Fractionation Of Milk For Trace Analysis Of Contaminants And Residues

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Aim

Developing a new **passive method** for **raw milk** to prepare samples for **trace analyses of residues** of chlorate and perchlorate (C/PC) with **IC** and **LC-MS/MS**.

Materials and Methods

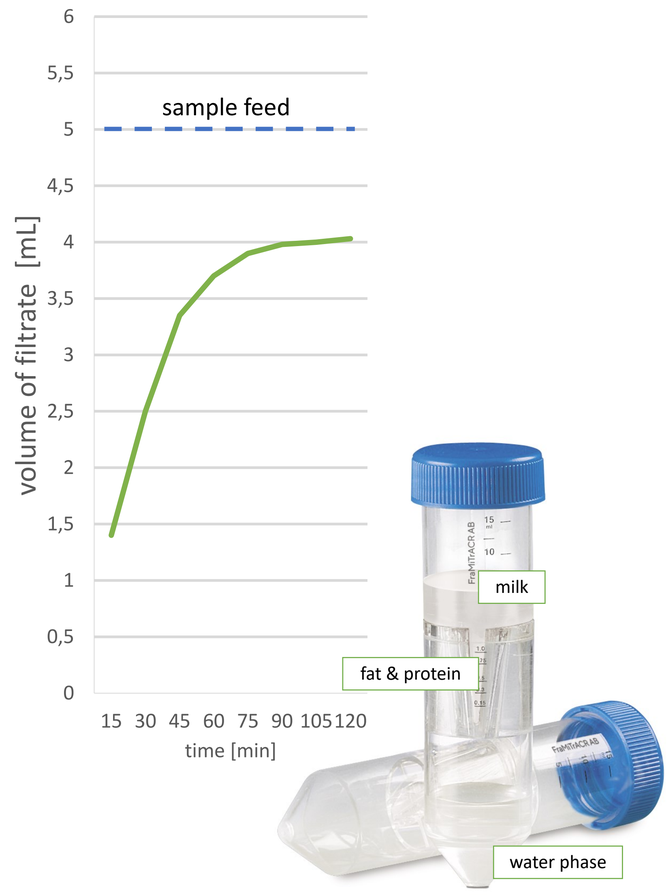
Unprocessed raw milk was separated into die phases **„water"** and **„fat & protein„** with a **fractionation unit (FraMiTrACR C/PC®)** and a standard **benchtop centrifuge**. Both, 10 mL raw milk using a fixed angle rotor at 30 minutes and 2,000 x g as well as 5 mL raw milk using a "swing out" rotor at 30 minutes and 4,000 x g resulted in **half of the volume of the feed added recovered as the "water phase"**.

For the analysis of the water phase, the **930 Compact IC Flex system (Metrohm)** with Dosin gradient technique was used for the determination of anions after sequential suppression and conductivity detection. For each determination, **0.25 mL of water phase** was injected into the analyser.

At the beginning of the development, the anion matrix in the water phase was collected from milk samples that were free of chlorate and perchlorate.

To **improve the detection limit**, the **characteristic anion matrix of the water phase has been subtracted** when evaluating the results.

The chlorate and perchlorate content was determined using **spiked samples in standard series** and also in **native samples**. In order to obtain **comparability** with previously used methods, the investigated raw milk samples were simultaneously analysed in a **contract laboratory** using the **modified Quppe method** and liquid chromatography with tandem mass spectrometry coupling **(LC-MS/MS)**.



Results

In the **water phase**, prepared with the fractionation unit (FraMiTrACR C/PC®) a **detection limit of 0.003 mg/kg chlorate and perchlorate** could be achieved by using the described **IC method**. Using a **LC-MS/MS method**, a **detection limit** of down to **0.001 mg/kg chlorate and perchlorate** could be achieved.

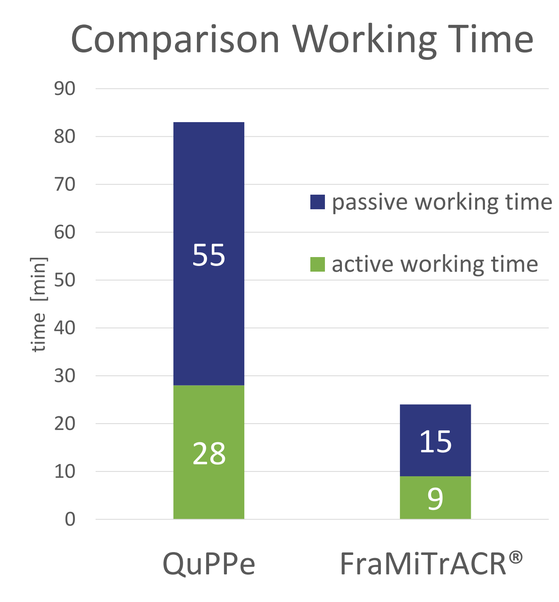
In direct **comparison** to well-established sample preparation methods, such as **Quechers** and **Quppe**, there is a **significant saving in working time (about 70%)** and thus in personell costs as well.

In addition, the complete **elimination of extraction solvents** or other additives leads to further cost savings in the laboratory process chain, simultaneously **excluding the risk of unwanted contamination**.

The **fractionation unit FraMiTrACR®** offers in various specifications **new opportunities** for trace analysis in milk and milk products.

Positive results for **antibiotics** are available as well.

Further studies for other analytes will be following soon.



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