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# Method development for arsenic analysis by modification in spectrophotometric technique

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## Abstract

Arsenic is a non-metallic constituent, present naturally in groundwater due to some minerals and rocks. Arsenic is not geologically uncommon and occurs in natural water as arsenate and arsenite. Additionally, arsenic may occur from industrial discharges or insecticide application. World Health Organization (WHO) and Pakistan Standard Quality Control Authority have recommended a permissible limit of 10 ppb for arsenic in drinking water. The arsenic at lower concentrations can be determined in water by using high tech instruments like Atomic Absorption Spectrometer (hydride generation). The arsenic concentration at low limits of 1 ppb could not be determined easily with simple spectrophotometric technique. Therefore, Spectrophotometric technique using the silver diethyldithiocarbamate was modified to achieve the better results, up to the extent of 1 ppb arsenic concentration.

## 1 Introduction

Arsenic is steel grey, very brittle, crystalline in nature and oxidizes on rapid heating to arsenous oxide with odor of garlic. Arsenic exists as inorganic or organic compounds. In the environment, it combines with oxygen, chlorine and sulfur to form inorganic arsenic compounds. Arsenic combines with carbon and hydrogen to form organo-arsenic compounds in animals and plants. Inorganic arsenic compounds are mainly used to preserve wood and organic arsenic compounds are used as pesticides, primarily for cotton crop (Carapella, 1973; Calvert, 1975). Therefore, arsenic can be released into the environment from sources such as pesticides applications, wood preservatives, mining activities and petroleum refining. It is found exclusively as arsenite (Arsenic-III) or arsenate (Arsenic-V) in groundwater. Arsenite can be converted to arsenate under oxidizing conditions (e.g. well-aerated surface water). Likewise, arsenate can become arsenite under reducing conditions (e.g. anaerobic groundwater).

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The groundwater pollution caused by arsenic in various countries of the world has led to major environmental issues. A number of networks supplying water in the United States particularly in the Midwest and West regions are containing arsenic. In West Bengal of India, arsenic found in groundwater at several places hundred times above the permissible limits set for drinking water. The situation in Bangladesh is even worse than the West Bengal. In many areas, arsenic contamination is found above 3000  $\mu\text{g/l}$  compared to the recommended level of 10  $\mu\text{g/l}$  (Guha Mazumder et al., 1988). Similarly arsenic contamination is observed in Argentina, Canada, Chile, China, Greece, Japan, Mexico, Mongolia, New Zealand, South Africa, Philippines, Taiwan, Thailand and USSR. Therefore, the arsenic poisoning is emerging as a global issue.

Arsenic monitoring is utmost important now a days. Palmer (2001) reported atomic spectroscopy is the most widely used method for the arsenic determination. Atomic spectroscopy involves the use of absorption characteristic of metals (Andreae, 1977; Christian and Feldman, 1970; Chu et al., 1972; Clement et al., 1973; Fishman and Spencer, 1977). USEPA (1999) reported that GFAAS is an approved method by USEPA for measuring arsenic in drinking water. The detection limit for this method is 1–5 ppb. USEPA (2003) reported about the use of an ICP-AES instrument for the same purpose. It utilizes optical spectrometry to measure the characteristic atomic emission spectra of the analyte.

For arsenic analysis, detection limits are to be required very low ( $\leq 1$  ppb). It can be achieved only on state of art and latest version of equipment like atomic absorption spectrometer or ICP. Such high-tech equipments facilities and trained technical manpower to operate this equipment are lacking in most of laboratories in the country. Spectrometers are available in most of water quality laboratories however; arsenic analysis in drinking water is quite difficult on these equipments due to requirement of low detection limits for arsenic. Therefore, a practicable method for the analysis of arsenic at low detection limit on spectrophotometer was developed which will be useful for all simple and modest laboratories.

## 2 Methodology

Two analytical methods for inorganic arsenic are reported in a reference handbook (Michael, 1982) "Evaluation of Water for Pollution Control". The reference procedure is a photometric measurement using silver diethyldithiocarbamate. Arsenic analysis with the help of atomic absorption spectrometer is reported as a secondary method. However, the reference method was modified to some extent to get better results owing to present research work. The principle of the modified method is based on reduction of arsenic to arsine gas by a mixture of zinc, stannous chloride, potassium iodide and hydrochloric acid in a specially designed distillation apparatus. The arsine ( $\text{AsH}_3$ ) is passed through a scrubber containing cotton saturated with lead acetate and then into an absorber tube containing silver diethyldithiocarbamate in pyridine. The arsenic reacts to form a red complex, which can be read on spectrometer.

Apparatus for the experiment consists of mainly the Arsine generator and absorption tube and Spectrophotometer (U-1100), Hitachi, Japan. Whereas reagents consists of Arsenic standard solution BDH, UK, Hydrochloric Acid (ASC), Lead acetate solution (10%), Potassium iodide solution (20%), Pyridine Silver diethyldithiocarbamate (SDDC), Stannous chloride solution (40%) and Zinc (0.3–1.5 mm or 14–50 mesh).

Reagent Preparation for this experiment is done in following way:

1. 1.25 g of Silver Diethyldithiocarbamate (SDDC) was transferred in 250 ml volumetric flask and mixed well. The reagent was stored in an amber bottle. This reagent was used as an absorber for Arsenic.
2. Standards of 0, 5, 15, 25, 35 and 45 ppb or  $\mu\text{g/l}$  arsenic were prepared from 1 ppm standard (BDH Cat. No. 455042K, Lot No. 105016109, UK).

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### 3 Procedure (modified technique)

For evaluation of this modified technique, standard addition technique was used to prepare arsenic fortified solutions. For the addition of 20 ppb arsenic in each sample, five ml of 1-ppm arsenic standard was taken in 250 ml measuring flask; it was made up to the mark with water sample. After proper mixing, this sample was transferred into arsenic generation or distillation apparatus (Fig. 1) under fume hood to vent toxic fumes. A cotton ball soaked with 10% lead acetate solution was placed in the gas scrubber. Prepared arsenic absorber solution (25 ml) was transferred into gas bubbler assembly and attached it to the distillation apparatus. Then, 25 ml HCl, 1 ml of stannous chloride solution and 3 ml potassium iodide solution were added to flask respectively. After 12 min, 6 g of 14–50 mesh sized zinc was also added to flask. The temperature was maintained at 40°C for 12 min in the beginning after adding potassium iodide solution, 60°C for another 12 min in the middle after adding zinc and then set at 40°C for 16 min with continuous stirring at the rate of 100 to 150 rpm.

After the completion of reaction time (about 40 min), a dry sample cell was filled with un-reacted arsenic absorber solution (the blank) and other one filled with reacted arsenic absorber. Finally, concentration was determined by using concentration mode on pre-calibrated spectrophotometer at 535 nm ( $\lambda_{\max}$ ). The added concentration i.e. 20 ppb was deducted from final concentration of each sample. Standard addition is a widely accepted technique for checking the validity of test results. It is known as “Spiking” and “Known addition”. This technique is also used to check the performance of the reagents, instrument, apparatus, procedure and also to enhance the lower detection limit.

It was observed that fumes of arsenic absorber solution present in 10 mm rectangular quartz cell with lid were responsible to fog the lenses installed in the sample compartment which ultimately responsible of undesired results. Another source of unwanted results especially in low concentration is electricity supplied to spectrometer (variation in voltage). Making amendments in the equipment as shown in Fig. 2 rectified the prob-

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lem of fogging. Amendments include suction tube at upper side of the lenses (window) followed by a suction pump and a stabilizer. However, the problem of voltage variation was controlled with the help of stabilizer as shown in Fig. 3.

#### 4 Results and discussions

5 According to procedure, arsenic is reduced to arsine gas and formed a red complex with silver diethyldithiocarbamate. For the selection of suitable wavelength, absorbances and transmittances (%) were noted on spectrophotometer for known standard solutions i.e. 0, 25, 35 and 45 ppb after adopting the above procedure. Results are presented in Table 1.

10 From the results, it was concluded that maximum absorbances were noted at 535 nm in all cases. However, absorbances were found almost same from 535 to 540 nm. The chances of interferences in this method are almost negligible except antimony salts, which may interfere in color development. Therefore, 535 nm was selected as  $\lambda_{max}$  as shown in Fig. 4.

15 Correlation between absorbance vs. known concentrations was developed with the help of regression model in the light of experimental data shown in Table 2. Graphical views (xy) are shown in Figs. 5 and 6. First and second views reflected relationship between absorbance and concentration with or without straight line. The degree of fitness or R square is near to 1, which indicated good relationship between x and y. The developed co-relation, degree of fitness or R square and value of constant is given below:

20

Concentration of As = 983.4 X Absorbance – 0.8014

Degree of fitness or R<sup>2</sup> = 0.9958

Constant = –0.8014

25 X Coefficient = 983.4

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Fifteen water samples were analyzed on pre-calibrated spectrophotometer at 535 nm ( $\lambda_{\text{max}}$ ) by using modified method on concentration mode. The same numbers of samples were analyzed on atomic absorption spectrometer. The results can be seen in Table 3 and graphical view is shown in Fig. 7. The difference in concentration is  $\pm 1$  to 2 ppb that is not noteworthy at very lower concentration. The comparison of this modified technique with other commonly used spectrometric techniques i.e. APHA Method, HACH and WHO is given in Table 4.

## 5 Conclusions

The combination of high toxicity and widespread occurrence of arsenic has created a pressing need for effective monitoring and measurement of arsenic in soil and groundwater. Technological advances in a variety of analytical instruments have made improvement in accuracy and detection limit. However, the development of cost effective and reliable technique for arsenic determination by using comparatively cheap instrument like spectrometer is the need of time especially in developing countries facing arsenic contamination problem. Most of laboratories in these countries have not state of art equipment like Atomic Absorption Spectrometer (AAS) and Inductive Coupled Plasma Spectrometer (ICP) to analyze arsenic low level. This paper presented a brief overview of the scientific literature on existing technologies used for arsenic analysis in the groundwater and also includes research developments in this area. World Health Organization (WHO) has recommended guideline value as 10 ppb for arsenic and is analyzed in most of laboratories on high tech equipment like AAS and ICP due to low detection limit. These instruments need trained technical manpower to operate and maintain. Many laboratories are unable to analyze arsenic due to lack of such facilities. Spectrophotometer is a commonly available instrument in most of laboratories. Considering this fact, a modified spectrophotometric method has been developed for the analysis of arsenic at low detection limit. The comparative analytical evaluation on spectrophotometer with modified technique and Atomic Absorption Spectrometer

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revealed almost similar result ( $\pm 1$  to 2 ppb) and hopefully this modified method will be useful to enhance the analytical capabilities in respect of arsenic determination in most of laboratories.

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**Table 1.** Absorbance of standard solution at different  $\lambda$  range.

Standard Solution (nm)	0.000 ppb		0.025 ppb		0.035 ppb		0.045 ppb	
	Abs.	T%	Abs.	T%	Abs.	T%	Abs.	T%
520	0	0	0.015	96.6	0.026	94.2	0.037	91.8
521	0	0	0.016	96.4	0.026	94.2	0.037	91.8
522	0	0	0.016	96.4	0.027	94	0.038	91.6
523	0	0	0.017	96.2	0.028	93.7	0.039	91.4
524	0	0	0.018	95.9	0.029	93.5	0.041	91
525	0	0	0.019	95.7	0.03	93.4	0.042	90.8
526	0	0	0.02	95.5	0.031	93.2	0.043	90.6
527	0	0	0.021	95.3	0.032	92.9	0.043	90.6
528	0	0	0.022	95.1	0.033	92.7	0.044	90.4
529	0	0	0.023	94.9	0.034	92.5	0.045	90.2
530	0	0	0.023	94.9	0.034	92.5	0.045	90
531	0	0	0.024	94.7	0.035	92.3	0.046	90
532	0	0	0.025	94.4	0.035	92.3	0.046	90
533	0	0	0.025	94.4	0.035	92.3	0.046	90
534	0	0	0.025	94.4	0.035	92.3	0.046	90
535	0	0	0.026	94.2	0.036	92.1	0.047	89.7
536	0	0	0.026	94.2	0.036	92.1	0.047	89.7
537	0	0	0.026	94.2	0.036	92.1	0.047	89.7
538	0	0	0.026	94.2	0.036	92.1	0.047	89.7
539	0	0	0.026	94.2	0.036	92.1	0.047	89.7
540	0	0	0.026	94.2	0.036	92.1	0.047	89.7

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**Table 2.** Arsenic conc. vs. absorbance.

Sr. No.	Concentration (ppb)	Absorbance
1	0	0
2	5	0.008
3	15	0.015
4	25	0.026
5	35	0.036
6	45	0.047

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**Table 3.** Spectrometer vs. AAS (as analysis).

Sr. No.	Spectrometer (ppb)	AAS (ppb)
1	2	2
2	5	4
3	2	1
4	5	4
5	2	1
6	0	0
7	2	1
8	5	3
9	16	15
10	3	4
11	2	1
12	0	1
13	5	4
14	2	2
15	3	2

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**Table 4.** Comparison of modified technique with other spectrometric methods.

Steps	Modified Analytical Technique (2002)	HACH Method (1997)	Standard Methods (APHA, 1992)	Reference Method by WHO (Michael, J. S., 1982) and Standard Methods (APHA, 1971)
I	Dampen a cotton ball in 10% lead acetate solution and put it at appropriate place.	Same	Same	Same
II	25 ml of prepared arsenic absorber solution into absorber tube.	25 ml prepared arsenic absorber solution into absorber tube.	4 ml of SDDC into absorber tube.	Same
III	5 ml of 1-ppm arsenic standard added into 250 ml flask and volume make up with sample.	No standard addition	No standard addition	No standard addition
IV	Volume of sample ~245 ml	250 ml sample taken.	70 ml sample taken.	35 ml sample taken.
V	Stirring (100–150 rpm) continued up to step-XII.	Stir control set to 5 and heat control to 0.	Stirring continued up to Step-XI.	No stirring.
VI	Added 25 ml conc. HCL into generation flask of 250 ml volume	Added 10 ml acetate buffer and flushed with Nitrogen gas at 60 ml/minute	Added 10 ml acetate buffer and flushed with Nitrogen gas at 60 ml/minute	Added 5 ml conc. HCL into generation flask.
VII	Added 1 ml of stannous chloride.	15 ml of 1% sodium borohydride injected within 2 min.	15 ml of 1% sodium borohydride injected within 2 min.	Added 0.40 ml of stannous chloride.
VIII	Added 3 ml of potassium iodide solution.	Additional N <sub>2</sub> gas flushing for 15 min.	Additional N <sub>2</sub> gas flushing for 15 min.	Added 2 ml of potassium iodide solution.
IX	12 min given as "Reaction Time" at 40°C.	15 min given as reaction time.	–	15 min given as reaction time without heat control
X	6 g zinc added (0.3–1.5 mm or 14–50 mesh size)	6 g zinc added (20 mesh size)	–	Added 3 g of zinc.
XI	12 min given as reaction time at 60°C.	15 min given as reaction time and heat control set at 3.	–	30 min given as reaction time without heat control.
XII	16 min given as reaction time at 40°C.	15 min given as reaction time and heat control set at 1.	–	–
XIII	Measurement of blank and sample on spectrophotometer at 520 nm.	Measurement of blank and sample on spectrophotometer at 520 nm.	Measurement of blank and sample on spectrophotometer at 520 nm.	Measurement of blank and sample on spectrophotometer at 535 nm.
XIV	Added 25 ml conc. HCL into generation flask of 250 ml volume	Added 10 ml acetate buffer and flushed with Nitrogen gas at 60 ml/minute	Added 10 ml acetate buffer and flushed with Nitrogen gas at 60 ml/minute	Added 5 ml conc. HCL into generation flask.

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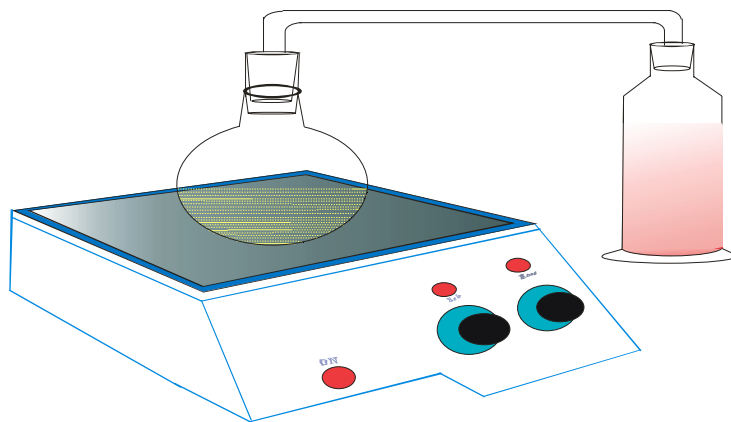
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**Fig. 1.** Arsine generation system.

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Fig. 2. Suction system to rectify fogging problem.

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**Fig. 3.** Suction system to rectify fogging problem and stabilizer to control voltage variation.

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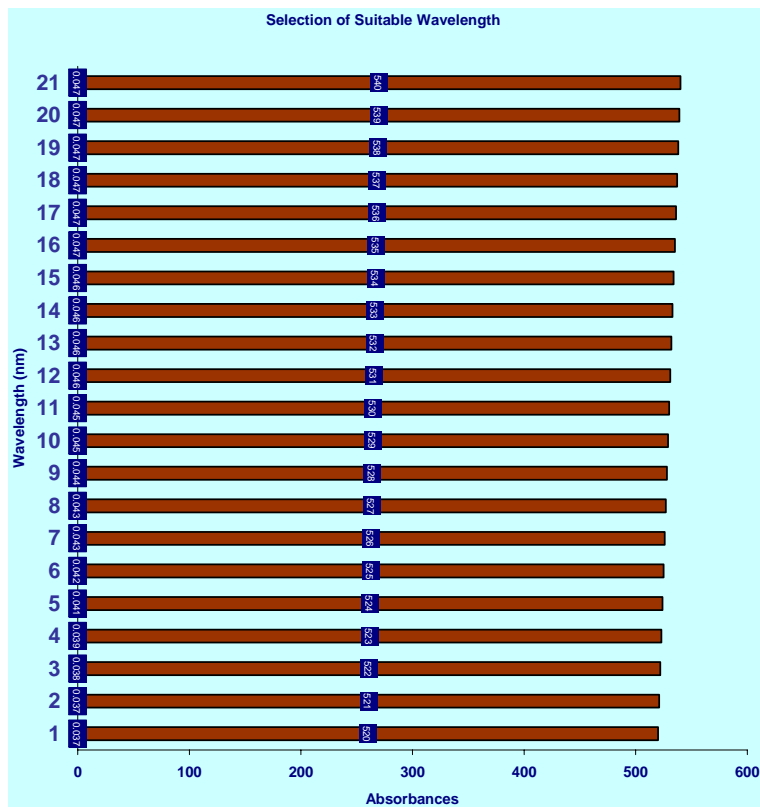


Fig. 4. 0.45 ppb arsenic standard at different wavelength.

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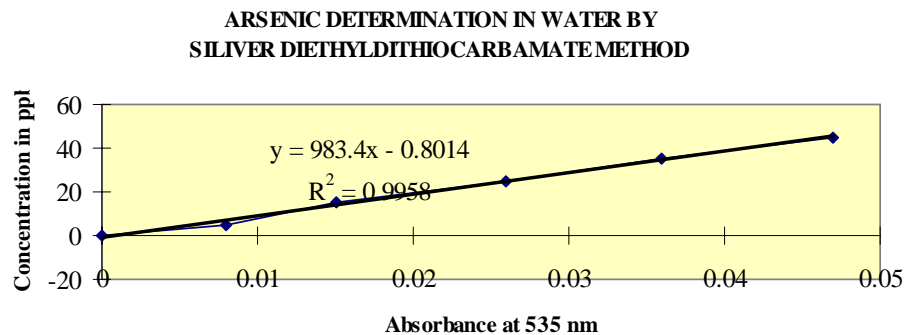
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**Fig. 5.** Absorbance vs. concentration with straight line.[Title Page](#)[Abstract](#)[Introduction](#)[Conclusions](#)[References](#)[Tables](#)[Figures](#)[I◀](#)[▶I](#)[◀](#)[▶](#)[Back](#)[Close](#)[Full Screen / Esc](#)[Printer-friendly Version](#)[Interactive Discussion](#)

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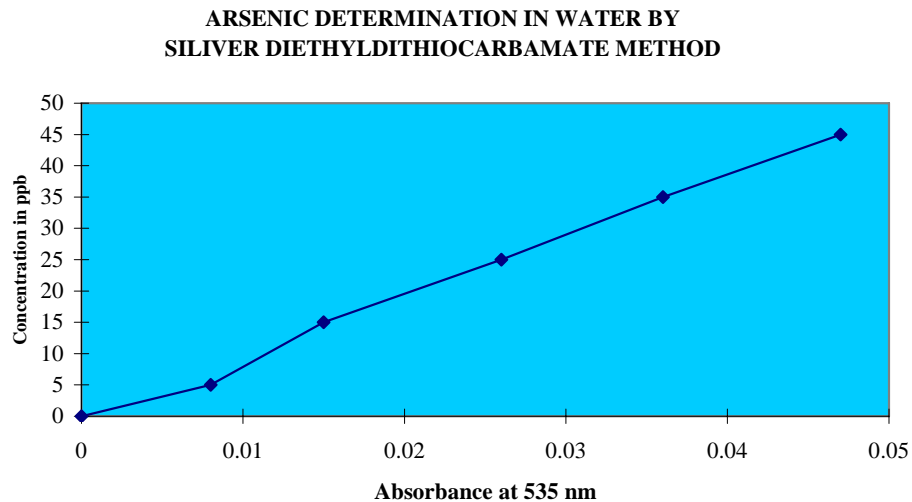


Fig. 6. Absorbance vs. concentration without straight line.

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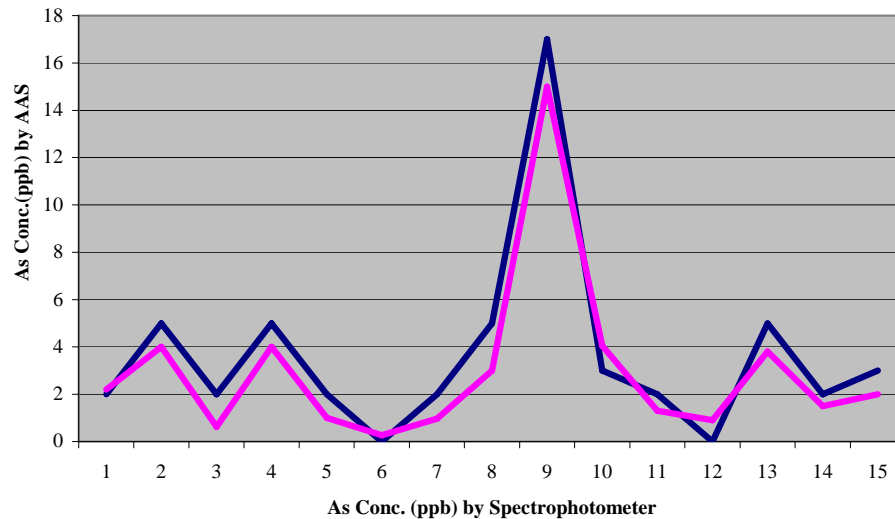


Fig. 7. Comparison of arsenic analysis.

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