

**Milkotronic Ltd**

# **LACTOSCAN S\_LP**

**MILK ANALYZER**

**Wide LCD display – 4 lines x 16 characters**

**Plastic box**

**Operation manual**

## Switching Adapter

- **Input:** 100 - 240 V ~1.6 A max.  
50-60 Hz
- **Output:** +12 V  $\overline{\text{---}}$  3 A min.
- **Output power:** 36 - 42 W

## Measurement modes

- cow milk
- sheep milk
- UHT milk
- goat milk
- buffalo milk
- cream
- whey
- recovered milk
- other /pasteurized milk/

### **CAUTION!**

Keep the switching adapter dry!  
Please, read and follow strictly all the instructions in the manual.

Due to continuous improvement in the device, information contained in this manual is subject to change without notice. Contact the company-producer for revisions and corrections

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## **SAFETY INSTRUCTIONS**

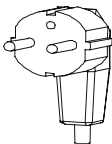
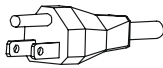
- 1. Read this manual carefully and make sure that you understand all the instructions.**
- 2. For safety purposes the device is equipped with grounded power cable. If there is no grounded electrical outlet where the device will be used, please, install such before using the device.**
- 3. Place the device on leveled and stable plate. In case it falls or is severely shocked it may be damaged.**
- 4. Connect to the electrical network in such a way that the power cable to stay away from the side for accessing the device and not to be stepped on.**
- 5. Every time before cleaning the device switch it off and unplug it from the electrical outlet. The device has to remain unplugged till the cleaning completion.**
- 6. Do not disassemble the unit in order to avoid possible electrical shock. In case of malfunction contact your local dealer.**
- 7. Handle the liquids the device works with carefully, following all the instructions for their preparation.**
- 8. Place the switching adaptor in such a way as to be protected from overflow and spillage of liquids.**

## PARTS AND ACCESSORIES

In the table below the standard delivery configuration of the milk analyzer is listed:

No	Description	Item No	pcs
1.	Ultrasonic portable milk analyzer	LSSLP001	1
1 sample measurement time		90 sec.	<input type="checkbox"/>
		60 sec	<input type="checkbox"/>
		30 sec	<input type="checkbox"/>
2.	Operation manual	LSSLP002	1
3.	Plastic sample holder	LSSLP003	2
4.	Spare Pipes	LSSLP004	2
5.	12 V DC Power Supply Cable	LSSLP005	1
6.	Alkaline cleaning solution Lactodaily	100 g	1
7.	Acidic cleaning solution Lactoweekly	100 g	1

In the table below the milk analyzer spares and accessories, which are delivered on customers request are listed:

No	Description	Item No	pcs	<input checked="" type="checkbox"/> / <input type="checkbox"/>
	a) included in the set: <input checked="" type="checkbox"/> b) not included in the set (may be additionally bought): <input type="checkbox"/>			
8.	RS232 Interface Cable - Analyser-IBM PC	LSSLP006		<input type="checkbox"/>
9.	Service Pack - CD	LSSLP007		<input type="checkbox"/>
10.	ECS POS Serial Printer	LSSLP017	1	<input type="checkbox"/>
11.	12 V Serial Printer Power Supply Cable	LSSLP018	1	<input type="checkbox"/>
12.	RS232 Interface Cable - Milk Analyser – Serial Printer	LSSLP019	1	<input type="checkbox"/>
13.	Plug type		1	<input checked="" type="checkbox"/>
			1	<input type="checkbox"/>

## **FUNCTION**

**The function of the milk analyzer is to make quick analyses of milk on fat (FAT), non-fat solids (SNF), proteins, lactose and water content percentages, temperature (°C), freezing point, salts, as well as density of one and the same sample directly after milking, at collecting and during processing.**

## **2. TECHNICAL PARAMETERS**

### **2.1. Working modes characteristics:**

The program of the milk analyzer has 5 (five) working modes.

#### **2.1.1. Measurement mode milk / dairy product – first type**

#### **2.1.2. Measurement mode milk / dairy product – second type**

#### **2.1.3. Measurement mode milk / dairy product – third type**

These modes have been calibrated on customers' request for 3 milk types from the following: cow, sheep, UHT, buffalo, goat, camel milk, cream, ice cream mixtures, whey, recovered milk, etc. before leaving the production facilities and the text on the display will be for the corresponding types, as is indicated on page 2 Measurement modes.

#### **2.1.4. Cleaning**

#### **2.1.5. Printing**

## 2.2. Measuring range:

Fat .....	.....from 0.01% to 25%
SNF .....	.....from 3% to 15%
Density * .....	.....from 1015 to 10 40 kg/m <sup>3</sup>
Proteins .....	.....from 2% to 7%
Lactose .....	.....from 0.01 % to 6 %
Water content .....	.....from 0 % to 70 %
Temperature of milk .....	.....from 1°C to 40°C
Freezing point .....	.....from – 0,400 to – 0,700°C
Salts .....	.....from 0,4 to 1,5%

\* Density data are shown in an abbreviated form. For example 27.3 have to be understood as 1027.3 kg/m<sup>3</sup>. To determine the milk density, write down the result from the display and add 1000.

Example: result 21,20; density = 1000 + 31,20 = 1031,2 kg/m<sup>3</sup>

The abbreviated form of the density is used also when entering data for samples in working mode **Recalibrate**, for example:

If the measured sample density is 1034.5 kg/m<sup>3</sup>, then in the menu for entering the samples parameters used for calibration, across the parameter Den = , you have to enter 34.5.

## 2.3. Maximum permissible absolute error:

Fat .....	.....± 0.10%
SNF .....	.....± 0.15%
Density .....	.....± 0.3 kg/m <sup>3</sup>
Proteins .....	.....± 0.15%
Lactose .....	.....± 0.20%
Water content .....	.....± 3.0%
Temperature of milk .....	.....± 1°C
Freezing point.....	.....± 0.001°C
Salts .....	.....± 0.05%

The difference between two consequent measurements of one and the same milk could not exceed the maximum permissible absolute error.

## 2.4 Correct ambient conditions:

Maximum permissible absolute error is guaranteed in case of normal ambient conditions:

Air temperature.....from 10°C to 40°C  
Relative humidity .....from 30% to 80%  
Power supply .....220V (110V)  
extent of contamination at normal environmental conditions.....2



Maximum permissible absolute error values in point 2.3 are in dependence on the correctness of the corresponding chemical method, used for component content determination. In point 2.3. are used the following reference methods: Gerber – for fat, gravimetric – for SNF, Kjeldahl – for protein. The boundary for maximum variation of repeatability when the power supply voltage is from +10 to – 15% from the nominal voltage values (220 V) have to be no more than 0.8 accuracy according point 2.3. The analyzer is used in conditions free of outer electrical and magnetic fields (except the magnetic field of the Earth) and vibrations.

## 2.5. Dimensions:

.....175/175/150 mm, mass 1,5 kg

## 2.6. Continuous working time:

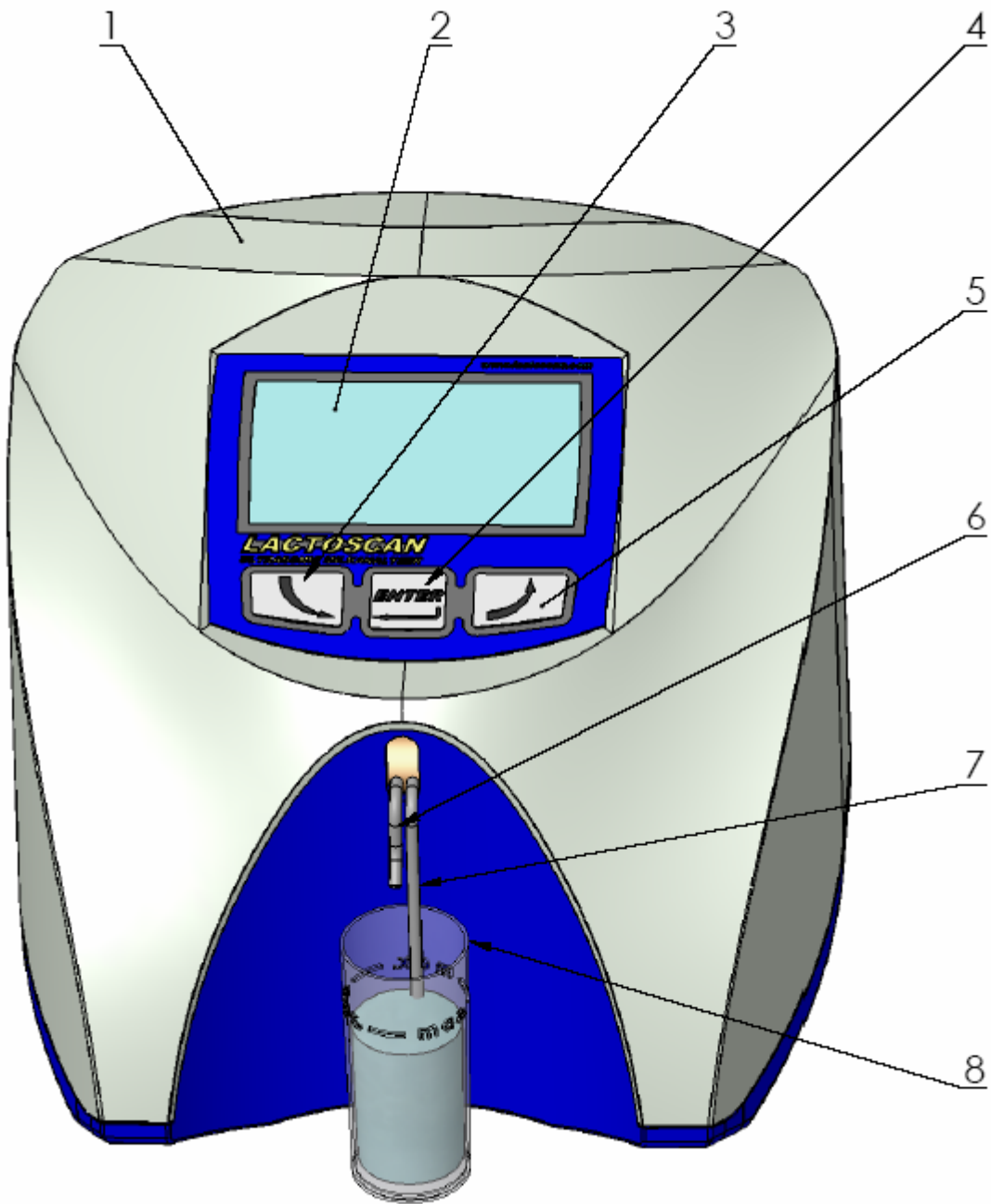
.....non-stop

## 2.7 Milk sample volume per one measurement:

.....15 cm<sup>3</sup> (= 25 ml)



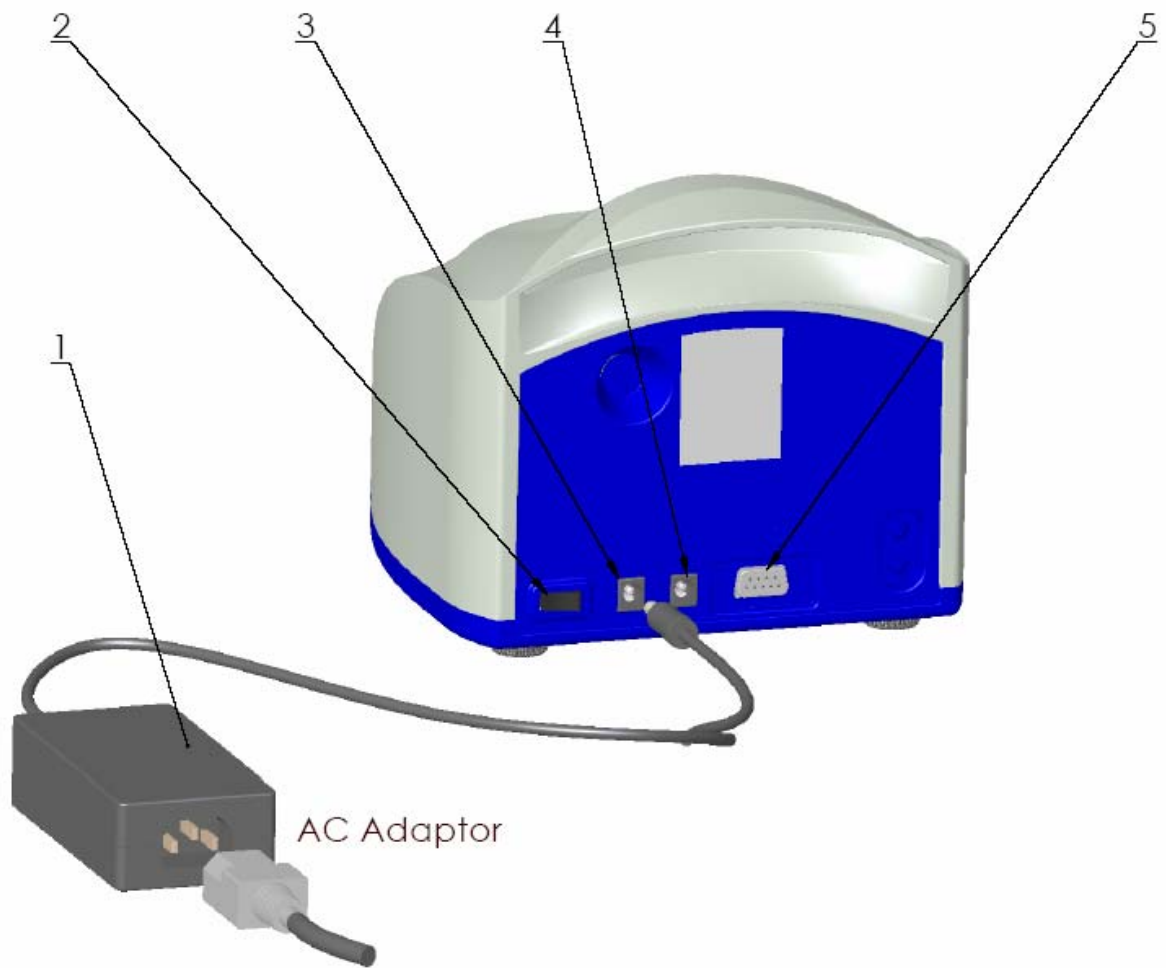
**Fig.1 Front panel**



- 1. Upper cover
- 2. Wide Display
- 3. Button "Down"
- 4. Button "Enter"

- 5. Button "Up"
- 6. Output pipe
- 7. Input pipe
- 8. Sample holder

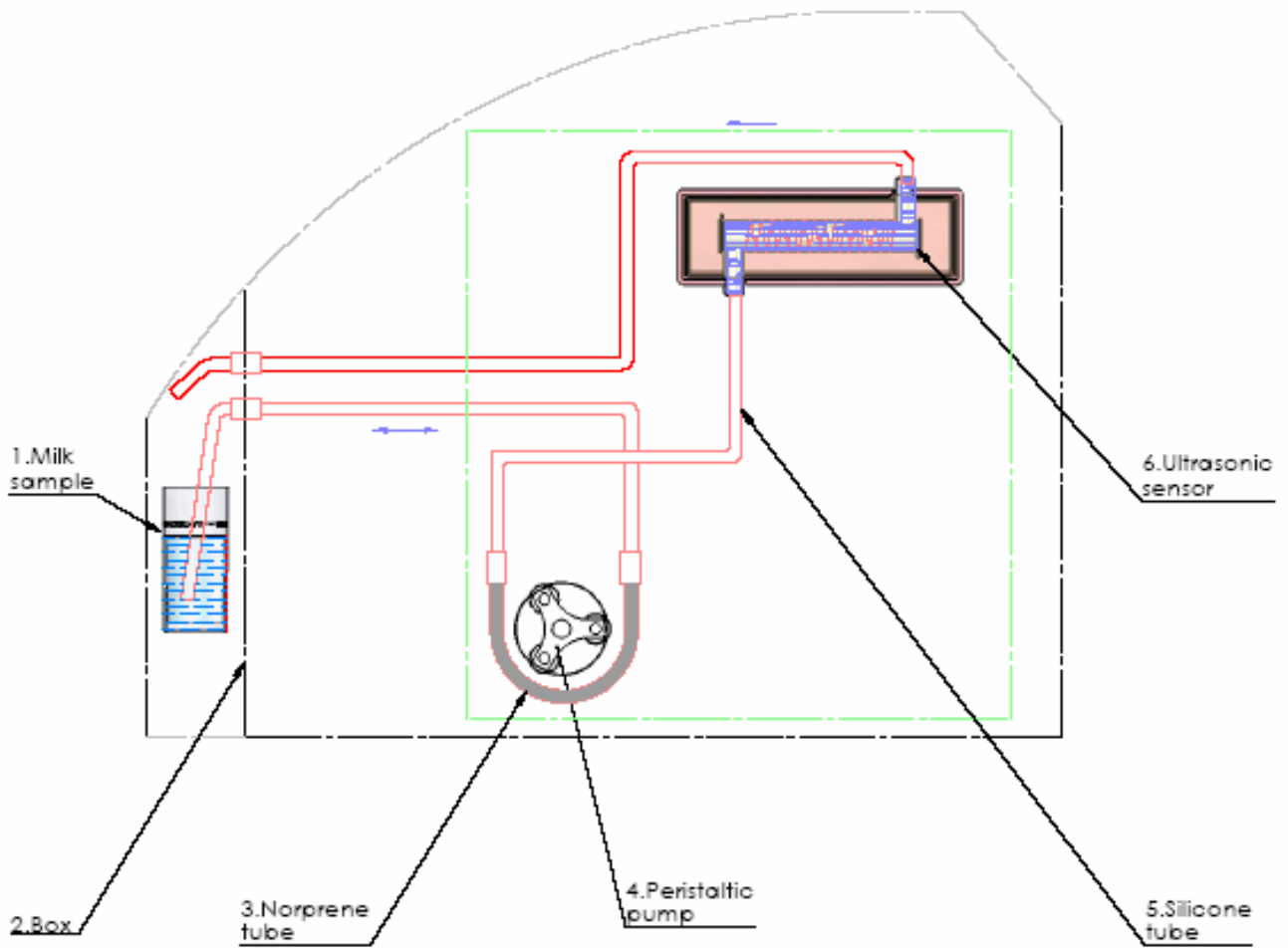
**fig. 2 Back panel**



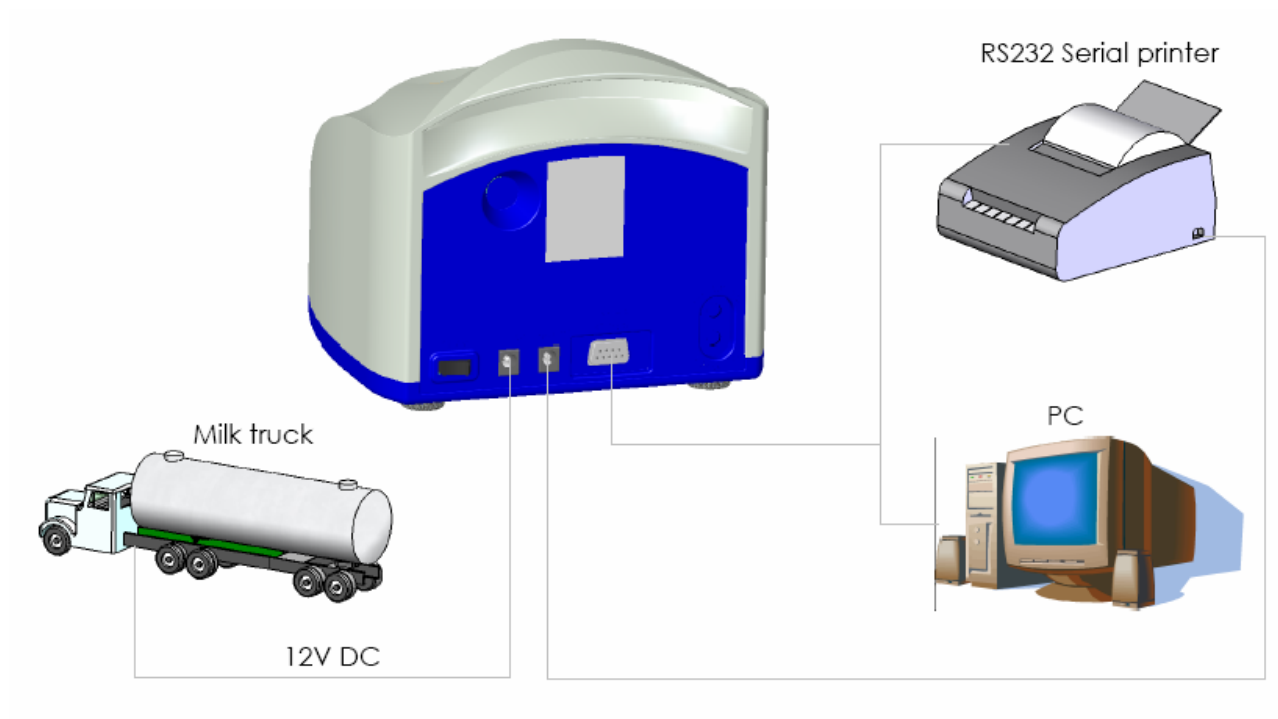
- 1 Switching adapter
- 2. Power switch
- 3. 12 V printer output

- 4. 12 V input
- 5. Serial interface (RS232/ printer)

**Fig. 3 Principle working scheme**



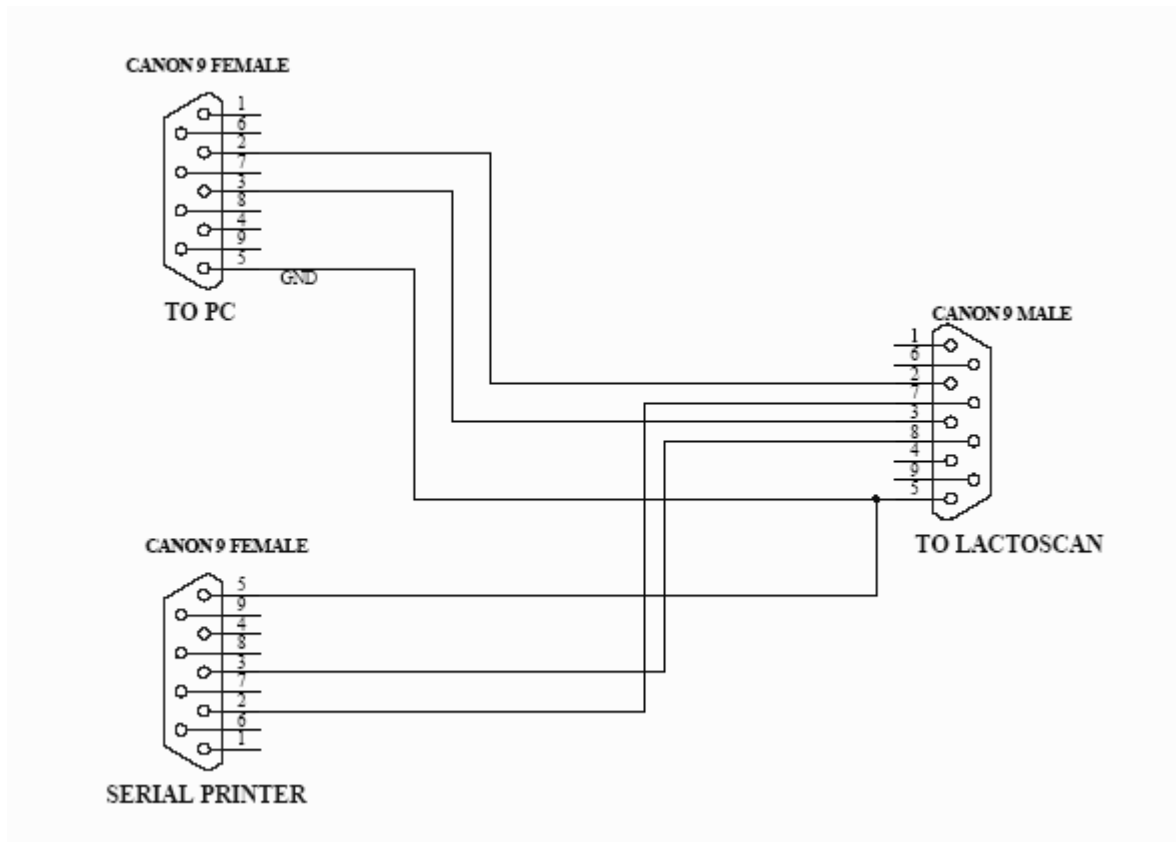
**Fig. 4**      **Peripherals connection**



## Fig. 5 Cable Description

### 90-1801-0008

RS232 Interface Cable - Milk Analyser – Serial Printer/ IBM PC



### 90-1801-0009

DC 12V Power Supply Milk Analyzer Cable

1. GND
2. No connection
3. No connection
4. 12V DC

### 3. QUALIFICATION OF RAW MILK, THERMALLY TREATED MILK, OTHER DAIRY PRODUCTS AND DERIVATIVES

#### 3.1. Taking samples and preparation for analyses

In order to receive reliable results in qualification of milk, dairy products and derivatives are needed: precise samples taking; correct samples storing (in need to be preserved); correct preparation before making measurement. The rules and requirements for this are described in details in *Appendix Preparing Samples*.

#### 3.2. Making the measurement.

##### 3.2.1. Preparing the analyzer for working mode

**3.2.1.1.** Put the analyzer on the working place, providing good ventilation and not in the vicinity of heat providing devices or sources. The temperature in the premises has to be in the boundaries 10-30°C.

**3.2.1.2.** Check if the power switch is in "0" position and that the outlet voltage complies with the voltage indicated on the rating plate of the analyzer. Connect the power supply cable to the electrical outlet.

**3.2.1.3.** Switch on the "**POWER**" button, which starts the identification procedure. For a short time the display shows the number of the software versions, for example:

**Milk analyzer xxx**  
**MB vers yy**  
**Ser. N. xxxx**

where:

**Milk analyzer xxx** is the time for measurement.

**MB vers YY** is the motherboard software version.

**Ser. N. xxxx** is the serial number – written on the rear panel of the analyzer.

These data are called analyzer's **Identity**



*If in the process of exploitation there is a need to ask a question the company-producer, you have to send the data, written on the display during the above described initialization procedure i.e. the analyzer's identity.*

**3.2.1.4.** Till the analyzer is prepared for work (at about 5 minutes) the following message is written on the display: “**Getting ready**”. Above pointed time is in dependence of the environmental temperature and increases with decreasing the temperature.

**3.2.1.5.** When the device is ready for work the display shows: “**Ready to start**”.

The analyzer is ready to make analyses in mode 1 (normally Cow)

**3.2.1.6.** If you want to pass to another mode press the button **Enter** and hold it pressed. The following message appears on the display:

**Release button to  
start menu**

Release the button **Enter**. The display shows the possible working modes:

**Milk selector**  
**Cal1 – Cow**  
**Cal2 – Sheep**  
**Cal3 – UHT**  
 -----  
**Cleaning**  
**Printing**

Using “up” ▲ and ”down” ▼ buttons, choose the working mode and press **Enter** in order to start it.

### **3.2.2. Making analyses**

To start measurement: pour the preliminary prepared sample in the sample holder of the analyzer; put the sample holder in the recess of the analyzer; press the button **Enter**.

The analyzer sucks the milk, makes the measurement and returns the milk in the sample-holder. During the measurement the temperature of the sample is shown on the display.

Ignore the results received immediately after switching on the analyzer and after measuring distilled water. Make a second measurement with new portion of the same sample.

### 3.2.3. Displaying the results

**3.2.3.1.** When the measurement is finished, the sample returns in the sample-holder and the display shows the results. For example:

<b>Results:</b> <b>F=ff.ff</b> <b>S=ss.ss</b> <b>D=dd.dd</b> <b>P=pp.pp</b> <b>L=ll.ll</b> <b>W=ww.ww</b>
--

Where:

<b>F= ff.ff</b>	- measured FAT in percentage;
<b>S= ss.ss</b>	- measured SNF in percentage;
<b>D= dd.dd</b>	- measured density in percentage;
<b>P= pp.pp</b>	- measured protein in percentage;
<b>L= ll.ll</b>	- measured lactose in percentage;
<b>W= ww.ww</b>	- measured sample's added water in percentage.

By pressing the button "Down" ▼ the display shows the second page, containing the results:

<b>Page 2 Results:</b> <b>T=tt.tC</b> <b>FP=-0.fff sol=0.sss</b>
--

Where:

<b>tt.tC</b>	- sample's temperature;
<b>-0.fff</b>	- measured sample's freezing point;
<b>0.sss</b>	- measured salts values;



By pressing the buttons “up” ▲ and ”down” ▼ , the operator has the possibility to pass from one page result to another.

**3.2.3.2.** Write down the results in the form. The results remain on the display till a new measurement is started. If the analyzer is connected to a computer or a printer, it sends the data to the computer or prints them.

### **Mode Printing**

Serves for control of the printing. There are 2 variants:

- after switching on the power supply of the device. Then the analyser’s parameters are printed (Identity).
- after completed measurement. Prints out the results from the last measurement.

## 4. CLEANING THE ANALYZER

### 4.1. Periodically cleaning (rinsing) the analyzer

It is done in the process of routine work of the analyzer. Its aim is to prevent drying up and adhesion of different milk components in the milk analyzer's measuring system.



The company-producer recommends usage of the chemicals, supplied with the analyzer – alkaline and acidic (Lactodaily and Lactoweekly). You may order them separately or together with the analyzer. Try to use only these chemicals for cleaning the analyzer.

In case you missed to order these chemicals the alternative is to use alkaline and acidic cleaning solutions for dairy, produced by one the companies, supplying such chemicals:

<http://www.johnsondiversey.com/Cultures/en-GB/OpCo/Your+Business/Dairies.htm>

[http://www.ecolab.com/SolutionGuide3.asp?name=1345&PS\\_GRP\\_CD=FDBV&BUS\\_NM](http://www.ecolab.com/SolutionGuide3.asp?name=1345&PS_GRP_CD=FDBV&BUS_NM)

[http://www.calvatis.com/documents/products/produkte\\_farm\\_melkhygiene.pdf](http://www.calvatis.com/documents/products/produkte_farm_melkhygiene.pdf)



Do not use chemicals not intended for usage in the milking systems or vessels in the dairy sector. Pay special attention to the concentration of the acidic chemical. **Increased concentration may damage the measuring sensor.**

#### 4.1.1. Periodical cleaning frequency.

It is easy to understand what is the period on which the rinsing could be done as the analyzer reminds you when it is necessary. This is done by a sound signal in 1-second cycle after the set time intervals elapse:

- 55 min. after switching on the power supply of the analyser, but idle work;
- 15 min. after the last measurement of real milk sample.

After cleaning completion, new measurement takes place in above described time intervals.

The following message appears on the display:

**Time to start  
cleaning**

#### 4.1.2. Making the rinsing

After above message is received put in the recess of the analyzer a glass filled with 120 ml water (in case 1 from p.4.1.1.) or alkaline cleaning solution (in case 2 from p.4.1.1.).

Press **Enter** to start the rinsing mode.

In this mode the analyzer makes 3 cycles and stops.

Already used solution is poured out of the analyser. Now the device is ready for the next measurement. In case of doubt that the analyzer is still not well cleaned, the procedure Cleaning may be executed repeatedly.

### 4.2. Complete cleaning

#### 4.2.1. Complete cleaning frequency

This cleaning is done after finishing the work with the analyzer at the end of the working day or if it is obvious that the measuring system of the analyzer is contaminated in case of intensive work with it. It is done with alkaline cleaning solution.

Preparation of 1 % alkaline solution of Lactodaily for circulation cleaning in the milk analyzer:

1. Take the package 100 g concentrated powder chemical Lactodaily
2. Carefully cut the upper end, paying attention not to spill it.
3. In appropriate vessel (for example bucket) pour 1 l water.
4. Add the powder and then again water up to 10 l.

Then follow the instruction for milk analyzer cleaning.

#### 4.2.2. Cleaning

##### 4.2.2.1. Rinsing milk residues

Fill in the glass with water. Put it in the recess of the analyser and start command Cleaning from the main menu. After finishing it pour out the contaminated water.

##### 4.2.2.2. Cleaning with alkaline cleaning solution

Fill in the glass with warm (50-60 C) alkaline cleaning solution. Put it in the recess of the analyser and start the command **Cleaning** from the main menu. After finishing it, pour out the contaminated liquid.

##### 4.2.2.3. Rinsing with water

Fill in the glass with water. Put it in the recess of the analyser and start command **Cleaning** from the main menu. After finishing it pour out the contaminated water. Now the device is ready for work.

##### 4.2.2.4. Cleaning with acidic solution

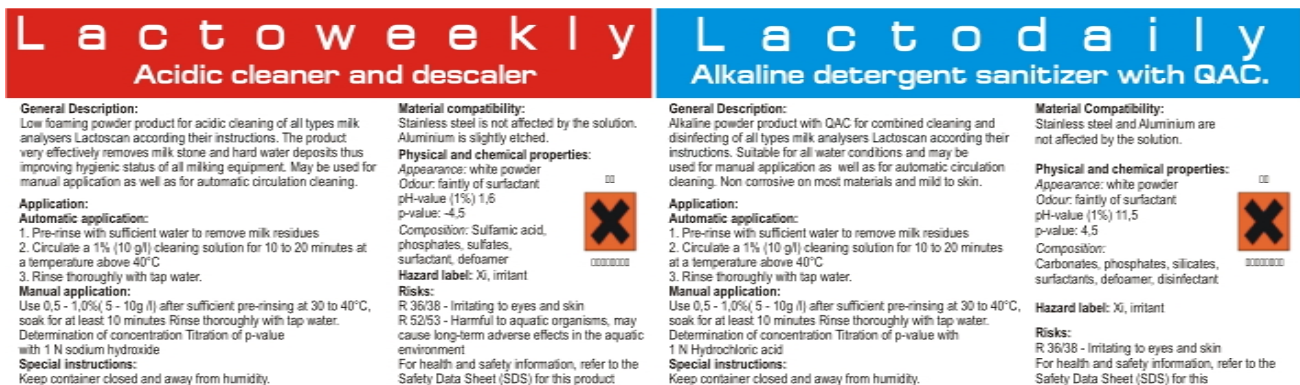
It is recommended to be done every day.

Preparation of 1 % acidic solution of Lactoweekly for circulation cleaning in the milk analyzer:

1. Take the package 100 g concentrated powder chemical Lactodaily
2. Carefully cut the upper end, paying attention not to spill it.
3. In appropriate vessel (for example bucket) pour 1 l water.
4. Add the powder and then again water up to 10 l.

Then follow the instruction for milk analyzer cleaning.

**Fig. 6 Labels for the cleaning chemicals**



The following procedure is executed:

1. Rinsing the milk residues:

Fill in the glass with water. Put it in the recess of the analyser and start command Cleaning from the main menu. After finishing it pour out the contaminated water.

2. Cleaning with acidic solution

Fill in the glass with warm (50-60 C) acidic cleaning solution. Put it in the recess of the analyser and start the command Cleaning from the main menu. After finishing it, pour out the contaminated liquid.

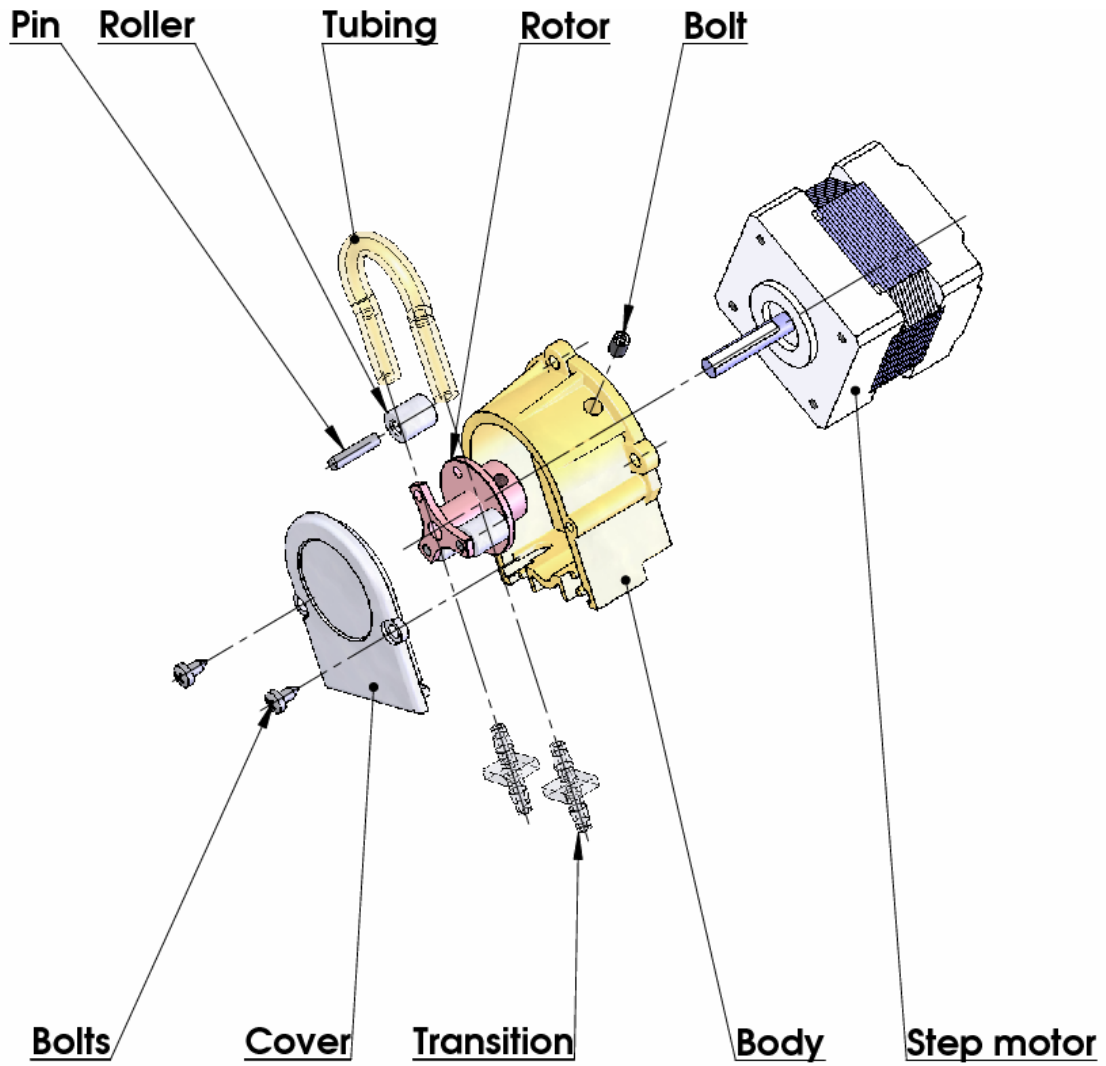
3. Rinsing with water

Fill in the glass with water. Put it in the recess of the analyser and start command Cleaning from the main menu. After finishing it pour out the contaminated water. Now the device is ready for work.

**IMPORTANT**  
**THE MAIN REASON FOR MALFUNCTIONING OF THE DEVICE IS THE BAD CLEANING OF THE SYSTEM AFTER MAKING ANALYSIS.**  
**In case of malfunction due to the bad cleaning of the analyser your guarantee is not valid anymore and any repair has to be paid.**

### 4.3. Peristaltic pump service

#### Fig.7 Peristaltic pump



## 5. POSSIBLE MALFUNCTIONS AND ERROR MESSAGES, TROUBLESHOOTING

In the table below are described the possible malfunctions during the milk analyzer's exploitation and ways for their repair/remedy. If the problem persists after all recommended measures are taken, please, connect the nearest service center for help. Do not forget to tell the analyzer's identity.



To receive the analyzer's identity, refer to point 3.2.1.3.

Error message	Possible problem /cause	Repair/remedy
<p><b>MA overheated</b> Accompanied by a continuous sound signal</p>	<p>Overheated milk analyzer</p>	<p>Immediately switch off the analyzer. Pay attention the analyzer to be situated away from direct sunlight or heating devices. Wait 5-10 minutes the device to cool down or to be normalized the ambient temperature and switch it on again.</p>
<p><b>Empty Camera</b></p>	<p>Insufficient quantity of the milk sample sucked in the system or air in the sample</p>	<p>The analyzer is ready to measure the next sample. In order to avoid the future appearance of the same error message, please, check the following:</p> <ul style="list-style-type: none"> <li>- The sample is prepared according the instructions and there aren't air bubbles in it.</li> <li>- There is a real suction of the sample after starting measurement, i.e. it is obvious that the level of the milk sample in the sample holder decreases. In other case – there is damage in the suction system.</li> <li>- Avoid the end of the suction pipe to be above the surface of the liquid (not dipped enough).</li> <li>- Avoid curdling of the milk sample. Clean immediately if there is a sample curdled in the system.</li> <li>- In mode Measurement, after starting the measurement, remove the sample holder and see if there is no milk poured</li> </ul>

		back in the sample holder.
<b>Sample Overheat</b>	Sucked overheated sample	<p>The analyzer is ready to measure the next sample. In order to avoid the future appearance of the same error message, please, check the following:</p> <ul style="list-style-type: none"> <li>-The sample is prepared according the instructions and its temperature does not exceed the maximum permissible sample's temperature.</li> <li>-Complete the procedure for checking the analyzer in case of error message <b>Empty Camera</b>.</li> </ul>

## **6. MAKING CORRECTIONS AND RECALLIBRATION OF THE DEVICE**

In the process of work with the analyser there is a possibility the results to start differing between the data for some of the measuring parameters when measured with the milk analyzer and the corresponding reference method of analyses (Gerber for fat, Kjeldhal for proteins etc). In order to establish the possible discrepancy and to correct the readings of the milk analyser do the following:

### **6.1. Taking samples and preparation of samples for checking the accuracy of the milk analyser, making corrections and recalibration**

This is a basic moment for the correct checking the accuracy of the analyser and for making correct and precise correction and calibration. It is accomplished according Appendix Sampling and preparation of samples for verification the accuracy of the milk analyzer, making corrections and recalibration.

### **6.2. Determination the type of the discrepancy:**

#### **6.2.1. Making measurements**

Make measurements with different samples (not less than 3) with known values of a separate parameter (for example fat content), determined by the known reference methods of analyses (for example Gerber's method for determination of fat content). For more accuracy it is recommended among these samples to be also such with values, close to the lowest and highest bounds for the measured parameters.

Make 5-time measurement for each of the samples. Calculate the average value for each sample parameter, without taking into consideration the first measurement for each sample.

#### **6.2.2. Analysing the measurement results**

Make comparison between the values of the parameter from the reference sample and measured with the analyser. Make analyses of the difference received.

**6.2.2.1.** If the received differences are relatively constant value for samples with different content of the analysed parameter, it is necessary to make correction.



For example

M% of the reference samples:	2,20	3,00	3,80	4,60	5,20
M% average when measuring with the analyser:	<u>2,38</u>	<u>3,17</u>	<u>4,01</u>	<u>4,79</u>	<u>5,42</u>
Difference:	0,18	0,17	0,21	0,19	0,22

**Conclusion:** the difference is relatively constant value and correction is possible to be done with – 0,2 % (see Corrections, p6.3.3)

**6.2.2.2.** If the differences are not a constant value it is necessary recalibration to be done.

For example.

M% of the reference samples:	2,20	3,00	3,80	4,60	5,20
M% when measured with the analyser:	<u>2,02</u>	<u>2,93</u>	<u>3,76</u>	<u>4,75</u>	<u>5,44</u>
Difference:	-0,18	-0,07	-0,04	0,15	0,24

**Conclusion:** It is obvious that the difference is variable value and recalibration have to be done (See Recalibration, p.6.4).

### 6.3. Making corrections

#### 6.3.1. Possible corrections, limits and changing steps

Every parameter from each calibration may be separately corrected. Below is the table with possible corrections, limits and changing steps:

Parameter	Increasing	Decreasing	Step
FAT	0.95%	0.95%	0.01%
SNF	4.75%	4.75%	0.05%
Density	4.75%	4.75%	0.05%
Lactose	0.95%	0.95%	0.01%
Salts	0.95%	0.95%	0.01%
Proteins	0.95%	0.95%	0.01%
Added water	9.00%	9.00%	1.00%
Sample's temperature	9.90°C	9.90°C	0.1°C

### 6.3.2. Preparing the analyzer for mode Corrections

6.3.2.1. Press the button **Enter** and without releasing it switch on the power supply of the device, wait for the starting identification messages and release the button after the following message appears on the display:

**Release button  
to start setup**

After releasing the button on the display is shown:

**Setup Menu**

followed by possible to be entered by the operator menus:

**Special modes  
Corrections  
Settings**  
-----  
**Tests  
Exit**

6.3.2.2. By using buttons “up” ▲ and “down” ▼ position on **Corrections** and press **Enter**.

### 6.3.3. Making correction

#### 6.3.3.1 Determining the calibration mode

When starting **Corrections**, the following appears on the display

**Corrections:  
Measurement  
Temperature  
Exit**

By using buttons “up”▲ and ”down”▼ position on the **Measurement** and press **Enter**.

### 6.3.3.2. Choosing correction parameter

After choosing calibration mode the display shows the following:

<b>Cal1 Cow</b>		
<b>Prev</b>	<b>OK</b>	<b>Next</b>

Using the buttons “up”▲ **Next** and ”down”▼ **Prev(ious)** choose the calibration you need to correct. The display shows:

<b>Cal:1 Cow</b>		
<b>Param:Fat</b>		
<b>Correct=00.00</b>		
<b>Edit</b>	<b>OK</b>	<b>Next</b>

Using the button **Next** you are choosing the next parameter for correction. Using the button **Edit** you are entering the mode for editing the current correction.

### 6.3.3.3. Making correction

It is done by using the buttons “up”▲ and ”down”▼. Now you have the possibility to increase or decrease the value of the measured parameter in the above mentioned boundaries

Leaving this mode means saving the correction value and activating it.

### 6.3.3.4. Making verification

After the corrections are made put the milk analyser in working mode following the sequence below:

1. Switch off the power supply.
2. Switch on the “**POWER**” button, which starts the identification procedure. For a short time the display(фиг.1, поз. 1) shows the versions of the software, for example:

**Milkanalyzer xxx  
MB vers yy  
Ser. N. xxxx**

Till the analyzer is in preparation mode (at about 5 minutes) the display shows: **“Getting ready”**.

3. After the analyzer is ready a sound signal is heard and display shows: **“Analyzer ready”**. Now the analyzer is ready to make analyses on its first working mode (usually Cow).
4. make several times measurement of reference samples with known values of the corrected parameter. If the difference between the values of the parameter from the reference methods and milkanalyser are in the limits for the parameter it may be considered that the correction is successfully made.

If the discrepancy between the measurements from the milk analyser and classical methods is bigger than is necessary to make second correction according above described way.

If after the second correction the results are unsatisfactory we recommend making a calibration of the analyser. In dependence of the conditions and your requirements you may make the calibration using a personal computer type IBM PC and the company's calibration program or autonomous - by recalibration.



When making corrections or calibrations be 100% sure in the accuracy of the reference methods result.

## **6.4. Recalibrating the milk analyser**

### **6.4.1. Running the analyser in mode Recalibrate**

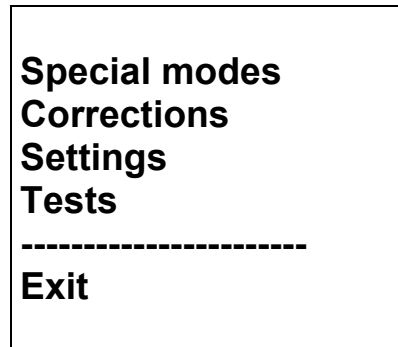
**6.4.1.1.** Press the button **Enter** and without releasing it switch on the power supply of the device, wait for the starting identification messages and release the button after the following message appears on the display:

**Release button  
to start setup**

After releasing the button on the display is shown:



Followed by the possible to be entered by the operator menus:



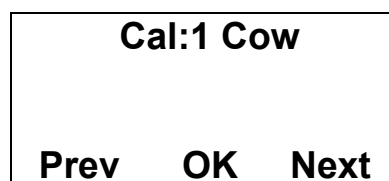
**6.4.1.2.** By using buttons “up” ▲ and “down” ▼ position on **Settings** and press button **Enter**.

**6.4.1.3.** Analogically, position on **Recalibrate** and press the button **Enter**.

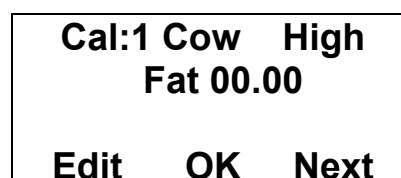
## **6.4.2. Making recalibration**

### **6.4.2.1. Determining the calibration mode**

After starting **Recalibrate**, the display shows the following:



Using the buttons “up” ▲ and “down” ▼ position on the corresponding calibration (for example if you need to recalibrate cow milk, which is assigned in the milk analyser as **Cal 1 - cow**) and press the button **Enter**. The following message appears on the display:



By pressing **Next** you are going to correction of another parameter, by using **Edit** – to set the value of the chosen sample

### **6.4.2.2. Entering values for the separate sample parameters**

After choosing the edit mode the display shows the following:

<b>Cal:1 Cow High</b>		
<b>Fat 00.00</b>		
-	<b>OK</b>	+

In this display is possible to be entered the results from the milk **with high FAT** chemical analyses, which were received by the methods from *Appendix Methods*.

In this mode:

- by using the buttons "up" ▲ and "down" ▼, choose the needed parameter to be entered;
- by pressing "**Enter**" the cursor will mark the number to be changed.

For example: FAT=01.29;

- using the buttons "up" ▲ and "down" ▼, enter the needed value;
- by next pressing of **Enter** pass towards the next number;
- after completion of entering the needed FAT value, press **Enter**;
- the cursor returns to its starting position.

#### 6.4.2.3. Making recalibration with the available samples

After entering the values for the separate parameters of the sample, the analyzer passes in mode preparation for work and the display shows:

<b>Cal: Cow</b>
<b>Put sample High 5</b>
<b>times</b>

which reminds us to put 5 times the sample with high **FAT**.



The sample has to be with temperature in the boundaries 15-25°C.

Stir 2-3 times the milk sample before each measurement by pouring it from one vessel to another. Pour the needed quantity in the sample-holder and put it in the recess of the device. Start the measurement. The sample is sucked. Appears the following menu:

**Recalibrate 1  
Put sample high 5  
times  
Samp T=16.8**

After the sample is measured, appears the following menu:

**Recalibrate 1  
Put sample High  
Cal meas=1/5**

which reminds to make the next measurement. Follow the procedure till the 5th measurement.

After 5th measurement completion automatically appears the menu, which reminds to enter the values for the sample with low FAT. Enter the values analogically to the procedure, described in the previous sample.

After the 5<sup>th</sup> measurement appears the menu:

**Recalibrated  
Analyzer ready**

This means that the calibration was completed successfully and the analyzer is recalibrated for cow milk, marked as “**Cal 1**”.

Switch off the power supply of the device and switch it on again.  
The device is ready to work with the new calibration.

If during work the sample's temperature exceeds the temperature range 15-25° C the following message appears:

**Temperature out of  
range**

Wait till the end of the measurement. When the message appears:

**Put sample again**

Put sample with temperature in the temperature range and continue the measurement till completion of 5-time measurement.



## 7. STARTING THE DEVICE IN A SERVICE TEST/SETUP OPERATIONAL MODE. MENUS DESIGNATION

### 7.1. Starting the device in a service Test/Setup operational mode.

In order to start the **Setup** of the device the operator has to press the button **Enter** and without releasing it to switch on the power supply of the device, to wait for the starting identification messages and to release the button after the following message appears on the display:

**Release button  
to start setup**

After releasing the button on the display is shown:

**Setup Menu**

Followed by possible to be entered by the operator menus:

**Special modes  
Corrections  
Settings**  
-----  
**Tests  
Exit**

You may move in the menus by using buttons “**up**” ▲ and “**down**” ▼. If by pressing the button **Enter** you choose a menu, each menu offers new points/submenus. When **Exit** is chosen the device leaves the **Setup** mode and returns to normal work.



Due to the continuous improvements made in the milk analyser or due to the type of the ordered product, it is possible some of the options in the device to be not active. In this case, if you try to enter the corresponding menu, the following message will appear: **Not available option**.

## 7.2. Menus Function:

### 7.2.1. Special modes.

Serve for choosing special (technological) working modes. After starting it the following appears on the display:

**Special modes**  
**Calibration**  
**Cycle**  
**Exit**

This mode is normally used in production conditions.

#### 7.2.1.2. Calibration mode

In mode **Calibration** the analyzer is ready to make measurement and to send the received results towards the technological milk analyzers calibration system. For this purpose you need personal computer type IBM PC, company's calibration system LSC.EXE and methods for calibration of milk analyzers (see the corresponding documents). To start measurement in this mode, the operator has to put a sample-holder containing milk sample in the recess of the analyzer and to press the button **Enter**.

#### 7.2.1.2. Cycle mode

Mode **Cycle** serves for training the analyzers. When you start this mode, the analyzer, without additional commands, sucks the sample, makes the measurement, pours the sample out in the sample-holder and displays the received results cyclically.

#### **Note:**

This mode is normally used in production conditions. It is recommended the customer to calibrate the device using the embedded function Recalibrate (i.e autonomous, without computer)

## 7.2.2. Corrections

Serves for entering corrections in the measured data. Detailed description in point 6.3.2 and 6.3.3.

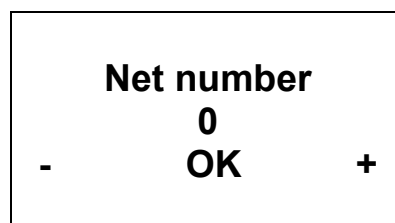
## 7.2.3. Settings.

Serve for assigning different working parameters (modes).

### 7.2.3.1. Net number.

Serves for assigning the device network number when connecting it in the production network. The possible numbers are from 0 to 15 including.

After starting this function the display shows the following:



By using the button “up” ▲ the operator has the possibility to increase the number, showing the channel’s number, and by button “down” ▼, to decrease it. Pressing the button **Enter** saves the chosen channel and exits the function.



When connected in the production network each device has to have a unique number.

### 7.2.3.2. Recalibrate.

Serves for changing definite calibration. Methods are described in point 6.4.

### 7.2.3.3. Save/Rest Cal.

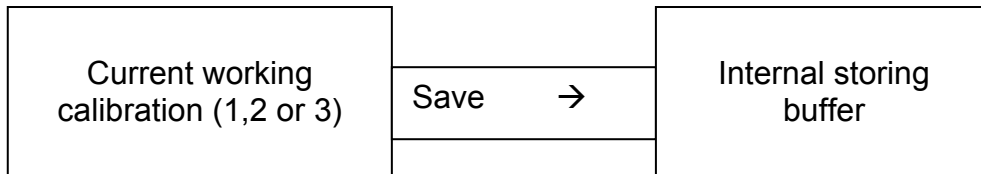
Through this menu you may save the new calibration in the device or to restore the old one (factory) calibration. This is necessary in case that you’ve calibrated the device for cow milk, but after that the device is not measuring correctly and you decide to restore the factory calibration settings. Position the cursor across “**Restore calibration**” and press “**Enter**”

Possibilities:

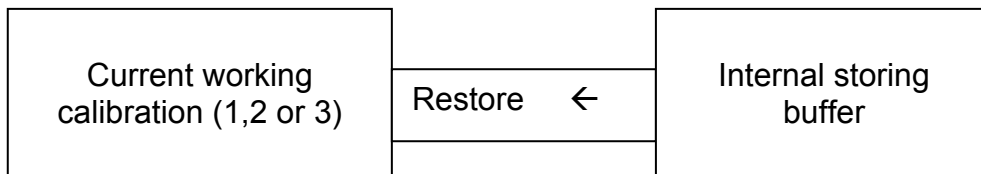
**Save calibration** – saves the chosen calibration in an internal buffer.

**Restore calibration** – restores the chosen calibration from the internal buffer.

The procedure **Save/Restore** is done for each calibration separately.



Current calibration content is not changed, the analyzer continues using it, but there is a reserve copy in an internal buffer.



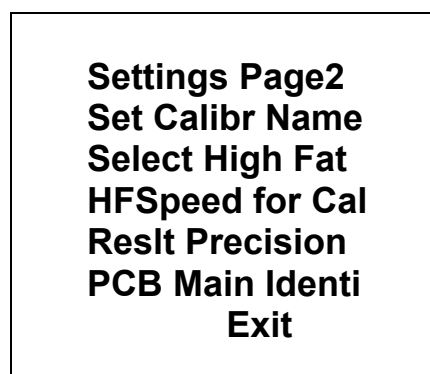
The current calibration is replaced with the calibration from the internal buffer and the analyzer starts working with it. The content of the internal buffer is not changed.



**If after recalibration “Save calibration” is pressed the new calibration settings will be saved over the factory settings. After that is impossible to restore the factory settings of the calibration. Save the newly made calibration only if you are sure about its correctness.**

#### 7.2.3.4. Settings Page 2.

After this menu is started the display shows the following:



Now there is a possibility one of the following options to be set:

##### 7.2.3.4.1. Set Calibr Name.

Sets the names of the separate calibrations. The name could be chosen from the group of predefined calibrations names or to edit a new one. When editing

the new name there is a possibility all ASCII codes to be used, as letters (caps and normal), numbers and punctuation marks and popular symbols. The calibration name consists of 8 symbols.

Example:

When it is suitable to us this possibility of the analyser? For example if you have a device factory calibrated for Cow milk, Sheep Milk and UHT milk, but you need often to measure camel milk. Using the methods, explained in details in Appendix Methods you may make a new calibration without need to send the analyser back to the producer for calibration. Using this procedure you may make calibrations for most often analysed milk and to write down the exact calibration name, which will be shown on the display and printed on the printer.

After starting this menu the display shows:

**Cal:1 Cow**

**\Prev   OK   Next**

There are the following possibilities:

With button **OK** – to start editing the name of the chosen calibration.

With button **Prev** – to choose the previous calibration, chosen for setting the calibration name.

With button **Next** – to choose the next calibration name for editing.

If the operator has chosen and confirmed calibration for change of the name, the display shows (example):

**Cal:Cow**

**PreDef Exit Edit**

There are the following possibilities:

With button **PreDef** – to choose a calibration name from the list of preliminary given names.

With button **Exit** – to leave the menu.

With button **Edit** – to edit the new calibration name.

If a name from the preliminary given names list is chosen, the display shows:

**Cal1: Cow  
New Cow**  
**Exit Yes Next**

There are the following possibilities:

With button **Exit** – to leave the menu.

With button **Yes** – to confirm the chosen from the list calibration name. Now the program returns to the beginning of the menu for setting calibration names.

With button **Next** – to show the next calibration name from the list.

If it is decided a new calibration name to be edited, the display shows:

**Cal1: Cow  
New Cow**  
**Prev Set Next**

There are the following possibilities:

With button **Prev** – to display the previous ASCII symbol.

With button **Set** – to confirm the ASCII symbol, shown on the display and passes to editing the next symbol from the calibration name.

With button **Next** – to show the next ASCII symbol.

The editing finishes by entering the eighth symbol from the name of the calibration.

#### **7.2.3.4.2. Select High Fat**

If the analyzer has embedded function for measurement of high fat products, by this menu the calibration, with which this measurement to be made is chosen. What is seen when this option is turned on is obvious slow down of the sample's suction speed.

#### **7.2.3.4.3. HFSpeed for Cal**

If the analyzer has embedded function for measurement of high fat products, and a new calibration for high fat measurement is needed, before starting the new calibration the operator has to start this menu. What is seen when this option is turned on is obvious slow down of the sample's suction speed during calibration.

Please, pay attention to the fact that switching off the power supply cancels this command action.

#### **7.2.3.4.4. Result Precision**

Serves for setting the precision of the measured results shown on the display. It is set separately for every parameter, the precision could be 0.01 (standard) or 0.1.

#### **7.2.3.4.5. PCB Main Identity**

Gives information about the type and the version of the analyser (LS Identity).

#### **7.2.3.5. Set Base Freezing Point.**

Serves for editing the Basic Freezing Point. It is used by the customer according to the Appendix for calculation of the added water and determination of the Freezing point of the sample.

### **7.2.4. Tests.**

Start different tests. Possibilities:

#### **7.2.4.1. Test pump.**

Starts pump's test. The number of the completed suction/display cycles is indicated.

#### **7.2.4.2. Ultrasound.**

Test for the ultrasonic system. Used in production conditions.

#### **7.2.4.3. Set Amplitude.**

Serves for ultrasound amplitude adjustment. It is used under production conditions or by the customer (after sensor change) according to the instructions in the document SetCell.pdf.



***Please, use this menu only after reading the above pointed document SetCell.pdf***

### **7.2.5. Exit**

By pressing the button you may leave the program and pass towards another menu.

## 7.2.6. Milk analysers' setup menu structure

### Analyzer Setup

Special  
modes

Calibration  
Cycle

Corrections

Measurement  
Temperature

Settings

Net number  
Recalibrate

Save Calibr  
Restore Calibr  
Settings Page2

Set Calibr Name  
Select High Fat  
HFSpeed for Cal  
Reslt Precision  
PCB Main Identi

Tests

Set Base FrPnt

Test pump  
Ultrasound  
Set Amplitude



## **8. ADDITIONAL POSSIBILITIES OF THE ANALYZER**

### **8.1. Connecting to 12 V DC power supply.**

If there is a need the analyzer to work on place without electrical supply available, then it could be powered by car battery or other 12 V DC external power supply. Use the 12 V power supply cable, supplied with the analyser.

### **8.2. Connecting to IBM PC**

The milk analyser can be connected to IBM PC using the RS232 interface cable. In order to make the connection: switch off both milk analyser and PC. Connect the RS 232 cable towards Serial interface and towards the computer. Turn on both milk analyser and PC. Now the milk analyser is ready to communicate with IBM PC. This is enough for starting the program for collection and archive of the measurement results

### **8.3. Connecting a printer (option).**

In order to print out the measurement results, a serial printer could be connected to the device – for example ESC/POS Serial printer, production of Datecs or Seiko. The interface connector for the printer is on the rear panel of the device – “Serial printer output”. The printer (if it is Datecs), should be connected to the “12 V printer output” on the device rear panel. Connect it via cables, delivered by the company-producer. If the printer is connected directly to the electrical network, then the analyzer and the printer should be connected to one and the same electrical phase.

Communication parameters: 9600 bps, No parity, 8 bits, 1 stop bit. It's one-way communication (uses one line) – the analyzer only sends and the printer only accepts data.

## APPENDICES

### APPENDIX 1 METHODS

#### 1. Determination of milk's density

##### 1.1. General

Milk density is defined as relation between the mass of definite milk volume at temperature 20 °C and the mass of equal volume distilled water at temperature 4 °C.

Density, alone, could not be used as a control parameter at milk quality control. Using the density the tentative figures for the SNF and salts could be determined.

##### 1.2. Sampling and preparation for analyses

Sampling milk or other milk derivatives and their preparation for analyses is done according corresponding Appendices.

Milk density is determined not earlier than 2 h after milking. The milk must be with temperature from 10 to 25 °C.

Before determination of density the milk must be well stirred. To avoid foam formation, it has to be carefully poured on the cylinder's walls. The cylinder must be slightly tilted.

Before taking the readings the cylinder, with the milk must be placed on an even surface, facing the light, so the readings could be easily seen.

##### 1.3. Basic principles.

The density of the milk is determined using aerometer, also called lacto-density-meter (milk density meter) and is expressed with a number, representing milk density meter degrees, decreased 1000 times or only with milk density meter degrees.

##### 1.4. Necessary devices and reagents

- Aerometer /lacto-density-meter, milk meter/.
- Cylinder – with inner diameter not less than 5 cm, and length, corresponding to the dimensions of the lacto-density-meter.
- Ammonium with preliminary defined relative density.

##### 1.5. Making the determination:

Dry and clean, the lacto-density-meter is slowly dipped in the milk till division 1,030, and then is left in free-floating state. The lacto-density-meter must not touch the cylinder's walls and to be on at least 5 mm from them.

When taking the readings the eyes must be on one and the same level with the meniscus. The reading is done in the meniscus' upper end with accuracy till 0,0005, and the temperature – with accuracy till 0,5 °C.



The difference between two parallel determinations must be not more than 0,0005.

### **1.6. Recalculating the values according lacto-density-meter at 20 oC.**

If the milk, when determining its density, has temperature, higher or lower than 20 °C, the readings from lacto-density-meter are recalculated towards 20 °C.

Density recalculation towards 20 °C is done on the following way:

for every temperature degree over 20 °C from the received by the milkmeter value are added 0,2 ° for the cow and goat milk and 0,25 ° for sheep and buffalo milk lacto-density-meter degressed or 0,0002, respectively 0,00025 towards density; and for every temperature degree under 20 °C from the readings of milkmeter value are deducted 0,2-0,25 lacto-density-meter degrees or 0,0002, (0,00025) from the density.

## **2. Determination of fat content in the milk and milk derivatives.**

### **2.1. General**

For making analyses are used pure reagents for analyses (pure reagents for analyses (p.r.a.) and distilled water or water with equivalent purity.

### **2.2. Sampling**

Milk and milk derivatives sampling is done according *Appendices Milk sampling and preparation of samples for analyses ad Sampling and preparation of samples for verification the accuracy of the milk analyser, making corrections and recalibration.*

### **2.3. Basic principles.**

The method uses dissolving the milk and dairy products proteins with sulphuric acid with definite concentration in butyrometer and separating the fat under the influence of amilic alcohol, heating and centrifuging in a form of dense transparent layer, the volume of which is measured in the graduated part of the butyrometer.

### **2.4. Necessary devices and reagents**

- Butyrometers for milk, special for skimmed milk and cream;
- Rubber stopples for butyrometers;
- Stand for butyrometers;
- Special pipettes or automatic for milk, sulphuric acid and изоамилов alcohol from 1, 10 and 11 cm<sup>3</sup>;
- Pipettes from 1 and 20 cm<sup>3</sup>;

- Glasses from 25 till 50 cm<sup>3</sup>;
- Centrifuge for Gerber;
- Water bath;
- Mercury thermometers up to 100 °C with value scale 1 °C;
- Sulphuric acid with density 1,82 at 20 °C for determination of fat content of the milk;
- Isoamilic alcohol for Gerber with density 0,811 to 0,812.

## **2.5. Making the determination:**

Preparation of samples for analyses.

The milk is mixed well in order to become homogenous mixture (if necessary it is slowly heated up to 35-40 °C ) and is carefully shaken and tempered to 20±2 °C. The samples from whey and buttermilk are preliminary filtered through double layer gauze and is then tempered to 20±2 °C. Cream samples are placed in water-bath at temperature 35 till 40 °C, stirred till homogenous sample is received and cooled down to 20±2 °C.

## **2.6. Making measurement**

*With butyrometer for milk*

For milk, whey and buttermilk.

With automatic or special for acids pipette are measured 10 cm<sup>3</sup> sulphuric acid with  $d=1,820 \text{ kg/m}^3$  at 20 °C in the milk butyrometer. Carefully on the butyrometer's walls are piled up 11 cm<sup>3</sup> from the prepared sample. The pipette is held till its full drainage.

For cream

From the prepared sample is measured 10 g with error up to 0,001 g and 50 cm<sup>3</sup> water are added. Mixture is well stirred and heated up to 30-35 °C, then is again stirred and cooled down to 20±2°C, and the following steps are as with the milk sample using sulphuric acid with  $d=1,789$  till  $1,790 \text{ kg/m}^3$ .

*With butyrometer for cream*

For cream

5 g from the sample are measured with butyrometer with error up to 0,0001 g and then 5 cm<sup>3</sup> water are added, 10 cm<sup>3</sup> sulphuric acid with  $d=1,780$  to  $1,790 \text{ kg/m}^3$  at 20 °C and 1 cm<sup>3</sup> изоамилов alcohol. The butyrometer is closed with rubber staple and is shaken till the proteins are fully dissolved.

## **2.7. Calculating the results**

*By using milk butyrometer*

Milk, whey, buttermilk.

Using the butyrometer's graded scale the grams fat in 100 g product are read directly. When the milk is curdled, the result is increased with 0,1 g for every degree.

*By using cream butyrometer.*

## Cream

Using the butyrometer's graded scale the fat content in the products is directly read in percentages.

### **2.8. Measurement accuracy**

#### *By using milk butyrometer*

The difference between two parallel determinations could not exceed:

For skimmed milk, whey and buttermilk - 0,05 g for 100 g product;

For cream - 0,5 g for 100 g product;

For milk - 0,1 g for 100 g product;

#### *By using cream butyrometer*

The difference between two parallel determinations could not exceed 0,5 g for 100 g cream.

## **3. Determination of water content and solids in the milk and milk derivatives.**

### **3.1. General**

The solids represent the fat content, proteins, carbohydrates and salts.

Sampling is done according *Appendices Milk sampling and preparation of samples for analyses ad Sampling and preparation of samples for verification the accuracy of the milk analyser, making corrections and recalibration.*

### **3.2. Basic principles.**

Water content is determined by weight when drying at temperature  $(102\pm 2)$  °C of the weighted product till constant mass, expressed in grams for 100 g product.

The solids/dry substance is the mass of the dry remainder, received after dehydration of determined quantity product at temperature  $(102\pm 2)$  °C till constant mass and is expressed in grams for 100 grams of the product.

### **3.3. Necessary devices and reagents**

- Assay balance with loading bounds 200 g and error 0,0002 g.
- Mercury thermometers from 0 to 100 °C and from 0 to 150 °C with value of scale division 1 °C;
- Pipettes from 5 to 10 cm<sup>3</sup>, class II;
- Glass banks with grind stopples with volume 100-200 cm<sup>3</sup>;
- Drying-oven with thermal regulator for keeping the temperature  $(102\pm 2)$  °C;
- Exicator with silicagel or another hygroscope material;
- Weight plates;
- Peg for the weight plates;
- Glass pods with rounded ends;
- Quartz, sea or river sands.

### 3.4. Making the determination:

Sample preparation for analyses.

The milk (whey, cream, and buttermilk) is well shaken. If needed, the sample is heated slowly up to 38-40°C, it is well mixed and cooled down to 20°C. Mixing and pouring are done at least three times in dry and clean vessel.

### 3.5. Making the measurement

The weight plate with 20-30 g washed out and tempered sand and glass rod is dried at 102±2 °C for 1 h, and then is taken out, covered with the cap, tempered with exicator (up to 30 min) and the mass is weighted with accuracy up to 0,0005 g. In the weight plate, using pipette, at about 10 cm<sup>3</sup> milk are poured, covered and weighted. With the help of the glass rod milk is well mixed with the sand and without a cap is heated on a water-bath till a homogenous mass is formed. Then the weight plate is put in a drying-oven at temperature 102±2°C, it is dried out for 3 h, it is taken out of the oven, covered with the cap, tempered in exicator (up to 30 min) and the mass is weighted. Weight-glass is placed in the drying-oven again and is dried 1 h, then is taken out, tempered and weighted. This procedure is repeated till the difference between two consequent measurements becomes not more than 0,004g. In case that at the second or following drying procedure mass increases, then for the calculation is taken the previous measurement.

### 3.6. Calculating the results

Water content in grams for 100 g product (milk or milk derivatives), is calculated by the formula:

$$X = \frac{M_2 - M_3}{M_2 - M_1} * 100$$

Where

M1 - the mass of the plate with the sand and the glass rod, g;

M2 - the mass of the plate with the sand, the glass rod, and the sample before drying, g;

M3 - the mass of the plate with the sand, the glass rod, and the sample after drying, g;

The dry substance (Y) is calculated using the formula:

$$Y = 100 - X,$$

Where:

X is the calculated water content.

### 3.7. Measurement accuracy.

The difference between two consecutive measurements of one and the same sample could not be more than 0,2 g for 100 g product.

#### **4. Determination of casein content in the milk.**

##### **4.1. General**

The methods are based on the Volker's method. For making the analyses are used pure reagents for analyses (p.r.a.) and distilled water or water with equal purity.

##### **4.2. Sampling**

According corresponding *Appendices*.

##### **4.3. Basic principles.**

Added to the milk formalin liberates acidic residues from the protein's end groups, which are titrated with soda caustic solution. The soda caustic quantity is proportional to the casein in the milk content.

##### **4.4. Necessary devices and reagents**

- Glass 250 cm<sup>3</sup>.
- Pipettes Foll - 25,5 cm<sup>3</sup>.
- Pipettes Mor from 1 cm<sup>3</sup>, with division 0,1 cm<sup>3</sup>.
- Soda caustic p.r.a. - 0,143 n solution.
- Formalin 40% p.r.a - freshly neutralized.
- Phenolphthalein - 2 % solution in 70 % ethyl alcohol.
- Potassium oxalate p.r.a. 28 % water solution.
- Cobalt sulphate p.r.a. 5 % water solution.

##### **4.5. Making the determination:**

For cow milk

Reference sample preparation.

20 cm<sup>3</sup> from the measured milk are poured in a glass vessel together with 1 cm<sup>3</sup> 3 % water solution of cobalt sulphate. The sample is shaken and a slight rose color of the solution is received, which serves as a standard in the research.

##### **4.6. Making the measurement**

20 cm<sup>3</sup> from the milk are measured in a glass and are titrated with 0,1 N soda caustic, using phenolphthalein as an indicator, till the color of the standard sample is reached. The volume of the used soda caustic is not taken into consideration.

4 cm<sup>3</sup> 38-40 % formalin are added towards the neutralized sample and the rose color disappears as a result of the liberated carboxylic groups. It is well

stirred and titrated with 0,1 N soda caustic, till slight rose color is recovered. At the second titration the volume of the used soda caustic is measured.

For sheep milk

Casein content in sheep milk is determined on the same way. The only difference is that instead of 4 cm<sup>3</sup> 38-40 % formalin in the milk are added 6 cm<sup>3</sup>, and the standard/reference sample is prepared with 1 cm<sup>3</sup> 4 % solution of cobalt sulphate.

#### 4.7. Calculations

The quantity of the 0,1 N soda caustic in cm<sup>3</sup>, used in the second titration, multiplied by the coefficient 0,7335 is equal to the casein content in the milk in percentages.

The following tables could be used for quicker readings of casein's percentage on the base of used cm<sup>3</sup> 0,1 N soda caustic:

**Table I**

Calculation of casein content in the cow milk on the base of used cubic centimeters 0,1 N soda caustic:

0,1 n NaOH cm <sup>3</sup>	Casein%	0,1 n NaOH cm <sup>3</sup>	Casein %	0,1 n NaOH cm <sup>3</sup>	Casein %
3,00	2,20	3,35	2,46	3,70	2,71
3,05	2,24	3,40	2,49	3,75	2,75
3,10	2,27	3,45	2,53	3,80	2,79
3,15	2,31	3,50	2,56	3,85	2,82
3,20	2,35	3,55	2,6	3,90	2,86
3,25	2,38	3,60	2,64	3,95	2,90
3,30	2,42	3,65	2,68	4,00	2,93



**Table II**

Calculation of casein content in the sheep milk on the base of used cubic centimeters 0,1 N soda caustic:

0,1 n NaOH cm <sup>3</sup>	Casein%	0,1 n NaOH cm <sup>3</sup>	Casein %	0,1 n NaOH cm <sup>3</sup>	Casein %
5,40	3,96	6,10	4,47	6,80	4,99
5,45	4,00	6,15	4,51	6,85	5,02
5,50	4,03	6,20	4,55	6,90	5,06
5,55	4,07	6,25	4,58	6,96	5,10
5,60	4,10	6,30	4,62	7,00	5,13
5,65	4,14	6,35	4,66	7,05	5,17
5,70	4,18	6,40	4,69	7,10	5,21
5,75	4,22	6,45	4,73	7,15	5,24
5,80	4,25	6,50	4,77	7,20	5,28
5,85	4,29	6,55	4,80	7,25	5,32
5,90	4,33	6,60	4,84	7,30	5,35
5,95	4,36	6,65	4,88	7,35	5,39
6,00	4,40	6,70	4,91	7,40	5,43
6,05	4,44	6,75	4,95	7,45	5,46

#### **4.8. Measurement accuracy.**

Two parallel samples are measured and the difference between them could not exceed 0,1 %.

The accuracy of the method require the work to be done at place with good natural illumination, titration to be done evenly, without interruptions, colorless formalin to be used, preliminarily neutralized with soda caustic and phenolphthalein indicator.

Formalin titration is easy method, but it is not enough precise. More accurate results for casein content are obtained using Kjeldhal's method, but it requires special appliances.

### **5. Determination of salts in the milk**

#### **5.1. General**

For the mineral substances in the milk conclusions can me made on the ashes content.

#### **5.2. Sampling**

According *Appendices Milk sampling and preparation of samples for analyses ad Sampling and preparation of samples for verification the accuracy of the milk analyser, making corrections and recalibration.*

### 5.3. Basic principles.

Milk is dried, carbonized and turned to ashes till constant mass. The ashes received are calculated in percentages.

### 5.4. Necessary devices and reagents

- Assay balance;
- Crucibles;
- Water-bath or infrared lamp;
- Hot plate or burner;
- Drying-oven with thermal regulator;
- Muffle furnace;
- Exicator;
- Quantity filter.

### 5.5. Making the determination:

In preliminary tempered and weighted crucible of the assay balance at about 10 g milk is weighted with accuracy up to 0,0005 g. The crucible with the sample is placed in a water-bath or infrared lamp till the evaporation of milk to dry state. Then it is carbonized with the burner or on a hot plate, paying attention not to be splashed out. The crucible is placed in a muffle oven and turns to ashes slowly, without the sample to be kindled, at temperature 500-550 °C till white or grey-white ashes. It is tempered in an exicator and is weighted till the appointed accuracy. Heating up in the oven is repeated till a constant mass is received.

### 5.6. Calculations

Ashes content is calculated using the formula

$$ashes = \frac{(C - A)}{(B - A)} * 100$$

Where:

A – the mass of empty, tempered crucible, g

B – the mass of the crucible together with the milk, g

C – the mass of the crucible with the received ashes, g

### 5.7. Measurement accuracy

The difference between tow parallel determinations could not be more than 0,02 %.

## **APPENDIX 2 REPRESENTATIVE SAMPLES FROM MILK AND OTHER MILK DERIVATIVES FOR MILK ANALYSER'S CALIBRATION**

### **1. General**

The samples used for analyser's calibration have to be representative for the corresponding milk type and have to be with known quality parameters: fat in percentage, SNF in percentage, density, lactose in percentage, total protein in percentage and salts in percentage. Changes in the analyzed parameters in the samples have, if possible, to cover the whole measuring range – i.e. used samples to be with low, middle and high content of the analyzed components.

The exact value of the parameters is decisive for correct and accurate calibration, because if the parameters are not set correctly during calibration the same parameter will not be measured correctly.

### **2. Necessary quality parameters values determination**

For more precise determination of above listed quality parameters of the milk and its derivatives is advisable they to be examined in an authorized laboratories, using the corresponding arbitration methods for this purpose.

#### **2.1. Laboratory methods**

##### **2.1.1. Determination of fat content**

Determination of fat content in the milk and its derivatives (cream, whey, buttered milk) is one of the most important analyses in the dairy production and milk processing. According this parameter the payment schemes are made and it is observed from the point of view correct production process and the basic economy balances are made with its help.

##### **A/ Röse-Gottlieb method**

The fat content is determined using the gravimetric method, fat extraction from ammonia-alcohol milk solution using diethyl and petroleum ether, evaporation of the solvent and weighting the residuum.

##### **B/ Gerber method**

The proteins in the milk and dairy products are dissolved with sulphuric acid with definite concentration in butyrometer and the fat is separated under the influence of amyl alcohol, heating and centrifuging in a form of dense,

transparent layer. The volume of this layer is measured in the divided part of the butyrometer.

This is quick, easy method with sufficient accuracy. We recommend it for usage. For more detailed description see *Appendix Methods*.

### 2.1.2. Milk density determination

A/ With picnometer and Mor-Vestval scales

This is the most exact method for determination of milk and its derivatives' density.

B/ with aerometer (lacto-density-meter)

Compared with the above method this is quick and easy readable with satisfactory accuracy. We recommend it. For more detailed description see *Appendix Methods*.

During the lactation period and under the influence of different zoo engineering factors the density of the different milk kinds varies in the following bounds:

Milk kind	Minimum	Maximum	Average
Cow	1,027	1,033	1,030
Buffalo	1,026	1,032	1,029
Goat	1,027	1,033	1,030
Sheep	1,031	1,040	1,034

### 2.1.3. Determination of total proteins

A/ Kjeldahl method

Heating with concentrated sulphuric acid in the presence of catalyst mineralizes a definite volume of the milk sample. The liberated ammonium combines with the sulphuric acid and forms ammonium sulphate. After adding surplus soda caustic ammonium is liberated. When distilled it combines with the boronic acid. The quantity of the combined ammonium is determined by titration with acid with determined titer. From the combined with the ammonium acid the initial nitrogen content is determined, and also the proteins in the milk.

B/ Titration with formalin

Formalin, added to the milk, combines with the amino group in the protein's molecule and forms methyl groups, which have no alkaline reaction. Milk acidity increases by the liberated carboxylic groups, which are titrated with soda caustic solution. The used volume soda caustic is proportional to the protein content in the milk.

#### **2.1.4. Determination of casein content in the milk**

##### **A/ Kjeldahl method**

The total nitrogen content in the milk is determined. Casein is precipitated with acetic acid (acetate buffer) and is filtrated. The content of nitrogen in the filtrate is determined. Casein content is the difference between the two results for nitrogen using the Kjeldahl's method.

##### **B/ Titration with formalin**

More details for this method – see *Appendix Methods*.

#### **2.1.5. Determination of salts in milk.**

For the salts in milk and its derivatives is judged by its ashes content. Milk dries, becomes carbonized and turns to ashes till constant mass. The ashes received are calculated in percentage.

#### **2.1.6. Determination of solids in milk**

Solids describe the content of fats, proteins, carbohydrates and salts. Its value may be used for determination of each of these parameters in case of known other values.

Salts are determined by drying till constant mass – see *Appendix Methods*.

#### **Express methods by using another milk analysers**

It is possible another device to be used for determination of some of the quality parameters of milk and its derivatives samples, intended for calibration, but it has to be noted that it is possible incorrect values to be received, that's why it is necessary to be completely sure in the accuracy of their readings.

Usage of Milkoscan and other milk analysers based on the infrared measurement principle.

By using it the fat, lactose and protein content may be determined. Problem may arise with determination of salts and SNF. This is due to the impossibility of the infrared method to determine the solids and in order to receive the solids in the sample their meaning is accepted as a constant.

## **APPENDIX 3 MILK SAMPLING AND PREPARATION OF SAMPLES FOR ANALYSES**

### **1. General**

Milk sampling and qualification of raw, thermally treated milk and its derivatives (cream, whey, buttermilk etc.) is accomplished for every separate homogeneous batch. As homogeneous batch is accepted:

- Milk, delivered by a separate producer (an individual farm, farm etc.), and received from one kind of animals after their complete milking, independently from the number of milk-cans and tanks.
- Milk, received from one or several farms or milk collecting centers, but delivered in a joint vessel.
- In the enterprise – from one and the same kind raw milk poured in one vessel.
- For cream, whey, buttermilk etc. – produced as a result of milk processing and its derivatives from one and the same kind and quality, poured in a separate vessel.

Milk is qualified not earlier than 2 hours after milking.

When the milk is frozen it has to be warmed up to 10-15 °C and stirred according the below-described procedure.

A sample is taken from every separate vessel proportionally to the quantity of the milk in it. Samples from the different vessels are mixed well and from the received medial sample are taken 200 - 250 cm<sup>3</sup> for accomplishing the needed analyses.

### **2. Stirring the milk and its derivatives before sampling**

#### **Milk stirring**

It is a very important condition for receiving exact results. Before taking samples from big vessels the milk (fresh or thermally treated, whole-milk or whipped) has to be well stirred for no less than 5 min., by vertical and circular slow movements. Mixing spoon with long handle is used, allowing the lowest layers of the liquid to be reached.

The milk in the milk-cans is stirred 5 to 8 times from the surface to the bottom and reverse with slow circular movements.

#### **Cream stirring**

Due to the fact that the cream is significantly thicker liquid than the milk and contains high percentage fat it has to be preliminary very well stirred from the surface to the bottom with reciprocation movements at about 20-25 times.

#### **Whey and buttermilk stirring**

It is analogical to milk stirring.

### 3. Sampling

Samples from milk, whey or buttermilk are taken with metal or glass pipe (dry, clean and stainless-steel) with diameter at about 10 mm, which is slowly dipped till the bottom of the vessel and its upper end remains open. In this way it is filled with milk simultaneously with its dipping. When the pipe is taken out of the vessel its upper end has to be tightly closed with a thumb. For a bigger reliability of the analyses results it is recommended the quantity of the taken sample to be no less than 200 ml.

Cream sample is carefully well stirred in order not to form foam. For taking a medial sample from milk-cans and tanks a sample pipe is used. Stuck to its outer surface cream has to be removed by using filter paper, napkin or clean cloth, preventing in this way the proportionality between the samples and the total amount of the cream to be disturbed.

### 4. Sample preservation

The vessels where the samples will be put have to be clean, dry, glass, metal or from other suitable material, to be tightly closed with rubber or other stopples. The stopples not to absorb water and fat and not to influence the analyses sample content.

In summer the sample fills up to the top the vessel, but in winter – at least 3/4 from the vessel's volume. Each sample for analyses has to be labeled and described in a way not allowing to be mixed up.

The samples are stored in conditions, assuring temperature, corresponding to the requirements for storing such kind of product (advisable – 1 °C).

If there is a need of longer sample storing they have to be preserved; the most commonly used preservative is potassium dichromate ( $K_2Cr_2O_7$ ) - 1 g for 1 000 ml. The samples have to be stored in a cold and dark place after the preservation. Have in mind that during the analyses the results for SNF% will be increased with 0,1 %. After adding the preservative the sample has to be well stirred.



Do not make analyses if the acidity of the milk is more than 25°T for cow and 28 °T for sheep milk.

### 5. Preparing the samples for analyses

Milk – raw and thermally treated

When examining samples taken immediately before analyses and shortly stored, the milk is poured several times from vessel to vessel in order to distribute the fat content uniformly. To avoid foam formation or separation of milk fat, the samples have to be carefully poured using the walls of the vessels, as they are tilted slightly. For a better mixing the sample it has to be poured at least 3 times. When needed the same is tempered to the temperature within the measuring range.

If there is fat stuck on the walls of the vessel and the stopple (when the samples were stored for a long time), the milk has to be slowly heated up to 35-40 °C. At the same time it has to be slowly shaken. The cream, stuck to the walls of the vessel is removed. The sample is poured several times and is cooled down (advisable up to 20 °C).



If there is separated liquefied fat or white particles with irregular form on the vessel's walls reliable results could not be received.

### **Whey**

Before making analyses the whey sample is filtered through double sheet gauze put over the glass funnel in order to separate the fat grains get into liquid by incidence and if it is needed the sample is tempered and carefully stirred.

### **Buttermilk**

Before making analyses the buttermilk sample is filtered through single sheet gauze put over the glass funnel in order to separate the big protein particles and if it is needed the sample is tempered and carefully stirred.

### **Cream**

The sample is slowly warm up to 35 – 40 °C in water bath. The fat is dispersed wholly by carefully shaking and if necessary, by stirring with glass stick. The sample is poured from vessel to vessel several times and is cooled down (advisable to 20 °C). If after this procedure the sample is not homogenous, the measurement is not carried out.

Sample for analyses is prepared from homogenized cream by diluting it with distilled water in degree, sufficient for the components of the diluted cream to be reached in the measuring range of the analyser.



## **APPENDIX 4 SAMPLING AND PREPARATION OF SAMPLES FOR VERIFICATION THE ACCURACY OF THE MILK ANALYSER, MAKING CORRECTIONS AND RECALIBRATION.**

### **1. Necessary consumables and devices**

- Distilled water;
- Minimum 3 milk samples with known content of fat, SNF, protein, density, lactose, solids;
- Heating water bath;
- Cooling water bath or chamber;

### **2. General**

Milk sampling and storage of samples of raw, thermally treated milk and its derivatives (cream, whey, butter-milk etc.) aiming verification the accuracy of the analyser, making corrections and recalibration is accomplished following the recommendations below:

- Sample to be taken from homogeneous batches, observing all the requirement;
- The sample's volume to be enough for making minimum 5 measurements for each sample or not less than 0.5 l;
- The samples to correspond to the standard physic-chemical and microbiological requirements, to be pure, without admixtures, without added cleaning or other unusual substances and without falsifications;
- Do not use samples with total acidity of milk more than 17<sup>o</sup>T;
- Vessels, where the samples will be handled have to be clean, dry, glass, metal or other suitable material, to be tightly closed with rubber or other stopples. The stopples not to absorb water and fat and not to influence the analyzed sample content;
- Till the start of the analyses the samples are stored in conditions, assuring preservation of their content and quantities (advisable low temperature – 1-3 °C).

For longer storage of the samples a preservative is added as was already described in p.9.1.1, and then the sample has to be well stirred.

### **3. Representative Samples**

The samples have to be representative for the corresponding milk type. Changes in the analyzed parameters in the samples have, if possible, to cover the whole measuring range – i.e. used samples to be with low, middle and high content of the analyzed components.

Exemplary recommended values:

#### Cow milk

Parameter	Low value	High value
% fat content	2,00	6,00
% Solids-Non-Fat content	8,00	9,00

The Lactose percentage content (4,0-5,5; average-4, 7), Protein (2,00-4,00; average-3, 3), salts (0,7-0,8) is proportional to the SNF content. When preparing samples these values vary within limited bounds.

#### Sheep milk

Parameter	Low value	High value
% fat content	5,50	10,00
% Solids-Non-Fat content	9,00	11,50

The Lactose percentage content (average - 4,6), Protein (average - 5,8), salts (average - 1,0) is proportional to the SNF content. When preparing samples these values vary within limited bounds.

#### Buffalo milk

Parameter	Low value	High value
% fat content	5,50	10,50
% Solids-Non-Fat content	9,00	11,00

The Lactose percentage content (average - 4,7), Protein (average - 4,3), salts (average - 0,8) is proportional to the SNF content. When preparing samples these values vary within limited bounds.

#### Goat milk

Parameter	Low value	High value
% fat content	2,00	6,00
% Solids-Non-Fat content	8,00	9,00

The Lactose percentage content (average - 4,6), Protein (average - 3,7), salts (average - 0,8) is proportional to the SNF content. When preparing samples these values vary within limited bounds.

## Cream

Parameter	Low value	High value
% fat content	8,00	20,00
% Solids-Non-Fat content	2,50	5,00

The cream samples are diluted with distilled water. Degree of dilution is 2-3 times, in dependence of the initial fat content in the cream.

## Whey

Parameter	Low value	High value
% fat content	0,20	0,80
% Solids-Non-Fat content	5,00	7,50

The content of fat and SNF in the whey depends on the kind of the dairy product as a result of which the whey is received.

## 4. Samples preparation

Milk – raw or thermally treated

For raw milk sample with average content of the analyzed components is advisable to be used milk, collected from at least 10 animals from the most commonly met breed in the region where the analyser will be functioning.

Low fat and high fat samples are prepared on the following way:

Available fresh or thermally treated milk is poured in a separating funnel, which is place in a refrigerator for at least 12 hours at temperature 4 - 6 °C in order to stratify. For a bigger stratification a longer time is required.

The layer at the bottom is poured in a vessel. It is well mixed by pouring it from vessel to vessel and is warmed up to 40 °C in a water bath.

The upper layer is poured in another vessel.

Using the certified methods the density and the concentration of the analyzed components - fat, protein, SNF, lactose, salts are determined.



The analyser's accuracy depends only on the correctness of the chemical analyses of the components in the samples and the normal acidity during calibration!

It is recommended the first cow milk sample with low fat content to be with the following parameters:

1.8-2% FAT; 8.7-9% SNF; 3,3-3,5 % Protein; 4,8-4,9% Lactose; 0,75 Salts; 1030-1033 kg/m<sup>3</sup> Density.

The second cow milk sample with high fat content to be with the following parameters:

5-5,5% FAT; 8.4-8,79% SNF; 3,1-3,2% Protein; 4,6-4,7% Lactose; 0,7 Salts; 1028-1029 kg/m<sup>3</sup> Density.

Samples with medial values of the separate parameters are received by mixing the two boundary values in a definite proportion.

Preserve the samples, using above described method for their longer storage.

When using samples, stored shortly, preliminary pour the sample from one vessel to another in order to distribute the milk components evenly paying attention not to form foam in the sample.

When the samples are stored for a longer period it is recommended to warm it up to 35-45 °C, and the vessel to be shaken carefully. In case that there is a cream stuck on the vessel's surfaces – remove it. The sample is poured from vessel to vessel several times and is cooled down (advisable to 20 °C /.



If there is separated liquefied fat or white particles with irregular form on the vessel's walls this sample could not be used.

### **Whey and buttermilk**

The samples are poured several times from vessel to vessel and if needed gradual heating with stirring with cooling down is done.

### **Cream**

The sample is slowly warmed up to 35 – 40 °C in water bath. The fat is dispersed wholly by carefully shaking and if necessary, by stirring and pouring it from vessel to vessel till its full homogenization.

From homogenized cream is prepared sample for analyses by diluting it with distilled water in degree, sufficient for the components of the diluted cream to be reached in the measuring range of the analyser.

## **5. Advisable scheme for independently determination the content of different parameters in milk and its derivatives**

When is not possible to use the help of authorized laboratories and above mentioned milk analysers we recommend you to follow the scheme:

**5.1. For cow milk (whole milk, low fat, skimmed milk) and UHT milk**

Determination of fat content – Gerber’s method, described in *Appendix Methods*.

Density determination – using aerometer, described in *Appendix Methods*.

SNF determination – by formula – p. 3.2.3.1.B

Determination of Lactose content – by formula – p.3.2.3.2.A

Determination of salts content – by formula – p. 3.2.3.3.A

Total protein content determination – by formula – p. 3.2.3.4.A

**Example:** Determination of the quality parameters for two samples cow milk (low fat and high fat), obtained and prepared according p. 2.3.1 and 2.4.1.

First – determine the fat content in the samples, using the Gerber’s method (p.3.2.)

Suppose that for the first sample the result is 2,0 %F, for the second – 5,9 %F.

Second – determine the milk density, using aerometer (p.3.1.)

Suppose that the results are 1,0316 for the first sample and 1,0274 for the second

Third – Calculate the SNF content using the formula (p.3.2.3.1.B)

$$SNF = \frac{0,075 * 2,0 + 100 - 100 / 1,0316}{0,378} = 8,50\%$$

$$SNF = \frac{0,075 * 5,9 + 100 - 100 / 1,0274}{0,378} = 8,23\%$$

Fourth – determine the lactose content by the formula (p.3.2.3.2.A)

$$Lact. = SNF * 0,55 = 8.50 * 0.55 = 4.67 \%$$

$$Lact. = SNF * 0,55 = 8.23 * 0.55 = 4.53 \%$$

Fifth – determine the salts content by formula (p.3.2.3.3.A /

$$Salts = SNF * 0,083 = 8.50 * 0.083 = 0.71 \%$$

$$Salts = SNF * 0,083 = 8.23 * 0.083 = 0.68 \%$$

Sixth – determine the total protein content by formula (p.3.2.3.4.A)

$$Proteins = SNF * 0,367 = 8.50 * 0.367 = 3.12 \%$$

$$Proteins = SNF * 0,367 = 8.23 * 0.367 = 3.02 \%$$

So, when calibrating the milk analyser we’ll use samples with the following parameters:

	<u>I st sample</u> (low fat)	<u>II nd sample</u> (high fat)

milk fat	2,00	5,90
SNF	8,50	8,23
density	1,0316	1,0274
lactose	4,67	4,53
salts	0,71	0,68
proteins	3,12	3,02

## 5.2. For sheep milk

Determination of fat content – Gerber’s method, described in Methods p. 3.4.

Density determination – using aerometer, described in Methods p. 3.3.

SNF determination – by formula – p. 3.2.3.1.B

Determination of Lactose content – by formula – p. 3.2.3.2.A

Determination of solids/salts content – by formula – p. 3.2.3.3.A

Total protein content determination – by formula – p. 3.2.3.4.A

**Example:** Determination of the quality parameters for two samples sheep milk (low fat and high fat), obtained and prepared according p. 2.3.1 and 2.4.1.

First – determine the fat content in the samples, using the Gerber’s method (p.3.2.)

Suppose that for the first sample the result is 5,6 %M, for the second – 9,8 %M.

Second – determine the milk density, using aerometer (p.3.1.)

Suppose that the results are 1,0352 for the first sample and 1,0300 for the second

Third – Calculate the SNF content using the formula (p.3.2.3.1.B)

$$SNF = \frac{0,075 * 5,6 + 100 - 100 / 1,0352}{0,378} = 10,11\%$$

$$SNF = \frac{0,075 * 9,8 + 100 - 100 / 1,0300}{0,378} = 9,65\%$$

Fourth – determine the lactose content by the formula (p.3.2.3.2.A)

Lact. = SNF \* 0,45 = 10.11 \* 0.45 = 4.55 %

Lact. = SNF \* 0,45 = 9.65 \* 0.45 = 4.34 %

Fifth – determine the solids content by formula (p.3.2.3.3.A /

Salts = SNF \* 0,075 = 10.11 \* 0.075 = 0.76 %

Salts. = SNF \* 0,075 = 9.65 \* 0.075 = 0.72 %

Sixth – determine the total protein content by formula (p.3.2.3.4.A)

Proteins = SNF \* 0,475 = 10.11 \* 0.475 = 4.80 %

Proteins = SNF \* 0,475 = 9.65 \* 0.475 = 4.58 %

So, when calibrating the milk analyser we'll use samples with the following parameters:

	<u>I 1st sample</u> (low fat)	<u>II nd sample</u> (high fat)
milk fat	5,60	9,80
SNF	10,11	9,65
density	1,0352	1,0300
lactose	4,55	4,34
salts	0,76	0,72
proteins	4,80	4,58

### **5.3. For wheat, buttermilk and cream**

Determination of fat content – Gerber's method, described in Methods p. 3.4.

Density determination – using aerometer, described in Methods p. 3.3.

SNF determination – using drying - p. 3.3. and formula – p. 3.2.3.1.A

## APPENDIX 5: EASY CALIBRATION OF THE MILK ANALYSER BY CALCULATING THE BASIC PARAMETERS VIA FORMULAS



DETERMINATION OF THE BASIC PARAMETERS IN THE MILK SAMPLE BY USING FORMULAS IS NOT AS PRECISE AS USING THE ARBITRARY METHODS, BUT IS SUITABLE FOR USAGE IN FIELD WORK.

### 5.1. Determination some of the parameters by formulas

There is dependence between the different parameters in milk and its density, which may be expressed with mathematical equation. On this base different formula, tested and confirmed by the classical laboratory methods for analyses, are developed. We recommend the following:

### 5.2. SNF determination.

For determination of SNF the correlation dependence exists between the milk's density, fat and SNF in the milk. When the density and the fat are known, the SNF can be calculated.

There are several formulas with different applicability.

A/ When the salts and fat are known

SNF is calculated by subtracting the fat percentage from the salts.

$$SNF = \text{Salts} - F (\%)$$

Where

Salts – salts in (%) ,

F – fat content in (%) ,

This formula is used for determination of SNF in whey, buttermilk, and cream.

B/ Known quantity of fat and density (most commonly used method when maximum accuracy is needed).

We recommend the following formula:

$$SNF = \frac{0,075 * F\% + 100 - 100 / \text{density}}{0,378}$$

This is a universal formula and actual for milk of almost all kind of cows and sheep all over the world.



### 5.3. Determination of lactose content

We recommend the following formulas:

A/ for cow milk

$$\text{Lact.} = \text{SNF} * 0,55 (\% )$$

Where

SNF – content of SNF in percentages (%),

0,55 – constant coefficient.

B/ for sheep milk

$$\text{Lact.} = \text{SNF} * 0,45 (\% )$$

Where

SNF –solids-non-fat content in percentages (%),

0,45 – constant coefficient.



This is an actual coefficient for sheep breeds on the territory of the Balkan Peninsula.

### 5.4. Determination of salts content

We recommend using the following formulas:

A/ for cow milk

$$\text{Salts} = \text{SNF} * 0,083 (\% )$$

Where

SNF – solids-non-fat content in percentages (%),

0,083 – constant coefficient.

B/ for sheep milk

$$\text{Salts} = \text{SNF} * 0,075 (\% )$$

Where

SNF – solids-non-fat content n percentages (%),

0,075 – constant coefficient.



This is an actual coefficient for sheep breeds on the territory of the Balkan Peninsula.

### **5.5. Determination of total proteins content**

We recommend using the following formulas:

A/ for cow milk

$$\text{Protein} = \text{SNF} * 0,367 (\% )$$

Where

SNF – solids-non-fat content in percentages (%),

0,367 – constant coefficient.

B/ for sheep milk

$$\text{Protein} = \text{SNF} * 0,475 (\% )$$

Where

SNF – solids-non-fat content in percentages (%),

0,475 – constant coefficient.



This is an actual coefficient for sheep breeds on the territory of the Balkan Peninsula.

## APPENDIX 6 FREEZING POINT DETERMINATION

### 1. Methods for determination.

The milk analyzer determines the freezing point of each sample and the quantity of added water. The milk analyzer does not measure the freezing point, but calculates it from the components it depends on. The basic components in the milk are water, solids, lactose, FAT, proteins, minerals (salts) and acids. The freezing point depends only on the diluted in the milk components and quantity of the solvent (in the milk it is water). The ultrasonic technology allows direct measurement of FAT, proteins, lactose + salts (the soluble components, only influencing the freezing point), and the quantity of the solvent in % is determined by  $100\% - \text{total solids \%}$ ,  $\text{total solids} = \text{lactose \%} + \text{FAT \%} + \text{proteins \%} + \text{salts \%} + \text{acids \%}$ .

Without understanding the meaning of the freezing point – determined or shown from the milk analyzer added water result easily may lead to a mistake for the value of this parameter.

### 2. The basic freezing point.

Milk freezes at lower temperature than water. The average freezing point of the raw milk in the most regions is at about  $-0,540^{\circ}\text{C}$ . The average reading for your region is called “basic” freezing point.

The freezing point of milk is a “physiological constant”. This does not mean that it will not vary. In fact feed, breed, season, time of lactation, climate, whether the sample is taken at the beginning, middle or end of lactation – all these factors will have an effect on the freezing point of the individual sample. This means that there is an average value of all these numbers. The more samples used in obtaining this average, the more reliable it is as a base. Or the basic freezing point is an average of freezing points of milk, taken from many cows. When a laboratory checks a producer, it is only comparing the average of the producer’s cows against a larger area average.

The Health authorities establish the basic freezing point or agriculture departments in some regions, sometimes by universities, separate dairy producers, or their associations. Frequently, tolerances have been established on top of a basic freezing point to allow some variations in the milk as well as device or operator variations.

Without mentioning the basic freezing point, the Association of Official Analytical Chemists now recommends an upper limit freezing point at  $-0,525^{\circ}\text{C}$  (2,326 standard deviations above the most recently determined North American average of  $-0,5404^{\circ}\text{C}$ ), below which there will be at 95%

confidence that will show 99% of all freezing point determinations on unwatered milk:

“if the freezing point is  $-0,525^{\circ}\text{C}$  or below, milk may be presumed to be free of water or may be confirmed as water free by tests, specified below. If the freezing point is above  $-0,525^{\circ}\text{C}$ , milk will be designated as “presumptive added water” and will be confirmed as added water or added water free by tests specified below. Evaluate extreme daily fluctuations in the freezing point of herd, pooled herd, or processed milk for presence of added water”.

“Presumed added water”, as described above, must be “confirmed” by means of tests on authentic milk samples obtained as specified in the AOAC METHODS.

After determination the freezing point of your sample via the milk analyzer, the added water is calculated using the following formula:

$$\text{AddedWater} = \frac{\text{FrPoint}_{\text{Base}} - \text{FrPoint}_{\text{Calc}}}{\text{FrPoint}_{\text{Base}}} * 100[\%]$$

Where:

FrPointBase is the basic freezing point

FrPointCalc is measured freezing point

**Note:**

If the freezing point is not correctly determined, the result for the added water is not valid. In this case results for FrPoint and AddWater are not shown on the display and on the printout from the printer. If the density of the measured sample is 0, the result for AddWater is not valid and is also not shown on the display and the printouts.

Sample:

First variant

If you've entered for milk analyzer basic freezing point  $-0.520^{\circ}\text{C}$  (according article 5.9 of the EU Milk Hygiene Directive 92/46/EEC), measured freezing point  $-0.540^{\circ}\text{C}$ , using the above pointed formula you'll receive  $-3,8\%$ . Because it is not possible the added water to be negative value, the milk analyzer indicates 0% added water. The reason for this is the tolerance in the basic freezing point, reasons for which are described below.

If in the same milk we add 3,8% water, and the basic freezing point is the same, the milk analyzer will measure freezing point  $-0.520^{\circ}\text{C}$ , and will indicate again 0% added water.

Second variant

If you've entered for the device basic freezing point  $-0.540^{\circ}\text{C}$ , measured freezing point  $-0.540^{\circ}\text{C}$ , the milk analyzer will indicate 0%. When you add 3,8% water, the device will indicate 3,8%-added water.

From the above mentioned follows that it is very important to enter correct basic freezing point in the device.

The device's results for added water may give information about doubt of added water in the milk and the exact value of this added water may be determined after a "cowshed sample" is taken and the result for the freezing point, measured by the milk analyzer of the "cowshed sample" is entered as basic freezing point in the formula for calculation of added water.

Then the result from this formula will give us the absolute value of the added water for the corresponding milk supplier.

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