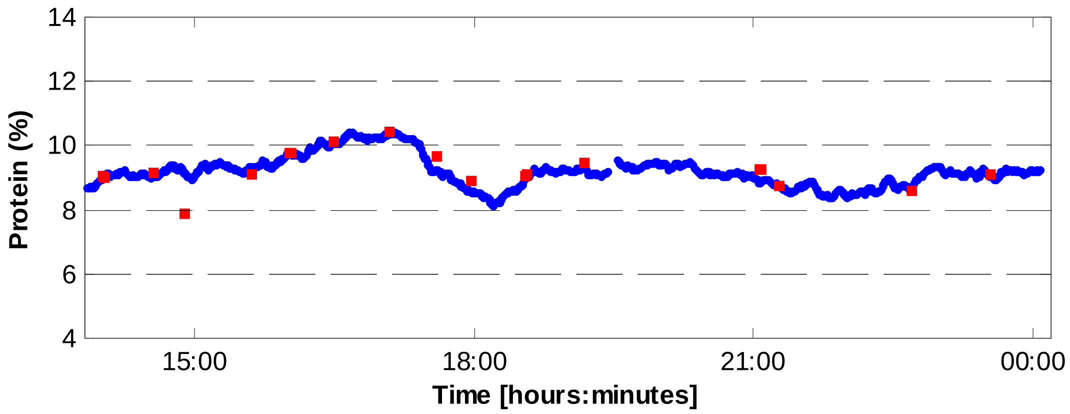


Fig. 6 In-line protein predicted results (blue line) compared to laboratory results (red).

Total Solids

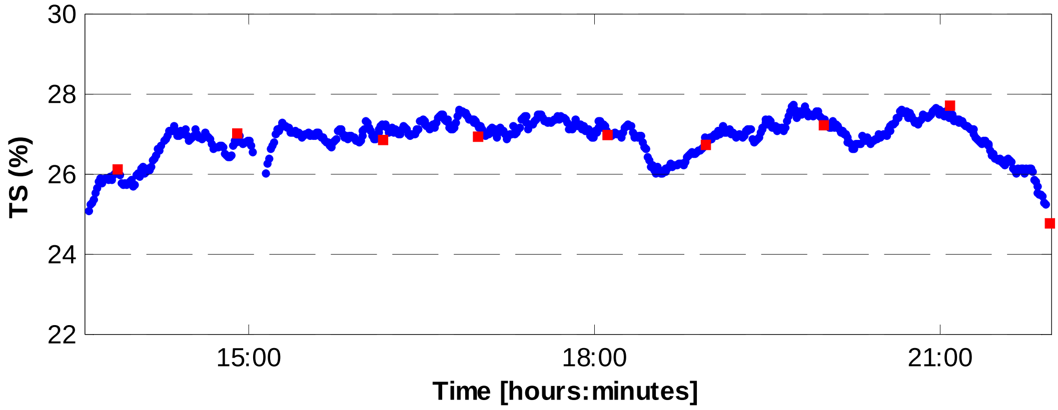


Fig. 7 In-line Total Solids predicted results (blue line) compared to laboratory results (red).

Fat

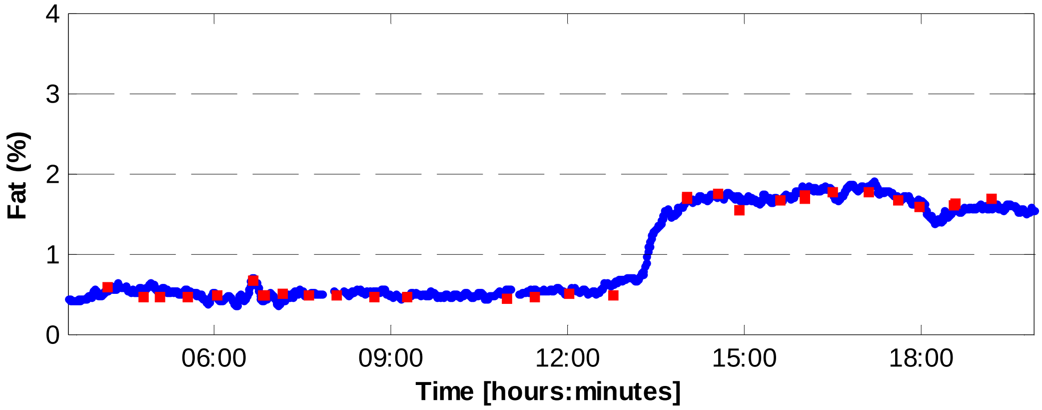


Fig. 8 In-line fat predicted results (blue line) compared to laboratory results (red).

Installation, Measuring Point, Sensors and Analysis

ProFoss is installed in the outlet of the filtration unit. Installation should preferably be made in a pipe with a constant upward flow

Measurements will be made with a lateral transmittance interface directly installed into the production line with a standard GEA-Tuchenhagen Access Unit.

For detailed information see the ProFoss WPC & MPC installation guide.

Reference Analysis Method

We recommend you to evaluate the performance of the calibrations using the joint ISO/IDF standards methods according to the procedure described in IDF201.

The test sets used for evaluation of the performance were analysed by means of the following methods:

- Sampling — ISO707/IDF50. Milk and milk products – Guidance on sampling.
- Validation — ISO21543/IDF 201: 2006. Milk products – Guidelines for the application of near infrared spectrometry.
- Total Solids — ISO 5534/ IDF 21B:1987. Milk, cream and evaporated milk - Determination of total solids content (reference method)
- Fat — IDF 13C:1987. Evaporated milk and sweetened condensed milk – Determination of fat content – Röse-Gottlieb (reference method)
- Protein — ISO 8968/IDF 20. Milk – Determination of nitrogen content.

If an indirect reference method such as the MilkoScan is used for calibration development, it has to be fully calibrated and validated against a primary reference method.

It is always recommended to use a primary method for calibration validation and calibration surveillance.

Ordering Information

- P/N 60042675 — ProFoss Calibration WPC/MPC Protein, TS and Fat.

Revision History

Rev.	Date of Issue	Revised Material	Approved by
1	2010-05-12	First issue.	IH
2	2010-12-08	Protein, Moisture and Fat Calibration update.	IH
3	2015-01-20	Protein, Moisture and Fat Calibration update.	IH

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