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Application Data Sheet	**ICPE-9000, AA-7000F and HVG-1**
	Analysis of Arsenic in Canned Fish with Hydride Vapour Generation Method
No. AD-0067	

The quantitative analysis of arsenic in food can be carried out using HVG with flame atomic absorption spectrophotometry (AAS)[1] and inductively coupled plasma-atomic emission spectrometry (ICP-AES) methods. Inorganic arsenic exists in trivalent and pentavalent forms, and in seafood, arsenic is mainly found in organic form[2]. In order to decompose this organic arsenic and any other co-existing organic substances, nitric acid is added to the sample followed by thermal decomposition. As a result of this process, arsenic is changed to pentavalent form. However, because arsenic reacts to form hydrides in trivalent form, the sample must be pre-reduced by adding potassium iodide in order to ensure all the arsenic is in trivalent form. On the other hand, reduction will not take place properly if there is any nitric acid left and so after thermal decomposition, the sample is heated until almost dry to evaporate all the nitric acid. This data sheet demonstrates the analysis of arsenic in canned fish using both AAS-HVG and (ICP-AES)-HVG methods.

❑ Sample Preparation

The 1000ppm Arsenic (III) standard solution and sodium borohydride were from Sigma-Aldrich (Fluka), USA whereas potassium iodide (KI) was from JT Baker, USA. Hydrochloric acid (HCl) and sodium hydroxide were purchased from Merck, Germany. Ultra pure water was produced by reverse osmosis, electrodeionisation, UV and finally filtered by a 0.22 mm filter to produce water with resistivity of 18 MΩ using the Milli-Q system from Millipore, USA. The Arsenic hollow cathode lamp was purchased from Hamamatsu, Japan.

The sample used in this analysis was Canned Fish Certified Reference Material (CRM), T07121QC, from the Food and Environment Research Agency (FAPAS), USA. It contains 2550 μg/kg arsenic and the satisfactory range is 1842-3259 μg/kg Arsenic. The Canned Fish sample, 0.25g was prepared using nitric acid-microwave system, followed by mild boiling to remove nitric acid and then topped up to 25ml with 1M HCl. There were two sets of preparation. Finally, the sample was diluted 10 times and added with KI to a final concentration of 1% before analysis.

The sodium borohydride and HCl reagents used in HVG were prepared as in Shimadzu HVG-1 Instruction Manual[3].

❑ Analytical Conditions

The flame AAS and ICP-AES analysis conditions were shown in Tables 1 and 2 respectively.

Table 1: AA-7000F Analytical Conditions

Wavelength	193.7nm
Lamp current	12mA
Slit width	0.7nm
Background correction	Deuterium lamp
HVG quartz cell heating	Air-Acetylene flame

Table 2: ICPE-9000 Analytical Conditions

Radio Frequency Power	1.20	(kW)
Plasma Gas Flow Rate	10.0	(L/min)
Auxiliary Gas Flow Rate	0.60	(L/min)
Nebulizer Gas Flow Rate	0.80	(L/min)
Plasma Torch	Mini-torch	
Observation	Axial	

❑ Results

The Arsenic calibration curves and profiles by AAS-HVG were shown in Figures 1 and 3 whereas Figures 2 and 4 are for ICP-HVG. The results were summarized in Table 3. Both the AAS-HVG and ICP-HVG results were in the satisfactory range.

Table 3: Results of AAS-HVG and ICP-HVG

	AAS-HVG	ICP-HVG	CRM (T07121QC)
Measured Concentration	2872 µg/kg 2966 µg/kg	2709 µg/kg 2712 µg/kg	1842-3259 µg/kg
Instrument Detection Limit	0.1 ppb[4] (3 σ blank)	0.1 ppb[2] (3 σ blank)	
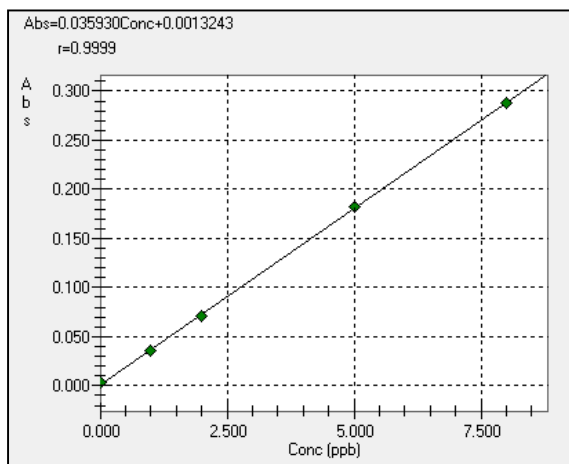
Figure 1: Arsenic calibration curve with AAS-HVG
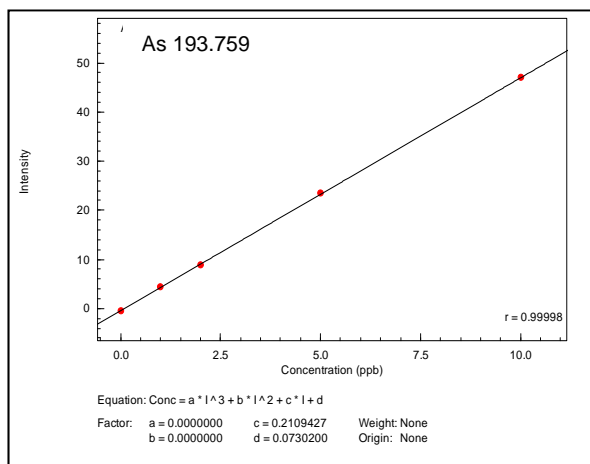

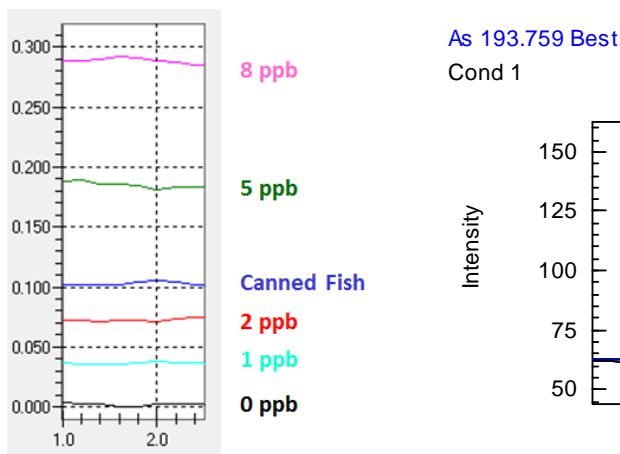Figure 2: Arsenic calibration curve with ICP-HVG
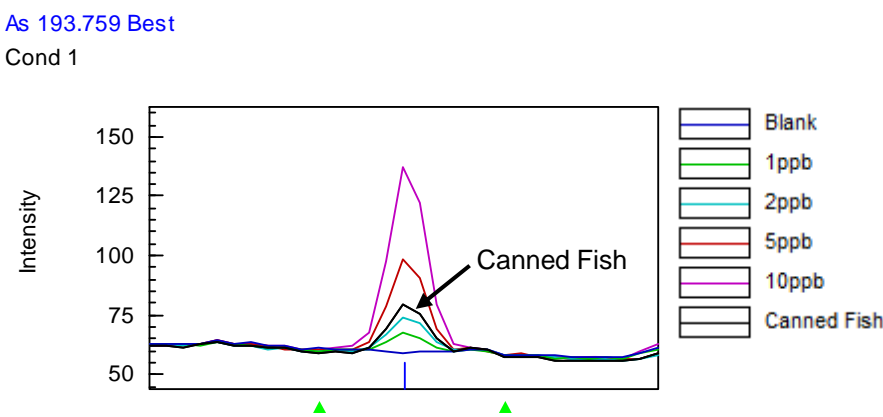

Figure 3: AAS-HVG Arsenic profiles

Figure 4: ICP-HVG Arsenic Profiles

❑ Summary

Arsenic can be analysed using both AAS-HVG and ICP-HVG methods. Arsenic reacts with nascent hydrogen to generate gaseous hydride. Introducing the hydride into the flame atomizer or into the plasma enables high sensitivity analysis. It is shown that with microwave digestion system, removal of nitric acid and pre-reduction with potassium iodide, both AAS-HVG and ICP-HVG can determine arsenic in canned fish at ppb level.

❑ Reference

1. AOAC Method No 986.15. Arsenic, Cadmium, Lead, Selenium, and Zinc in Human and Pet Foods.
2. Training Material for ICPE-9000 Operation
3. Shimadzu HVG-1 Instruction Manual
4. AA Cook Book No 3 – Flame AAS Parameters for Each Element (AA-7000)

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