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Separation of Sb (V) and Sb (III) antimony compounds using anion exchange chromatography technique

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Abstract: Separation of Sb (V) and Sb (III) antimony compounds using anion exchange chromatography technique have been done. To obtain the separation of Sb (V) and Sb (III) antimony compounds which is good in this study have been studied several parameters of separation in anion exchange chromatography technique. Parameters that influence the process of separation of Sb (V) and Sb (III) antimony compounds is the concentration and pH of the mobile phase (eluent) have been evaluated. The separation of Sb (V) and Sb (III) antimony compounds is good and optimum obtained using an eluent 200 mM phosphate buffer at pH 7 with a flow rate of 1 mL/min. Based on the optimum conditions for the separation of Sb (V) and Sb (III) antimony compounds with anion exchange chromatography method has generated value the capacity factor (k ') Sb (V) and Sb (III) obtained are respectively 1.77 and 3.01. While the value of selectivity (α), Number of theoretical plates (N) and Resolution (Rs) obtained are respectively 1.70; 369.48; and 1.48.

Keywords: Anion exchange chromatography, Capacity factor, Eluent, Resolution, Selectivity

1. Introduction

Antimony is a non-essential element found in plants, animals and humans (De Gregori et al. 2007). Antimony is also released into the environment through human activities such as ashes, mining, smelting and burning of fossil fuels. Smelting and mining activities occur at high Sb levels in locations such as South China. The toxicity of the antimony compound depends on the shape of the species. Generally, antimonite (Sb (III)) is ten times more toxic than antimonate (Sb (V)), and inorganic antimony species are more toxic than organic antimony species (Ge and Wei, 2012).

To evaluate the toxicity and understand the biogeochemical cycle of Sb in the environment, a precise analytical method is needed because of the different toxicity and migration of Sb species in the environment (Zheng et al. 2000).

Several methods of analyzing the speciation of antimony compounds using the hyphenated chromatography technique with various detection techniques have been reported, including the analysis of the speciation of Sb (III) and Sb (V) compounds using the HPLC-ICP-MS method (Zheng et al. 2000; Muller et al. 2009; Liu et al. 2010), HPLC-HG-AAS method (Satiroglu et al., 2000), the HPLC-HG-AFS method (Quiroz et al. 2011), analysis of the speciation of Sb (III) compounds and Sb (V), and TMSb (V) with the HPLC / pre-reduction / HG-AFS method (Quiroz et al. 2011), the HPLC-(UV)-HG-AFS method (De Gregori et al. 2007), the HPLC-HG-AFS method Quiroz et al. 2011).

In environmental samples apart from two inorganic antimony species, namely Sb (III) and Sb (V) the methylated form has also been detected. The presence of methyl antimony species is about 10% of the total dissolved antimony found in seawater. The antimony speciation method based on anion exchange chromatography has succeeded in separating Sb (III) and Sb (V) or Sb (V) and (CH3)3SbCl2 (De Gregori et al. 2007).

HPLC has been used for Sb speciation analysis since ten years ago. Many attempts have been made to attempt to determine inorganic Sb (III) and Sb (V) in liquid samples using anion exchange column. The organic Sb compounds including monomethyl antimony, dimethyl antimony, and trimethyl antimony are generally found in soil. However, the instability of the Sb species in the elution process needs consideration, because there is no CRM (Certified Reference Material) for the Sb species. The effect of mobile phase concentration, pH value and mobile phase organic modifier on separation efficiency has been estimated. These factors in the chromatography system are optimized to obtain better separation with shorter retention times (Ge and Wei, 2012).

The separation of Sb (V) and Sb (III) antimony compounds using the anion exchange chromatography technique carried out in this study is a preliminary analysis to find the optimum conditions for separation of Sb (V) and Sb (III) antimony compounds, namely by looking at the influence of flow rate, concentration and pH of the mobile phase used. The optimum conditions for separation obtained will then be used for the speciation analysis of Sb (V) and Sb (III) antimony compounds using the HPLC-HG-AAS hyphenated technique.

2. Methods

The tools used in this study were: HPLC Waters 2487 equipment, PRP-X 100 250 x 100 mm.id anion exchange column and glassware commonly used in the laboratory. The main ingredients used in this study were: Potassium antimonyl tartrate trihydrate (Sb (III)), potassium hexahydroxy antimonate (V) (Sb (V)), phosphate buffer, and aquabidest.

The separation of Sb (V) and Sb (III) antimony compounds was carried out by varying the various parameters that affect the measurement results such as the

concentration and pH of the phosphate buffer eluent. Variations in the concentration and pH of the eluent were carried out to optimize the separation of Sb (V) and Sb (III) antimony compounds. The concentrations of phosphate buffer eluent that were varied were 20 mM, 30 mM, 50 mM, and 100 mM.

Meanwhile, the pH of the phosphate buffer varied at pH 4. 5. 6. and 7 with a flow rate of 1 mL/minute. From the experiments that have been done, it will be obtained the optimal separation of Sb (V) and Sb (III) antimony compounds supported by the value of the capacity factor (k'), selectivity (α), and resolution (Rs) obtained.

3. Results and Discussion

Antimony compounds Sb (III) and Sb (V) can be detected using a UV detector at a wavelength of 220 nm. Optimization of the separation of Sb (III) and Sb (V) antimony compounds using anion exchange chromatography method is necessary to obtain good separation conditions. The optimum conditions for the separation of Sb (III) and Sb (V) antimony compounds in the anion exchange chromatography that need to be done are variations in the concentration and pH of the mobile phase.

3.1 Mobile Phase Concentration

Optimization of the separation of the Sb (III) and Sb (V) antimony compounds in this study was carried out by varying the concentration of the mobile phase of the phosphate buffer to obtain a good separation. The effect of the mobile phase concentration on the capacity factor (k ') of the antimony compounds Sb (III) and Sb (V) can be seen in Fig 1.

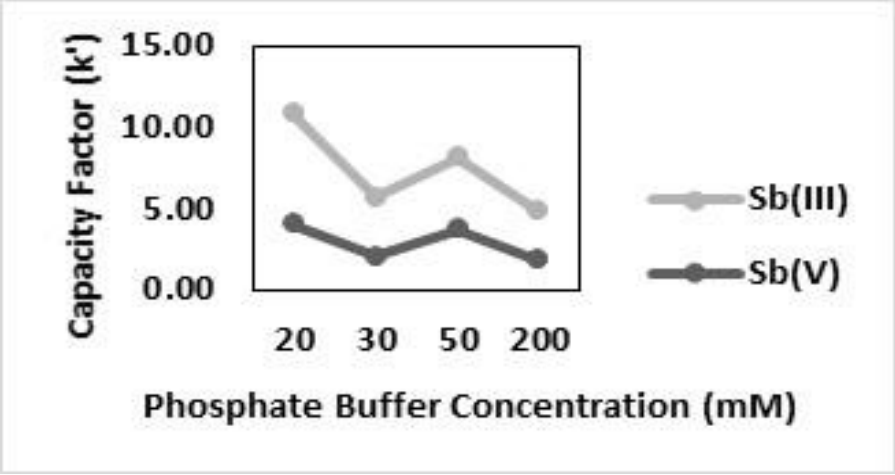

Fig 1. Effect of mobile phase concentration on the capacity factor (k ') of antimony compounds Sb (III) and Sb (V) using phosphate buffer eluent pH 7

From Fig 1. it can be estimated that at 200 mM phosphate buffer concentrations.This is supported by the calculation of the capacity factor (k ') and the value selectivity (α) as can be seen in **Table 1.**

Table 1. Data on retention time measurement, capacity factor and selectivity calculation of antimony compounds Sb(V) and Sb (III) using phosphate buffer eluent at a flow rate of 1 mL / minute

Conc. Buffer Fosfat (mM)	Retention time (t_R) (min)		Capacity factor (k')		Selectivity (α)
	Sb (V)	Sb (III)	Sb (V)	Sb (III)	
20	16.03	24.85	4.01	6.77	1.69
30	15.00	23.00	2.03	3.64	1.79
50	15.26	17.77	3.67	4.45	1.21
200	15.21	22.03	1.77	3.01	1.70

3.2 Effect of pH

The effect of pH on the capacity factor (k ') o thef antimony compounds Sb (III) and Sb (V) can be seen in Fig 2.

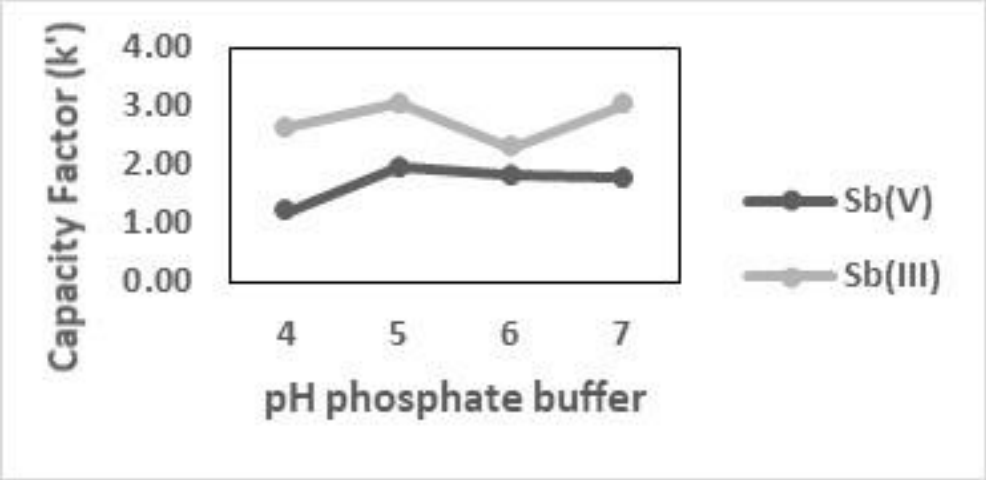

Fig 2. Effect of pH eluent on the capacity factor (k ') of antimony compounds Sb (III) and Sb (V) using 200 mM phosphate buffer eluent pH 7.

From figure 1 and figure 2. it can be estimated that at pH 7 the antimony compounds Sb (V) and Sb (III) can separate well as can be seen in the chromatogram in Fig 3. This is supported by the calculation of the capacity factor value. (k ') and the selectivity value (α) as can be seen in Table 2**.**

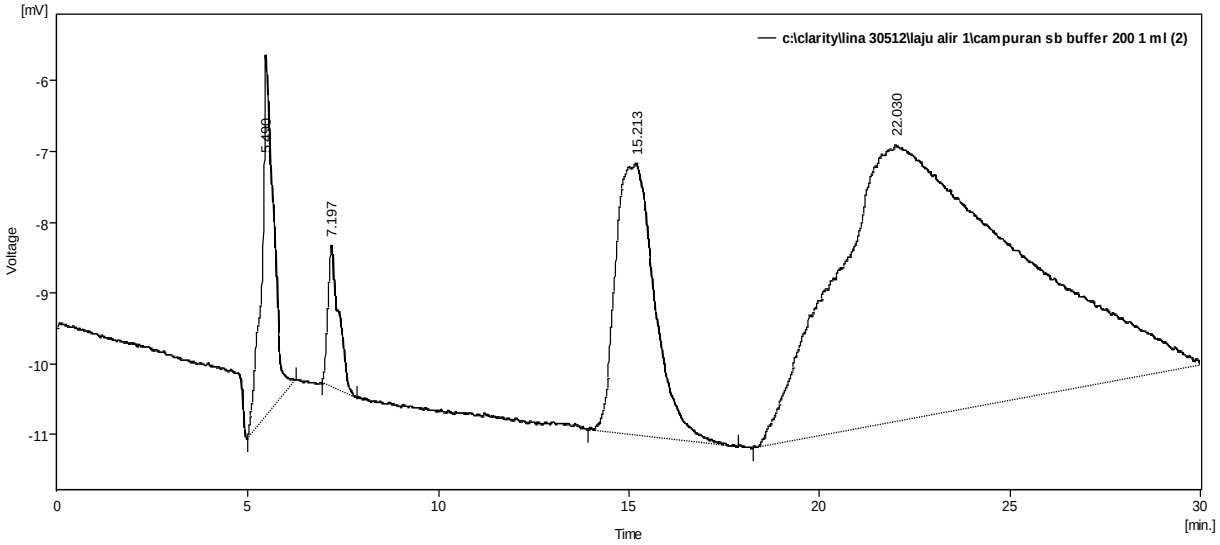

Fig 3. Chromatogram profile of the separation of antimony compounds **(1)** Sb (V) 15.213 min and **(2)** Sb (III) 22.030 min using phosphate buffer eluent 200 mM pH 7.

From the results obtained, the optimal separation conditions for the antimony compounds Sb (V) and Sb (III) is to use a mobile phase of 200 mM phosphate buffer at pH 7 with a flow rate of 1 mL / minute. This is supported by the chromatogram and the calculation of the capacity factor (k '), selectivity (α), and resolution (Rs) obtained in this study. The result is that the optimum value of the capacity factor (k ') for the Sb (V) and Sb (III) compounds are 1.77 and 3.01, respectively. While the selectivity value (α), the number of theoretical plates (N) and the resolution (Rs) obtained were 1.70; 369.48; and 1.48.

Table 2. Retention time measurement data, calculation of capacity factor and selectivity of antimony compounds Sb (V) and Sb (III) at 200 mM phosphate buffer eluent pH variation, flow rate 1 mL / minute

Buffer Fosfat pH	Retention time (t_R) (min)		Capacity factor (k')		Selectivity (α)
	Sb (V)	Sb (III)	Sb (V)	Sb (III)	
4	12.42	20.44	1.21	2.63	2.17
5	15.67	21.56	1.94	3.05	1.57
6	15.62	18.15	1.83	2.28	1.24
7	15.21	22.03	1.77	3.01	1.70

4 Conclusion

The results showed that the optimum separation of Sb (V) and Sb (III) antimony compounds were obtained using a mobile phase of 200 mM phosphate buffer at pH 7 with a flow of 1 mL / minute. While the retention time (tR) obtained on the separation of Sb (V) and Sb (III) antimony compounds were 15.21 minutes and 22.03

minutes, respectively. Based on the optimum conditions obtained in the separation of Sb (V) and Sb (III) antimony compounds, the separation performance is obtained which is indicated by the value of the chromatographic basic quantities obtained, namely: the capacity factor (k ') for the Sb (V) and Sb (III) antimony compounds obtained respectively 1.77 and 3.01 with the selectivity value (α) is 1.70. While the number of theoretical plates (N) and resolution (Rs) obtained were 369.48 and 1.48, respectively.

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