Measurement by Flame Micro Sampling Method

■ Introduction

The AA-6300 combined with an autosampler ennables the automated flame micro sampling method. In the flame micro sampling method, flame atomic absorption analysis is conducted with small sample quantities (2 - 100µL), while in the conventilnal flame method (hereafter "flame continuous suction method"), the sample is continuously aspirated so rather large amount of sample is necessary.

Table 1 shows the comparison of the flame micro sampling method with the flame continuous suction method. The flame micro sampling method has sevral advantages over the flame continuous suction method: analysis is possible with a small amount of sample,

and when an autosampler is used, automatic dilution of the sample and automatic addition of interference inhibitor are possible. 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 by the flame continuous suction method.

We introduce here an example of analysis of Na and Mg in the standard river water sample, using the flame micro sampling method. (The standard riverwater; JAC0031, unspiked, cetified values for Na;4.2±0.1mg/L and Mg;2.83±0.06mg/L, provided by the Japan Society of Analytical Chemistry)

Table 1 Comaprison of flame continuous suction method and flame micro sampling method

	Measure-ment time	Volume of Sample Consumption (per analysis)	Sensitivity (Absorbance)	Coefficient of Variation (%) (Simple repetition)	Auto dilution
Continuous Suction method	10 – 20sec	1 – 2mL	–	Approx. 1%	Not possible
Micro sampling method	Approx. 100sec	2 – 100µL	Approx. 80% of suction method	2 – 3%	Possible (50 times max.)

The maximum dilution factor refers to maximum value when the mixing port is not used (2µL/100µL).

■ Instrument and Analytical Conditions

Table 2 indicates the instrument and main analytical conditions. For both elements, the conditions were set so that if the measured value exceeded the 1mg/L

upper limit concentration of the calibration curve, the sample amount injected would be automatically decreased and the sample reanalyzed.

Table 2 Instrument and Analytical Conditions

Instrument	Atomic Absorption Spectrophotometer AA-6300	
Autosampler	ASC-6100F, ASK-6100, ASK-6300, Micro sampling kit	
Measurement element	Na	Mg
Measurement wavelength	589.0nm	285.2nm
Ignition mode	NON-BGC	BGC-D$_2$
Flame type	Air / acetylene	
Burner angle	0˚	
Sampling time	30 sec.	
Interference inhibitor	None	Lanthanum (La)
Standard solution concentration (mg/L)	0.2, 0.5, 1.0	
Max. conc. (mg/L) for auto dilution/reanalysis	1.0	

■ Measurement Results

The measurement results for Na and Mg are shown in Table 3 and 4, respectively. Measurements were repeated twice for the standard sample and three times for the actual sample. The calibration curves were generated for both Na and Mg by injecting different volumes of 1mg/L solutions using an au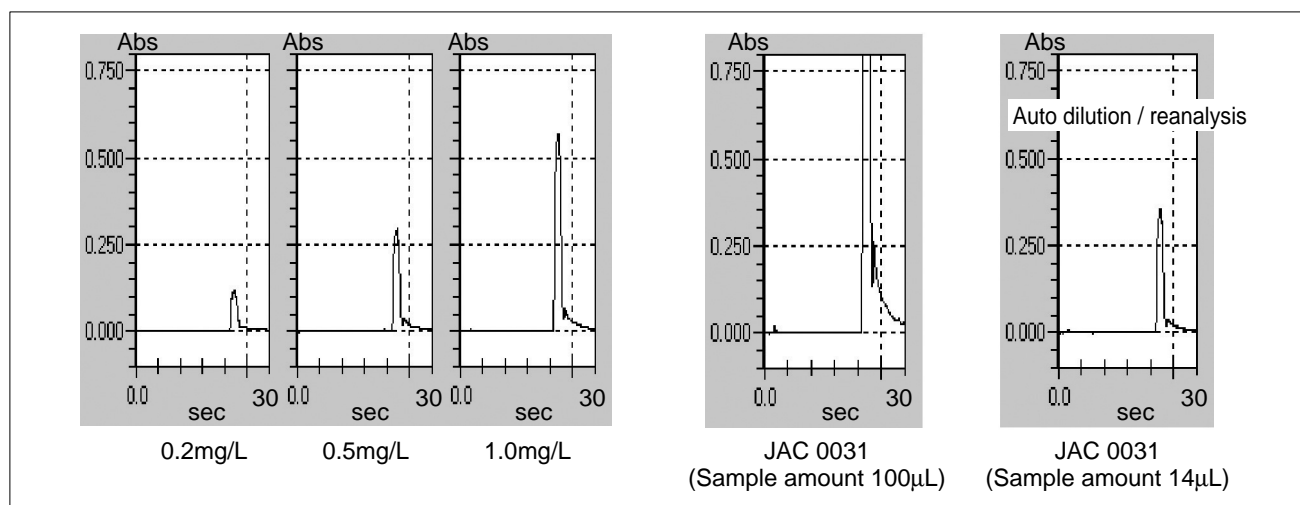tosampler. For the Mg, lanthanum was also added as the interference inhibitor using the autosampler. In the analysis of the actual samples, since the first measurement value exceeded the set upper limit concentration, dilution and reanalysis were automatically conducted with smaller volumes of samples. The obtained values agreed well with certified values for both elements.

Table 3 Measurement result for sodium (Na)

Operation	Sample ID	Exclude	Set concentration (mg/L)	Concentration (mg/L)	Abs.	Position	Sample amount (μL)	Diluent (μL)	Total volume (μL)	Auto dilution	Actual concentration (mg/L)	CV (%)
STD-AV	0.2mg/L		0.2		0.1184	R2	20	80	100			1.31
STD-AV	0.5mg/L		0.5		0.2954	R2	50	50	100			0.55
STD-AV	1.0mg/L		1.0		0.5652	R2	100	0	100			1.58
UNK1	*JAC0031*	X		*3.3821*	*1.8933*	*2*	*100*	*0*	*100*		*3.38*	
UNK2-1	JAC0031			0.5972	0.3433	2	14	86	100	7.143	4.27	
UNK2-2	JAC0031			0.6121	0.3516	2	14	86	100	7.143	4.37	
UNK2-3	JAC0031			0.6074	0.3490	2	14	86	100	7.143	4.34	
UNK2-AV	JAC0031			0.6056	0.3480	2	14	86	100	7.143	4.33	1.22

Table 4 Measurement result for magnesium (Mg)

Operation	Sample ID	Exclude	Set concentration (mg/L)	Concentration (mg/L)	Abs.	Position	Sample amount (μL)	Diluent (μL)	La5% (μL)	Total volume (μL)	Auto dilution	Actual concentration (mg/L)	CV (%)
STD-AV	0.2mg/L		0.2		0.1946	R2	16	64	20	100			3.05
STD-AV	0.5mg/L		0.5		0.4750	R2	40	40	20	100			0.61
STD-AV	1.0mg/L		1.0		0.9388	R2	80	0	20	100			1.25
UNK1	*JAC0031*	X		*2.0887*	*1.9516*	*2*	*80*	*0*	*20*	*100*		*2.09*	
UNK2-1	JAC0031			0.7446	0.7016	2	20	60	20	100	4	2.98	
UNK2-2	JAC0031			0.7272	0.6854	2	20	60	20	100	4	2.91	
UNK2-3	JAC0031			0.7377	0.6952	2	20	60	20	100	4	2.95	
UNK2-AV	JAC0031			0.7365	0.6941	2	20	60	20	100	4	2.95	1.18

Auto dilution / reanalysis

0.2mg/L 0.5mg/L 1.0mg/L JAC 0031 (Sample amount 100μL) JAC 0031 (Sample amount 14μL)

Fig.1 Examples of peak profiles for sodium (Na)

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