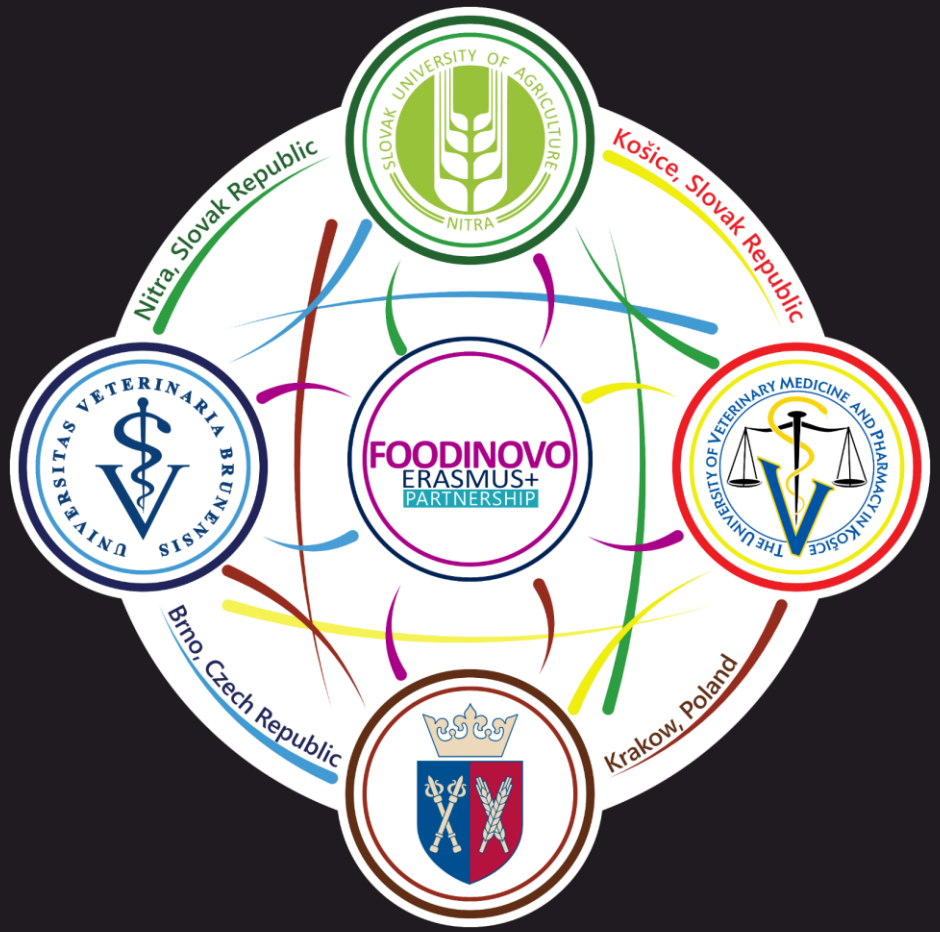
Sampling of Raw and Consumers Milk for Quality and Safety Analysis



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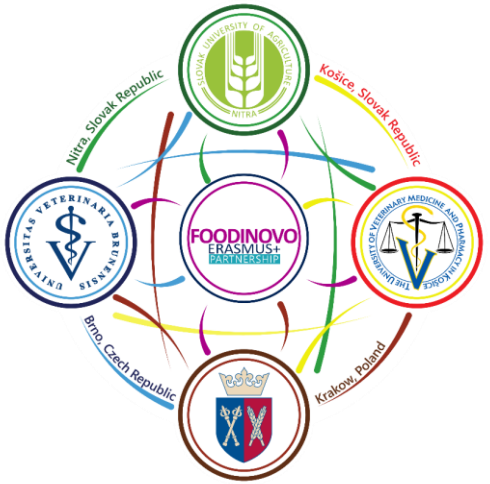
Milk Sampling

- the properties of raw milk have a fluctuating character
- the raw milk sample should be proportional and average, and therefore it is **very important to mix** the entire contents of storage tanks, containers etc.



Milk Sampling

- the sampling methods and the method of their transport and storage are applicable to raw milk from producers' deliveries and to raw milk and heat-treated milk in storage tanks and transport tanks
- it is necessary to follow the procedure according to the specific purpose of sampling, when sampling milk

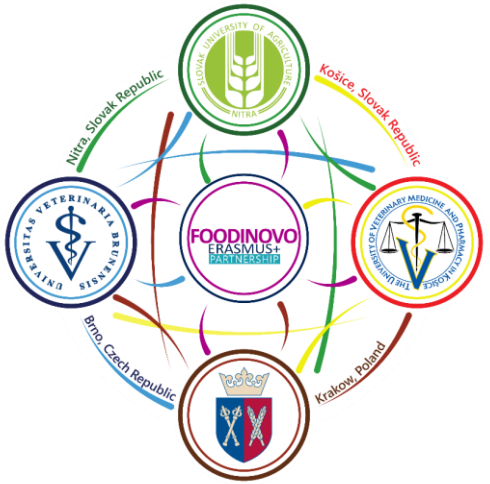
Milk Sampling

- raw milk samples are most often taken in order to:
  - dairy performance checks,
  - hygiene and technology controls in primary milk production,
  - monitoring of milk quality indicator values, mainly for the purpose of reimbursing milk,
  - milk infringement detection,
  - for research purposes and the like.

Milk Sampling

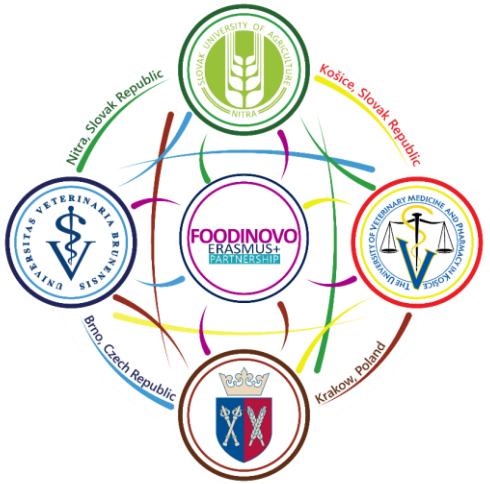
- the samples of heat-treated milk and final milk products in the form of drinking milk are taken in order to:
  - targeted and interoperational control, phase samples respecively,
  - quality control of final products,
  - product health control.

Types of Milk Samples



- according to their nature, milk samples are divided into private and official samples
- **private samples** are samples taken by an individual employee or a company laboratory officer for for their own analysis of milk quality
- **official milk** samples are taken in the public interest or in disputes

Types of Milk Samples



- according to the source from which the samples are taken, milk samples are divided into:
  - quarter samples taken from the mammary gland,
  - individual samples from one dairy cow,
  - pool and tank samples of raw milk,
  - phase samples of milk taken during the technological process of production of drinking milk in the dairy during the entry,
  - interoperational and output control,
  - consumer retail packaging and wholesale drinking milk packaging.

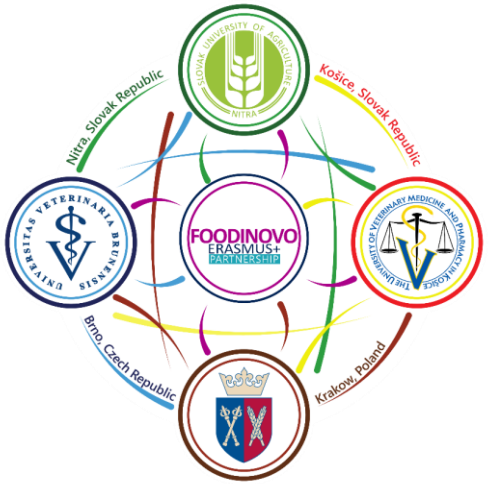
Types of Milk Samples

- phase samples of milk are taken at the place of primary milk production or in the dairy in order to control the quality of production in a targeted and interoperational control, while samples are taken from the milk receiving tank, pasteuriser (thermosector, cooler), storage tanks, milk filler, bottles and samples of intermediate products and finished dairy products

Types of Milk Samples

- if milk samples are taken from the drain or valve, it is important to release a sufficient quantity of milk first, otherwise samples are taken according to the requirements of the laboratory and the objectives of quality analysis

Pool Milk Sample

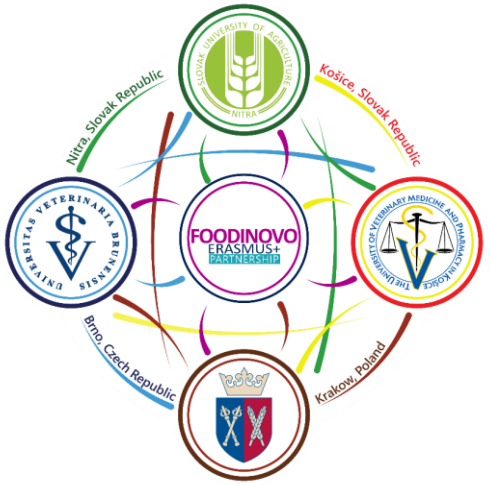


- the pool milk sample is a sample obtained from one or more storage tanks, canisters, or milk tanks that is ready for delivery to a milk processing plant
- the pool sample of milk have to meet the requirement that the pool sample taken have to be **proportional** and **average**

Pool Milk Sample

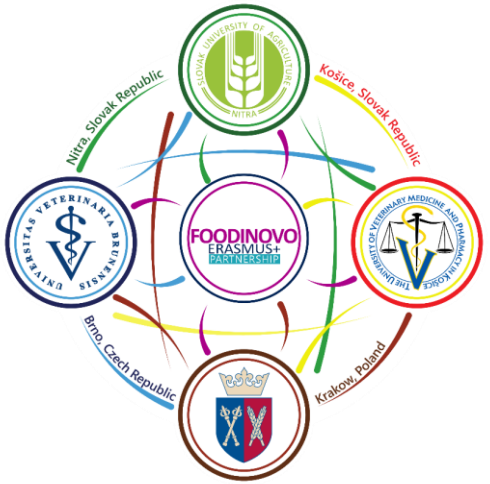
- at the place of primary milk production, the milk should be handed over to the tank for transport from several refrigeration tanks respectively storage containers, the samples have to be taken individualy and proportional from containers (total sampled volume into the mixing container have to be of at least 2 - 3 L)

Pool Milk Sample



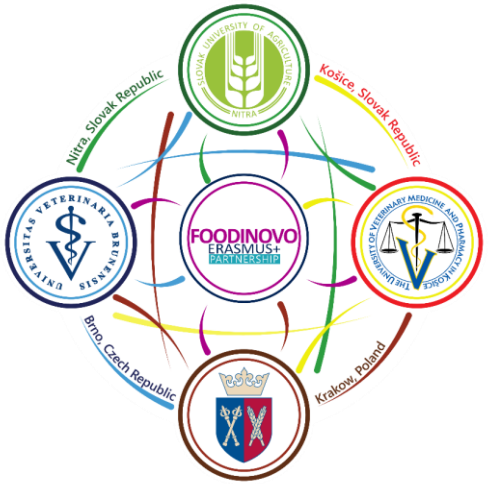
- the volume of milk is mixed in a mixing vessel and then a pool sample is taken from it into the prepared sample container, thus achieving its **averageness**
- **the proportionality of the pool sample** is obtained by taking an equal proportion of milk from each container or tank into the mixing container for the pool sample
- the proportion of milk means that the individual volume of milk tank in each container must be taken into account

Milk Tank Sample



- the milk tank sample is a sample taken from a milk tank which transports milk from the place of primary production to a milk processing plant
- the tank sample is taken by an authorized milk reception officer at the dairy plant
- the sample is taken manually or using the so-called autosampler, which is a device for automatic sampling of milk tank sample and is part of the transport tank

Milk Sampling Technique



- the sampling of raw and heat-treated milk from kettles, cisterns, etc., it must be performed by qualified person who have been demonstrably previously trained
- the person taking the milk sample must be properly acquainted with the relevant technique
- from a health safety point of view, the person taking the milk sample must not suffer from an infectious disease

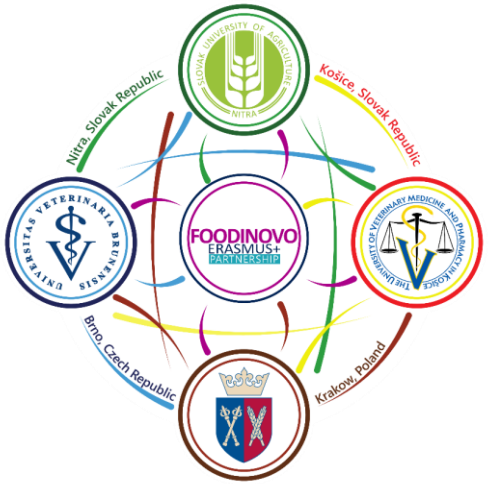
Picture reference:
https://sc01.alicdn.com/kf/Ha44441f9b0a540edbe7c907fba3e85f7K/225368811/Ha44441f9b0a540edbe7c907fba3e85f7K.jpg_.webp (cited 15.4.2022)




Milk Sampling Technique

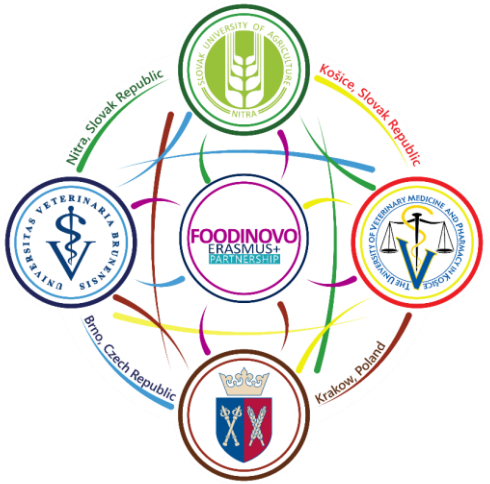
- in the case of official milk samples, interested participants must be allowed to take part in the collection
- the testing laboratories or the competent authorities shall inform the sampling staff of the sampling techniques so as to ensure that the sample is representative of the whole lot of milk
- also inform staff with the methods of marking the milk sample so as to ensure the unambiguous identity of the sample taken

Milk Sampling Equipment



- the device for milk sampling must be made of stainless steel or other suitable material of adequate strength and must have a construction suitable for the purpose (mixing, sampling, etc.)

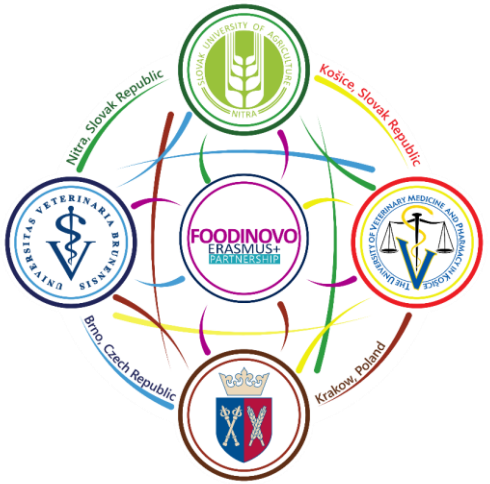
Milk Sampling Equipment



- plungers and agitators for mixing liquids in tanks must have a sufficiently large area to achieve adequate mixing of the product, without causing deterioration in the sensory quality of the milk, e.g. formation of swollen aroma and taste
- the ladles must have a rigid handle of sufficient length to allow a sample to be taken from any depth of the milk tank, while the volume of the ladle must not be less than 50 mL

Picture reference: https://5.imimg.com/data5/MH/IW/MY-6771446/ss-milk-sampler-500x500.png; https://5.imimg.com/data5/BL/TD/MY-6771446/milk-can-plunger-500x500.jpg (cited 15.4.2022)

Milk Sampling Equipment



- sample containers and closures must be made of glass, suitable metals or plastics
- the materials of which the sampling device and the sample boxes with closures are made must not cause any change in the sample which could affect the results of the milk quality and quantity analyzes
- all surfaces of the sampling equipment and sample boxes must be clean and dry, smooth and free of any cracks or fissures, the corners must be rounded

Milk Sampling Equipment

- in addition, in the case of sampling for **microbiological analysis**, the sampling equipment and sample boxes must be sterile, including milk sampling devices
- if the same sampling device is used for further sampling, it must be cleaned and sterilized after each sampling
- if the sample container is transparent, it must be stored in a dark place to avoid exposing the sample to direct sunlight

Milk Sampling

- sampling can be **manual**, **semiautomatic** and **automatic**
- the milk to be sampled must be thoroughly mixed, the sample being taken immediately after mixing while the milk is still being mixed
- if several milk samples are taken from the tanks for different tests, it is always necessary to take a milk sample for microbiological analysis as first sample

Milk Sampling

- the procedure and volume of the milk sample is given by the methodical procedures of the individual tests
- the volume of the used sample bottles have be such that the containers are almost completely filled with the sample to allow the contents to be properly mixed before testing, but to prevent the cream from coagulating during transport during transport

Milk Sampling

- automatic or semiautomatic devices for raw milk sampling from primary producers have to meet the requirements of the testing laboratory or the competent authority
- this facilities have to be suitable for the purpose of milk sampling and have to be inspected regularly
- they have to meet the requirement for measuring the minimum volume of milk
- they have to also be able to take a representative sample of the entire volume of milk after mixing

Milk Sampling

- samples of heattreated milk for direct consumption in retail packs have to be taken from tightly sealed and intact packs
- if possible, samples have to be taken from the packaging machine or from the refrigeration chamber of the dairy processing plant immediately after processing
- in the case of pasteurized milk, this is on the day of processing

Milk Sampling

- samples shall be taken from each type of heattreated milk (pasteurized, UHT, sterilized, etc.) in numbers corresponding to the tests to be carried out and in accordance with the instructions issued by the testing laboratory or other competent authority

Milk Sampling

- in products filled in small consumer packaging, samples are taken according to their size, one or more unopened packaging
- the homogenity of the sample is achieved by mixing, pouring or tempering before the milk quality analysis
- however, it is important not to froth the milk, as inaccurate analysis results could be obtained

Milk Sampling

- according to the specific method of milk analysis, the samples have to be tempered to the required temperature, prior to the determination procedure
- raw milk samples are usually tempered to a temperature of 20 – 25 ° C, unless a different temperature is prescribed in the methodological procedure
- if the sample shows a hardly dispersible milk fat deposit on the walls of the container or consumer packaging, the sample must be heated to a temperature of 35 – 40 ° C and then rapidly cooled to 20 ° C with constant stirring

Milk Sampling

- the milk samples have to be firmly sealed in the sample containers or sealed, correctly labeled with the type of product and its nature, weight or volume of the product from which the sample was taken, product condition, storage conditions, identification number, name, surname and signature
- the transport of milk samples is carried out together with a sampling protocol

Milk Sampling

- the milk samples are transported according to the requirements of the testing laboratory in accordance with the type of milk and the analytical methods to be used for the analysis of the milk sample
- special safety measures for some analyzes are given in the methodological procedures of individual analyses

Milk Sampling

- the raw milk samples taken for microbiological analysis must be transported and stored at a temperature between 0 and 4 ° C
- the time between sampling and analysis must be as short as possible, in no case more than 36 hours
- if the time between sampling and analysis does not exceed 24 hours, the competent authority may accept a storage temperature in the range 0 to 6 ° C

Milk Sampling

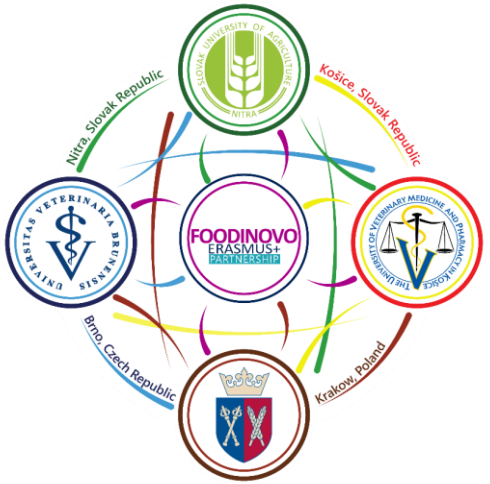
- pasteurized milk samples taken for microbiological analysis have to be transported and stored at a temperature between 0 and 4 ° C
- the time between sampling and analysis must be as short as possible, in no case longer than 24 hours
- milk samples, other than raw milk and pasteurized milk for microbiological analysis, must be stored in a laboratory in a refrigerator and the time between sampling and analysis must be as short as possible

Sample Analysis Time

Sample for analysis	Sample analysis time
Residues of inhibitory substances	up to 30 hours since sampling
Microbiological analysis	up to 48 hours since sampling
Evidence of the presence of cow 's and goat' s milk in sheep 's milk	up to 48 hours since sampling
Freezing point of milk	up to 48 hours since sampling
Milk components and number of somatic cells in milk	up to 72 hours since sampling

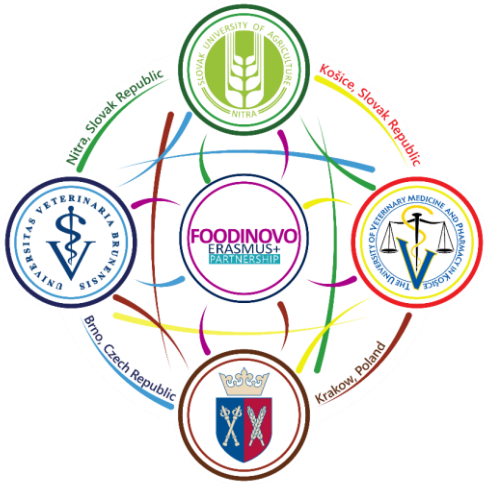
If the testing laboratory finds that the specified time since milk sampling has been exceeded, this shall be recorded in the analysis report. If time exceeds significantly, such samples are excluded from testing upon receipt.

Frequency of Purchased Milk Sampling



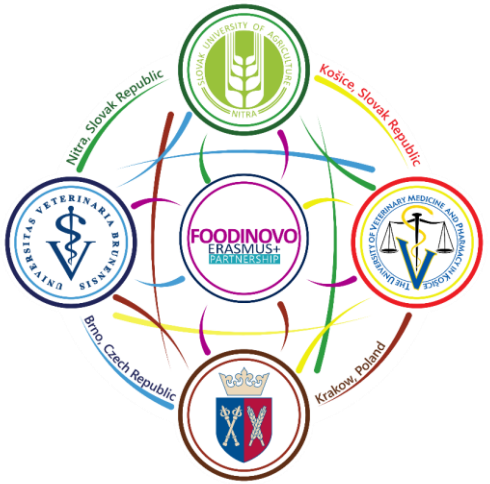
- Milk buyers from primary producers regularly check the quality of the milk purchased.
- Checking:
  - total number of microorganisms (TPC), at least twice a month, always with the simultaneous determination of dairy growth inhibitory substances, and is expressed as a moving geometric mean of the values over a period of two months with at least two samples per month,
  - the number of somatic cells (PSB), at least twice a month, always with a simultaneous determination of fat content, and is expressed as a moving geometric mean of the values over a period of three months when at least one sample is taken per month,
  - the presence of dairy growth inhibitors, at least twice a month,
  - the freezing temperature of the milk at least once a month, is expressed as the arithmetic mean of the determinations over the last 2 months.

Preservation of Milk Samples



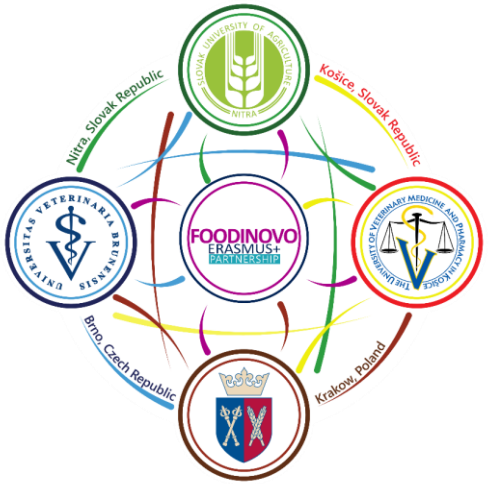
- it is optimal to analyze the quality of the milk sample as soon as possible after its collection, resp. according to the instructions and limits of the testing laboratory, which will evaluate the quality parameters of the tested milk sample
- for the purpose of selected methodological procedures for determining the quality parameters of milk, it is possible to preserve samples
- on the other hand, in some analyzes, such as sensory evaluation, sample preservation is excluded

Preservation of Milk Samples



- the selection of a suitable preservative must take into account the methodological procedures that apply to the samples
- the necessary requirement for preservatives is that they keep the milk sample unaltered
- the relevant testing laboratory may determine the preservative, resp. preparation

Preservation of Milk Samples



- it is necessary to correctly label the sample as well as information on the possible addition of a preservative
- several preservatives in different concentrations can be used for preservation
- a combination of chemical preservation and heat treatment can also be used in the preparation of reference samples

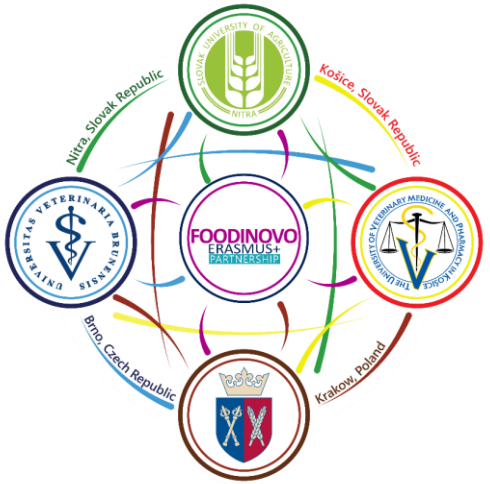
Preservation of Milk Samples

- The preservative used should not affect the milk and the result of the laboratory analysis of the milk sample. The optimal preservative should have the following properties:
  1. broad spectrum applicability;
  2. efficacy at low concentrations;
  3. high water solubility;
  4. stability under most storage conditions;
  5. coloring for security purposes;
  6. compatibility with high-fat or low-fat milk as well as stand-alone drinking milk;
  7. reasonably long shelf life;
  8. hypoallergenicity, non-toxicity and respect for the environment;
  9. low cost and easy availability;
  10. ease of dispersion (preferred tablet form).

Preservation of Milk Samples

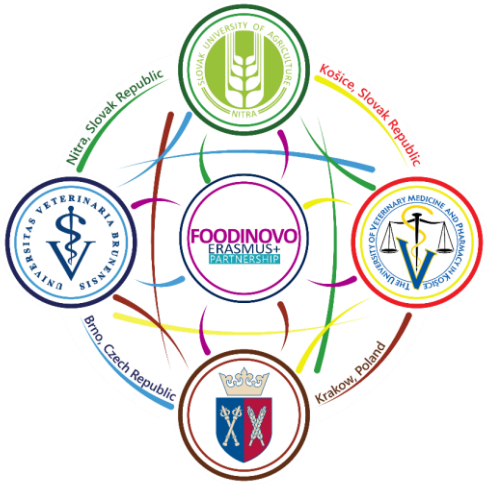
- No milk quality analysis preservatives on the market currently meet all these requirements at the same time
- Nonpreserved samples are used to measure the freezing point of milk and to prove the presence of cow's and goat's milk in sheep's milk
- If samples intended for measuring the freezing point of milk are preserved, they will be excluded from testing upon receipt.
- If the samples are preserved otherwise and it is known that the preservative does not have a significant effect on the test result, the testing laboratory shall record this fact in the test report

Preservation of Milk Samples



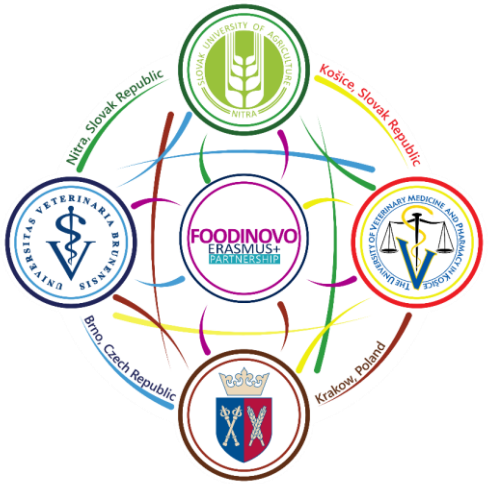
- The following chemicals can generally be used to preserve the raw milk samples for laboratory examination:
- Potassium dichromate (K_2Cr_2O7)
- Bronopol (2-bróm-2-nitropropán-1,3-diol)
- Sodium azide (NaN_3) a sodium azide preservative
- Boric acid preservative (H_3BO_3)

Potassium dichromate ($K_2Cr_2O_7$)



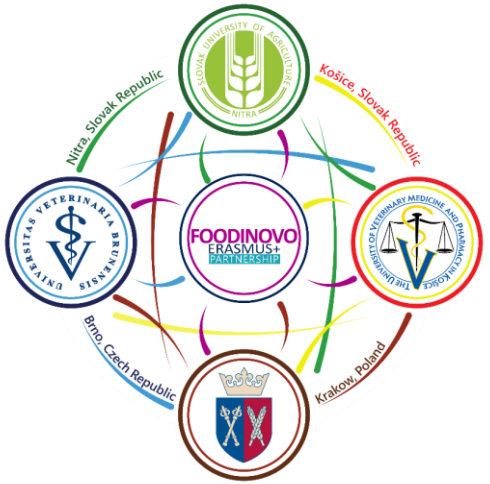
- it is used in various forms (powder, tablet or solution).
- 0.1 g or more is added to the milk sample taken. 1 drop per 10 mL milk sample if a saturated aqueous solution is used
- it is used for preserving milk samples in analyzes to determine the milk fat content and evidence of heat treatment of milk, respectively. alkaline phosphatase activity (max. amount 0.1%)

Bronopol (2-bróm-2-nitropropán-1,3-diol)



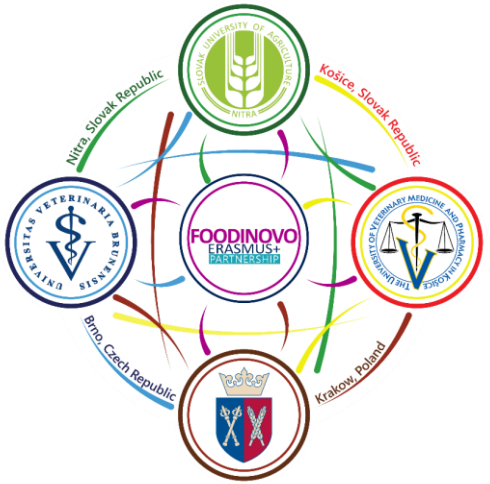
- used when using instrumental methods of analysis of a milk sample in a concentration of 0.1 - 3.0 g/L milk
- the most common form in which it is used as a preservative are tablets, for example also together with natamycin (antibiotic) in the form of so-called bronopol microtablets
- one tablet is used to preserve a 20 - 40 mL milk sample in determining the chemical composition of milk and somatic cells
- not used for the determination of residues of inhibitory substances in milk

Sodium azide (NaN3)



- Samples preserved using Acidol (sodium azide) at a concentration of 0.1 - 3 g/L milk are used to measure CPM, additional microbiological features, somatic milk cell count, milk composition, evidence of the presence of residues of inhibitory substances and their identification
- it is a substance classified as a poison and therefore the principles of safe handling must be followed when handling sample cards

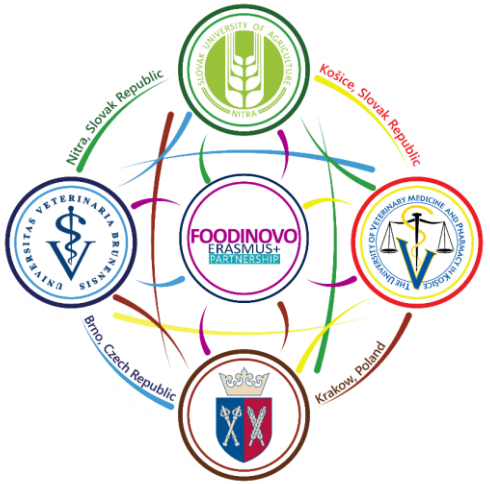
Sodium azide preservative (NaN_3)



- Azidiol contains the azide and the antibiotic chloramphenicolused to determine the total number of microorganisms in milk, has a bacteriostatic effect

Boric acid preservative (H_3BO_3)

- It is used in the determination of microorganisms in milk because it has a bacteriostatic effect



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