

## Analysis of Na, K, Ca and Mg in mineral water using Microsampling method

The AA-7000 in combination with the ASC-7000 sample preparation station allows the automated flame micro sampling method (Figure 1). In this method, the flame atomic absorption analysis is conducted with small sample volumes (2 – 90  $\mu\text{L}$ ), while in the conventional flame method (hereafter “flame continuous method”), the sample is continuously aspirated with a flow rate of approximately 8 ml/min and larger sample volumes are needed for aspiration.

The flame micro sampling method has several advantages over the flame continuous method: analysis is possible with a small amount of sample, and when the autosampler is used, automatic dilution of the sample and automatic addition of buffer solutions are possible in order to compensate interferences. Moreover, since only a small amount of sample is introduced, the flame micro sampling method is effective for analysis of high matrix samples which may cause clogging of the burner in the flame continuous method. So the method is the right choice for determination of alkaline and alkaline earth elements in mineral water.

Sodium, Potassium, Calcium, and Magnesium belong to the essential mineral substances in the human organism. These elements take influence in the generation of enzymes and hormones, control the osmotic pressure in tissues and body fluids and are important for the exchange procedures in the cell membranes.<sup>[1]</sup> The recommended daily amounts (Na: 550, K: 2000, Ca: 800-1000, Mg: 350 mg/L<sup>[1]</sup>) are possible to be partly

covered by consumption of mineral waters. But the composition of mineral waters according to the essential elements has a wide variety, and depending on the origin the composition might be quite different.

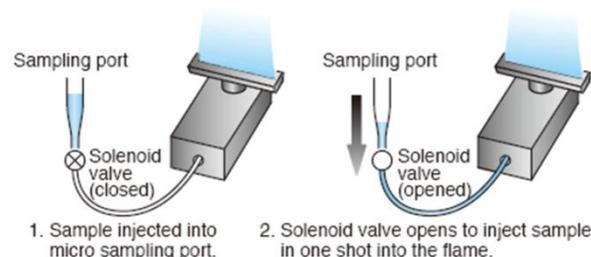


Figure 1: Microsampling method

### ■ Method

The control of Na, K, Ca and Mg in a variety of mineral waters has been performed according to the actual DIN/EN regulations<sup>[2]</sup>, with the Shimadzu atomic absorption spectrophotometer AA-7000 in a fully automatic multi element sequence.

The blank, standards and the water samples are all placed in the autosampler and then will be mixed automatically with the corresponding reagents which have to be added according to the DIN/ EN method. In case of Sodium and Potassium 40  $\mu\text{L}$  of CsCl-solution (12,65 g CsCl + 50 ml HCl (d=1,16) filled up to 500 ml volume with  $\text{H}_2\text{O}$ ) will be added for a 400  $\mu\text{L}$  mixing volume of standard and sample solution which is homogenized before injection to the flame. In case of Calcium and Magnesium a  $\text{La}_2\text{O}_3$ -solution (5,875 g  $\text{La}_2\text{O}_3$  + 50 ml HCl (d=1,12) filled up to 250 ml volume with  $\text{H}_2\text{O}$ ) has been used.

The instrumental parameters and measuring conditions are listed in Table 1 below. These conditions are automatically set for each element including optimized burner high and gas flow rates.

### ■ Results and Conclusion

Under these conditions, a series of more than 20 drinking water and mineral water samples has been analyzed. A reference material (NIST SRM 1640) was measured along with this series as a laboratory control sample. The excellent recoveries are listed in Table 2.

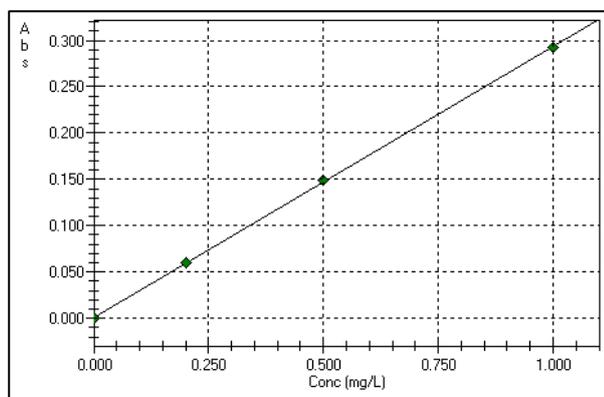


Figure 1: Potassium calibration curve

Elem.	Certified [mg/L]	Result [mg/L]	Recovery [%]
Ca	0.7045 ± 0.089	6.980	99.08
K	0.994 ± 0.027	1.015	102.11
Mg	5.819 ± 0.056	5.850	100.53
Na	29.35 ± 0.31	29.19	99.42

Table 2: Comparison of the certified and measured concentrations of NIST SRM 1640 control sample

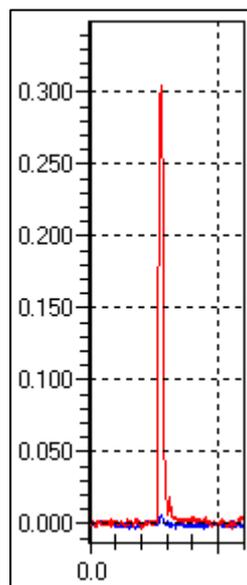


Figure 2: Potassium peak profile (50 µL injection)

Instrument	AA 7000 with autosampler ASC 7000 incl. Microsampling Kit			
Measurement element	Na	K	Ca	Mg
Wavelength	589,0 nm	766,5 nm	422,7 nm	285,2 nm
Slit width	0,2 nm	0,7 nm	0,7 nm	0,7 nm
Lamp current	8/600 mA	8/600 mA	10 mA	8 mA
Backgroundcorrection	SR	SR	D <sub>2</sub>	D <sub>2</sub>
Flame type	Air-C <sub>2</sub> H <sub>2</sub>	Air-C <sub>2</sub> H <sub>2</sub>	Air-C <sub>2</sub> H <sub>2</sub>	Air-C <sub>2</sub> H <sub>2</sub>
Gas flow rate	1,8 l/min	2,0 l/min	2,0 l/min	1,8 l/min
Sampling time	20 sec	20 sec	20 sec	20 sec
Interference buffer	CsCl-solution	CsCl-solution	La <sub>2</sub> O <sub>3</sub> -solution	La <sub>2</sub> O <sub>3</sub> -solution
Calibration range [mg/L]	0,1 – 1,0	0,1 – 1,0	0,25 – 5,0	0,025 – 0,5

Table 1: Instrument and Analytical Conditions

- [1] Mineralstoffe und Spurenelemente, Verlag Bertelsmann Stiftung, 1991
- [2] Deutsche Einheitsverfahren (DEV) zur Wasser-, Abwasser- und Schlamm-untersuchung, Verlag Chemie, Weinheim; DIN 38406-E14 (Natrium), DIN 38406-E13 (Kalium), DIN EN ISO 7980-E3-1 (Calcium, Magnesium)

For Research Use Only. Not for use in diagnostic procedures. Shimadzu Corporation ("Shimadzu") reserves all rights including copyright in this publication. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to, or arising out of the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

