

Introduction of HFS-4 hydride formation system

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1. Introduction

The hydride formation system is an instrument that connects to a flame atomic absorption spectrometer to enable high-sensitivity measurements of arsenic (As), selenium (Se), and antimony (Sb) in aqueous solutions. These instruments are widely used to analyze As, Se, and Sb present in environmental water, waste water, foodstuffs, and other substances.

Our newly developed HFS-4 hydride formation system (Figure 1) retains the key performance feature of its predecessor instrument (the HFS-3), namely, that it uses flame Zeeman atomic absorption spectrometry with no baseline drift to enable highly stable measurements. In addition, this new hydride formation system significantly reduces the volume required for samples and reagents while increasing the sensitivity and analytical throughput. Moreover, the instrument is equipped with a new technology, a 4-stage peristaltic pump. As discussed in this note, this allows for the automated addition of preliminary reducing agents such as potassium iodide (KI) when analyzing As.



Fig. 1 The HFS-4 hydride formation system.

2. Principles of measurement

In the hydride formation-atomic absorption method, As, Se, Sb, and similar elements in a sample aqueous solution react with sodium borohydride (NaBH_4) and hydrochloric acid (HCl) to yield gaseous hydrides. These are atomized using argon gas and introduced into a heated quartz cell. The elements that can be treated using the hydride-formation method are limited, but it allows highly sensitive measurements.

Caution is required when using the hydride formation method because the efficiency of hydride production depends on the chemical state of the element. Inorganic As exists in trivalent and pentavalent states. Because the production of AsH_3 is lower for the pentavalent state than the trivalent state, KI must be introduced into the sample in advance to reduce the pentavalent state to the trivalent state.

In foodstuffs, large quantities of As are present in the form of organic As. Because the majority of organic As does not form hydrides, organic As must be decomposed into inorganic As in advance.

3. Key product features

3-1 The HFS-4 flow pathways

The HFS-4 is equipped with (1) a three-liquid-blending pathway in which the sample, HCl , and NaBH_4 are blended; and (2) a four-liquid-blending pathway in which KI for As analysis is blended by pumping.

Figure 2 shows the HFS-4 flow pathways. In the four-liquid-blending pathway, the preliminary reducing agent is automatically added via pumping, ensuring that pentavalent As is reduced to trivalent As; this reduces the effort required to prepare samples.

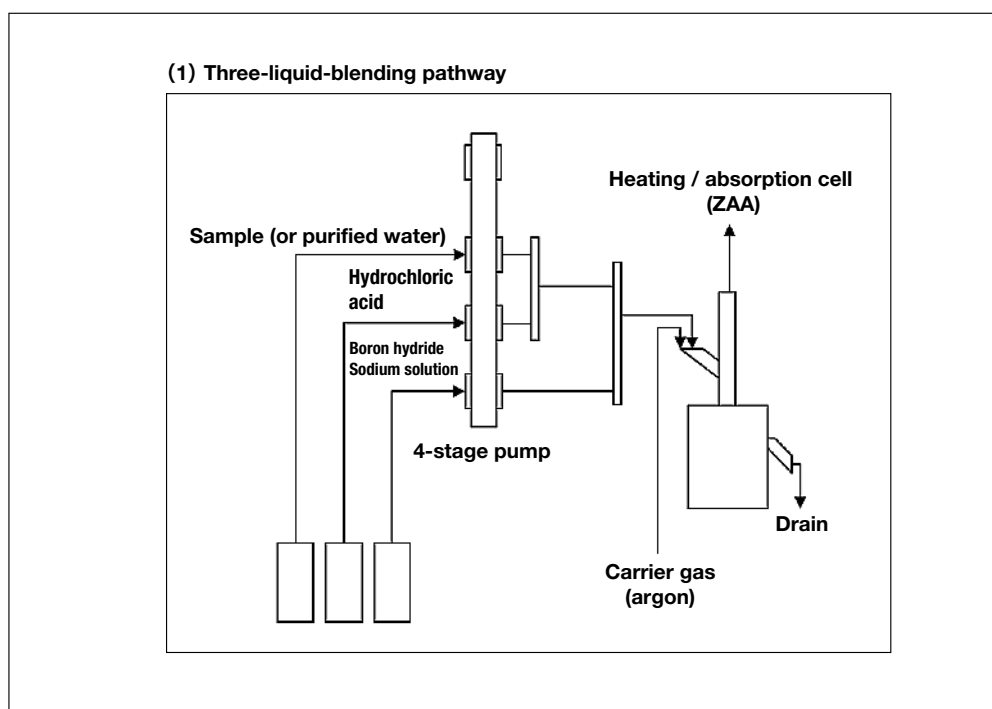


Fig. 2 THFS-4 flow pathways.

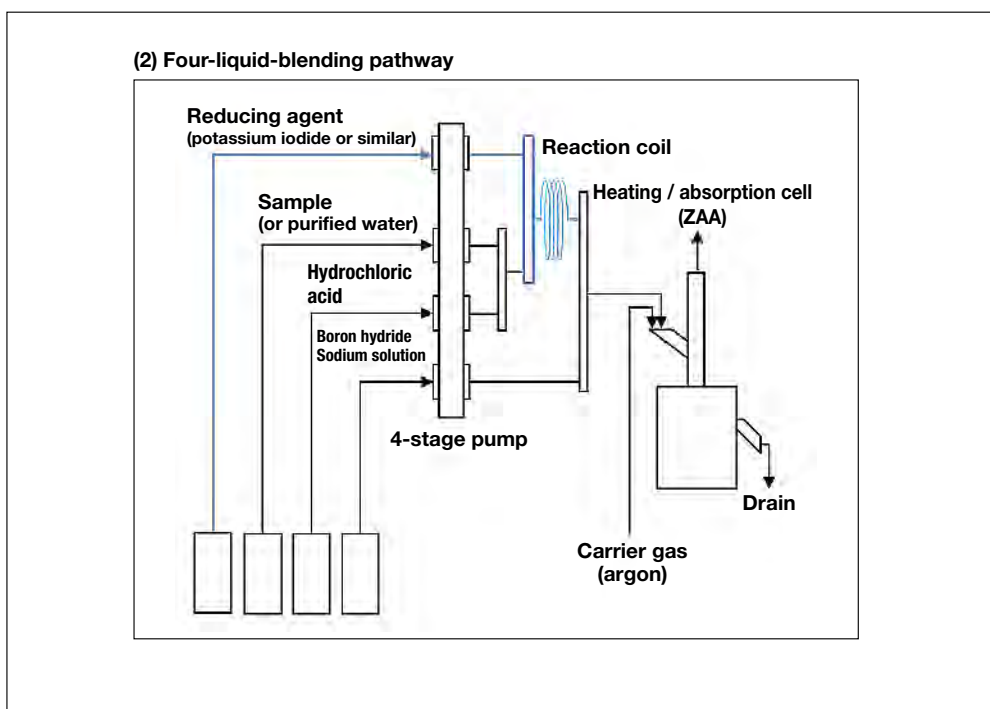


Fig. 2 THFS-4 flow pathways.

3-2 Stability through flame Zeeman atomic absorption spectrometry

We connected the SSC-230 autosampler to the HFS-4 to perform consecutive measurements of As. We prepared calibration curves for As concentrations of 0, 1, 5, and 10 $\mu\text{g/L}$, and then performed 50 consecutive measurements of the 5 $\mu\text{g/L}$ solution. We did not perform autozeroing or resloping of the calibration curves during sample measurements. The measurement conditions are listed in Table 1, and the measurement results are summarized in Table 2. Figure 3 shows the atomic absorption signal after the 50 consecutive measurements. The results indicate that stable, accurate, and quantitative measurements were obtained, with a mean value of 5.02 $\mu\text{g/L}$ and an RSD of 1.22%.

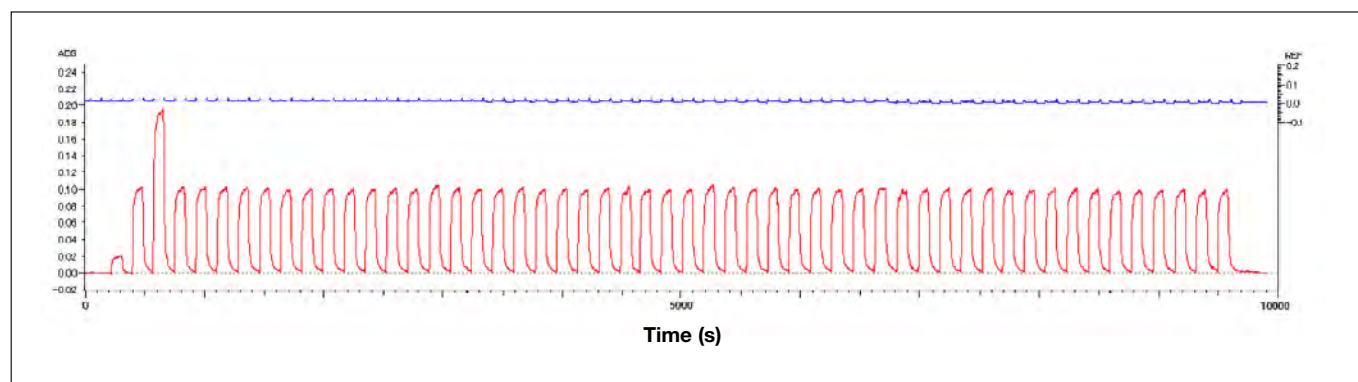


Fig. 3 Atomic absorption signal for 5 $\mu\text{g/L}$ As.

Table 1 Conditions for measurement of 5 $\mu\text{g/L}$ As.

Element	As	Atomizer	STD Burner	Meas. Mode	Working Curve
Instrument	ZA3000	Flame	Air-C ₂ H ₂	Signal Mode	BKG Correction
Atomization	Flame/ Auto-sampler	Fuel (C ₂ H ₂)	1.2 L/min	Curve Order	Linear
Wavelength	193.7 nm	Oxidant (Air)	160 kPa	Calculation	Integration
Lamp Current	12.0 mA		15.0 L/min	Delay Time	70.0 sec
Slit Width	1.3 nm	Burner Height	7.5 mm	Calculation Time	5.0 sec

Table 2 Results of measurements of 5 µg/L As.

	Concentration	Absorptivity
Mean value	5.02	0.0980
SD	0.06	0.0012
RSD	1.22%	1.23%

4. Applications

4-1 Measurement of Se in a standard sample of river water

As specified by the JIS K 0102 standard, KI should not be added for Se measurements. Therefore, we performed measurements using the three-liquid-blending pathway with the sample, HCl, and NaBH₄. Here, we present measurements of Se in a standard sample of river water. Figure 4 shows the Se atomic absorption signal, while Figure 5 shows the calibration curve used. Table 3 lists the measurement conditions, while Table 4 summarizes the measurement results. The measured values lie within the accepted range of values.

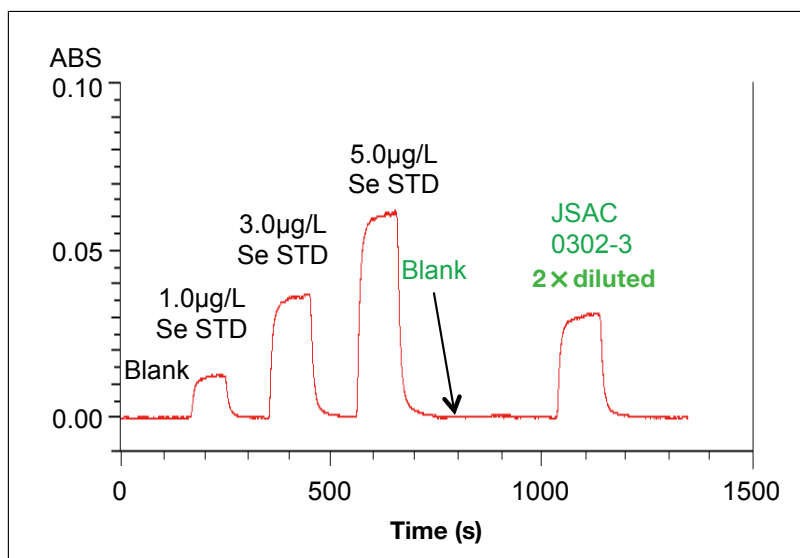


Fig. 4 Atomic absorption signal for Se.

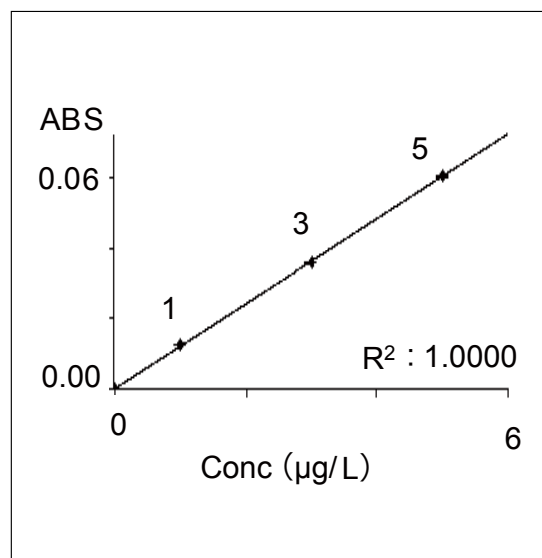


Fig. 5 Se calibration curve.

Table 3 Se measurement conditions.

ZA3000 conditions			
Element	Se	Atomizer	STD Burner
Instrument	ZA3000	Flame	Air-C ₂ H ₂
Atomization	Flame	Fuel (C ₂ H ₂)	1.0 L/min
Wavelength	196.0 nm	Oxidant (Air)	160 kPa
Lamp Current	12.5 mA		15.0 L/min
Slit Width	1.3 nm	Burner Height	7.5 mm
HFS-4 reagent		Sample preparation	
Reagent	Flow Rate	Sample	10 mL
1mol/L HCl	1 mL/min	HCl	4 mL
1% NaBH ₄	1 mL/min	Total	20 mL

Table 4 Measurement results for Se in a standard sample of river water

Sample name	Measured value (µg/L)	Accepted range (µg/L)
JSAC 0302-3	5.11	5.2±0.1

Measured values reported here are values converted to the concentration of the original liquid

These example measurements of As (AA150004) and Sb (AA150005) in standard samples of river water may be found on the S. I. navi application page; please consult that reference for further details.

5. Summary

The HFS-4 hydride formation system retains the high stability of previous-generation models while offering significant reductions in the quantities of samples and reagents consumed. We expect that these instruments will find an increasingly broad array of applications, primarily in environmental science and food analysis.

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