

The Hand in Hand Solution for Milk and Dairy Products

Relevant for: Dairy industry

Dairy products are subject to the strongest quality control regulations throughout production and in the packaged product. Both Anton Paar's laboratory and process instrumentation are reliable solutions to fulfill these high demands.



1 Introduction

Milk and dairy products are very complex substances that are composed of an expansive diversity of different molecular species. Being the product of mammary glands, milk is the primary food source for neonatal mammals. The milk from cows is the main source of dairy products and will therefore subsequently be looked at in more detail.

Many factors affect the composition of raw milk such as the cow's breed, age, physical state and seasonal variations in the animal's diet [1]. Therefore, only an approximate milk composition of 87 % - 88 % water and 12 % - 13 % total solids can be given. The total solids consist of approx. 4 % fat and 9 % solids-non-fat (SNF) such as proteins, lactose, minerals, vitamins, and many more [2].

2 Why density measurement?

The determination of physical parameters, such as the density of milk, plays an important role in the dairy industry. The density measurement of raw milk with Anton Paar density meters has been successfully used for quality control for many years [3]. Milk is composed of many different constituents with different densities. The density measurement of milk proves useful as a fast and precise method for the detection of deviations of milk composition, e.g. the addition of water.

Density measurement is a simple and robust method which provides a simple analysis parameter which is measured in process as well as in the laboratory.

The oscillating U-tube principle, or digital density measurement, is applied in Anton Paar density meters. It replaces older methods, such as hydrometers, pycnometers and lactometers and is recognized by many agencies as the laboratory standard for good density measurement.

The advantages of digital density instruments are:

- Highest level of accuracy
- Ease of operation
- Rapid results
- Robust, long life.

3 The density of milk

The density of raw milk depends on its composition, temperature and previous handling and can usually be found in the range of 1.026 g/cm^3 - 1.034 g/cm^3 at 20°C , although literature data varies.

The inclusion of air strongly influences the density of milk and dairy products. Included air is "trapped" in viscous dairy products like yogurt and escapes only very slowly or not at all. The amount of dissolved air in fresh milk is around 6 %, but may be up to 10 % after transport [4]. This entrapped air may influence the density of milk and dairy products and lead to erroneous measurement results and bad repeatabilities. Thus, samples are pretreated before measurement for consistent results.

A phenomenon observed in 1883, named after Recknagel, describes the fact that milk density increases slowly after milking by up to 0.001 g/cm^3 . This increase takes up to two days at 15°C but completely stops at 5°C after only six hours. The density increase is attributed to the removal of air and the slow solidification of milk fat [5]. As a consequence, small differences in density may be observed due to the temperature history of the milk or dairy product. For example, different densities may be found for the same sample, depending on whether it was held at 40°C or 20°C before measuring at 20°C .

Table 1: Density of various dairy products as a function of fat and solids-non-fat (SNF) content [6]

Product	Product Composition		Density [g/cm ³] at			
	Fat [%]	SNF [%]	4.4 °C	10 °C	20 °C	38.9 °C
Producer milk	4.00	8.95	1.035	1.033	1.030	1.023
Homogenized milk	3.6	8.6	1.033	1.032	1.029	1.022
Skim milk, pkg	0.02	8.9	1.036	1.035	1.033	1.026
Fortified skim	0.02	10.15	1.041	1.040	1.038	1.031
Half and half	12.25	7.75	1.027	1.025	1.020	1.010
Half and half, fort.	11.30	8.9	1.031	1.030	1.024	1.014
Light cream	20.00	7.2	1.021	1.018	1.012	1.000
Heavy cream	36.60	5.55	1.008	1.005	0.994	0.978

Table 1 [6] gives an overview of the density results of various dairy products as a function of fat and the solids non-fat (SNF) content, obtained at different temperatures.

4 The temperature dependence of milk

It can be seen from **Table 1** the density of milk decreases with increasing temperature. The higher the fat content of milk, the more the density changes with increasing temperature because the volume of fat changes more with temperature than the volume of water. The temperature coefficient of milk is in the range of 0.003 g/(cm³K), the temperature coefficient of cream is between 0.006 g/(cm³K) and 0.008 g/(cm³K).

5 The calculation of total solids in milk

As mentioned before, raw milk contains approx. 12 % to 13 % total solids (TS) that consist of approx. 4 % fat and 9 % solids-non-fat (proteins, lactose, minerals, vitamins, etc.). There is a direct relationship between milk density, fat content and solids-non-fat.

The fat content of milk is routinely determined in dairies. In combination with the milk density it can be used for the calculation of total milk solids (TS) according to the Fleischmann formula [7]:

$$TS [\%] = 1.2 * f + 266.5 * (SG - 1)/SG$$

TStotal solids content

ffat content

SG ... specific gravity SG15/15.

The formula is based on the specific gravity SG at 15 °C [8]. The fat content of milk can be determined in different ways, e.g. according to the Roesse Gottlieb reference method or the Babcock or Gerber tests (butyrometric procedure) [9]. Provided that the fat content of milk is known, the measurement results of the DMA™ density meter can be transferred to a PC program, such as Excel, via a LIMS system where the total solids can be calculated automatically.

6 Quality control of milk

As milk is a multi-component system it is not possible to determine the concentration of one component by density measurement alone. Yet, the density measurement of milk quickly indicates deviations from the normal milk composition due to the addition of water. The addition of 10 % water to the milk will result in a density decrease of approx. 0.003 g/cm³.

Considering the fairly large natural variations in milk composition, the addition of water to milk can only be detected by density measurement if at least 10 % water are added.

Skimming, i.e. the removal of fat, causes the milk density to increase. If skimming (causing a density increase) and water addition (causing the density to decrease) are done at the same time, a "normal" milk density can be observed. For this reason density measurement alone does not represent a method for quantitative quality analysis.

While Infrared Analysis (IR) is a well-established and widely used method for routine analyses of milk proteins, fat or carbohydrates [10], density measurement remains a very useful control method

for indicating deviations from the normal milk composition. Especially for quick quality checks of the delivered raw milk, the portable DMA™ 35 and the laboratory density meters DMA™ 501 and DMA™ 1001 are well suited.

7 Volume to weight conversion with proper milk density

On delivery, the volume of the milk is usually measured using volumetric flow meters. However, account settlement is often based on the weight. Additionally, for declarations of the package quantity by volume, the exact density is determined using an appropriate instrument.

For the conversion of volume to weight the density of the milk is required.

Mass = Density x Volume

Milk density is influenced by different factors. As the density of milk changes over time as mentioned before, milk density must be measured on the spot to calculate an average density that represents the actual conversion factor. Experimental results obtained in the laboratory under different conditions cannot be compared with on-the-spot measurements.

The on-site determination of density as a conversion factor from volume to weight can be carried out with the portable DMA™ 35 and the laboratory density meters DMA™ 501 and DMA™ 1001 as they measure milk density with high accuracy

To achieve the most accurate weight measurement, a combination of density and volumetric flow is used. Mass flow sensors, while very popular, are not the most accurate. The most accurate mass flow sensors claim an accuracy of $\pm 0.0001 \text{ g/cm}^3$, however this is for the top of the line sensor which will cost significantly more than a combination of density and volumetric flow meter.

Anton Paar process density sensors for milk and dairy applications provide a 4-digit accuracy. They offer, combined with a very compact yet precise volumetric flow sensor, highly accurate mass flow results at a very competitive price.

8 Density measurement during production and in the final package

8.1 General

Portable density meters have proven to be ideally suited for the initial quality control of delivered raw milk. However, for measurements in the laboratory and for products with higher viscosity values, bench-top density meters are better suited.

All subsequently described density meters provide integrated tables or formulas for the conversion of

density results to various concentrations and product specific parameters. These can be selected by the user for the measurement of specific samples. In addition to the large number of pre-installed conversion tables, custom specific measuring units can also be programmed. For example, for sweetened products, the relative Brix value can be determined directly.

8.2 Laboratory instruments

Anton Paar laboratory density meters have been used successfully for quality control of dairy products for many years. The relative density of milk, in combination with the fat content, can be used to calculate the total solids [11].

Considering the relatively large natural variations of milk density, an uncertainty of measurement of 0.001 g/cm^3 may be considered sufficient for routine measurements. For these applications, the density meters DMA™ 35 (**Figure 1**), DMA™ 501 or DMA™ 1001 (**Figure 2**) represent the ideal solution.



Figure 1: DMA™ 35 Portable Density Meter

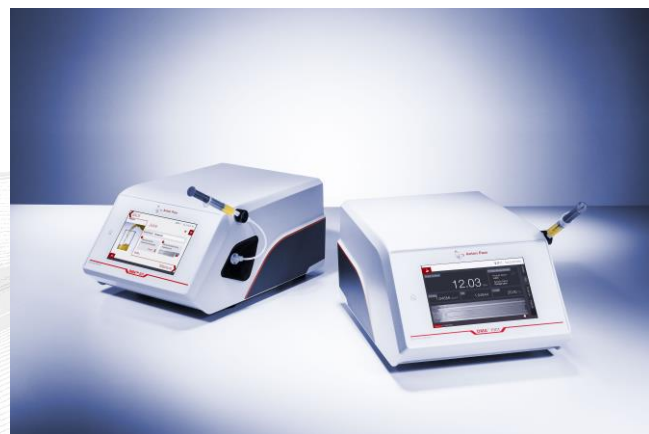


Figure 2: DMA™ 501 and DMA™ 1001 Density Meters

For university institutes, national dairy institutes and larger dairies the more accurate density meters such as DMA™ 4100 M, DMA™ 4500 M and DMA™ 5000 M [12] (**Figure 3**) might be required.



Figure 3: DMA™ 4100 / 4500 / 5000 M Density Meters

8.3 Process milk standardization using density measurement

Online density measurement is routinely used for process control in the milk industry. The process density sensor L-Dens 7400 (**Figure 4**) coupled with the mPDS 5 evaluation unit (**Figure 5**) is a most suitable solution for reliable continuous process monitoring due to their long and successful history in the milk industry.



Figure 4: Inline installation of the density sensor L-Dens 7400

Milk standardization is a process in which a specified amount of milk fat is added to skim milk to produce the desired product, i.e. whole milk, reduced fat milk, etc. The densities of the skimmed milk and milk fat are known. The change in density following addition of the milk fat is monitored and the fat content of the standardized milk is determined.



Figure 5: mPDS 5 Evaluation Unit

9 A new milk application from Anton Paar

9.1 General

A new application program developed by Anton Paar incorporates several standard milk industry formulas into the DMA™ M digital laboratory density meter and the process milk fat monitor (mPDS 5).

Together with the fat content obtained with the Gerber or Babcock tests, the total solids (TS), solids non-fat (SNF) and corrected lactometer reading (CLR) are calculated.

9.2 Laboratory solutions

In certain countries, milk farmers are paid based on the milk's fat and solid-non-fat content. Sometimes, milk collection centers use lactometers instead of density meters, but the use of a density meter proves more accurate, reliable, repeatable and user-friendly. Therefore, the readings between two different instruments have to agree and deliver uniform test results. This can be achieved with a customer specific method for DMA™ M (**Figure 6**).

The formula [13]

$$\text{SNF [\%]} = 0.25D + 0.22f + 0.72$$

for solid non-fat applies for liquid milk at 20 °C where

f = fat content in %w/w and

D = (1000*density-1000).

Formulas for the calculation of SNF can also be found elsewhere [14]. The milk fat is determined in the laboratory with the Gerber or Babcock test and subsequently entered into the DMA™ M for the SNF calculation (Figure 6). If the milk is warmer or colder, the SNF has to be corrected adding to D a factor of 0.24 for each degree above 20 °C and subtracting 0.24 for each degree below [13].

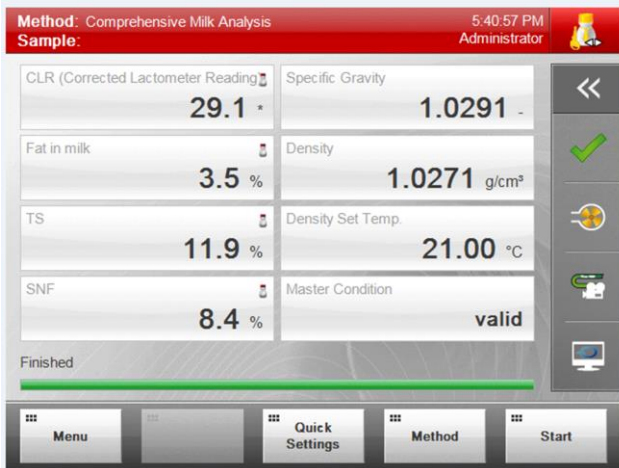


Figure 6: DMA™ M Milk fat Corrected Lactometer Reading

9.3 Process solutions

As described above, the fat content determined in the laboratory is entered into the DMA™ M, and the CLR, TS, SNF and SG are automatically calculated and displayed. Anton Paar process instrumentation is able to perform the same calculations as shown in Fig. 7, without the need to enter the laboratory milk fat content. This is accomplished by performing a skim milk adjustment.

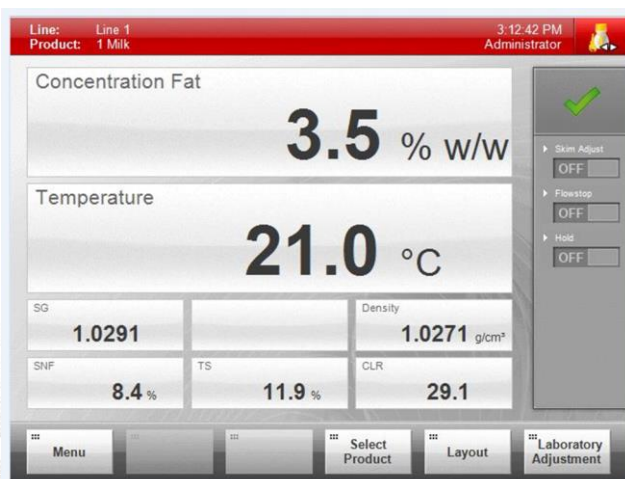


Figure 7: Screen shot of an mPDS 5 Evaluation Unit

The calculation of the fat content of cream/whole milk is based on the density difference between skim milk and cream/whole milk. As the density of skim milk changes with seasons and depends on the supplier of the raw milk, it is necessary to carry out a skim milk adjustment on a regular, automated basis.

To accomplish this, the separator is briefly set to remove all fat from the milk and skim milk is run through the L-Dens 7400 density sensor for 1 minute. Once the value is stable, the temperature and density are automatically stored, the separator returns to normal production and the density difference is monitored to calculate the fat content. Knowing the temperature, density and fat content, all other values are calculated; density at 20 °C, density at a customer specific temperature, specific gravity, solids-non-fat (SNF), corrected lactometer reading (CLR), and total solids (TS). Because both lab and process measurements use the same method, results are comparable, repeatable and reliable.

The frequency of the skim milk adjustment must be found out empirically. As long as the deviation to the laboratory reference lays within acceptable limits no skim milk adjustment is necessary. Adjustment frequency depends on the customer, production size blending capabilities and milk quality and is typically performed automatically every 20-40 minutes. In addition to the skim milk adjustment, it is possible to enter a constant milk fat content for the calculations. A product specific adjustment is also possible.

9.4 A process example

The raw milk flows to the separator and the pasteurizer and then to the milk or cream tank, respectively. The skim milk density and temperature are measured and the values stored automatically. Once the skim milk values are saved, the separator allows some of the cream to remain in the standardized milk and the density difference of the saved skim milk and standardized milk is used to calculate the fat content. If the fat content, density and temperature are known, the CLR, SNF and TS are calculated using standard industry formulas.

9.5 Lab and process working together

The following two examples illustrate the agreement between data obtained with laboratory and process instrumentation. The laboratory results for % SNF and % TS were recorded with a DMA™ density meter, the laboratory result for fat was obtained with a different method.

Figure 8 illustrates the calculated linear regression.

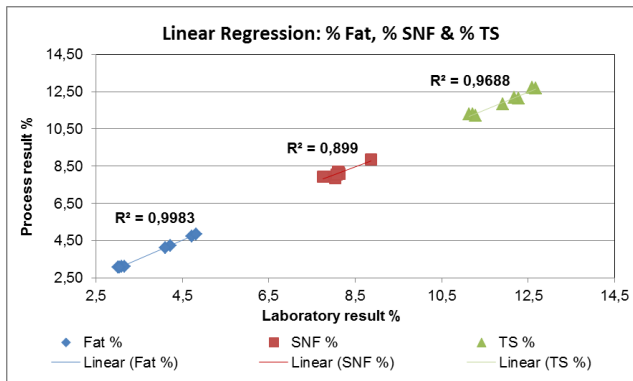


Figure 8: Calculated linear regression

Figure 9 compares laboratory and process data.

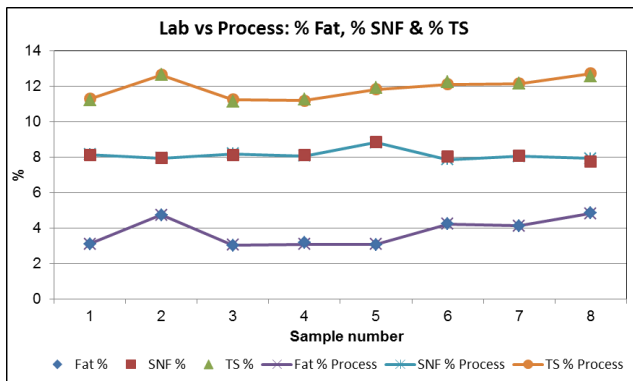


Figure 9: Laboratory and process data comparison

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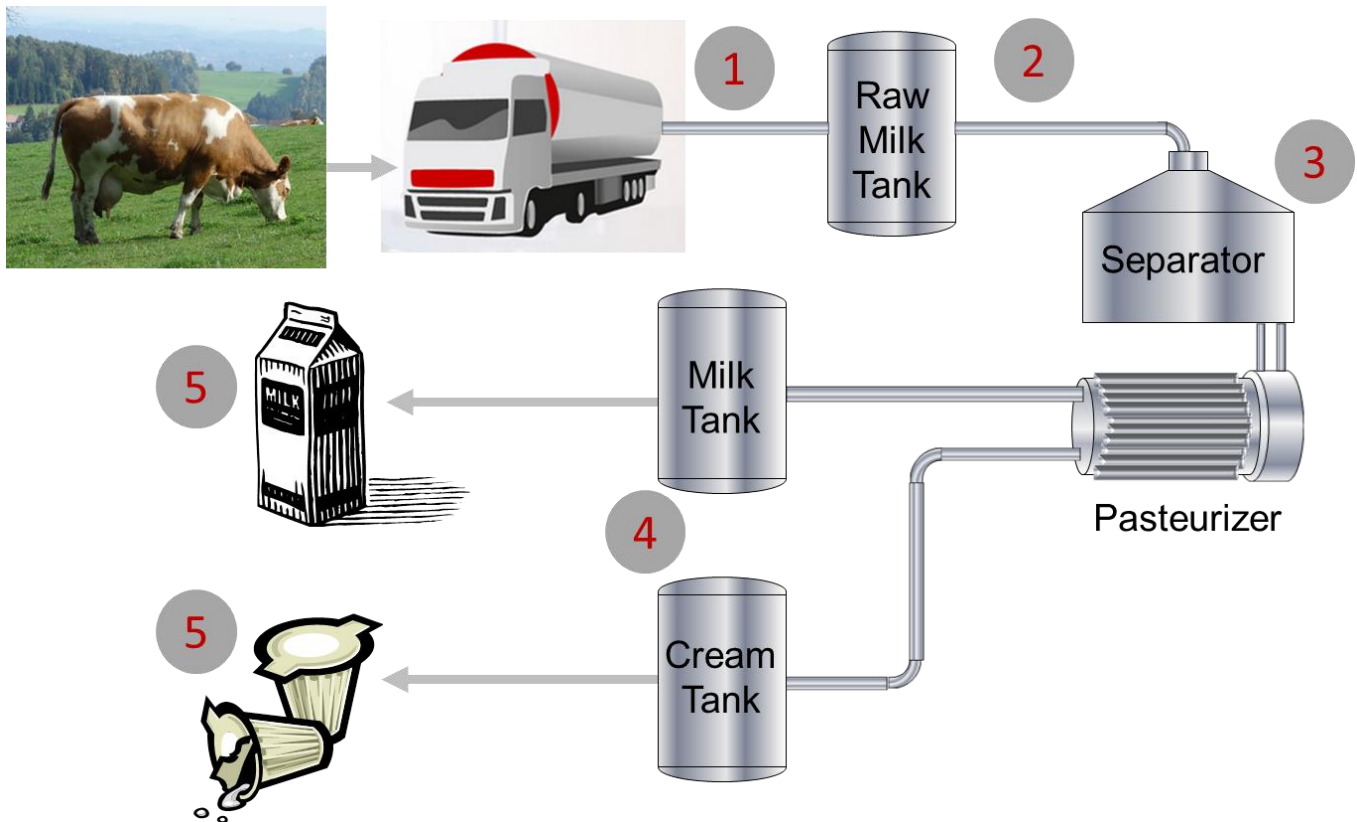
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Appendix: The milk production process



Measuring point 1: Raw milk receiving

Why? Adulteration test (addition of water)

What and how?

Laboratory instrumentation: Density with DMA™ 35, DMA™ 501, DMA™ 1001, and (with pre-determined fat content) **CLR, SNF, TS and fat** with DMA™ 4100/4500/5000 M

Process instrumentation: Density with L-Dens 7400; mPDS 5

Measuring point 2: Raw milk tank

Why? Quality check before processing

What and how?

Laboratory instrumentation: Density with DMA™ 35, DMA™ 501, DMA™ 1001, and (with pre-determined fat content) **CLR, SNF, TS and fat** with DMA™ 4100/4500/5000 M

Measuring point 3: Standardized milk process

Why? Process control, quality control and assurance

What and how?

Laboratory instrumentation: Density with DMA™ 35, DMA™ 501, DMA™ 1001, and (with pre-determined fat content) **CLR, SNF, TS and fat** with DMA™ 4100/4500/5000 M

Process instrumentation: Density and (with skim milk adjustment) **CLR, SNF, TS and fat** with L-Dens 7400, mPDS 5

Measuring point 4: Standardized milk tank, pre-packaging

Why? Quality control pre-packaging

What and how?

Laboratory instrumentation: Density with DMA™ 35, DMA™ 501, DMA™ 1001, and (with pre-determined fat content) **CLR, SNF, TS and fat** with DMA™ 4100/4500/5000 M

Measuring point 5: Packaged products

Why? Quality control of packaged products

What and how?

Laboratory instrumentation: Density with DMA™ 35, DMA™ 501, DMA™ 1001 and (with pre-determined fat content) **CLR, SNF, TS and fat** with DMA™ 4100/4500/5000 M