

SEA NO. 19 AUG.1998

Arsenic Detection Lower Limits of X-ray Overhead Beam Method (SEA5120) and X-ray Underside Beam Method (SEA2120)

1. Overview

The energy dispersion fluorescent X-ray analyzer is simple to use, accurate, fast, and can be used with all sample types including solids, powders, colloids, and aqueous solutions. In this application brief we report on findings about the detection lower limit of arsenic in an aqueous solution using the overhead beam method and underside beam method.

In fluorescent X-ray analysis, a cause of measurement error is whether a sample thickness is infinite in terms of X-rays. The effect on the fluorescent X-ray intensity by changing the sample amount also applies to changing the shape of the sample container. As a result, we experimentally found a sample amount that will allow a uniform detected X-ray intensity.

2. Sample Thickness Experiment

- (1) Instrument: SEA2120 (incident angle of 60° and output angle of 50°)
- (2) Sample: 2.0 ml of arsenic standard solution for atomic absorption (1000 mg/l) diluted in 10 ml of distilled water. This is an As 200ppm solution.
- (3) Measurement method: 2.5 μm mylar film was stretched over a solution container (inner diameter 24 mm, height 22 mm) of specification, and 2 to 10 ml of sample solution is added and measured by fluorescent X-ray analysis.
- (4) Measurement conditions: Rh X-ray tube, applied voltage of 50 kV, tube current of 3 μA, 10mm collimator, atmosphere is air, measurement time is 100 seconds.
- (5) Result: the following table shows the relationship between sample thickness and fluorescent X-ray intensity. Figure 1 shows the fluorescent X-ray spectrum.

Table 1 Sample Amount and Fluorescent X-ray Intensity

Amount (ml)	Thickness (cm)	Rate gross (cps)
2.0	0.44	36.2
4.0	0.88	38.9
6.0	1.32	39.8
8.0	1.77	40.5
9.0	1.98	40.6
10.0	2.21	40.0

Results show that measurement error caused by sample amount can be controlled at a thickness of about 1.5 cm and if the amount used is 7 ml or greater.

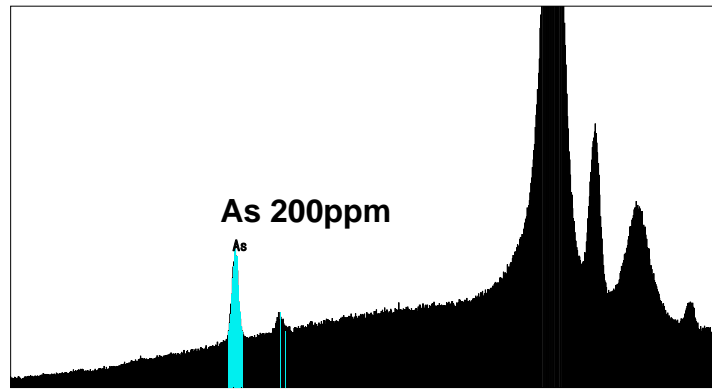


Figure 1 As 200ppm Fluorescent X-ray Spectrum

3. Arsenic Detection Lower Limit with X-ray Overhead Beam Method (SEA5100) and X-ray Underside Beam Method (SEA2100)

- (1) Standard solution: 2 ml of arsenic standard solution for atomic absorption (1000 ppm) diluted in 10 ml of distilled water. Water distilled by RO membrane + ion replacement was used as the blank.
- (2) Measurement method: 2.5 μm mylar film was stretched over a solution container (inner diameter 24 mm, height 22 mm) of specification and 9 ml of sample solution is added and measured by fluorescent X-ray analysis.
- (3) Measurement Conditions:

	Overhead Beam (SEA5120)	Underside Beam (SEA2120)
X-ray tube	Mo	Rh
Applied Voltage	50 kV	45 kV
Tube Current	8 μA	3 μA
Collimator	1.8 mm	10 mm
Atmosphere	Air	Air
Measurement Time	1000 sec	1000 sec
Sample Amount	9 ml	9 ml

- (4) Detection Lower Limit: Detection lower limit shows As density has a statistical error three times (3X) the background.

SEA5120 Detection Lower Limit

Standard As 500ppm Ave gross intensity (cps)	BKG (n=10) Ave gross intensity (cps)	BKG Statistical Error (cps)	SEA5120 As Detection LL
58.208	14.05	0.128	4.35 ppm

SEA2120 Detection Lower Limit

Standard As 500ppm Ave gross intensity (cps)	BKG (n=10) Ave gross intensity (cps)	BKG Statistical Error (cps)	SEA5120 As Detection LL
62.631	15.684	0.142	4.54 ppm

4. Summary

Many samples for fluorescent X-ray analysis are solids but aqueous solutions can also be easily and simultaneously analyzed for multiple elements. As our test results of arsenic show, accurate measurement of a 7ml sample can be done. However, for lesser amounts, a fixed amount of sample must be added to sample containers of identical size in a measurement sequence.

The detection lower limit of arsenic in an aqueous solution is about 4.5 ppm for both the SEA2100 and SEA5100.