

Quantitative analysis of Na, K and Ca in milk and yogurt using the MH-5000 Ultra Compact Elemental Analyzer

Introduction

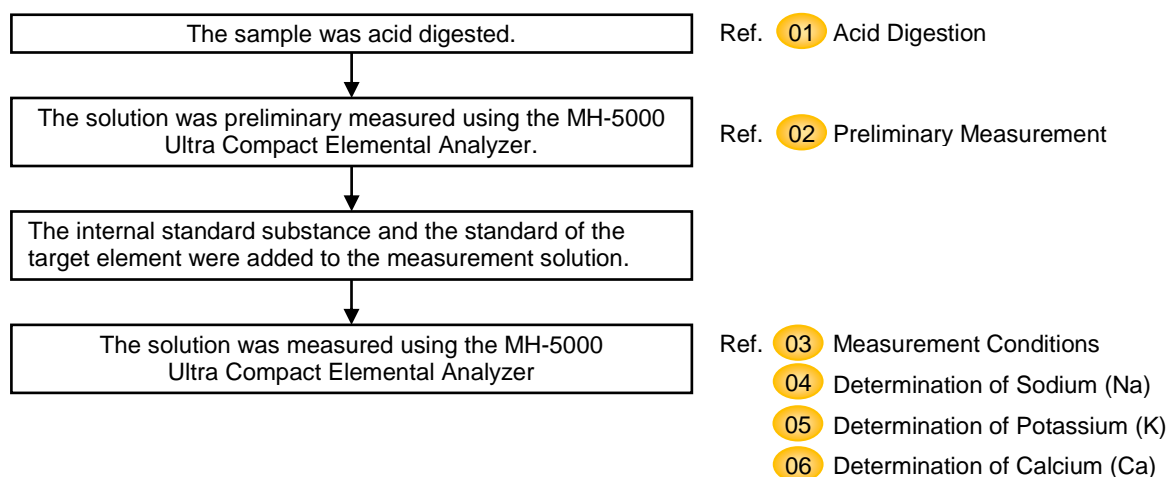
This application note describes the quantitative analysis of Sodium (Na), Potassium (K) and Calcium (Ca) in milk and yogurt using the MH-5000 Ultra Compact Elemental Analyzer.

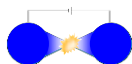
Procedure

Following five samples were used.

No.	Sample	Category	Ingredients
1	Milk	Fluid milk	100% raw milk
2	Calcium and iron fortified milk drinks	Milk drinks	Dairy products, Milk calcium, Whey minerals, Cellulose, Iron pyrophosphate, Emulsifier, Vitamin D, Folic acid
3	Plain yogurt	Fermented milk	Raw milk, Dairy products
4	Plain fat-free yogurt	Fermented milk	Dairy products, Milk proteins
5	Sweetened yogurt	Fermented milk	Raw milk, Dairy products, Sugar, Stevia

Following figure shows the procedure of analysis:

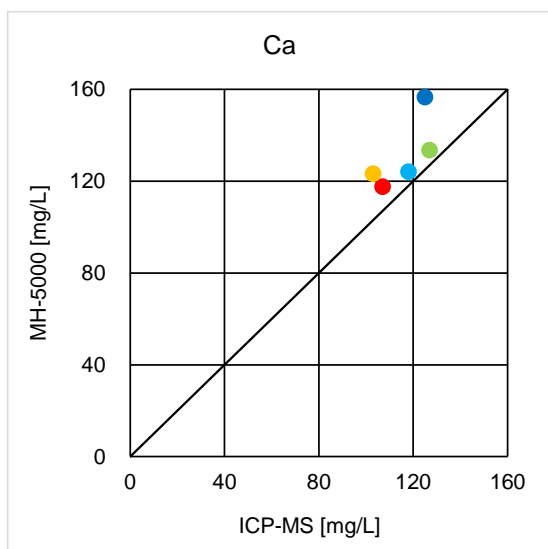
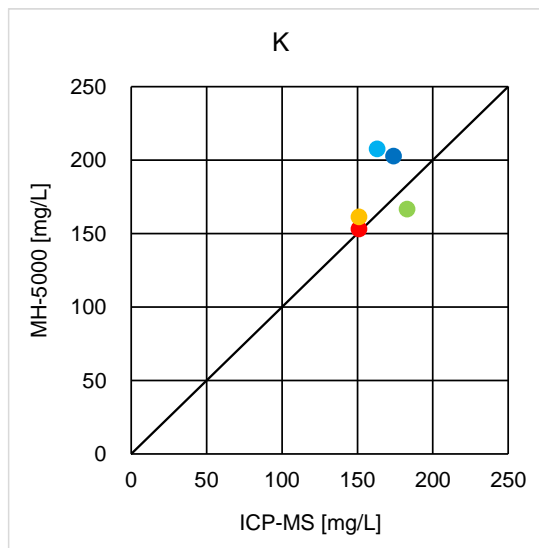
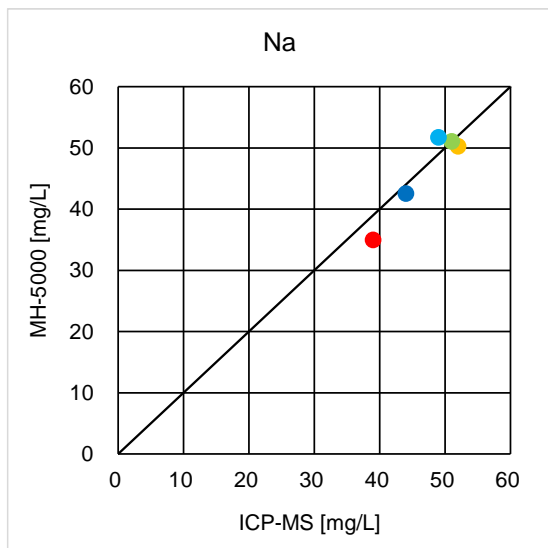




Results

The measurement results were compared with that of ICP-MS certified by the accredited measurement laboratory to guarantee accuracy of the MH-5000.

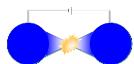
● Sample 1 ● Sample 2 ● Sample 3 ● Sample 4 ● Sample 5



Note: These are the concentrations of Na, K and Ca after acid digested and diluted and not the content per 100-gram sample.

Please refer to the following sections for further information.

- 04 Determination of Sodium (Na)
- 05 Determination of Potassium (K)
- 06 Determination of Calcium (Ca) ●



01

Acid Digestion

Following reagents, equipment, apparatus were used and following procedure was applied for acid digestion.

Reagents: Following two reagents were used.

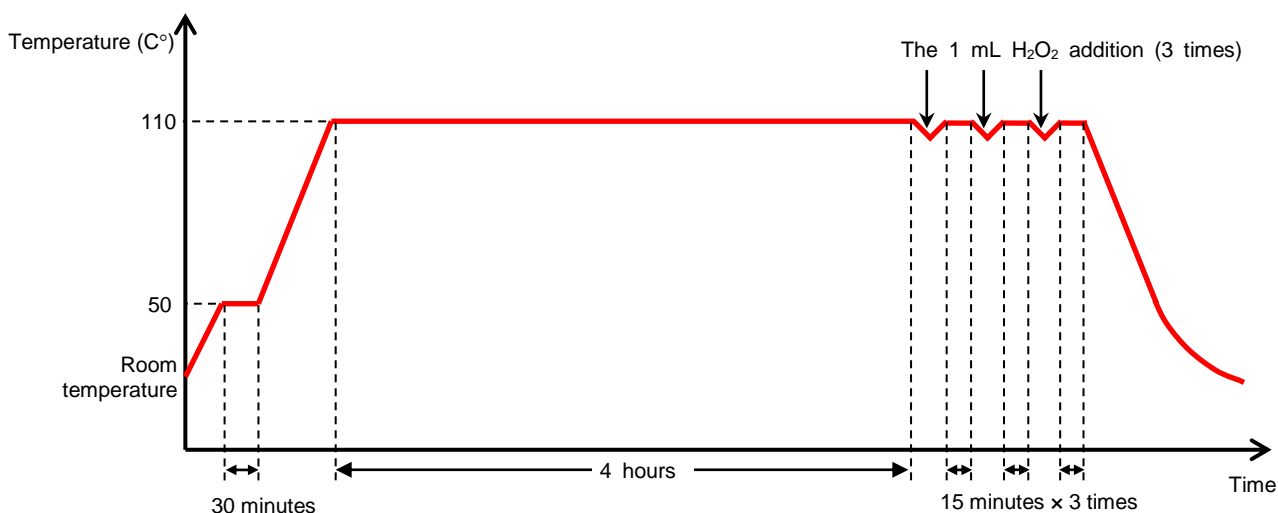
Product Name	Grade	Specification	Manufacturer
Nitric acid 1.38	For atomic absorption spectrometry	60 - 61% (T)	KANTO CHEMICAL CO., INC.
Hydrogen peroxide	For atomic absorption spectrometry	35.0 - 35.5% (T)	KANTO CHEMICAL CO., INC.

Equipment: Electrical balance, heating block (*Digi PREP CUBE*)

Apparatus: Disposable digestion tube (*Digi TUBEs* 65 mL), disposable watch glass, 10 mL measuring cylinder, 20 mL measuring flask, beaker and piston pipette

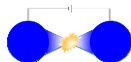
Procedure:

- 1) A 2-gram sample was measured into a digestion tube.
- 2) 10 mL of nitric acid was added to the tube.
- 3) The tube was inserted into the heating block and disposable watch glass was placed on it.
- 4) The tube was heated at a preset temperature of 50°C for 30 minutes.
- 5) After that, it was heated at a preset temperature of 110°C for 4 hours.
- 6) The tube was removed from the heating block and was added 1 mL of hydrogen peroxide.
- 7) After strong bubbling was fizzled out, the tube was reinserted to the heating block and heated at a preset temperature of 110°C for 15 minutes.
- 8) The step 6) to 7) was repeated three times.
- 9) The tube was allowed to cool to room temperature and the solution was diluted to 20 mL with water.



The solution after diluting contained 5 mol/L HNO₃.

Note: The solution after acid digested and diluted to 20mL is hereinafter called “acid digested solution”. ●



02

Preliminary Measurement

The acid digested solution was preliminary measured.

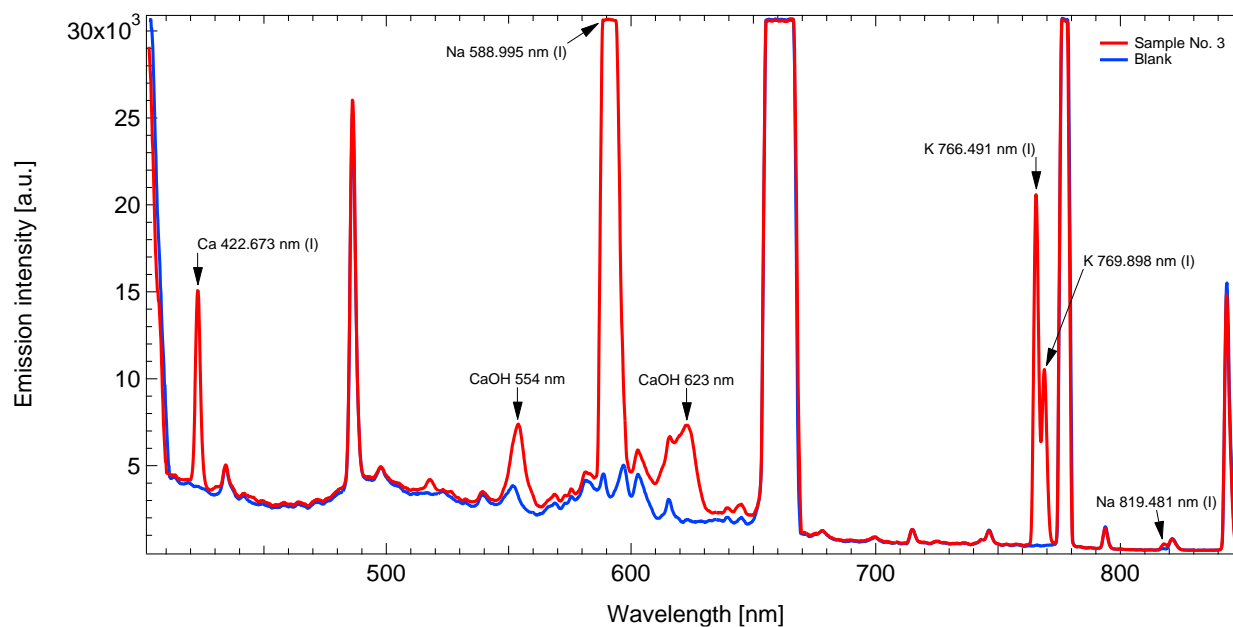
Solution: Acid digested solution of the sample
Acid digested water compared with the sample (Blank)

Cuvettes: LepiCuve-C, quartz cuvette

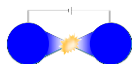
Instrument model: MH-5000 s2086

Instrument parameters: 700 V, (ON: 2 ms / OFF: 80 ms) $\times 25$ pulses

Spectra: Following figure shows the spectra of sample 3, plain yogurt, and the others are omitted because of no significant difference.



Several emission lines of Sodium (Na), Potassium (K) and Calcium (Ca) were detected. 



03

Measurement Conditions

Instrument models and parameters

The rate of acid digested solution was determined depending on the emission intensity of the target element at the test measurement. Instrument parameters were determined depending on the emission intensity of the target element in the solution and the acid concentration of the solution.

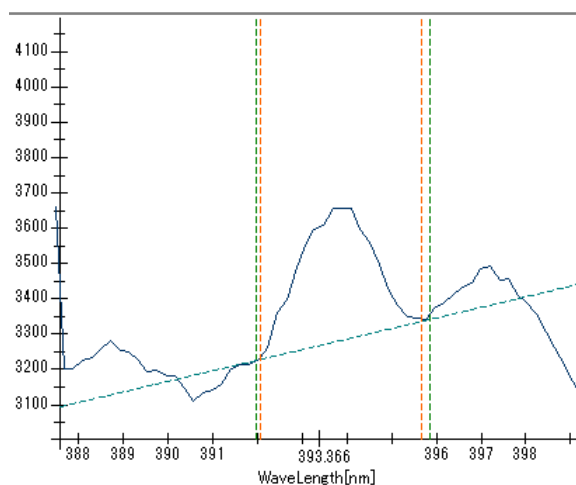
Target element	Internal standard element	Model	Rate of acid digested solution	Instrument parameters
Na 588.995 nm (I)	Rb 794.760 nm (I)	s2086	2 v/v%	900 V, (ON: 2 ms / OFF: 40 ms) × 20 pulses
K 766.491 nm (I)	Rb 794.760 nm (I)	s2086	2 v/v%	900 V, (ON: 2 ms / OFF: 40 ms) × 40 pulses
Ca 393.366 nm (II)	Sr 407.771 nm (II)	s2086	20 v/v%	700 V, (ON: 2 ms / OFF: 60 ms) × 25 pulses

Following sections describe two important functions on the LepiSuite LEP_Analyzer particularly for quantitative analysis.

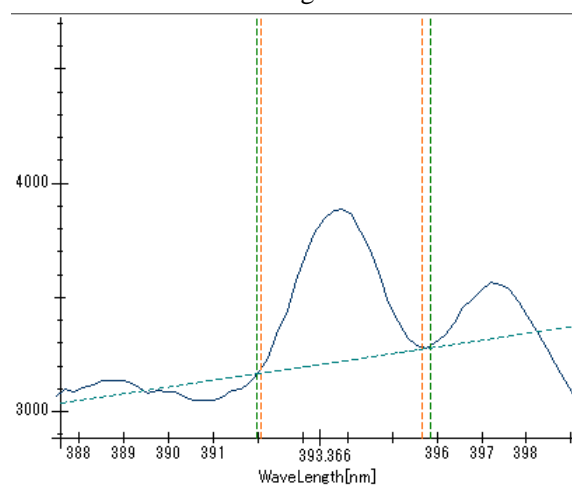
Baseline correction

Baseline correction is required for calculation of the emission intensity. The emission intensity is verified at several concentrations after measurement to allow the baseline to be adjusted as needed.

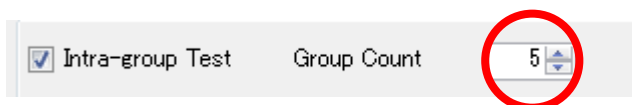
E.g. Without addition of Ca



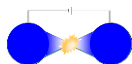
With the addition of 21 mg/L Ca

*Postprocessing*

Following picture shows the settings of postprocessing on the LepiSuite Analyzer. When the Group Count was set to 5, five raw data was treated as one group, thereby performing 15 iterative measurements as three groups.



Concentration of the target element was calculated from the calibration curves obtained by the average and the standard deviation of three groups (n = 3). ●



04

Determination of Sodium (Na)

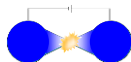
The rate of acid digested solution is 2 v/v%. The standard addition method was used to prevent effects of coexistence elements. Rubidium was used as an internal standard substance.

Measurement conditions

Sample:	2 v/v% acid digested solution, 0 - 1.2 mg/L Na as the standard, 250 mg/L Rb as the internal standard substance
Instrument model:	MH-5000 s2086
Cuvette:	LepiCuve-C, quartz cuvette
Instrument parameters:	900 V, (ON: 2 ms / OFF: 40 ms) × 20 pulses
Emission line of target element:	Na 588.995 nm (I)
Emission line of internal standard substance:	Rb 794.760 nm (I)

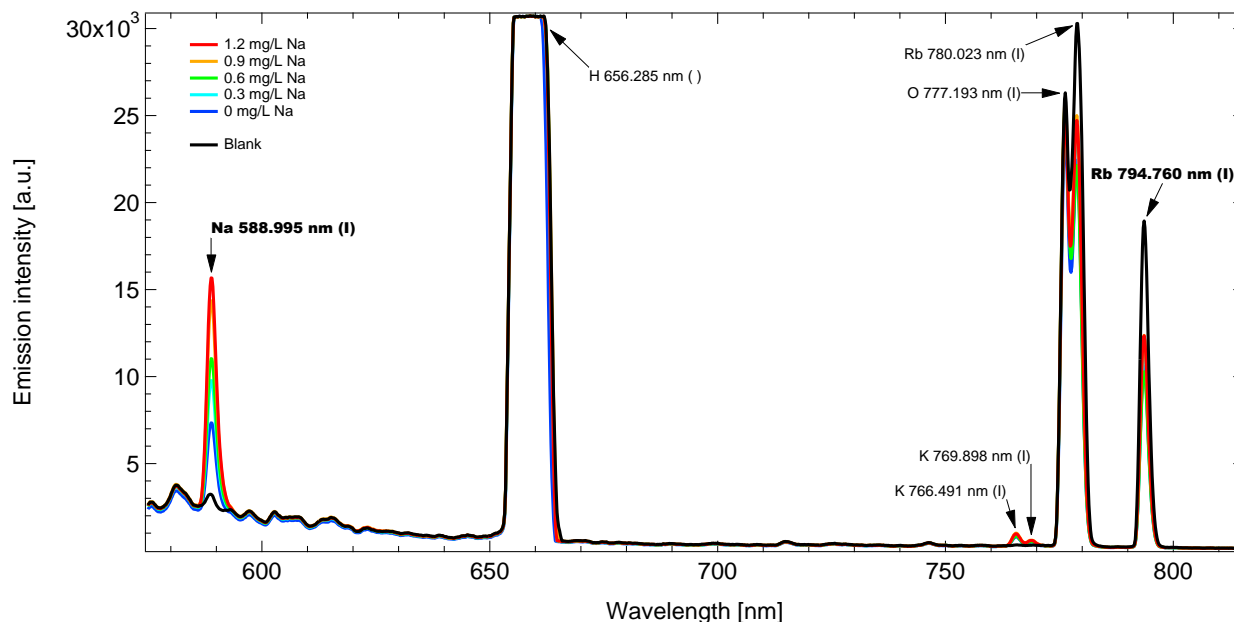
Results

No.	Sample	MH-5000 mg/L	ICP-MS mg/L	Percentage of the ICP-MS %
1	Fluid milk	35	39	90
2	Calcium and iron fortified milk drinks	50	52	97
3	Plain yogurt	51	51	100
4	Plain fat-free yogurt	52	49	106
5	Sweetened yogurt	43	44	97

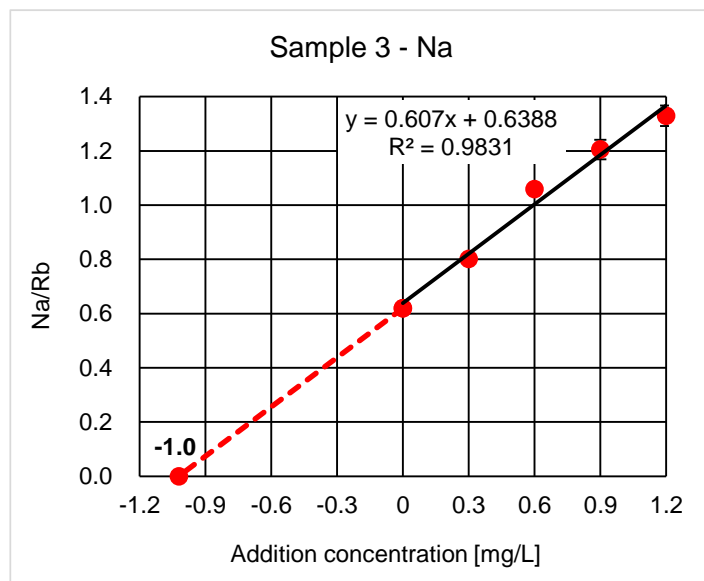


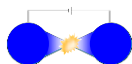
Following figures show the spectra and calibration curve of sample 3, plain yogurt, and the others are omitted because of no significant difference.

Spectra



Calibration curve





05

Determination of Potassium (K)

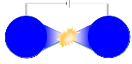
The rate of acid digested solution is 2 v/v%. The standard addition method was used to prevent effects of coexistence elements. Rubidium was used as an internal standard substance.

Measurement conditions

Sample:	2 v/v% acid digested solution, 0 - 4 mg/L K as the standard, 15 mg/L Rb as the internal standard substance
Instrument model:	MH-5000 s2086
Cuvette:	LepiCuve-C, quartz cuvette
Instrument parameters:	900 V, (ON: 2 ms / OFF: 40 ms) × 40 pulses
Emission line of target element:	K 766.491 nm (I)
Emission line of internal standard substance:	Rb 794.760 nm (I)

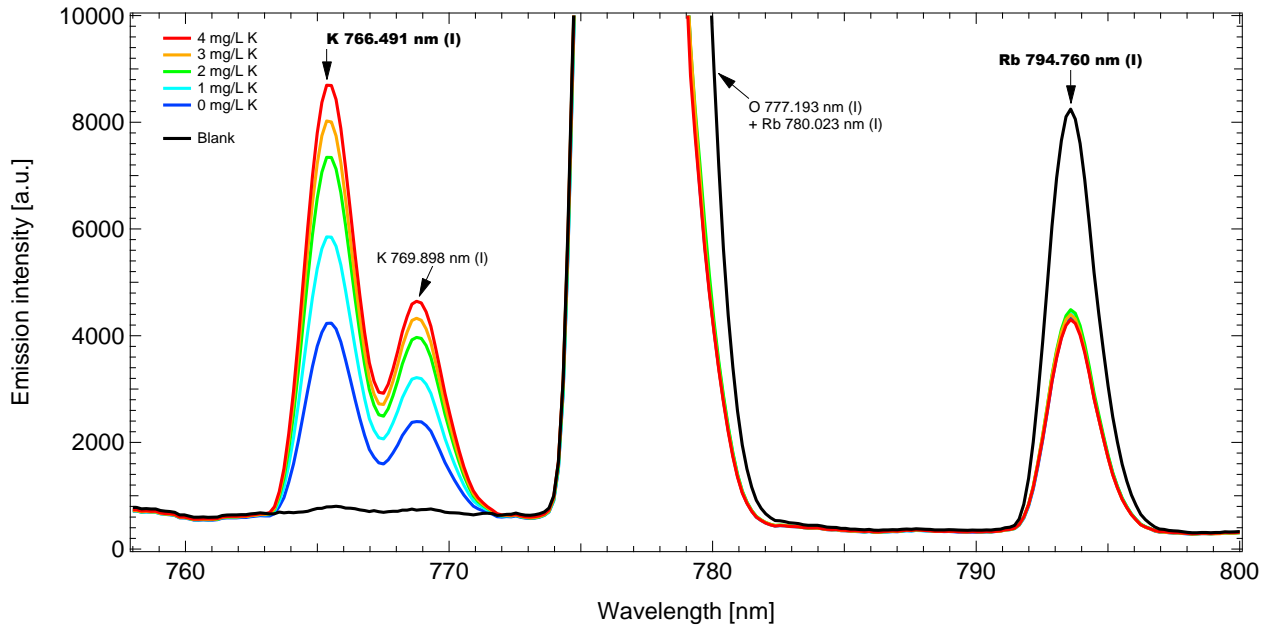
Results

No.	Sample	MH-5000 mg/L	ICP-MS mg/L	Percentage of the ICP-MS %
1	Fluid milk	153	151	101
2	Calcium and iron fortified milk drinks	161	151	107
3	Plain yogurt	167	183	91
4	Plain fat-free yogurt	208	163	127
5	Sweetened yogurt	203	174	117

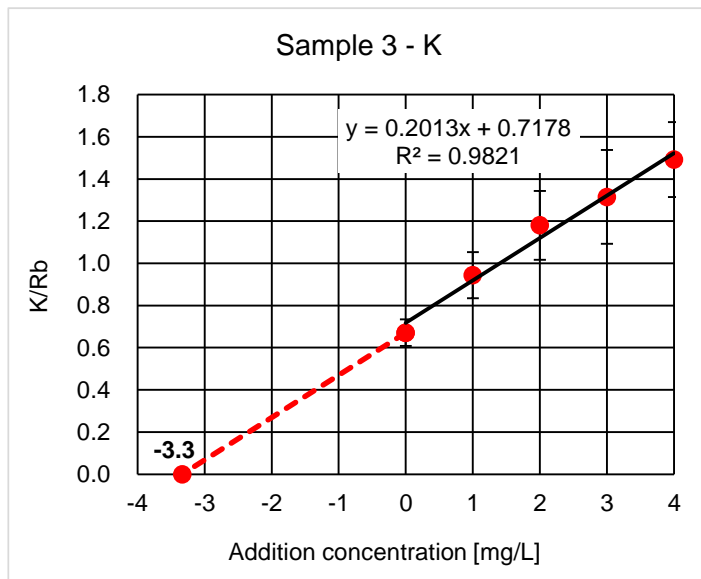


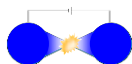
Following figures show the spectra and calibration curve of sample 3, plain yogurt, and the others are omitted because of no significant difference.

Spectra



Calibration curve





06

Determination of Calcium (Ca)

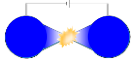
The rate of acid digested solution is 20 v/v%. The standard addition method was used to prevent effect of coexistence elements. Strontium was used as an internal standard substance.

Measurement conditions

Sample:	20 v/v% acid digested solution, 0 - 28 mg/L Ca as the standard, 40 mg/L Sr as the internal standard substance
Instrument model:	MH-5000 s2086
Cuvette:	LepiCuve-C, quartz cuvette
Instrument parameters:	700 V, (ON: 2 ms / OFF: 60 ms) × 25 pulses
Emission line of target element:	Ca 393.366 nm (II)
Emission line of internal standard substance:	Sr 407.771 nm (II)

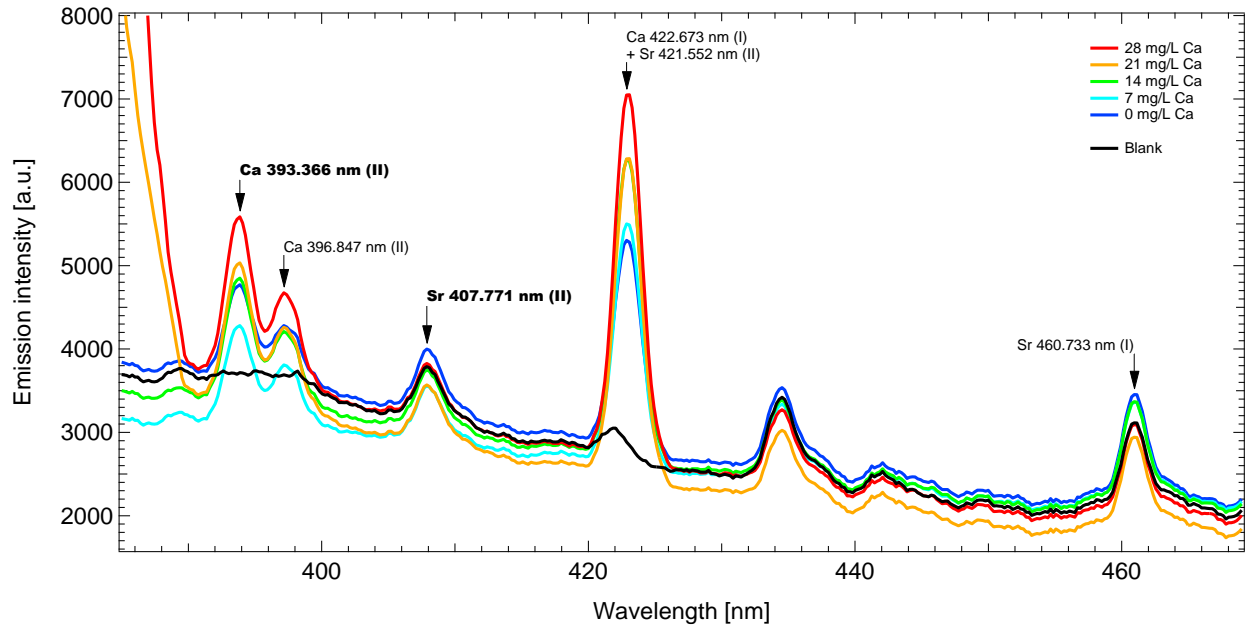
Results

No.	Sample	MH-5000 mg/L	ICP-MS mg/L	Percentage of the ICP-MS %
1	Fluid milk	118	107	110
2	Calcium and iron fortified milk drinks	123	103	120
3	Plain yogurt	134	127	105
4	Plain fat-free yogurt	124	118	105
5	Sweetened yogurt	157	125	125

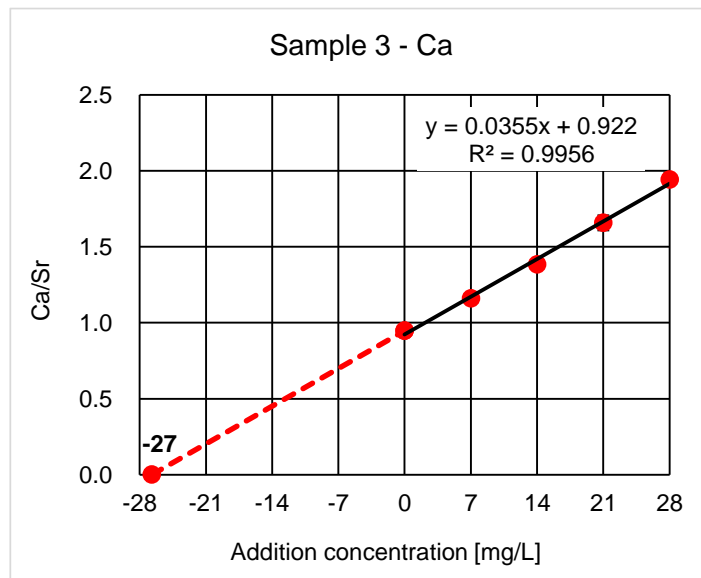


Following figures show the spectra and calibration curve of sample 3, plain yogurt, and the others are omitted because of no significant difference.

Spectra



Calibration curve



MICRO EMISSION

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