

Determination of Zirconium (IV) Based on Flow Injection Analysis with The Xylenol Orange as A Complexing

Wa Ode Rustiah¹, Sulistyani²

¹Medical Laboratory Technology, Politeknik Kesehatan Muhammadiyah, Makassar, South Sulawesi

²Department of Chemistry Education, Faculty of Mathematics and Natural Sciences, Yogyakarta State University, Yogyakarta

*Corresponding Author: waoderustiah79@gmail.com

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Abstract: The development of spectrophotometry method based on Flow Injection Analysis (FIA) to determine Zr (IV) in tin ore accompanying mineral has been done using xylenol orange as complexing agent. The complex between Zr (IV) and xylenol orange 0,05% was formed at pH 2 (1:1). The complex formed having maximum absorbance at wavelength 554 nm. The mineral decomposition has been done by fusion method using Na_2CO_3 and Na_2O_2 as fluxes for open system and the close system decomposition using HNO_3 , HClO_4 and HF as solvents. FIA-spectrophotometry that has been developed was able to show good analytical performance, with precision of %KV not more than 5% at concentration level mg/L, was 3,98%, with sensitivity was 2,79 ppm and the detection limits of calculated ($S/N = 3$) was 0,21 ppm. This method has been applied for analyzing Zr (IV) in zircon mineral sample with analysis result 20,26% of ZrO_2 and recovery percentage was 100,7%.

Key word: Zr(IV), xylenol orange, decomposition, spectrophotometry UV-Vis, FIA

INTRODUCTION

Tin ore mining is one of the major export commodities for Indonesia, which is derived from the mining sector. Tin ore mining is still being carried out, recently, to be encouraged to utilization the other elements that are associated with minerals of tin ore due to high efficiency (Gasik, 2013; Renganathan, Tanneru, & Madurai, 2018). Generally, the processing of these elements is relatively difficult, either because the measure is little or the impurities are difficult to remove, thus, to obtain a fair condition and the highest standard is required in certain ways (Journal, 2006; Parissi-Poulou & Panderi, 1999).

From the several associated minerals of tin ore, one of which is zircon. Zircon is formed as associated minerals in rocks containing Na-feldspar. Mineral zircon is a mineral that mostly contains elements of zirconium. This element is a lot of uses including electronic raw materials, ceramics, as well as masking materials atomic reactors (Rustiah, Noor, Maming, Lukman, & Nurfadilah, 2020). The techniques for the separation and purification of zirconium has been done, one of which is solvent extraction. Solvent extraction with the butyl acid phosphate method was first used to separate zirconium from the element fission products are then determined by γ ray liveliness of zirconium, which isolated from the aqueous phase as barium fluozirkonat. This extraction method can be used for separating hafnium from zirconium, despite the results are unsatisfactory because the levels of hafnium are much smaller than zirconium in zircon minerals (Armagan, Soylak, Elci, & Dogan, 2002; Gasik, 2013; Saracoglu, Soylak, & Elci, 2002).

The techniques of analysis to determine the zirconium, such as ICP-IDMS is costly and not available in all laboratories (Chiba, Soukura, & Tatsuta, 2011; Tang et al., 2014). Therefore, the development of sensitive and economical technology is needed, such as the

procedures in visible spectrophotometry. This research explored the decomposition method to determine the metal level of zirconium in zircon mineral sample from Bangka island. The zirconium metal contained in the mineral zircon sample in the form of a specific structure and there are some fairly large, accordingly, use a dry decomposition method is still considered as a standard method (Chadajah, Rustiah, & Munir, 2018). Another method to analyze is the decomposition of the wet method, which includes the decomposition of open and closed ways using strong acids and mixtures there of (Analysis & Peat, 1988; De Oliveira, 2003). Xylenol orange is complexing with optimal results in the determination level of zirconium, as well as providing high sensitivity and selectivity in visible spectrophotometry (Abdeldaim & Mansour, 2018; Pikkemaat et al., 2007). Selection techniques of flow injection analysis (FIA) due to simple analytical techniques and transform the ordinary into the automated analysis (McKelvie, 2008), based on the manipulation of the chemical and physical characteristics of the analyte zone dispersed distribution of the sample which injected into the flow of gas/liquid carrier and the flow detection (Ali et al., 2018; Chiba et al., 2011; Li et al., 2016).

RESEARCH METHODS

Decomposition Procedure

Decomposition technique of zircon mineral using the dry method

Decomposition with the sodium carbonate (Na_2CO_3)

A total of 1.00 grams of mineral zircon sample and 7.00 grams of Na_2CO_3 was weighed carefully, then closed. The cup and its contents were heated in an electric furnace at $1000^\circ\text{C} \pm 1$ hour until the mixture is well fused. The heated mineral zircon sample was cooled and diluted with the hydrochloric acid (HCl) 1:1. The filtrate was filtered, then diluted with distilled water to a volume of 100 mL solution (sample M-1A).

Decomposition with the sodium peroxide (Na_2O_2)

Equal treatment with the Na_2CO_3 , but the fuser replaced using Na_2O_2 and heated at 800°C (sample M-1B).

Decomposition technique of zircon mineral using the wet method

Decomposition of the open way

Mineral zircon of sample weighed as much as 0.8 grams and then entered in a Teflon vessel, subsequently, added in succession: 3 mL of HNO_3 , 3 mL of HClO_4 and 10 mL of HF. Furthermore, the sample solution is heated using hotplate for 1-1.5 hours. After cooling for 20 minutes, added 2 mL of HF and heated again ± 4 hours. After cooling, added 2 mL of HClO_4 and heated again until slightly dry. HCl has then added 5 mL of 1:1 ratio until dissolve completely. The solution was transferred into a flask of 50 mL and diluted with distilled water to mark boundaries (sample M-2A).

Decomposition of the close way

A total of 0.08 grams of the smoothed mineral zircon sample is weighed precisely, then entered in a Teflon bomb. Added in succession: 1 mL of HNO_3 , 1.5 mL of HClO_4 and 5 mL of HF. The Teflon bomb was closed and heated at 130°C for ± 2 hours. After cooling, the solution was transferred into a flask of 50 mL and diluted with distilled water to mark boundaries (sample M-2B).

Analysis of Zr (IV) with the FIA-spectrophotometry

Determining the optimum conditions of FIA

The construction of FIA-spectrophotometry conducted volume-based (Safavi, Haghighi, & Peiravian, 2003; Yapar, Dal, Tuncel, & Uysal, 2004), as shown in Figure 1.

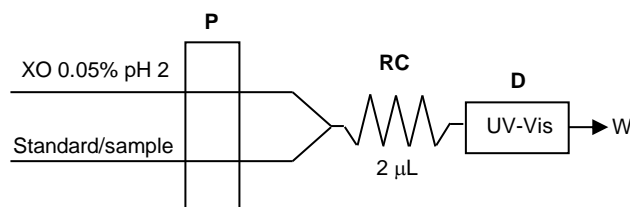


Figure 1. Construction of FIA-spectrophotometry (P: the peristaltic pump; RC: reactor coil; D: detector)

Optimization of flow rate

Two pieces of 25 mL beaker glasses are filled with the Zr (IV) 10 ppm and 0.05% solution of xylenol orange pH 2.0, respectively. Subsequently, it determined the absorbance with the FIA-spectrophotometry systems on the variation of the flow rate of 0-10 mL/min at λ 554 nm (Noroozifar, Khorasani-Motlagh, & Akbari, 2007).

A calibration curve with FIA-spectrophotometry system

The variation concentration of Zr(IV) solution in the ppm level are 5-40 ppm into a beaker, then diluted with distilled water to a volume of 50 mL. Subsequently, as many as 2 mL pipette inserted into the flask 10 mL and diluted with distilled water to mark boundaries, then transferred into 2 pieces of 50 ml beaker glasses and adjusted to pH 2.0, then the standard solution incorporated into the FIA system (channel 1). Another beaker glass filled with a solution of 0.05% xylenol orange at pH 2 (channel 2), which is equipped with a coil reactor at a flow rate obtained in previous measurements for determining the flow rate. The solution that comes out of the circuit FIA kinetically measured absorbance with a UV-Vis spectrophotometer at λ 554 nm (Trenggamayunelgi, Sulistyarti, & Retnowati, 2019).

Analysis of Samples

Analysis of samples decomposition using the dry method

Two pieces of 10 mL flask (L1 and L2) that already contains 2 ml samples of M-1A and M-1B sequentially diluted with distilled water to mark boundaries and transferred into 2 pieces of 50 mL beaker glass (G1 and G2) and adjusted to pH 2. Another beaker glass (G3) filled with a solution of 0.05% xylenol orange at pH 2. Subsequently, we analyzed with the FIA-spectrophotometry method and a flow rate of 4 mL/min at λ 554 nm.

Analysis of sample decomposition using the wet method

Equal treatment with the decomposition procedure of the dry method, but the sample was changed to M-2A and M-2B.

Determination of analytical performance

Precision

A solution of Zr(IV) with a concentration of 10 ppm was measured repeatedly with the same condition, subsequently, measured absorbance as a high signal. Precision is indicated by % KV.

Level of Sensitivity

Sensitivity analysis is determined from a calibration curve profile created using FIA-spectrophotometry.

Limit of detection

The detection limits determined by measuring the smallest of value absorbance can be distinguished from the signals given by the blank with some measurements. The detection limits expressed as a standard comparison signal (S) to the blank signal (N) or $S/N=3$.

Recovery (% Recovery)

Sample decomposition with Teflon bomb system, filtered into a 50 mL flask, then diluted with distilled water to mark boundaries. A total of 2 mL samples were diluted pipetted into a 10 mL flask, which already contains 5 mL of Zr(IV) standard 5 ppm. On the other hand, it made the solution of 0.05% xylenol orange at 0.1 M sulfuric acid. The two solutions were analyzed by the FIA-spectrophotometry method at λ 554 nm.

RESULTS AND DISCUSSION**Decomposition of Zirconium Mineral*****Decomposition of dry methods***

Decomposition by smelting way in an electric oven, show that footage zircon which forms brown solids melts at 1000 °C by the fuser Na_2CO_3 and 800 °C by the fuser Na_2O_2 . Observations on footage zircon, showing the decomposition by the two fuser, that is Na_2CO_3 and Na_2O_2 produce a white solution (Ramkumar & Chandramouleeswaran, 2017; Silva, Tóth, & Rangel, 2006). From the literature data, it is known that Na_2CO_3 has a melting point at 851 °C. Thus, the expected heating at 1000 °C for one hour is mineral sample would be destroyed. Meanwhile, the Na_2O_2 has a melting point at 480 °C, but in this study used the heating temperature up to 800 °C by raising the temperature slowly to ensure the melting process can take place properly (Analysis & Peat, 1988; Yang et al., 2013).

Decomposition of the wet method

Decomposition of the wet method carried out by two different systems, namely the decomposition of an open way, hereinafter called M-2A procedure, and decomposition using the Teflon bomb called M-2B procedures. Both procedures are based on the principle of dissolving the mineral sample using a mixture of a strong acid. From those results was found that the decomposition by the open or closed way still leaves a little bit of insoluble mineral with HCl (De Oliveira, 2003; Yang et al., 2013). The result has been reinforcing the notion that the concentrate of zircon still contains other minerals, such as cryolite, alvite and eucaliote, which are difficult to dissolve in strong acids. According to Potts (Potts, P.J., *A Handbook of Silicate Rocks Analysis, Blackie Academic & Profesional, 1985, 47-75., n.d.*), minerals such as zircon (ZrSiO_4), cassiterite (SnO_2) and tourmaline [$\text{Na}(\text{Mg,Fe,Mn,Li,Al})_3\text{Al}_6\text{SiO}_{18}(\text{BO}_3)(\text{OH,H})_4$] resistant to acid, moreover existence of tetrahedral and monoclinic from both mineral in such a way, affect to the acid molecule that harder to enter or move between crystal lattices, thus, it prefers to decompose.

The problem that arises is almost equal to the dry method, which is the mineral sample that not dissolve perfectly-remaining despite added with the hydrochloric acid, sulfuric acid, and hydrofluoric acid. The selection of the fuser must be careful not to

dissolve the cup material that used because it could be affected by the negative error, such as the analysis results obtained will be lower than it is.

Analysis of Zr(IV) with the FIA-spectrophotometry

The optimum condition of FIA-spectrophotometry

Flow rate

Analysis of FIA-spectrophotometry volume-based influenced by the system flow rate that constitutes the function of the volume of reagent in the analysis (Noroozifar et al., 2007; Trenggamayunelgi et al., 2019). The selection of the optimum flow rate is made to look for a complex equilibrium state formed on a flow injection system because the zone area of that is an instantaneous equilibrium measured by the visible light of the detector (Noroozifar et al., 2007; Yapar et al., 2004).

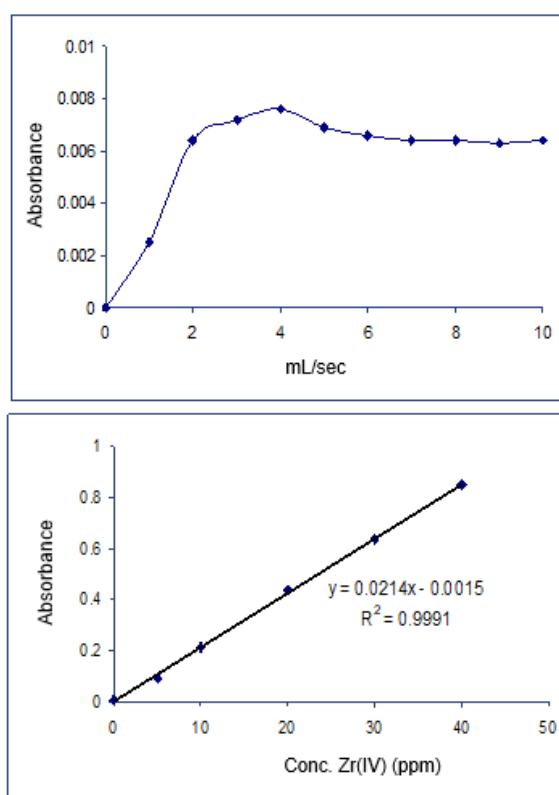


Figure 2. A: Evaluation of flow rate; B: A calibration curve by direct spectrophotometry system

Figure 2A indicates the maximum flow rate, which is used to achieve the optimum absorbance is at a flow rate of 4 mL/min. The flow rate of 4 mL/min has been selected for the absence of significant optimum differences in absorbance at a flow rate which over 4 mL/min. Moreover, the selection of flow rate, which over 4 mL/min, will affect to backpressure.

A calibration curve by FIA-spectrophotometry system

Construction of FIA-spectrophotometry with a flow rate of 4 mL/min calibrated with the various concentrations, as shown in Figure 3A. The linearity of a measurement can be obtained from the calibration curve. The calibration curve obtained from the signal profile of the standard solution with a concentration of 0-40 ppm. From the measurement results

using the FIA-spectrophotometry system, the calibration curve obtained by the equation: $y = 0.0013x + 0.002$, and the correlation coefficient was 0.9948. The results of the calibration curve with the FIA-spectrophotometry system is used to determine the levels of Zr(IV) in the sample.

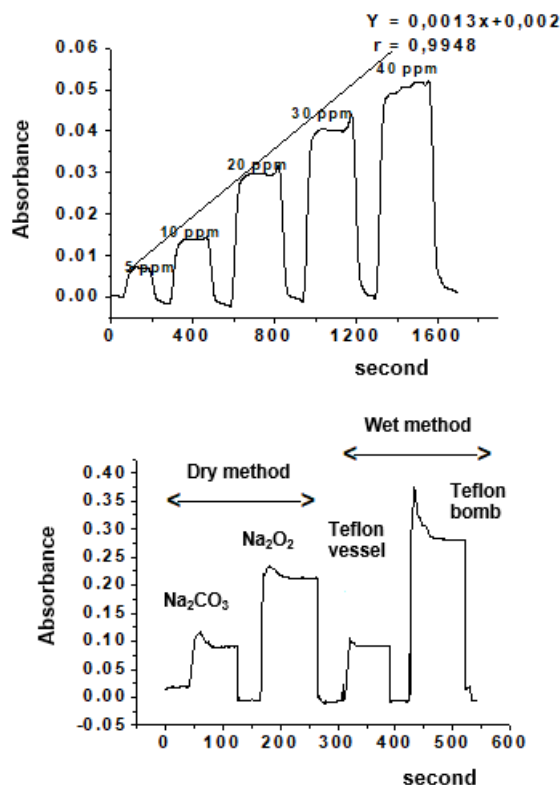


Figure 3. A: Profile and calibration curve by FIA-spectrophotometry system, B: Signal profiles with the various methods of decomposition

Analysis of sample with the FIA-spectrophotometry

Mineral samples zirconium that has decomposed, such as M-1A, M-1B, M-2A, and M-2B, further analyzed to determine the levels of Zr(IV). Signal profile analysis of FIA-spectrophotometry of four treatments can be seen in Figure 3b, shows that the dry method using Na₂O₂ is better than Na₂CO₃ on the total weight of the same sample. Whereas, the signal decomposition using the Teflon bomb is better than the signal with the fuser Na₂O₂ because the decomposition of Na₂O₂ obtained aliquot of 1 g in 100 ml, while the decomposition of Teflon bomb gained aliquots of 0.16 g in 100 mL.

The results of decomposition with the FIA-spectrophotometry methods for M-1A procedures is obtained 0.99% of ZrO₂ in the sample. Whereas, the results of the M-1B procedure amounts to 2.31% of ZrO₂. M-2A Sample is obtained 0.61% of ZrO₂ in the sample. While the results of M-2B procedures amounted to 20.26% of ZrO₂. The percentage of ZrO₂ were obtained from the analysis of the FIA-spectrophotometry is also compared with the direct spectrophotometric method using complexing xylenol orange (Yu, Wang, Zhou, Zhao, & Qiu, 2013). The calibration curve was done by varying the concentration of the standard solution of 0-40 ppm and obtained the equation, Absorbance = $0.0214 [Zr(IV)] - 0.0015$ with the correlation coefficient of 0.9991 that shown in Figure 2b. The results of decomposition with the direct spectrophotometric method for the M-1A

procedures obtained 0.125% of ZrO_2 in the sample. Whereas, the results of M-1B procedures amounted to 0.34% of ZrO_2 . M-2A sample is obtained 0.071% of ZrO_2 in the sample. While the results of the M-2B procedure amount 2.53% of ZrO_2 .

From the results, the analysis of FIA-spectrophotometry with the decomposition of the Teflon bomb method is better. Determination of the FIA-spectrophotometry method always shown the higher levels of ZrO_2 compared to the determination of the direct spectrophotometric method indicates that the necessity of more in-depth learning to the decomposition process and determination method.

Determination of analytical performance by using the FIA-spectrophotometry method

Precision

The precision indicates the ability of the analytical method provides the same data in the different analyses of time and place. Commonly, we used the variation coefficient of (% CV) for the stated precision (Ramkumar & Chandramouleeswaran, 2017; Safavi et al., 2003). The best data or the high precision for giving the 95% confidence level, the meaning shows a small mistake (% CV no more than 5%). The measurement results of this method at a concentration of ppm, providing precision with a variance coefficient of 3.98%. In this case, it indicates the excellent accuracy of the FIA-spectrophotometry method (Figure 5).

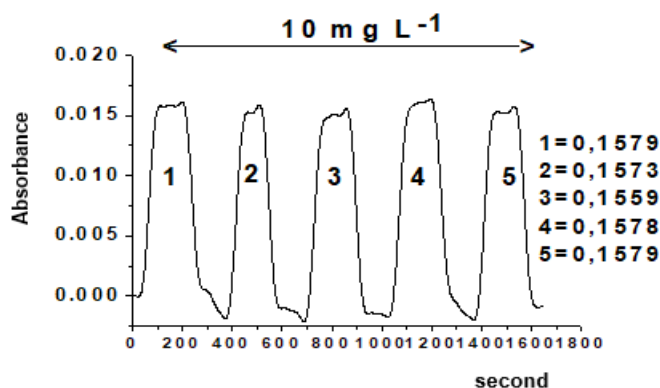


Figure 5. The curve of the absorbance precision relationship by FIA-spectrophotometry method at a concentration of 10 ppm

Sensitivity

The sensitivity of an analytical method showed the ability of an analytical method that is used to distinguish the concentration of the analyte. Two factors which may indicate the sensitivity of the technique is the slope of the calibration curve and precision (Rustiah et al., 2019; Silva et al., 2006). In case, the principles of light absorption used according to the Lambert-Beer verdict, the sensitivity can be defined as the smallest concentration changes can give the higher absorbance alteration, amounted to 0.0044 of absorbance units. The sensitivity value of the FIA-spectrophotometry method was 2.79 ppm.

Limit detection

Limit detection that measured indicates the concentrations determined from signals that can be differentiated signals given by the blank ($S/N = 3$) with several measurements. The limit detections obtained 0.21 mg L^{-1} .

Percent Reacquisition (% Recovery)

The percentage recovery obtained is equal to 100.71%, as shown in Table 1. The amount percentage indicates that the % recovery of the measurement method is still relatively good, and the matrix does not interfere with the measurement of this study.

Table 1. Percent Reacquisition (% Recovery)

	Absorbance	ZrO ₂ ** (Mg)	Recovery (%)
Samples	0.03319	8.101	
Sample + standards*	0.03354	8.193	100.71

Information :

* mg standard ZrO₂ = 0,034 mg

** Absorbance = 0.0013 [Zr(IV)] + 0.002

CONCLUSION

The results of this study show that Zr(IV) can be formed complexes with the xylenol orange at pH 2. Xylenol orange as complexing can be developed for the measurement of Zr(IV) by the FIA-spectrophotometry method at λ 554 nm.

Analytical performance by FIA-Spectrophotometry shows an excellent method to develop. The percentage recovery of 100.71% shown the matrix does not interfere with the measurement. The technique that emerged has been applied to a mineral containing ZrO₂ and gives the results 20.26% for the decomposition using the Teflon bomb system.

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