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THE FACULTIES SHALL HAVE THE CONFERENCE PROCEEDINGS AND POSTER PRESENTATIONS.

- HUMANITIES AND SOCIAL SCIENCES
- SCIENCE AND TECHNOLOGY
- HEALTH SCIENCES

PREFACE

Bangkokthonburi University is the institution of education that focuses on increasing the academic knowledge by supporting the researchers, scholars and the graduate students to create and develop their works for developing the new knowledge in the community and country level which responds to the objective of creation the academic strength, and to support for creating the development, spreading the knowledge, exchanging the experiences, and creating the cooperation in creating the interdisciplinary.

Bangkokthonburi University shall arrange the eighth national symposium and the fourth International symposium Schedule of online conference program. under the title of “**Research study and innovation for the socially sustainable development**” which will be cooperated from the public and private section, such as, National research council of Thailand, The association of researchers of Thailand, Rajamangala university of technology Rattanakosin, Chandrakasem rajabhat University, Phanomwan college of technology, Sport authority of Thailand, Kent state University, Fo guang University, Guizhou normal University, Universitys negeri medan, Bauman Moscow state technical University, Riga technical University, Western Sydney University.

Bangkokthonburi University hopes that this conference will be the opportunity in creating the beneficial knowledge for developing the interdisciplinary in various fields, and lead to the academic development in the future.



(Associate Professor Dr. Bangon Benjatikul)

President of Bangkokthonburi University

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Determination of Antimony Compounds With Aas and Hg-Qfaas Technique for Environmental Analysis

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Abstract

The determination of antimonite (Sb(III)) and antimonate (Sb(V)) compounds using AAS and HG - QFAAS have been done. The advantage of this HG-QFAAS detection technique is that it can increase the detection limit and measurement sensitivity when compared to measurement with AAS directly. To increase the sensitivity of the measurements, a gas-liquid reactor - separator integrated into the HG-QFAAS system has been successfully prepared. In fact, this gas-liquid reactor-separator is able to increase the sensitivity of determination of Sb ions in solution. The optimum conditions of parameters measurement in the determination of Sb ion species with HG method are influenced by the type of acids, the acid concentrations, and the reductant concentrations have been determined, and can increase the measurement of analytical performance simultaneously. The result obtained showed that the antimony compounds provide a good measurement signal. The developed HG-QFAAS method can increase the detection limit and sensitivity of the measurement compared to the direct measurement with AAS which has been proven from the Sb (III) and Sb (V) calibration curves obtained and has also been proven by t test. The analytical performance obtained is quite good, indicated by the detection limit (LOD), sensitivity (S), reproducibility as indicated by % CV (Coefficient of Variance), and linearity (concentration range) measurements. Based on the analytical performance obtained shows that the developed HG-QFAAS method is very well used for the analysis of Sb ions in microscopic concentrations. The Sb concentration in environmental samples (volcanic ash) was determined by measuring directly with HG-QFAAS. The highest Sb concentration found in the A volcanic ash sample of 0.65 $\mu\text{g} / \text{kg}$, volcanic ash taken directly on the edge of the Sibolangit volcano in North Sumatra Indonesia compared with B volcanic ash of 0.24 $\mu\text{g} / \text{kg}$ taken at the periphery road a little far from the location of Mount Sibolangit.

Keywords: Antimony compounds, hydride generation, antimonite (Sb(III)), antimonate (Sb(V)), volcanic ash.

Introduction

Antimony is one element that is toxic and can be in the environment through natural events and human activities (Smichowski, P, 1995). Antimony can be found in soil, water and air in small amounts and mostly pollutes land (Thomas.T, 1830). Antimony is in the form of Sb^{3+} and Sb^{5+} in biological and geochemical environments. These forms exist in a variety of different environmental matrices (Smichowski.P, 2008). The content is not more than $1 \mu g / L$ in clean water and $500 mg / kg$ in rocks. Besides antimony is also anthropogenic pollution.

Antimony speciation is important in environmental and clinical analysis as a toxic element that has bioavailability and reactivity which depends not only on the oxidation state but also on the character of the compound in particular. Generally, inorganic antimony compounds are more toxic than organic compounds. Sb (III) compounds are 10 times more toxic than Sb (V) compounds. In addition, the toxicity of antimony compounds is approximately 10 times lower than arsenic compounds but depends on the oxidation state and its structure (Nordberg.G.F, 2007).To evaluate the toxicity and biogeochemistrySb in the required environmental speciation analysis (Zheng J.,2000).

Speciation of antimony and its compound include the determination of antimonite (Sb(III)) and antimonate (Sb(V)) and antimony organic compounds. The speciation has been done by gas chromatography, liquid chromatography and capillary electrophoresis method (B. Michalke, 1999). Determination by HG-AAS is a sensitive analytical method for the elements As, Bi, Ge, Pb, Sb, Sn, and Te. Because of low interference, more selective, and more sensitive,Hydride Generation (HG) combined with AAS detection has been selected for the determination of elements in the form of hydrides (Kumar, A.R., 2005).

Several methods of analysis speciation of antimony compounds using various detection techniques have been done before, among others, the analysis of the speciation of Sb(III) and Sb(V) compounds by HG-ICP-OES method (Cabral, et al., 2008), detection of methylantimony compounds by QFAAS (Craig, et al., 1999), analysis of antimony speciation by HG-AFS method (Long deng, et al., 2001).HG-QFAAS detection techniques for antimonite (Sb(III)) and antimonate (Sb(V)) determination has been described in this paper. Theoptimization has been studied include the type and concentrations of acid, and concentrations of reductor ($NaBH_4$) (Herlinawati, 2017).

In addition, the determination of antimony levels by the AAS method is not able to provide sensitivity and good detection limits. Based on the weaknesses of previous studies, it is necessary to do further research for the separation and determination of antimony compound species. In this case, it is restricted to Sb (III) and Sb (V) determination. The analysis will be carried out using AAS direct and HG-QFAAS detection system as a comparison to determine antimony compounds in environmental samples, namely volcanic ash from Mount Merapi.

Materials and Methods

The instruments used in this study are: Hydride Generator (HG) and Quartz Flame Atomic Absorption Spectrophotometer (QFAAS) double beam-Avanta GBC@6506 which is equipped with 100 x 5 mm quartz tube cells. In this study, weighing the material was carried out with Mettler AE 200 analytical scales. To determine the maximum wavelength of antimony species, a UV / Vis Agilent Spectrophotometer HP-8453 A (Hewlett Packard Diode Array) is used.

All chemicals and reagents used in this study are: Potassium antimonyl tartrate trihydrate (Sigma-Aldrich), Potassium hexahydroxoantimonate (V) (Sigma-Aldrich), HCl 37% p.a (Merck), HNO₃ 65% (Merck), NaOH, NaBH₄ p.a (Merck), and aquadest.

Experimental Procedure

1. Measurement of Sb(III) and Sb(V) ions with AAS

The determination of Sb(III) and Sb(V) ions is performed to obtain the optimum AAS measurement conditions by varying of solvent. The variation of solvent that affect the measurement results. In this case the types of solvent studied were aquadest and HCl.

2. Measurement of Sb(III) and Sb(V) ions with HG-QFAAS

The determination of Sb(III) and Sb(V) ions is performed to obtain the optimum HG-QFAAS measurement conditions by varying the various parameters that affect the measurement results such as the concentration and type of acid. In this case the types of acids studied were HCl, HNO₃, and H₂SO₄. Also studied the concentration of HCl and NaBH₄ (Herlinawati, 2017).

3. Analytical Performance Determination of AAS

Various measurements include linear range (calibration curve) and measurement detection limits using the optimum conditions obtained. Measurements

to find the measured linear range of measurements were made by varying the concentrations of standard ion solutions of Sb(III) and Sb(V) ions are 20, 40, 60, 80 and 100 mg/L. From this experiment we will get the equation of regression line for the measurement of Sb(III) and Sb(V) with the concentration of standard series solution. The detection limit is determined by measuring the smallest absorbance pricing that can still be determined and differentiated from the signal given by the blank with multiple measurements. The detection limit is expressed as a standard signal comparison (S) against the blank signal (N) or $S/N=3$.

4. Analytical Performance Determination of HG-QFAAS

Various measurements include measurement repeatability, linear range (calibration curve), and measurement detection limits using the optimum conditions obtained. Repeatability is determined by measuring the absorbance of standard solutions of Sb 50 and 100 mg/L repeatedly with the same measurement conditions under the optimum conditions as above. Resonance is represented by % CV (coefficient of variance). Measurements to find the measured linear range of measurements were made by varying the concentrations of standard ion solutions of Sb(III) and Sb(V) ions, where the standard injection volume parameters, concentrations and types of acids, NaBH_4 concentrations as well as acid and NaBH_4 flow rates were fixed. The concentration variations of Sb(III) are 2, 4, 6, 8, and 10 $\mu\text{g/L}$ and Sb(V) used are 50, 100, 150, 200, and 250 $\mu\text{g/L}$. From this experiment we will get the equation of regression line for the measurement of Sb(III) and Sb(V) with the concentration of standard series solution. The detection limit is determined by measuring the smallest absorbance pricing that can still be determined and differentiated from the signal given by the blank with multiple measurements. The detection limit is expressed as a standard signal comparison (S) against the blank signal (N) or $S/N = 3$ (Herlinawati, 2017).

5. Preparation of Volcanic Ash Samples

Sample preparation was carried out with two treatments, namely for the analysis of total Sb in the sample using the destruction method using microwave with HNO_3 . Destruction of volcanic ash samples with HNO_3 was done by weighing ± 0.5 g of volcanic ash samples, added 4 mL of 65% HNO_3 in a microwave container and closed. Then digest with microwave for 1 hour. The solution was filtered and the filtrate obtained was put into a 10 mL flask, added aquabidest to the mark mark. The

solution was analyzed directly with HG-QFAAS for total Sb analysis in volcanic ash samples.

Results and Discussions

1. Determination of Sb Concentrations Directly with AAS

The metal concentrations of Sb (III) and Sb (V) can be determined by directly measuring the solution of these ions with an AAS device. Sb (III) and Sb (V) solutions in water produce a calibration curve as shown in Figure 1.

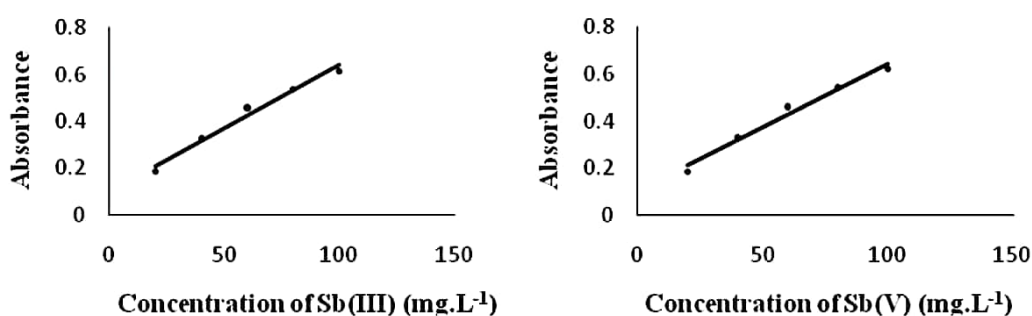


Figure 1 Sb (III) and Sb (V) calibration curves by AAS method using water solvent.

From the calibration curve in Figure 1 for the concentration ranges of Sb (III) and Sb (V) 20-100 mg. L⁻¹ obtained the regression equation: $0.0054 (C) = A - 0.1024$ and $0.0054 (C) = A - 0.1056$ ($C = \text{Sb ion concentration}$ and $A = \text{absorbance}$) with the correlation coefficient (R^2) = 0.9792 and $R^2 = 0.9792$. By using a 5 M HCl solvent obtained calibration curves Sb (III) and Sb (V) as in Figure 2.

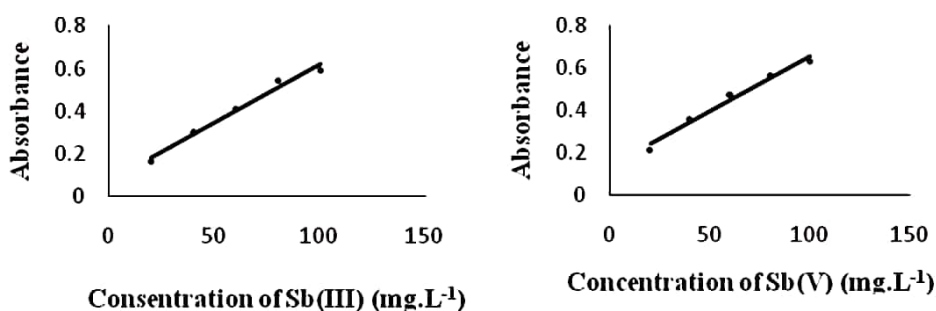


Figure 2 Sb(III) and Sb(V) calibration curves by AAS method using HCl solvent.

From the calibration curve in Figure 2 for the concentration ranges Sb (III) and Sb (V) 20-100 mg. L⁻¹ obtained a regression equation: $0.0055 (C) = A - 0.071$ and $0.0051 (C) = A - 0.1405$ ($C = \text{Sb ion concentration}$ and $A = \text{absorbance}$) with the correlation coefficient (R^2) = 0.9809 and $R^2 = 0.9784$. The linear regression equation

can be seen in Table 1. From the two calibration curves obtained almost similar curve gradient values. This shows that there is no significant difference between the sensitivity of measurement of Sb (III) and Sb (V) ions directly to AAS. Nevertheless there are differences in the intercept values of the curve as shown in Table 1.

Table 1 Linear regression equations measuring Sb (III) and Sb (V) directly with AAS using water and HCl solvents.

Sblon	Water Solvent		HCl Solvent	
	Without passing through zero point	passing through zero point	Without passing through zero point	passing through zero point
Sb(III)	$Y = 0,0054x + 0,1024$	$Y = 0,0068x$	$Y = 0,0055x + 0,071$	$Y = 0,0065x$
Sb(V)	$Y = 0,0054x + 0,1056$	$Y = 0,0068x$	$Y = 0,0051x + 0,1405$	$Y = 0,0071x$

From Table 1 it can be seen that the use of HCl in Sb (III) and Sb (V) solutions gives a fairly high background absorption value which is indicated by the large value of the intercept. This can occur if the measured absorbance value is affected by the absorption of light by multi-atomic species or by other species that are difficult to be atomized in flame. The analytical performance of this direct method can be shown by the detection limit and sensitivity values for each Sb ion (III) and Sb (V) as in Table 2.

Table 2 Detection limits (LOD) and sensitivity (S) ions Sb (III) and Sb (V) by the AAS method using water and HCl solvents.

Sblon	Water Solvent		HCl Solvent	
	LOD (mg.L ⁻¹)	S (mg.L ⁻¹)	LOD (mg.L ⁻¹)	S (mg.L ⁻¹)
Sb(III)	1,24	0,79	2,81	0,78
Sb(V)	1,24	0,79	3,04	0,84

Based on the results from Table 2 shows that the detection limit and sensitivity of the determination of Sb (III) and Sb (V) ions by the AAS method using water solvents are better than using HCl solvents.

2. Determination of Ion Concentration Sb(III) and Sb(V) with the HG-QFAAS technique

Evaluation of the method of determining Sb directly with AAS cannot be used for the purposes of Sb analysis at low concentrations. This method does not have sufficient detection and sensitivity limits to determine the Sb concentration found at the level of microscopic concentration, as commonly found in environmental compartments.

To increase detection limits and measurement sensitivity, hydride generation methods can be an option. In this method, Sb (III) and Sb (V) ions are first derivatized into hydride form and then atomized in quartz cells in AAS. Through the hydride formation stage the atomizing process becomes easier and more effective because the species to be atomized are already in the form hydride gas. In addition to this, the hydride formation stage can also simplify the matrix of the solution to be measured because only hydrides are in the gaseous form of diatomic system while the remaining liquid reaction will be wasted. Various hydride-forming reagents have been used, but NaBH_4 as a reductant is a reagent that has been chosen because it provides good hydride formation performance.

Determination of the total antimony content or its species by the hydride generation method requires various conditions that need to be optimized. The condition of hydride formation is strongly influenced by the concentration of reductant solution (NaBH_4), pH, and the type of acid used (Quevauviller, 1995). For this purpose, a study has been conducted on the effect of these parameters on the performance of hydride formation.

3. HG-QFAAS Analytical Performance

Based on the optimum conditions obtained in the determination of Sb (III) and Sb (V) by the HG-QFAAS method, then the analytical performance can be determined in the form of the determination of repeatability, linearity, detection limits, and sensitivity measurement.

3.1. Determination of the Repeatability, Linearity, and Limit of Detection Measurement

Precision is the repeatability of the results of an analysis carried out in the same laboratory at different times, in close time lags by the same analyst using the same equipment /instrument (Eurachem, 2014). Repeatability is a measure that shows the degree of concordance between individual test results, measured through

the spread of individual results from the average if the procedure is applied repeatedly to samples taken from a homogeneous mixture. Repeatability can be seen from the relative standard deviation of absorbance of a standard solution at a particular concentration which is measured repeatedly. The smaller the relative standard deviation, the better the repeatability of the measurement. Repeatability is indicated by the % CV (Coefficient of Variance).

From the research results obtained % CV for the determination of Sb (III) $6 \mu\text{g.L}^{-1}$ ($n = 7$) with the HG-AAS method was 2.33%. While the repeatability for determining Sb (V) $150 \mu\text{g.L}^{-1}$ ($n = 7$) with the HG-QFAAS method is 4.40%. The relatively small % CV value indicates that this measurement method has high precision and can be used for micro analysis (Herlinawati, 2017). The linearity measurement can be shown from the Sb (III) and Sb (V) calibration curves obtained from the curve regression equation, namely the correlation coefficient (R^2) which is close to 1. The intercept generated in the regression equation shows the accuracy of the measurement method used (Eurachem, 2014). If the linear regression equation produces an intercept with a value close to zero, it can be said that the measurement method used is quite accurate.

From the calibration curve for the concentration ranges Sb (III) and Sb (V) $2-10 \mu\text{g.L}^{-1}$ and $50-250 \mu\text{g.L}^{-1}$ the regression equation is obtained: $0.0625 (C) = A - 0.073$ and $0.0034 (C) = A - 0.0027$ ($C =$ ion concentrations of Sb and $A =$ absorbance) with correlation coefficients (R^2) = 0.9936 and $R^2 = 0.9942$. These results indicate good linearity for the analysis of Sb (III) and Sb (V) by the HG-QFAAS method.

The detection limit is determined by measuring the smallest absorbance that can still be determined and is distinguished from the signal given by the blank by several measurements. According to IUPAC the detection limit can be calculated based on the concentration or mass that can be detected at a minimum of the lowest concentration. This detection limit shows the smallest concentration of the analyte that can still be detected (generating a signal). From the results of experiments that have been conducted, the detection limit for the determination of Sb (III) by the HG-QFAAS method is $0.45 \mu\text{g.L}^{-1}$ with sensitivity (S) is $0.07 \mu\text{g.L}^{-1}$. The detection limit for the determination of Sb (V) by the HG-QAAS method is $8.42 \mu\text{g.L}^{-1}$ with sensitivity (S) is $1.26 \mu\text{g.L}^{-1}$ (Herlinawati, 2017).

Based on the t test obtained showed that the measurement of Sb (III) and Sb (V) directly with the AAS method and the HG-QFAAS method using aquadest and

HCl solvents when compared there was a significant difference (significantly effect) from the results of $t_{exp} > t_{table}$ obtained. While the measurements of Sb (III) and Sb (V) by the HG-QFAAS method using aquadest and HCl solvents with acidic conditions in hydride formation using H_2SO_4 and HNO_3 when compared there were no significant differences (no significant effect) from the results of $t_{exp} < t_{table}$ obtained. This shows that the method developed is very well used for the analysis of Sb ions in microscopic concentrations and can be used as a detector to detect the results of the separation of antimony compounds.

4. Determination of Antimony Compounds in the Environmental Sample

Volcanic ash samples were taken from the Sibolangit mountains in North Sumatra Indonesia. Samples were taken from the edge of the Sibolangit mountains from several points which were carried out randomly. The collected samples are put in plastic bags that have been prepared in advance, and are ready to be brought to the laboratory for analysis.

Samples were prepared by destruction by microwaves using HNO_3 solvents for total Sb analysis in volcanic ash samples and by extracting the samples into citric acid solutions for the analysis of species of antimony compounds in volcanic ash samples. The filtrate obtained is ready to be analyzed directly with HG-QFAAS technique.

5. Sb Analysis in Volcanic Ash Samples

Before specifying antimony compounds in volcanic ash samples, the total Sb concentration in the sample is determined. The total Sb concentration was determined by measuring directly with HG-QFAAS. The measurement results is found that the highest total Sb concentration is found in samples of volcanic ash A (0,65 $\mu\text{g}/\text{kg}$), namely volcanic ash taken directly on the edge of Mount Sibolangit, North Sumatra Indonesia compared with volcanic ash B (0,24 $\mu\text{g}/\text{kg}$) taken on the edge of the road some distance from the location of Mount Sibolangit.

Conclusion

Based on the analytical performance obtained shows that the developed HG-QFAAS method is very well used for the analysis of Sb ions in microscopic concentrations. The Sb concentration in environmental samples (volcanic ash) was determined by measuring directly with HG-QFAAS. The highest Sb concentration found in the A volcanic ash sample of 0.65 $\mu\text{g} / \text{kg}$, volcanic ash taken directly on the

edge of the Sibolangit volcano in North Sumatra Indonesia compared with B volcanic ash of 0.24 $\mu\text{g} / \text{kg}$ taken at the periphery road a little far from the location of Mount Sibolangit. However, the speciation analysis method is needed to determine antimony compounds in environmental samples so that the concentration of each antimony compound species, namely antimonite (Sb (III)) and antimonate (Sb (V)) species can be obtained.

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