

# A rapid method for simultaneous determination of arsenic, cadmium and lead in drinking water by inductively coupled plasma mass spectrometry

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## ABSTRACT

The raw water source of drinking water in most areas in the Philippines is typically river water and in some cases groundwater. These sources are prone to elevated levels of metals and metalloids that may cause exposure of the general population when the treatment of the water is inadequate. This work presents a simple method based on EPA Method 200.8 for the determination of total concentrations of arsenic (As), cadmium (Cd) and lead (Pb) in drinking water using inductively coupled plasma-mass spectrometry (ICP-MS) as the element-selective detector. This was applied in the determination of these elements in the water supply in Metro Manila, Philippines. The method detection limits were  $0.095 \mu\text{g L}^{-1}$ ,  $0.043 \mu\text{g L}^{-1}$ , and  $0.114 \mu\text{g L}^{-1}$  for total As, Cd and Pb, respectively.

The method was validated using National Institute of Standards and Technology (NIST) 1643e certified reference material for trace elements in water and determined values were  $60.4 \pm 0.5 \mu\text{g L}^{-1}$ ,  $6.7 \pm 0.1 \mu\text{g L}^{-1}$ , and  $19.6 \pm 0.5 \mu\text{g L}^{-1}$  for As, Cd and Pb, respectively. These determined values were in good agreement with the certified values in the reference material. Analysis of actual drinking water samples showed that most samples did not exceed the limit of the Philippine drinking water standard for the elements.

*Keywords:* As, Cd, Pb, ICPMS, Metro Manila, Drinking Water

## INTRODUCTION

Consumption of drinking water contaminated with toxic metals such as As, Cd and Pb has deleterious effects on human health (Kazi et al. 2009; Jain and Ali 2000; Massó et al. 2007; Satarug et al. 2003). Arsenic, which exists in different oxidation states, causes acute and chronic effects and is a suspected carcinogen that is responsible for skin and lung cancer (Hughes 2002). It attacks the nervous, cardiovascular, hepatic, endocrine, skin, renal and hematological systems which may cause skin lesions, encephalopathy, cirrhosis, hepatomegaly, diabetes and bone marrow depression (Hughes 2002). Bangladesh is said to have the world's biggest As contamination where high levels of As species have been found in groundwater in around 80% of the country's area and has placed an estimated 40 million people at risk (Karim 2000). Cadmium, for its part, may cause renal tubular dysfunction in kidneys and hepatotoxicity in livers after chronic exposure to it (Fowler 2009). In Southeast Asia, the highest cadmium exposure has been shown to be through dietary intake with rice as the major pathway (Ikeda et al. 2000). The possible mechanisms for Pb toxicity includes generation of reactive oxygen species, alteration of lipid metabolism, damage to the mitochondria, disruption of calcium homeostasis and substitution for zinc in various zinc-mediated processes. These may eventually result to neurological disorders, cancer, birth defects, and growth retardation especially in children (Ahamed and Siddiqui 2007).

Recent trends have made people more aware of the risks associated with trace metal toxicity and this led various regulatory agencies to set maximum allowable concentrations of these contaminants in the water to evaluate quality (Haider et al. 2002; Roccaro et al. 2005). These guidelines define the mandatory quality standards of water intended for human consumption. The guideline values in drinking water being implemented by the World Health Organization (WHO) are as follows: 10.0  $\mu\text{g L}^{-1}$  for As, 3.0  $\mu\text{g L}^{-1}$  for Cd, and 10.0  $\mu\text{g L}^{-1}$  for Pb (WHO 1996). In the Philippines, standard values of these trace metals in drinking water have been established by the government's Department of Health (DOH). According to the Philippine National Standards for Drinking Water 2007, the threshold limits in water intended for human consumption are 50.0  $\mu\text{g}$

$\text{L}^{-1}$  for As, 3.0  $\mu\text{g L}^{-1}$  for Cd, and 10.0  $\mu\text{g L}^{-1}$  for Pb (DOH 2007).

Equally important as setting the allowable concentration level for these metals is the development of techniques and methods that accurately quantify them at trace concentrations. Standard methods used for trace metal determination such as those from the US Environmental Protection Agency (US-EPA) include graphite furnace atomic absorption spectroscopy (GF-AAS, EPA Method 213.2, 1994), inductively coupled plasma optical emission spectroscopy (ICP-OES, EPA Method 200.7, 1994) and ICP-mass spectrometry (ICP-MS, EPA Method 200.8, Creed et al. 1994). Each of the methodologies employed offer unique advantages over others but also come with disadvantages and for this study, ICP-MS was used as the element selective detector due to the wide linear range (up to nine orders of magnitude), ultra trace and multi-elemental capability, and very low detection limits offered by the instrument (Thomas 2001). This paper presents a simple, rapid and more sensitive method utilizing ICP-MS for element detection in drinking water which was applied to survey the occurrence of As, Cd and Pb in the drinking water of all major cities comprising Metro Manila. The method described is simpler compared to EPA Method 200.8 (Creed et al. 1994) which is the standard method for determination of waters and wastes by ICPMS because of the absence of any pre-treatment step in the current method as opposed to the acid digestion step necessary in EPA Method 200.8. The lack of a pre-treatment step in the optimized method limits its applicability to determination of total concentrations of the analytes in drinking water samples due to possibility of higher dissolved solids in other matrices. This direct technique, however, is very useful for a direct and rapid determination of As, Cd and Pb in drinking water samples with minimal waste generated during the analysis of these contaminants for routine monitoring.

## EXPERIMENT

### Standard Solutions and Reagents

All reagents used in this work were of analytical grade. Single element standards of As, Cd, Pb and germanium (Ge) were purchased from CPI International (Santa Rosa, CA, USA). A certified reference material (NIST

1643e, Trace Elements in Water) by making appropriate dilutions of the stock solutions with ultrapure water and were acidified to have an acid concentration of 2%  $\text{HNO}_3$  (v/v) in polyethylene tubes. The material NIST 1643e was diluted 10-fold and was also acidified prior to validation experiments. Analytical blanks were prepared using ultrapure water in the same manner as the sampled tap water.

### Sample collection

Metro Manila, also called National Capital Region, is composed of sixteen cities and one municipality (shown in Fig. 1) and is home to about 12 million inhabitants.



**Figure 1.** Map of Metropolitan Manila showing the sixteen cities and one municipality comprising the metropolitan area.

Tap water was sampled from all the sixteen cities and municipality of Metro Manila as described by the Philippine National Standards for Drinking Water (DOH, 2007). Samples were collected in 250 mL polyethylene containers and kept at about 4°C during transport. The samples were acidified to have an acid concentration of 2%  $\text{HNO}_3$  (v/v) and maintained at 4°C before analysis. In total, 50 tap water samples were taken from households and establishments all of which are connected to one of the two major drinking water treatment plants supplying the cities. The sampling was conducted in August 2009. The samples were collected directly from the tap after allowing the water to flow freely for one minute. Field fortified samples were prepared using ultrapure water. Aliquots of the acidified samples were then placed in the autosampler vials and subjected to analysis to determine the total concentrations of As, Cd and Pb.

### Instrumentation

An Agilent 7500cx ICPMS (Agilent Technologies, Germany) equipped with a MicroMist glass concentric nebulizer and an integrated autosampler (I-AS with type A vials, 89 x 6 mL capacity) was used as the element-selective detector. The instrument was tuned to get the maximum sensitivity for all the analytes and reduce the interferences from oxides and doubly-charged species. The optimum operating conditions are summarized in Table 1. Instrument drift was monitored by analyzing test solutions containing 1.00  $\mu\text{g L}^{-1}$  of As, Cd, and Pb at repeated intervals throughout the analysis sequence. Ge (100.0  $\mu\text{g L}^{-1}$  Ge in 2%  $\text{HNO}_3$ ) was added online using the peristaltic pump and used as internal standard for As. The analysis of one sample

**Table 1.** Optimum Instrument Conditions for the Agilent 7500cx ICP-MS used in the analysis.

Instrument Parameters	
Rf power	1550 W
Carrier gas flow rate	0.85 L/min
Makeup gas flow rate	0.25 L/min
Nebulizer pump	0.01 rps
Sampling depth	8.2 mm
S/C Temp	2.0 °C
Peripump	30 rps

took about 3 min from drawing the sample until the rinses were finished prior to drawing of the next sample.

## RESULTS AND DISCUSSION

### Spectral Interference

Potential spectroscopic interferences that may be encountered were corrected using the built-in interference equations in the Agilent ICPMS ChemStation System (G1834B). The primary disinfection process employed in the Philippines is chlorination, thus the drinking water samples may have quantities of chlorine that may introduce spectral interferences in the form  $^{40}\text{Ar}^{35}\text{Cl}$  and influence accuracy of  $^{75}\text{As}$  determination. During analysis, counts for  $m/z$  75 (As), 77 (Se) and 82 (Se) were monitored. Comparing raw counts ( $n=3$ ) for  $m/z = 75$ , 77 and 82 from samples of drinking water showed that the average detectable counts for 77 and 82 were 2.84% and 4.41% of the counts corresponding to  $m/z$  75. These results indicate that interference from chloride was corrected using the mathematical equations in the ChemStation software as these are capable of correcting signals of the analyte if the interfering element contributes  $\leq 20\%$  of the total signal (D'Ilio et al. 2008).

### Method Validation

Linearity, internal standard response, detection limits, accuracy and repeatability were evaluated to assess method performance. Linearity was demonstrated for all the investigated elements (As, Cd, Pb) in the range 0.01 to 100.0  $\mu\text{g L}^{-1}$ , with correlation coefficients of at least 0.999 for all three elements. The method detection limits were estimated by analyzing 7 replicates of a solution containing 1  $\mu\text{g L}^{-1}$  of all three elements. The detection limits were calculated by multiplying the

standard deviation of the calculated elemental concentrations in the test solutions by the Student's  $t$ -value (3.143 for seven replicates, Wisconsin Department of Natural Resources Laboratory, 1996). The estimated values were 0.095  $\mu\text{g L}^{-1}$ , 0.043  $\mu\text{g L}^{-1}$ , and 0.114  $\mu\text{g L}^{-1}$  for As, Cd and Pb, respectively. These values are better compared to the reported detection limits in EPA Method 200.8: 1.4  $\mu\text{g L}^{-1}$  for As, 0.5  $\mu\text{g L}^{-1}$  for Cd and 0.6  $\mu\text{g L}^{-1}$  for Pb (Creed et al. 1994). For validation, the method was applied to determine the total As, Cd and Pb in NIST 1643e reference water. The results of repeated analysis of the reference material in different days showed good agreement against the certified values for all elements investigated (values summarized in Table 2). Ge was used as internal standard for As determination and was found to be ineffective for correction of Cd and Pb levels. This was expected since internal standard correction requires that the analyte and internal standard used should have relatively close  $m/z$  ratios for effective correction (Eickhorst and Seuber 2004). Concentrations of Pb and Cd, therefore, were determined by external calibration without the use of the internal standard. The signals for Ge (taken as counts per second) were monitored throughout the run and showed a relative standard deviation (RSD) of 1.22% indicative of a stable response and hence, improving the analysis for As. The RSD for Ge signals also indicates stable signals for the three analytes and this was validated by the RSD determined for the drift standard (1.0  $\mu\text{g L}^{-1}$  measured at repeated intervals throughout the analysis sequence) which was determined to be in the range 1.8% to 2.3% for the three analytes.

### Analysis of actual samples

The determined total As, Cd and Pb in drinking water samples are summarized in Table 3. The analysis of the field, laboratory and calibration blanks reflected

**Table 2. Determined total concentrations of As, Cd and Pb in certified reference material NIST 1643e (mean  $\pm$  sd,  $n=3$ ) in different days.**

Element	Measured Value ( $\mu\text{g L}^{-1}$ )			Certified Value ( $\mu\text{g L}^{-1}$ )
	Day 1	Day 2	Day 3	
As	60.4 $\pm$ 0.5	60.6 $\pm$ 0.5	60.1 $\pm$ 0.7	60.45 $\pm$ 0.72
Cd	6.7 $\pm$ 0.1	6.8 $\pm$ 0.2	6.6 $\pm$ 0.1	6.568 $\pm$ 0.073
Pb	19.6 $\pm$ 0.5	19.3 $\pm$ 0.4	19.7 $\pm$ 0.6	19.63 $\pm$ 0.21

City/ Municipality	Sample Number	As (ig L <sup>-1</sup> )	Cd (ig L <sup>-1</sup> )	Pb (ig L <sup>-1</sup> )
Marikina	1	1.47 ± 0.01	0.05 ± 0.02	0.68 ± 0.02
	2	0.38 ± 0.02	0.07 ± 0.02	0.30 ± 0.07
	3	0.76 ± 0.03	<MDL	0.25 ± 0.02
Pasig	4	0.65 ± 0.04	<MDL	0.55 ± 0.02
	5	0.49 ± 0.02	<MDL	0.26 ± 0.04
	6	0.45 ± 0.01	<MDL	<MDL
Pateros	7	0.41 ± 0.03	<MDL	0.28 ± 0.01
	8	0.38 ± 0.02	<MDL	0.66 ± 0.01
Makati	9	0.35 ± 0.01	<MDL	0.24 ± 0.01
	10	0.36 ± 0.01	<MDL	0.75 ± 0.02
	11	0.37 ± 0.02	<MDL	0.41 ± 0.01
Taguig	12	0.32 ± 0.02	<MDL	1.35 ± 0.01
	13	0.33 ± 0.04	<MDL	0.34 ± 0.01
Muntinlupa	14	5.47 ± 0.16	<MDL	<MDL
	15	12.46 ± 0.06	<MDL	0.60 ± 0.01
Parañaque	16	4.61 ± 0.15	<MDL	0.67 ± 0.01
	17	5.06 ± 0.06	<MDL	<MDL
	18	3.93 ± 0.15	<MDL	0.28 ± 0.01
Las Piñas	19	0.31 ± 0.02	<MDL	0.25 ± 0.01
	20	3.27 ± 0.04	<MDL	1.77 ± 0.01
	21	4.07 ± 0.14	<MDL	0.12 ± 0.01
Pasay	22	3.18 ± 0.04	<MDL	0.12 ± 0.01
	23	0.32 ± 0.02	<MDL	0.60 ± 0.01
	24	0.29 ± 0.01	<MDL	0.25 ± 0.01
Mandaluyong	25	0.31 ± 0.05	<MDL	0.71 ± 0.01
	26	0.29 ± 0.02	<MDL	0.49 ± 0.02
	27	0.29 ± 0.02	<MDL	1.15 ± 0.01
Quezon	28	0.26 ± 0.03	0.11 ± 0.01	8.62 ± 0.04
	29	0.27 ± 0.01	<MDL	0.53 ± 0.01
	30	0.68 ± 0.02	0.05 ± 0.01	0.68 ± 0.02
	31	0.37 ± 0.02	<MDL	0.48 ± 0.01
Valenzuela	32	0.34 ± 0.01	<MDL	1.51 ± 0.02
	33	0.54 ± 0.05	<MDL	0.69 ± 0.01
	34	0.50 ± 0.04	<MDL	0.43 ± 0.01
Malabon	35	0.39 ± 0.02	<MDL	0.59 ± 0.01
	36	0.28 ± 0.03	<MDL	<MDL
	37	0.27 ± 0.03	<MDL	0.44 ± 0.01
Navotas	38	0.25 ± 0.03	<MDL	0.51 ± 0.00
	39	0.28 ± 0.01	<MDL	0.65 ± 0.01
	40	0.26 ± 0.01	<MDL	0.17 ± 0.01
Caloocan	41	0.30 ± 0.04	<MDL	0.14 ± 0.01
	42	0.25 ± 0.02	<MDL	0.16 ± 0.01
	43	0.23 ± 0.01	<MDL	1.28 ± 0.01
Manila	44	0.27 ± 0.02	<MDL	0.13 ± 0.01
	45	0.23 ± 0.03	<MDL	1.15 ± 0.01
	46	0.21 ± 0.03	<MDL	0.64 ± 0.01
	47	0.25 ± 0.02	<MDL	1.04 ± 0.01
San Juan	48	0.30 ± 0.02	<MDL	1.07 ± 0.01
	49	0.27 ± 0.04	<MDL	18.98 ± 0.08
	50	12.96 ± 0.12	0.05 ± 0.01	6.72 ± 0.05

Table 3. Determined total concentrations of As, Cd and Pb in drinking water samples (mean ± sd, n=3). (The method detection limits were 0.095 µg L<sup>-1</sup>, 0.043 µg L<sup>-1</sup>, and 0.114 µg L<sup>-1</sup> for total As, Cd and Pb, respectively.)

elemental concentrations below the method detection limits. The validation of the method using the certified reference material and drift standard was carried out alongside the analysis of actual samples (results discussed in previous sections). For the actual samples, except for the measured Pb in a tap water sample taken from San Juan City, the determined concentrations of all elements monitored in all the samples passed the standard limits prescribed for drinking water in the Philippines. Notably, one sample from Muntinlupa City was above the guideline value set by the WHO for As in drinking water. Concentrations of As in the samples ranged from  $0.21 \pm 0.03 \mu\text{g L}^{-1}$  to a maximum of  $12.96 \pm 0.06 \mu\text{g L}^{-1}$ . Also, it can be observed that compared to most of the cities in Metro Manila, a relatively high As concentration resulted from samples that were taken in a set of neighboring cities (Las Piñas, Parañaque and Muntinlupa). It is suspected that possible sources of contamination would be intrusion from groundwater in which natural levels of As largely depends on the area's geochemical features as what was observed in similar studies (Cavar et al. 2005, Peters et al. 2006). However, it is deemed that our observation on the slightly elevated As in these neighboring cities, certainly, warrants further investigation.

Most of the measured Cd concentrations were either below the method detection limit or were in the range  $0.05 \pm 0.01$  to  $0.11 \pm 0.01 \mu\text{g L}^{-1}$ . Cd was only detected in several sampling sites. The Pb concentrations were measurable but were low compared to the allowable concentration except for one sample (San Juan City, site 49). Although Pb may be naturally occurring in certain areas, it rarely enters the water supply as a result of its dissolution from natural sources. Pb contamination in drinking water is largely caused by corrosion in pipes that make up the distribution network or household pipelines (Conio et al. 1996). This is probably the reason why elevated levels of this trace metal were observed in two samples from San Juan ( $18.98 \pm 0.08 \mu\text{g L}^{-1}$  and  $6.72 \pm 0.05 \mu\text{g L}^{-1}$ ) and one in Quezon City ( $8.62 \pm 0.04 \mu\text{g L}^{-1}$ ). In recent years, massive rehabilitation of the distribution lines were carried out by the water concessionaires but some areas may still be using old pipes.

There is little information about the occurrence of trace metals in the drinking water in most cities in Southeast

Asia. In the Philippines, one research reported on the influence of the presence of a dumpsite on the quality of the water (Sia Su 2007). Total Cd was included as a parameter to be considered in the study but the focus was entirely on the probable relationship of the occurrence of Cd, sulfate and coliform to the observed incidence of diarrhea in the population nearby the dumpsite. Another research looked at the exposure of women living in Metro Manila toward Pb and Cd as reflected in the results of the analysis in blood and urine samples taken from the volunteers (Zhang et al. 1998). The results showed that Pb exposure was through inhalation of air contaminated by vehicular exhausts and that Cd exposure through air accounts for only 15% of the body burden suggesting other pathways of exposure.

Recent studies have reflected that the main source of As exposure is via the drinking water. But rice, which is the staple food of most people in Southeast Asia, has been shown to accumulate As and is now posing an increased risk to the population consuming it (Meharg 2004). Cd has also been reported to be accumulated in rice (Ikeda et al. 2000). The major pathway for Pb exposure is still through inhalation of air but recent regulations on the use of Pb as anti-knock agent in fuels have reduced the Pb concentration in air significantly (Suk et al. 2003). The results on the total concentrations of As, Cd and Pb in the drinking water supply in Metro Manila show that the population inhabiting this metropolitan capital is at least safe from exposure to these elements via the drinking water source. These results also validate that the optimized method may be used for routine monitoring of As, Cd and Pb in the drinking water supply.

## CONCLUSION

The study presented in this paper demonstrates a simple, rapid and accurate method based on EPA Method 200.8 for the simultaneous determination of As, Cd and Pb in drinking water using inductively coupled plasma mass spectrometry as element selective detector. The estimated detection limits are very low suggesting that the method may be applied for trace analysis of these analytes in drinking water. The method may be used for routine monitoring or random analysis of these contaminants in the drinking water supply. Application

of the method in the analysis of drinking water from all cities in Metro Manila indicated that all except for one sample passed the drinking water standards for these trace contaminants set by the regulatory agencies in the Philippines.

## ACKNOWLEDGEMENTS

The authors would like to thank the Natural Sciences Research Institute, University of the Philippines Diliman for funding this work.

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