Milkotronic Ltd

LACTOSCAN SLC

MILK ANALYZER
4 characters LED display

Operation manual

Switching Adapter

- **Input**: 100 - 240 V ~1.6 A max. 50-60 Hz
- **Output:** +12 V ⎓ 4.17A min.
- **Output power:** 50 - 65 W

Measurement modes

- cow milk ☒
- sheep milk ☒
- UHT milk ☒
- goat milk ☐
- buffalo milk ☐
- camel milk ☐
- cream ☐
- whey ☐
- ice-cream mixtures ☐
- recovered milk ☐
- other ☐

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 producing company 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.

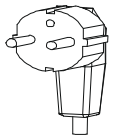
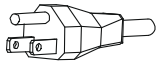
7. Handle the liquids the device works with carefully, following all the instructions for their preparation.

PARTS AND ACCESSORIES

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

№	Description	Item №	pcs
1.	Ultrasonic portable milk analyzer		1
2.	Operation manual		1
3.	Sample holder		2
4.	Syringe		1
5.	Spare Pipes		2
6.	12 V DC Power Supply Milk Analyser Cable	90-1801-0009	1
7.	Alkaline cleaning product Lactodaily 100 g		1
8.	Acidic cleaning product Lactoweekly 100 g		1

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

№	Description a) included in the set: ☒ b) not included in the set (may be additionally bought): ☐	Item №	pcs	☒ / ☐
9.	Handle		1	☐
10.	ECS POS Serial Printer		1	☐
11.	12 V Serial Printer Power Supply Cable	90-1801-0007		
12.	RS232 Interface Cable - Milk Analyser – Serial Printer	90-1801-0008	1	☐
13.	RS232 Interface Cable-Milk Analyser-IBM PC	90-1801-0010	1	☐
14.	Carrying case – plastic		1	☐
15.	Plug type		1	☐
			1	☐

1. 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 (^{o}C), freezing point, solids, as well as density of one and the same sample directly after milking, at collecting and during processing.

2. TECHNICAL CHARACTERISTICS

2.1. Working modes characteristics:

The program of the milk analyzer has four working modes.

2.1.1. r1.1 Measurement mode milk / dairy product – first type

2.1.2. r1.2 Measurement mode milk / dairy product – second type

2.1.3. r1.3 Measurement mode UHT 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. r2. Cleaning

Serves for cleaning the analyzer's measurement system. To clean the analyzer put a glass filled with cleaning solution (type 1) or water in the recess of the analyzer. In mode **r2. cleaning** the analyzer makes 8 suction cycles and stops. After cleaning or switching on the analyzer's power supply, before starting measurement, suction and releasing the sample is accomplished.

The operator will be reminded to start this mode – by a sound signal in 1 sec. period in the following cases:

- Idle mode, without taking measurements (switched on power supply without taking measurement) – after 55 min.

- 15 min after the last measurement of the milk sample, but no more than 55 minutes after switching on the power supply. After cleaning or water measurement, a new measurement is started in the above-described intervals.

2.2. Measuring range:

Fat	from 0.01 % to 20 %
SNF	from 3 % to 15 %
Density *	from 1015 to10 40 kg/m^3
Proteins	from 2 % to 7 %
Lactose	from 0.01 % to 6 %
Water content	from 0.0 % to 70 %
Temperature of milk	from 1 °C to 40 °C
Freezing point**	from –0,400 to –0,700 ° C
Solids**	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^3. To determine the milk density, write down the result from the display and add 1000.
Example: result 31,20; density = 1000 + 31,20 = 1031,2 kg/m^3

** Measured values for freezing point and solids are printed out.

2.3. Maximum permissible absolute error:

Fat	± 0.10%
SNF	± 0.15%
Density *	± 0.3 kg/m^3
Proteins	± 0.15%
Lactose	± 0.20%
Water content	± 3.0%
temperature of milk	± 1 °C
Freezing point	± 0.001°C
Solids	± 0.05%

The difference between two consequent measurements of one and the same milk may not exceed the maximum permissible absolute error. This difference is ensured when working under correct ambient conditions.

2.3.1 Correct ambient conditions for optimal results of analysis.

air temperature	from 10 °C to 35 °C
relative humidity	from 30% to 80%
power supply	220V (110V)

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 methods: Gerber – for fat, gravimetric – for SNF, Keldhal – for protein. The boundary for maximum variation of repeatability when the power supply voltage is from +10 to – 15% from the nominal voltage values (220V) 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.

2.4. Dimensions:.....……...……240/220/100 mm, mass 3,0 kg

2.5. Continuous working time:.....…...……..nonstop

2.6. Milk sample volume per one measure:..……25 cm^3 (= 25 ml.)

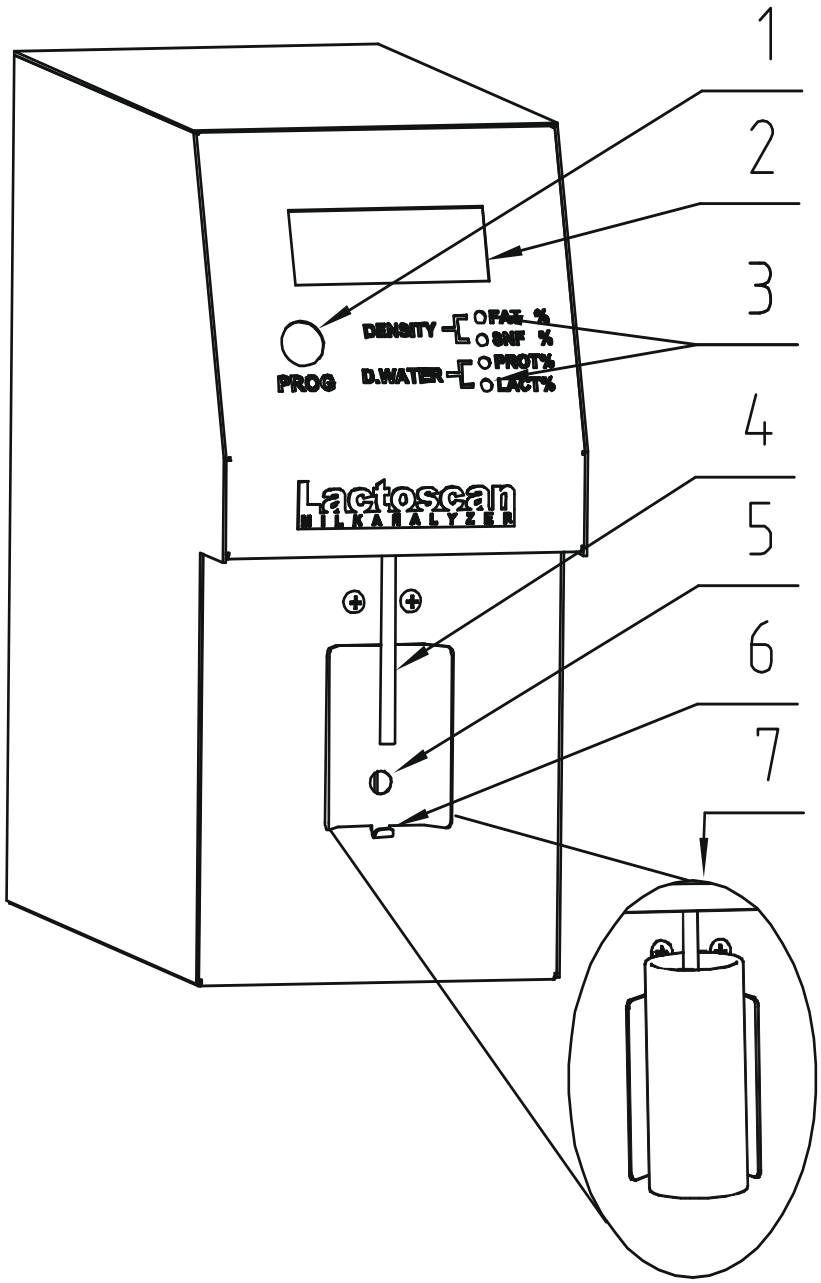
2.7. Connecting to 12 V DC power supply.
If there is a need the analyzer to work on place without electrical supply available, then it could be powered by car battery or other 12 V DC external power supply. Use the 12 V power supply cable (art. number 90-1801-0009, Parts and Accessories, point 13).

2.8. Connecting to IBM PC.
The milk analyser can be connected to IBM PC using the RS232 interface cable (art. number 90-1801-0010, p.6 Parts and Accessories, point 10). In order to make the connection: switch off both the analyser and PC. Connect the RS 232 cable towards Serial interface (fig. 2, point 9) and towards the computer. Turn on both analyser and PC. Now the milk analyzser is ready to communicate with IBM PC.

2.9.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 (see fig.2) – “Serial printer connector”. The printer should be connected to the “12 V printer output” on the device rear panel, fig.2. Connect it via cables, delivered by the company-producer. If the printer is connected directly to the 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.

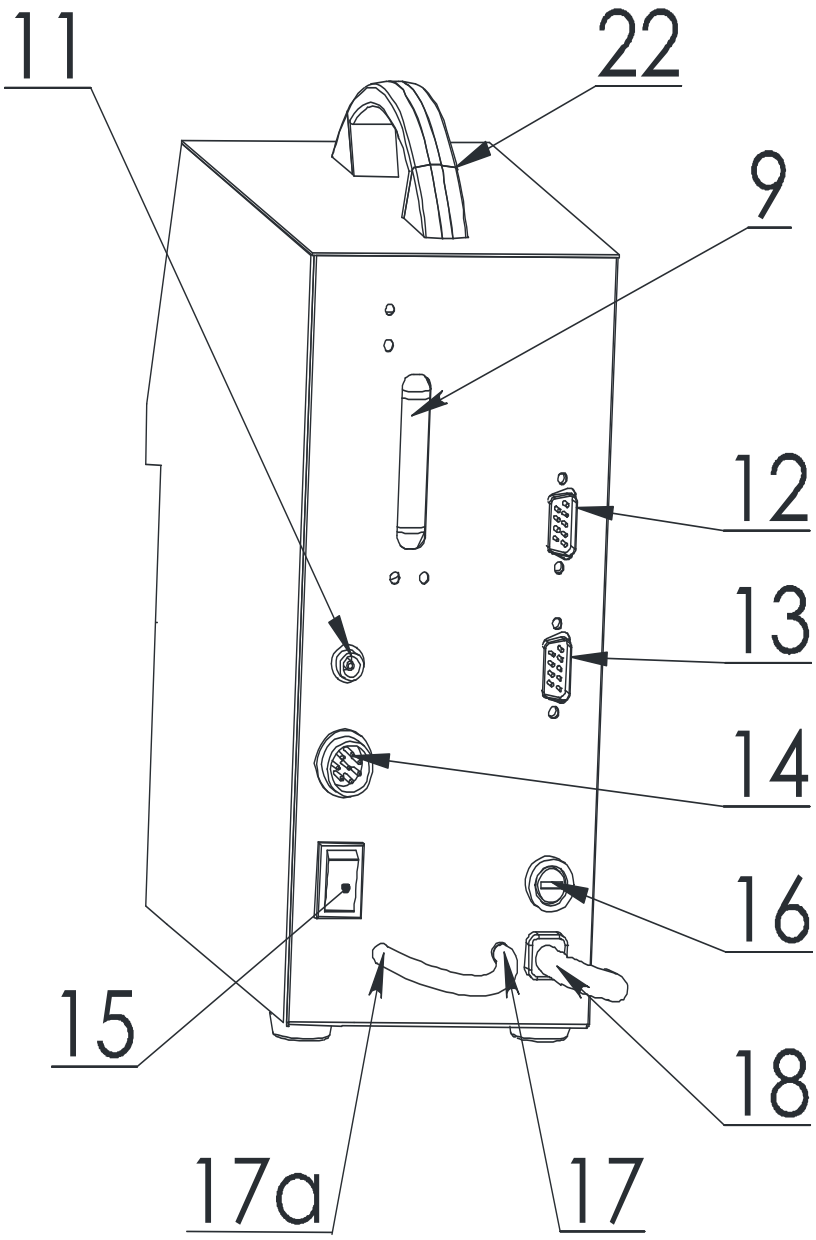
2. THE ANALYZER AND ITS COMPONENTS

Fig. 1 Front panel



1. Program Button
2. Digital Indicator (LED display)
3. LEDs
4. Input pipe
5. Start button
6. Hanger
7. Sample holder

Fig. 2 Back panel



9. Glass cuvette

11. 12 V printer output

12. Serial printer connector

13. Serial interface RS232

14. 12 V input

15. Power switch

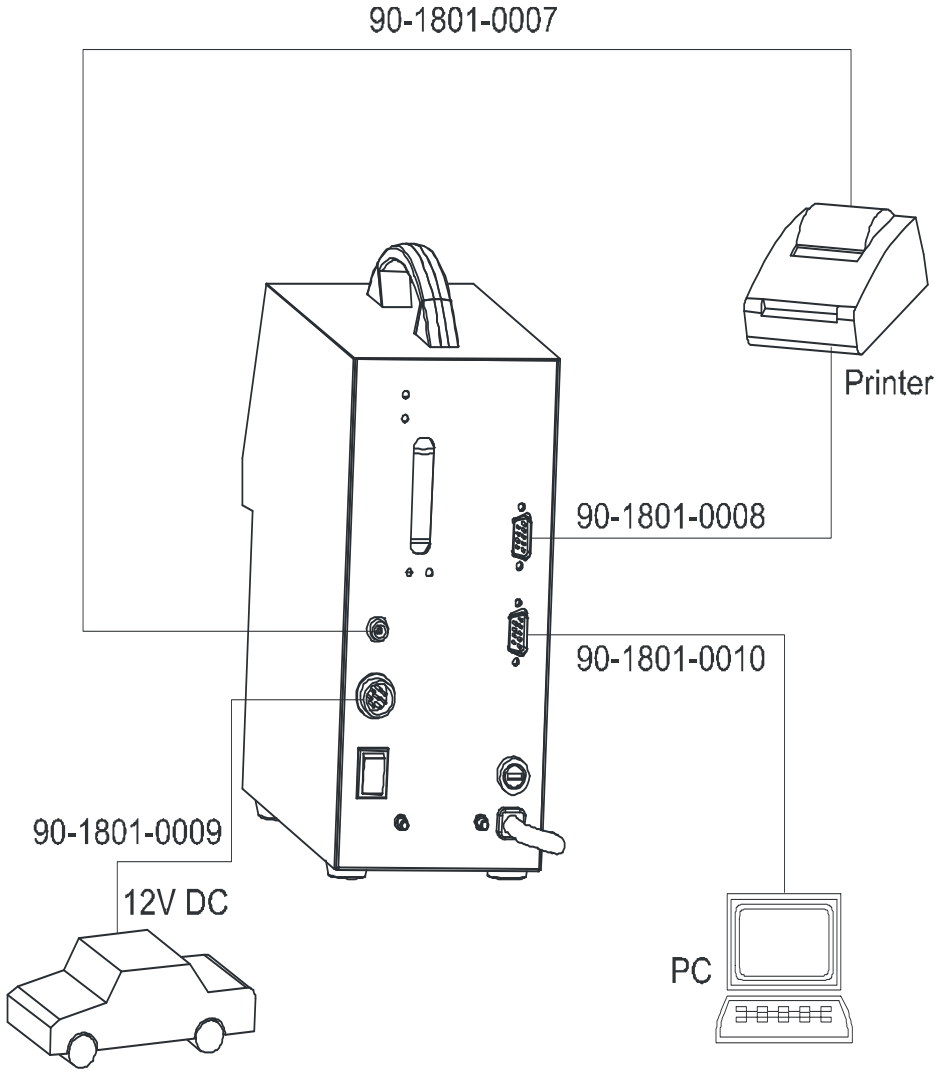
16. Fuse

17. Connecting
17a. pipe orifices

18. Power cable

22. Handle

fig.3. Cable Description

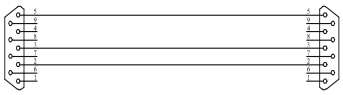


90-1801-0007

12 V Serial Printer Power Supply Cable (Thermal printers type EP-50 see http://www.datecs.bg)

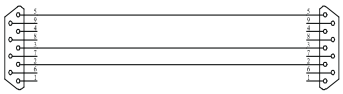
90-1801-0008

RS232 Interface Cable - Milk Analyser – Serial Printer standard for EP-50 (for other Printer, see printer's manual)

2. TxD 3. RxD 5.GND	Milk Analyser DB 9-pin male		Serial Printer DB 9-pin male	2. Receive Data (RxD) 3. Transmit Data (TxD) 5. Signal Ground (GND)

90-1801-0010

RS232 Interface Cable - Milk Analyser – IBM PC

2. TxD 3. RxD 5.GND	Milk Analyser DB 9-pin male		PC DB 9-pin female	2. Receive Data (RxD) 3. Transmit Data (TxD) 5. Signal Ground (GND)

90-1801-0009

12V DC Power Supply Milk Analyser Cable

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

4. ANALYZER INSTALLATION

4.1. Put the analyzer on the working place, providing good ventilation and not in the vicinity of heat providing devices or sources.

4.2. Check if the switch **"Power"** (fig.2, 15) 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 (fig.2, 18) to the electrical outlet.

4.3. Set the switch **"Power"** to "I" position. For a short time the version of the analyzer's software is shown on the display (fig.1, 2), then the modes r1.1, etc. and at the end after about 5 minutes, the serial number of the analyzer appears. When the device is ready for work a beep can be heard and the indicator **r1.1** lights up. The information, indicated during this procedure identifies the device.

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

4.4. If you wish to work in the other working mode press and hold the button **PROGRAM** (fig.1, 1) on the front panel of the device. Then, without releasing the button **PROGRAM**, put the sample-holder (fig. 1, 7) on its place. The possible operation modes will be displayed "**r1.1**"- mode 1; "**r1.2**"- mode 2; "**r1.3**"-mode 3; "**r2**"-cleaning mode. Release the button **PROGRAM** when the indicator shows the desired mode of operation. The device starts the chosen mode.

5. MAKING ANALYSES

5.1 The milk sampling.

When taking samples follow the below mentioned standard procedures to avoid any air enclosure in the milk.

5.1.1. Before taking the sample from a quantity of milk that has to be qualified, the milk has to be stirred with special mixing tool.

5.1.2 Taking a sample from vessels, which do not allow good stirring of milk is not acceptable. First the milk has to be poured in standard milk measuring vessel and stirred several times with the floating level indicator. If there is no floating level indicator, then the milk has to be poured 4-5 times from vessel to vessel before taking the sample.

5.1.3. Taking a sample has to be done with special tube made from aluminum, stainless steel or glass with inner diameter of 8-10 mm. **The sample must be at least 100-150 ml.**

5.1.4. If transportation of the sample to a laboratory is necessary then it has to be preserved with potassium bichromat or transported in a cooling bag.

5.1.5. Prepare the milk samples at least two hours after milking in order to avoid any air in the samples.

5.1.6. Just before making measurement with the milk analyzer, pour the sampling milk from vessel to vessel 4-5 times. Pour it in the plastic sample-holder (fig.1, 7) included in the set of the analyzer and start measurement.

5.1.7. Before analyzing the milk sample check whether the acidity of the milk is lower than 22^{o} T.

5.1.8. If the sample has been cooled leading to butter on the surface, it has to be heated to 40 ^{o}C and then cooled to 20-25 ^{o}C, in order to distribute the fat in an evenly way before the measurement takes place.

5.2. Making measurement

5.2.1. For working with the modes r1.1, r1.2 and r1.3 the sample has to be well stirred before pouring it in the sample-holder.

Put the sample-holder with the required quantity of milk in the recess (fig.1, 6) of the analyzer in such a way that the starting button (fig.1, 5) is activated.

The analyzer sucks the milk, makes the measurement and returns the milk in

the sample-holder. The values of the first milk sample achieved after the required start up procedure with distilled water have to be ignored. Make a second measurement with a fresh sample of the same milk, so not using again the first milk sample that already passed the analyzer. From now on one measurement per sample is sufficient during the whole series of analysis.

5.3. Displaying the results

5.3.1. When the measurement is finished, the sample returns in the sample-holder and the display (fig. 1, 2) shows consequently the results for fat **(FAT%)**, nonfat milk solids **(SNF %)** and density **(DENSITY).**
- When fat is displayed the diode **"FAT%"** lights (fig.1, 2)
- When nonfat milk solids are displayed the diode **" SNF %"** lights (fig.1, 3)
- Both diodes **" DENSITY"** light (fig.1, 3) when the density results are displayed

When there is a content of water in the milk both diodes across ADD. WATER light permanently.

*If you need the results of proteins, lactose and water to be displayed, press the button **PROGRAM** (fig. 1, 1) for 1-2 seconds and release without removing the sample-holder.*

- When proteins are displayed the diode **" PROT%"** lights (fig.1, 3)

- When lactose is displayed the diode **" LACT %"** lights (fig.1, 3)

- When water is displayed both diodes **"ADD.WATER %"** light. If the content of water is below 3%, both diodes light permanently, but the indicator shows the value 0,00%.

By pressing the PROGRAM button you may again show the Fat results.

Measured freezing point value is printed out.

5.3.2. Write down the results in the form. The results follow one after the other till the sample-holder with a new sample of milk has been positioned in the recess. If the analyzer was connected to a computer or a printer, it sends the data to the computer or prints them.

6. CLEANING THE ANALYZER AT THE END OF WORKING DAY

6.1. Cleaning the analyser

This cleaning must be done when working with the analyzer will be stopped for hours.

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: www.johnsondiversey.com, www.ecolab.com www.calgonit.de

6.1.1. Set the power-switch (fig.2, 15) in the position “0”. Disconnect the power supply cable (fig.2, 18) from the electrical outlet. Disconnect the tube from the orifice **“INPUT”** (fig.2, 17a) at the back panel and connect the tube with the syringe (see drw. Cleaning).

6.1.2. Fill the sample-holder with cold water and put it in the recess (fig.1, 6). Let the water pass through the measuring system by pumping with the syringe.

6.1.3. Repeat this procedure with:

alkaline 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.

When the glass level-indicator is intensively contaminated you need to clean it several times with **hot** alkaline solution. If there are still residues of milk in the glass level-indicator (fig. 2, 9) leave the **hot** detergent solution for 7-8 hours in the analyzer and then clean the device with **hot** water.

acidic solution

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.

Lactoscan Daily

Alkaline detergent sanitizer with QAC.

General Description:
Alkaline powder product with QAC for combined cleaning and disinfecting of all types milk analysers Lactoscan according their instructions. Suitable for all water conditions and may be used for manual application as well as for automatic circulation cleaning. Non corrosive on most materials and mild to skin.

Application:
Automatic application:
1. Pre-rinse with sufficient water to remove milk residues
2. Circulate a 1% (10 g/l) cleaning solution for 10 to 20 minutes at a temperature above 40°C
3. Rinse thoroughly with tap water.

Manual application:
Use 1,0%(10g /l) after sufficient pre-rinsing at 30 to 40°C, soak for at least 10 minutes Rinse thoroughly with tap water.
Determination of concentration Titration of p-value with 1 N Hydrochloric acid

Special instructions:
Keep container closed and away from humidity.

Material Compatibility:
Stainless steel and Aluminium are not affected by the solution.

Physical and chemical properties:
Appearance: white powder
Odour: faintly of surfactant
pH-value (1%) 11,5
p-value: 4,5
Composition:
Carbonates, phosphates, silicates, surfactants, defoamer, disinfectant

Xi
Irritant

Hazard label: Xi, irritant

Risks:
R 36/38 - Irritating to eyes and skin
For health and safety information, refer to the Safety Data Sheet (SDS) for this

Lactoscan Weekly

Acidic cleaner and descaler

General Description:
Low foaming powder product for acidic cleaning of all types milk analysers Lactoscan according their instructions. The product very effectively removes milk stone and hard water deposits thus improving hygienic status of all milking equipment. May be used for manual application as well as for automatic circulation cleaning.

Application:
Automatic application:
1. Pre-rinse with sufficient water to remove milk residues
2. Circulate a 1% (10 g/l) cleaning solution for 10 to 20 minutes at a temperature above 40°C
3. Rinse thoroughly with tap water.

Manual application:
Use 1,0%(10g /l) after sufficient pre-rinsing at 30 to 40°C, soak for at least 10 minutes Rinse thoroughly with tap water.
Determination of concentration Titration of p-value with 1 N sodium hydroxide

Special instructions:
Keep container closed and away from humidity.

Material compatibility:
Stainless steel is not affected by the solution. Aluminium is slightly etched.

Physical and chemical properties:
Appearance: white powder
Odour: faintly of surfactant
pH-value (1%) 1,6
p-value: -4,5
Composition: Sulfamic acid, phosphates, sulfates, surfactant, defoamer

Xi
Irritant

Hazard label: Xi, irritant

Risks:
R 36/38 - Irritating to eyes and skin
R 52/53 - Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment
For health and safety information, refer to the Safety Data Sheet (SDS) for this product

6.1.4. Rinse with pure water of about 50° C applying 3-4 suctions with the syringe and three consequent changes of the water in the glass.

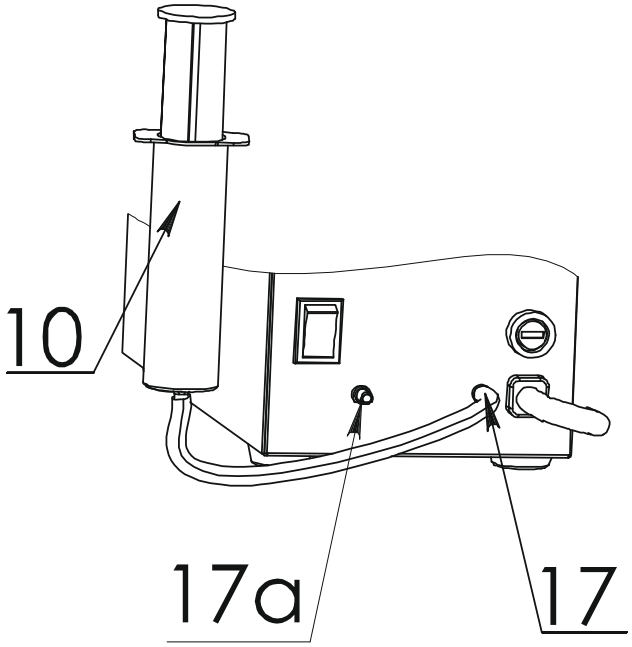
6.1.5. Disconnect the syringe and fill with air. Connect the syringe again and blow the air through the device in order to remove remaining water out of the device.

6.1.6. Connect the tube with the orifice.

If there is an interruption in the power supply during analyzing and milk remains in the device, immediately blow the milk out of the system and clean the device as explained above.

When cleaning with the syringe make sure that there is no excessive pressure during pumping. In that case there is a possibility of disconnecting a tube in the system. If you notice that the cleaning liquid does not enter the glass level-indicator (fig.2, 8), call the service center for help.

6.2. Rinsing
Clean the front panel (fig.1) with wet cloth and dry it.
Fig.4 Cleaning



Cleaning:
10. Syringe
17. Connecting
17a. pipe orifices

IMPORTANT!

THE MAIN REASON FOR MALFUNCTIONING OF THE DEVICE IS THE BAD CLEANING OF THE SYSTEM AFTER MAKING ANALYSIS!

When the glass level-indicator (fig.2, 8) is not clean your guarantee is not valid anymore and any repair has to be paid.

7. POSSIBLE MALFUNCTIONS AND ERROR MESSAGES, TROUBLESHOOTING

In the table below are described the possible malfunctions during the milk analyzer's exploitation. Also are described the ways of their repair / remedy. If the problem persists after all recommended measures are taken, please, connect the nearest service center.

Error message	**Possible problem /cause**	**Repair/remedy**
"Er.01" or **"Er.03"** Accompanied by a continuous sound signal	Overheated milk analyzer	Immediately switch off the analyzer. Pay attention the analyzer to be situated away from direct sunlight or heating devices. Wait 5-10 minutes till the device to cool down or to be normalized the ambient temperature and switch it on again.
"Er.02"	Insufficient quantity of the milk sample sucked in the system or air in the sample	The analyzer is ready to measure the next sample. In order to avoid the future appearance of the same error message, please, check that: - The sample is prepared according the instructions and there aren't any air bubbles in it. - 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. - Avoid the end of the suction pipe to be above the surface of the liquid (not dipped enough). - Avoid curdling of the milk sample. Clean immediately if there is a sample curdled in the system. - Check the state of the level indicator (point 9, fig.2). - Check the pipe, connecting cleaning inputs on the analyzer's rear panel.

		- In mode **Measurement**, after starting the measurement, remove the sample holder and see if there is no milk poured back in the sample holder.
"Er.04"	Sucked overheated sample	The analyzer is ready to measure the next sample. In order to avoid the future appearance of the same error message, please, check that: - The sample is prepared according the instructions and its temperature does not exceed the maximum permissible sample's temperature. - Complete the procedure for checking the analyzer in case of error message **Empty Camera.**

8. Test mode

8.1. Special (service) functions

Used for assigning different device parameters or starting the tests. To start them switch on the power supply while pressing the **Program** button (fig. 1, 1). On the display the number of the function **F.xx** is changing in one-second period. Release the button when the desired function is reached. This starts its execution.

8.2. Function descriptions on their numbers:

1. Calibration. After choosing this function the indication **CAL** appears on the display (fig. 1, 2). The device makes the measurement and sends the data towards the calibration system via the technological network. It is used in the production conditions when working with personal computer and program **LSC.EXE**. Starting this function automatically directs the analyzer **RS232** output towards a personal computer (on the analogy of function F.07 with computer output).

2. Display test. Periodically are shown the numbers from 0000 to 9999, and letters AAAA, bbbb, CCCC, dddd, EEEE, FFFF. LEDs (fig. 1, 3) are switched on consequently one after the other in direction from the top to the bottom.

3. Pump test. Max. number of executed sample suction and release cycles is 9999. When it is reached the program stops.

4. **Assigning the network number**. The device is indicated in the calibrating system under this number in case there is a connection with a personal computer. Valid numbers are from 0 to 15 inclusive. When the function is already selected (release of the Program button), the program stops, waiting new pressing of the button. When it is pressed, the valid numbers are displayed in one-second period, starting from currently stored. When needed number appears on the display, release the button. This stores the number in the device.

For correct work of the calibration system it is not allowed two or more devices to have one and the same network number.

Assigning the network number is necessary in production conditions when working with personal computer, program LSC.EXE or collecting data computer network.

5. Ultrasound test. Used in production conditions.

6. Device training – the sample is taken from the sample holder. Used in production conditions. The results are sent to the computer via the technological network.
Starting this function automatically directs the analyzer **RS232** output towards a personal computer (on the analogy of function **F.07** with computer output).

7. Choosing the data format towards output RS 232. Two possibilities – PC (personal computer) – measuring data collection program or Prn (serial printer) – printing the measurement results.
Serial exchange parameters are: **9600 bps, No parity, 8 bits data, 1 stop bit.**
Starting functions **F.01, F.06** automatically directs the analyzer **RS232** output towards a personal computer (on the analogy of function **F.07** with computer output). This means that after device test or calibration, in case of needed printing out the measuring results, the output **RS 232** has to be directed again towards the serial printer.

8. Reserve.

9. Sample's temperature correction. Allows increasing/decreasing the temperature from +9.9 to –9.9 degrees centigrade. Consequence of work is the same as it was already described in the correction functions for **FAT, COM** etc. Used in production conditions.

From 10 to 27. Measured parameters corrections in every separate calibration. Parameters are corrected independently one from the other for each calibration. Possibility to display the correction, to set to zero, to increase or decrease results with the steps pointed in the table below.
Consequence of work:
Release the **Prog.** button when the number of the needed function is reached (see table with functions below), which starts the corresponding function.
Press again the button **Prog.** The display shows the current value correction. Follows the measured value, which is possible to be increased with the corresponding step. When the maximum positive correction is reached the display shows values, with which the measured value can be decreased. This procedure is cyclically executed. When the needed correction value is reached, release the button to save the correction. Next pressing the button passes to the next function.

Correction functions table:

Function number						Calibration N:
Fat	SNF	Den	Lac	Sol	Kas(Pro)	
10	11	12	13	14	15	1
16	17	18	19	20	21	2
22	23	24	25	26	27	3

Below is the table with possible corrections, limits and changing steps:

Parameter	Increasement	Decreasement	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 %
Solids	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 ^{o}C	9.90 ^{o}C	0.1 ^{o}C

Example:
Choose (prepare) two milk samples: the first one near the lowest limit of the measuring range, the second one – near to the upper limit of the measuring range.
Measure these samples, using the reference methods (Gerber or Rose Gotlib for FAT, drying for SNF, Keldhal for Proteins).
And you receive:
First sample data from the reference methods: 2,3 FAT, 8,9 SNF, 3,3 Prot, 4,8 Lact.
Second sample data from the reference methods: 6 FAT, 8,5 SNF, 3,15 Prot, 4,7 Lact.
Make 5 time measurements of the same two samples with The milk analyser. Leave the results from the first measurement and calculate the average value of the rest 4 measurements for each parameter.
For example:
for the first sample measured with The milk analyser you've received the following results: 2,45 FAT, 8,9 SNF, 3,4 Prot, 4,8 Lac;
for the second sample, measured with The milk analyser you've received the following results: 6,15 FAT, 8,5 SNF, 3,25 Prot, 4,7 Lac.
It is obvious that the values for FAT and Protein differ from the reference values with +0,15 FAT; +0,1 Prot.

This means that you may correct the results by entering –0,15 for FAT using above described method and -0,1 for Prot.
After the correction the device must show:
First sample: 2,3 FAT, 8,9 SNF, 3,3 Prot, 4,8 Lact
Second sample: 6 FAT, 8,5 SNF, 3,15 Prot, 4,7 Lact.
On the same way you may correct the rest of the parameters.
Second readings:
If you receive the following results from the first sample, measured with The milk analyser: 2,1 FAT, 8,9 SNF, 3,4 Prot, 4,8 Lac;
second sample, measured with The milk analyser 6,2 FAT, 8,5 SNF, 3,25 Prot, 4,7 Lac.
It is obvious that the lower range is –0,2 , the upper +0,2. In this situation it is not possible to use a correction, but the device needs a new calibration according 8.2.3.4. Recalibrate using the same two milk samples.

When making corrections or calibrations you have to be 100% sure in the correctness of the results from the reference methods of analyses.

From 28 to 30. Functions for measured water corrections for each separate calibration. Possibility to display the correction, to set to zero, to increase with 9% and decrease with –9% the result. The consequence of work is corresponding to the above-described functions (10 to 27).

Water correction functions table:

Function number			**Purpose**
Calibr. 1 r1.1 - Cow	Calibr. 2 r1.2 – Sheep	Calibr. 3 r1.3 - UHT	
28	29	30	Water correction

New storage of the calibration coefficients values (recalibration) sets to zero the previous values of the corresponding calibration.

31 to 33. Functions for increasing the basic freezing point absolute value for each separate calibration. Serves for basic value editing. After the function is chosen (releasing the **Program button**), the program stops, waiting new pressing the button. After pressing it, in one-second period, the increased absolute value is shown on the display, starting at currently saved one. When the needed value is displayed, the operator has to release the button, which saves the new value in the device.

Functions table for increasing the absolute value of the basic freezing point:

Function number			**Purpose**
Calibration. 1	Calibration. 2	Calibration. 3	
31	32	33	Basic freezing point value absolute value increasing:

34 to 36. Functions for decreasing the basic freezing point absolute value for each separate calibration. Serves for basic value editing. After the function is chosen (releasing the **Program button**), the program stops, waiting new pressing the button. After pressing it, in one-second period,
the decreased absolute value is shown on the display, starting at currently saved one. When the needed value is displayed, the operator has to release the button, which saves the new value in the device.

Functions table for decreasing the absolute value of the basic freezing point:

Function number			**Purpose**
Calibration. 1	Calibration. 2	Calibration. 3	
34	35	36	Basic freezing point value absolute value decreasing:

Test table

Number	Function
1	Calibration/CAL/
2	Test display
3	Pump test
4	Assigning network number
5	Ultrasound test
6	Device preparation /CICL/
7	Choosing the data format towards output RS 232
8	Reserve
9	Temperature correction
10	Fat Correction, calibration N:1
11	SNF Correction, calibration N:1
12	Den Correction, calibration N:1
13	Lac Correction, calibration N:1
14	Sol Correction, calibration N:1
15	Kas(Pro)Correction, calibration N:1
16	Fat Correction, calibration N:2
17	SNF Correction, calibration N:2
18	Den Correction, calibration N:2
19	Lac Correction, calibration N:2
20	Sol Correction, calibration a N:2
21	Kas(Pro)Correction, calibration N:2
22	Fat Correction, calibration N:3
23	SNF Correction, calibration N:3
24	Den Correction, calibration N:3
25	Lac Correction, calibration N:3
26	Sol Correction, calibration N:3
27	Kas(Pro)Correction, calibration N:3
28	Water Correction, calibration N:1
29	Water Correction, calibration N:2
30	Water Correction, calibration N:3
31	Increasing the basic freezing point, calibration N:1
32	Increasing the basic freezing point, calibration N:2
33	Increasing the basic freezing point, calibration N:3
34	Decreasing the basic freezing point, calibration N:1
35	Decreasing the basic freezing point, calibration N:2
36	Decreasing the basic freezing point, calibration N:3

9. APPENDICES

9.1. Appendix 1 Milk sampling and preparation of samples for analyses

9.1.1. 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.), 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 have to be warmed up to 10-15 ^{O}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^3 for accomplishing the needed analyses.

9.2.1. 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.

9.1.3. 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.

9.1.4. 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 ^{O}C).
If there is a need of longer sample storing they have to be preserved; the most commonly used preservative is potassium dichromate /K2Cr2O7/ - 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 17^{o}T.

9.1.5. 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^{o} 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 ^{O}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 ^{O}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 ^{O}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 analyzer.

9.2. Appendix 2 Sampling and preparation of samples for verification the accuracy of the milk analyzer, making corrections and recalibration.

9.2.1. 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;

9.2.2. 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 analyzer, making corrections and recalibration is accomplished following the recommendations below:

- Sample to be taken from homogeneous batches, observing all the requirements in p.9.1.1.
- 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°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.

9.2.3. 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.

9.2.4. 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 analyzer 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 analyzer'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 Solids; 1030-1033 kg/m^3 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 Solids; 1028-1029 kg/m^3 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 ^{O}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 ^{O}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 ^{O}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 analyzer.

9.3. Appendix 3 Representative samples from milk and other milk derivatives for milk analyzer's calibration

9.3.1. General

The samples used for analyzer'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 to, if possible, 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.

9.3.2. 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.

9.3.2.1. Laboratory methods

9.3.2.1.1. 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 – Methods p.9.4.3.4.

9.3.2.1.2. 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 – Methods p.9.4.3.3.
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

9.3.2.1.3. 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 formaline
Formaline, 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.

9.3.2.1.4.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 formaline
More details for this method – in Methods p.9.4.3.6.

9.3.2.1.5. 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.

9.3.2.1.6. 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 – Methods p. 9.4.3.5.

9.3.2.2. Express methods by using another milk analyzers
It is possible another devices 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 analyzers 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.

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

9.3.2.3.1. 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 solids and fat are known
SNF is calculated by subtracting the fat percentage from the solids.
SNF = Solids – F (%), where
Solids – solids 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:

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

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

9.3.2.3.2. Determination of lactose content
We recommend the following formulas:
A/ for cow milk
Lact. = SNF * 0,55 (%), where
SNF – content of SNF in percentages (%),
0,55 – constant coefficient.
B/ for sheep milk
Lact. = 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.

9.3.2.3.3. Determination of salts content
We recommend using the following formulas:
A/ for cow milk
Salts = SNF* 0,083 (%), where

SNF – solids-non-fat content in percentages (%),
0,083 – constant coefficient.
B/ for sheep milk
Salts = 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.

9.3.2.3.4. Determination of total proteins content
We recommend using the following formulas:
A/ for cow milk
Protein = SNF * 0,367 (%), where
SNF - solids-non-fat content in percentages (%),
0,367 – constant coefficient.
B/ for sheep milk
Protein = 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

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 analyzers we recommend you to follow the scheme:
For cow milk (whole milk, low fat, skimmed milk) and UHT milk
Determination of fat content – Gerber's method, described in Methods p. 9.4.3.4.
Density determination – using aerometer, described in Methods p. 9.4.3.3.
SNF determination – by formula – p. 9.4.3.2.3.1.B
Determination of Lactose content – by formula – p. 9.4.3.2.3.2.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 = $\frac{0,075 \times 2,0 + 100 - 100/1,0316}{0,378}$ = 8.50 %
SNF = $\frac{0,075 \times 5,9 + 100 - 100/1,0274}{0,378}$ = 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 solids 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 analyzer we'll use samples with the following parameters:

	Ist sample (low fat)	II nd sample (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

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 = $\frac{0,075 \times 5,6 + 100 - 100/1,0356}{0,378}$ = 10.11 %
SNF = $\frac{0,075 \times 9,8 + 100 - 100/1,0300}{0,378}$ = 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 /
Solids = SNF * 0,075 = 10.11 * 0.075 = 0.76 %
Solids. = 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 analyzer we'll use samples with the following parameters:

	Ist sample (low fat)	II nd sample (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

For wheat, buttermilk and cream
Determination of fat content – Gerber's method, described in Methods p. 9.4.3.4.
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

9.4 Appendix 4 Methods

9.4.1. Determination of milk's density

9.4.1.1. General

Milk density is defined as relation between the mass of definite milk volume at temperature 20 ^{O}C and the mass of equal volume distilled water at temperature 4 ^{O}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 solids could be determined.

9.4.1.2. Sampling and preparation for analyses

Sampling milk or other milk derivatives and their preparation for analyses is done according Appendices № 1 and № 2.

Milk density is determined not earlier than 2 h after milking. The milk must be with temperature from 10 to 25 ^{O}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.

9.4.1.3. 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.

9.4.1.4. 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.

9.4.1.5. 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 ^{O}C.

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

9.4.1.6. Recalculating the values according lacto-density-meter at 20 ^{O}C.

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

Density recalculation towards 20 ^{O}C is done on the following way:

for every temperature degree over 20 ^{O}C from the received by the milkmeter value are added 0,2 ^{O} for the cow and goat milk and 0,25 ^{O} for sheep and buffalo milk lacto-density-meter degreed or 0,0002, respectively 0,00025 towards density; and for every temperature degree under 20 ^{O}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.

9.4.2. Determination of fat content in the milk and milk derivatives.

9.4.2.1. 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.

9.4.2.2. Sampling

Milk and milk derivatives sampling is done according Appendices № 1 and № 2.

9.4.2.3. 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.

9.4.2.4. 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 isoamilic alcohol from 1, 10 and 11 cm^3;
- Pipettes from 1 and 20 cm^3;
- Glasses from 25 till 50 cm^3;
- Centrifuge for Gerber;
- Water bath;
- Mercury thermometers up to 100 ^{O}C with value scale 1 ^{O}C;
- Sulphuric acid with density 1,82 at 20 ^{O}C for determination of fat content of the milk;
- Isoamilic alcohol for Gerber with density 0,811 to 0,812.

9.4.2.5. 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^{o}C) and is carefully shaken and tempered to 20±2^{o}C. The samples from whey and buttermilk are preliminary filtered through double layer gauze and is then tempered to 20±2^{o}C. Cream samples are placed in water-bath at temperature 35 till 40^{o}C, stirred till homogenous sample is received and cooled down to 20±2^{o}C.

9.4.2.6. Making measurement

With butyrometer for milk

For milk, whey and buttermilk.

With automatic or special for acids pipette are measured 10 cm^3 sulphuric acid with d=1,820 кg/m^3 at 20 ^{o}C in the milk butyrometer. Carefully on the butyrometer's walls are piled up 11 cm^3 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^3 water are added. Mixture is well stirred and heated up to 30-35 ^{o}C, then is again stirred and cooled down to 20±2^{o}C, and the following steps are as with the milk sample using sulphuric acid with d=1,789 till 1,790 кg/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^3 water are added, 10 cm^3 sulphuric acid with d=1,780 to 1,790 кg/m^3 at 20 ^{o}C and 1 cm^3 isoamilic alcohol. The butyrometer is closed with rubber stapple and is shaken till the proteins are fully dissolved.

9.4.2.7.Calculating the results

By using milk butyrometer

Milk, whey, buttermilk.

Using the butyrometer's graded scale the gramms 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.

9.4.2.8. 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.

9.4.3. Determination of water content and solids in the milk and milk derivatives.

9.4.3.1. General
The solids represent the fat content, proteins, carbohydrates and salts.
Sampling
Sampling is done according Appendices № 1 and № 2.

9.4.3.2. Basic principles.
Water content is determined by weight when drying at temperature (102±^{o}2) ^{o}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± ^{o}2) ^{o}C till constant mass and is expressed in grams for 100-grams of the product.

9.4.3.3. Necessary devices and reagents
- Assay balance with loading bounds 200 g and error 0,0002 g.
- Mercury thermometers from 0 to 100 ^{O}C and from 0 to 150 ^{O}C with value of scale division 1 ^{O}C;
- Pipettes from 5 to 10 cm^3, class II;
- Glass banks with grind stopples with volume 100-200 cm^3;
- Drying-oven with thermal regulator for keeping the temperature (102±2) ^{O}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.

9.4.3.4. 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^{o}C, it is well mixed and cooled down to 20^{o}C. Mixing and pouring are done at least three times in dry and clean vessel.

9.4.3.5. Making the measurement
The weight plate with 20-30 g washed out and tempered sand and glass rod is dried at 102±2 ^{o}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^3 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^oC, 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.

9.4.3.6. 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.

9.4.3.7. Measurement accuracy.

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

9.4.4. Determination of casein content in the milk.

9.4.4.1. General

The methods are based on the Volker's method.

For making the analyses are used pure reactives for analyses (p.r.a./ and distilled water or water with equal purity.

9.4.4.2. Sampling

According Appendices № 1 and № 2.

9.4.4.3. Basic principles.

Added to the milk formaline liberates acidic residuum 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.

9.4.4.4. Necessary devices and reagents

- Glass 250 cm3.
- Pipettes Фол - 25,5 cm3.
- Pipettes Mor from 1 cm3, with division 0,1 cm3.
- 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.

9.4.4.5. Making the determination:

For cow milk

Reference sample preparation.

20 cm^3 from the measured milk are poured in a glass vessel together with 1 cm^3 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.

9.4.4.6. Making the measurement

20 cm^3 from the milk are measured in a glass and a 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 taking into consideration.

4 cm^3 38-40 % formalin are added towards the neutralized sample and the rose color disappears as a result of the liberated carboxilic 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^3 38-40 % formalin in the milk are added 6 cm^3, and the standard/reference sample is prepared with 1 cm^3 4 % solution of cobalt sulphate.

9.4.4.7. Calculations

The quantity of the 0,1 n soda caustic in cm^3, 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^3 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^3	Casein%	0,1 n NaOH cm^3	Casein %	0,1 n NaOH cm^3	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^3	Casein%	0,1 n NaOH cm^3	Casein %	0,1 n NaOH cm^3	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

9.4.4.8. 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.

9.4.5. Determination of salts in the milk

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

9.4.5.2. Sampling
According Appendices № 1 and № 2.

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

9.4.5.4. 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.

9.4.5.5. 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 ^{O}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.

9.4.5.6. 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

9.4.5.7. Measurement accuracy

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

9.5. Appendix 5 Freezing point determination

9.5.1. Methods for determination.

The milk analyser determines the freezing point of each sample and the quantity of added water. The milk analyser 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 % – total solids %, total solids = lactose % + FAT % + proteins % + salts % + acids %.
Without understanding the meaning of the freezing point – determined or shown from the milk analyser added water result easily may lead to a mistake for the value of this parameter.

9.5.2. 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°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°C (2,326 standard deviations above the most recently determined North American average of –0,5404°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°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°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 analyser, the added water is calculated using the following formula:

$$AddedWater = \frac{FrPo\,\mathrm{int}_{Base} - FrPo\,\mathrm{int}_{Calc}}{FrPo\,\mathrm{int}_{Base}} * 100[\%]$$

Where:
FrPointBase is the basic freezing point
FrPointCalc is measured freezing point

Sample:
First variant
If you’ve entered for the milk analyser basic freezing point -0.520°C (according article 5.9 of the EU Milk Hygiene Directive 92/46/EEC), measured freezing point –0.540°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 analyser 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 analyser will measure freezing point –0.520°C, and will indicate again 0% added water.
Second variant
If you’ve entered for the device basic freezing point –0.540°C, measured freezing point –0.540°C, the milk analyser 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 analyser 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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