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Design And Implementation Of Spectrophotometry For Iodine Determination Based On Flow Injection Analysis

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Abstract : *Iodine is an element nonmetal and essential micronutrient needed by human body in a trace amount, iodine deficiency may cause brain damage, mental retardation, cretinism and endemic goiter (GAKI). Analysis method of detecting iodine has widely been used, where iodometry and spectrophotometry analysis is a standard method to determine iodine content. The analytical performance of iodine determination with a sensitive and selective flow injection analysis had been developed and evaluated. Iodate reacted with the excessive iodide in an acid medium to form tri-iodide, which can be detected with spectrophotometer at 352 nm. The result of analytical performance evaluation of that developed method indicated a linearity of calibration curve at the range of 0.1-1.0 mg/L, with the R^2 value approached one and detection limit is 0.01 mg/L. Through the flow injection analysis method, the precision of iodine determination was evaluated, which was revealed as 0.08% variant coefficient for the concentration of 0.5 mg/L. This method have been successful developed for the iodine determination in iodized sample.*

Key words*: iodate, iodine, flow injection analysis, spectrophotometry, triiodate.*

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I. Introduction

Iodine is essential micronutrients for body in fine number, lack of iodine result in brain damage, mental retardation, cretinism and endemic goiter (Xie and Zhao, 2004). Beside iodine as supplements, iodine also added into medicines as multivitamins and antiseptic. Iodine can be found in the compounds, e.g. potassium iodate (KIO_3). KIO_3 is a stable compound so that in the storage process the compound is not easily broken, but if in the form of potassium iodate (KI) it will be easily broken or unstable especially for a long storage time. The stability of iodine in KIO_3 is recommended for prevention iodine deficiency in some tropical countries (Choengchan et al, 2002).

Iodometry is the standard method to known the content of iodine species, where this method based on the occurrence of color change after titrated with sodium tiosulfate. The use of spectrophotometry for iodate determination at 350 nm has also been widely utilized and developed (Jakmunee and Grudpan, 2001). Beside iodometry and spectrophotometry, several analysis methods have also been used for the determination of iodate, like: electrochemical, gas chromatography-mass spectrophotometry, ion chromatography, and x-ray fluorescence. Flow Injection Analysis (FIA) method has been developed for the determination of iodate acid in the atmosphere using red pirogalol. This method is successfully applied to water sample (Ensafi and Chamjangali, 2002). Choengchan et al, has developed spectrophotometry based flow injection method based on blue complex of triiodide-starch ion (Xie and Zhao, 2004).

Flow Injection Analysis-FIA based on the iodate reduced by iodide in acid condition, iodine as a produced reacts with an excess of iodide ion to form triiodide. This method can be used for analysis of waters with high salt levels, where this method is very selective and without the influence of the matrix. Flow injection methods are easy to use, a few required sample volumes, short time analysis with high sampling frequency. In the other side, this method can be modified with different concentration ranges or by changing the sample injection volume. This research does to design a flow injection method which can be used for determination iodine species quickly, selectively, and sensitive in salt iodine sample.

II. Research Methods

Equipments and chemicals

FIA equipment: peristaltic pump, injector valve, chrome recorder power, coil reaction, and detector (HP 1050 series). UV-Visible spectrophotometer, magnetic stirrer and glassware. Chemicals used: potassium iodate, phosphoric acid, potassium iodide, sodium chloride, aquades, and sample of salt.

Procedures

Make standard solutions of KI, KIO_3 and H_3PO_4. Then create potassium iodate absorption spectrum, and optimize the concentration of H_3PO_4, KI, and KIO_3. The influence of NaCl concentration to absorbance make by varying NaCl 10-50% (w/v) with added H_3PO_4 0.05 M and KI 0.014 M, then make 100 mL of 5.0 KIO_3 ppm.

Optimization of FIA-UV

Flow injection analysis system used consists of two streams. The first current as carrier, which flow KI 0.07 M with H_3PO_4 0.25 M on 3.1 mL/sec.

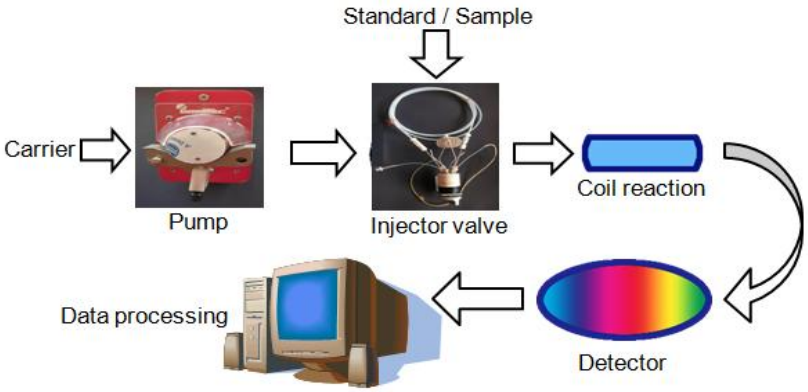

Figure 1. The series of flow injection analysis

The carrier flow until stable and recorded. Then, 100 micro liter standard solution and sample injected into the carrier, it react in reaction coil, where the tri-iodide ion is formed. The result solution were transferred to the flow cell, and measured at 352 nm.

Continuation study on the FIA-UV

Influence of reaction coil and optimization of standards concentration to high peak (AU) uses KIO_3 standard solution injected into the injection valve that flowed carrier through the pump. By using coil reaction and without coil reaction, studied the high-profile peak. Then, made calibration curve and calculate the concentration (ppm). The influence of NaCl concentration to high peak is done by injected NaCl 1% and 10% (w/v), then studied the resulting signal profile.

Variation KIO_3 concentration with the addition of NaCl is done by varying KIO_3 1.0-10.0 ppm and then added 1% and 10% NaCl into each solution. The solution injected into the valve that flowed carrier, next studied high peak and made calibration curve.

Along with the standard solutions injection, the salt sample injected (made 1%-20%) that flowed carrier (carrier containing NaCl 1%). Made standard curve on the addition of % NaCl and calculated concentration of iodine species (ppm).

III. Results and Discussion

Determination of iodate in salt sample can be made by using flow injection analysis method (Flow Injection Analysis). The principle used is iodate (IO_3^-) reacts with iodide (I^-) in an acid condition (H^+), iodine produced reacts with an excess of iodide ion to form triiodide (I_3^-). Optimizations of potassium iodate concentration done on phosphoric acid concentration and optimize potassium iodide by varying KIO_3 0.1-1.0 ppm.

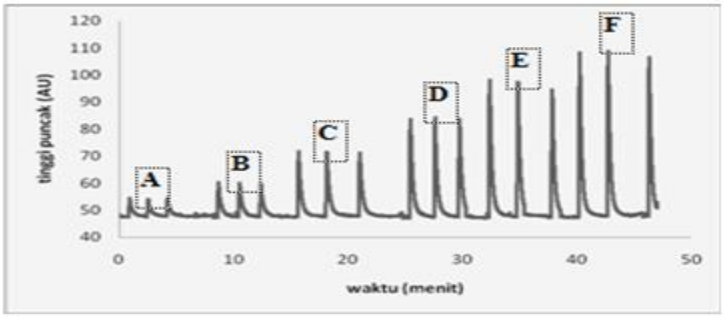

Figure 2. Signal profile of KIO_3 0.1-1.0 ppm standard solution

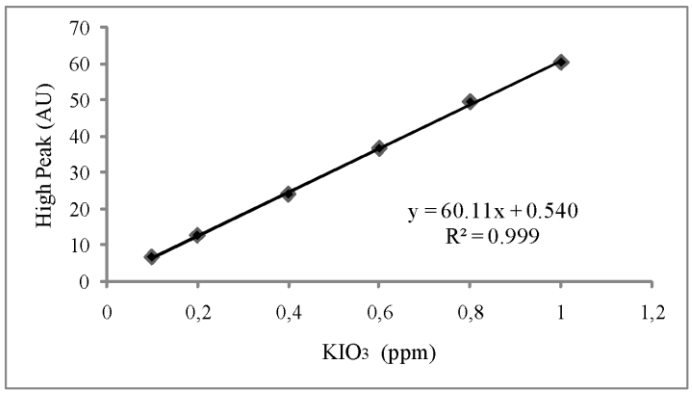

Figure 3. Calibration curve of standard KIO_3

Standard calibration curves obtained at optimum condition using flow injection analysis method. The results showed a variation of the concentration of iodate in aqueous solution with high peak (AU) is linear, so that this condition can be applied for the determination of iodate in sample.

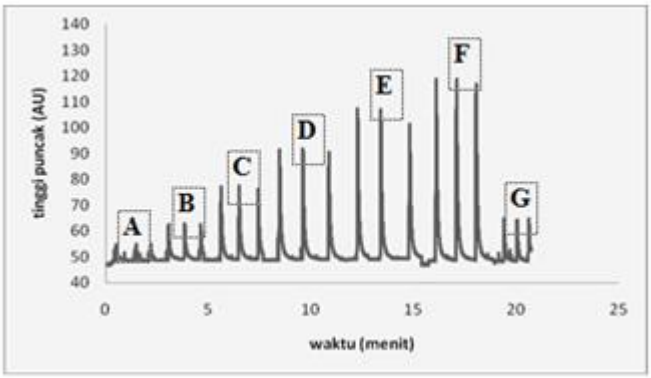

Figure 4. Signal profile of KIO_3 standard solution and salt sample 1%

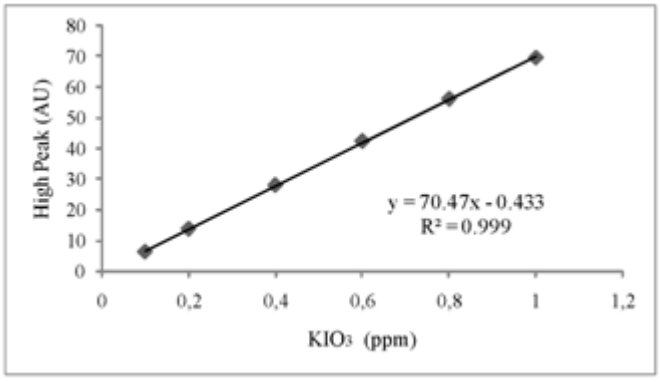

Figure 5. KIO_3 standard curve

From the equation y = 70.47x - 0.433, so the iodate concentration in salt sample can be calculated. On salt 1% concentration with AU = 16.05 obtained the iodate concentration is 24.0 ppm.

The Influence of Sodium Chloride

Sodium chloride optimization in spectrophotometry was done on optimum concentration of iodide and acid. Optimization of NaCl addition influence was done on KIO_3 5.0 ppm.

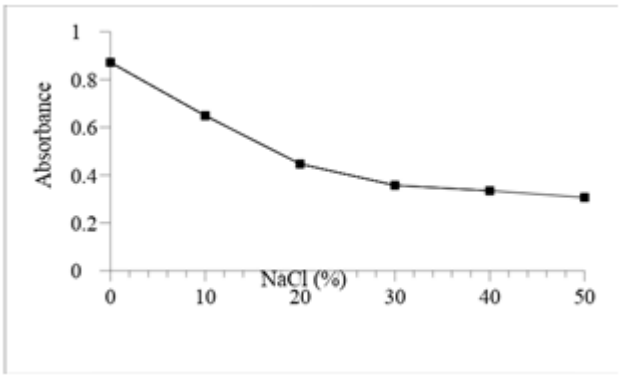

Figure 6. The influence of NaCl effect

The addition of NaCl was influence the absorbance, when added more concentration of NaCl, the absorbance will increasingly decline. This is because the present of iodine bound with chloride ion to form ICl or I_2Cl^-, so I_2 that reacts with I^- will be decreased. Optimization with FIA-UV method was also performed, by injecting NaCl 1% and NaCl 10% into the carrier solution (carrier is contains NaCl 1%).

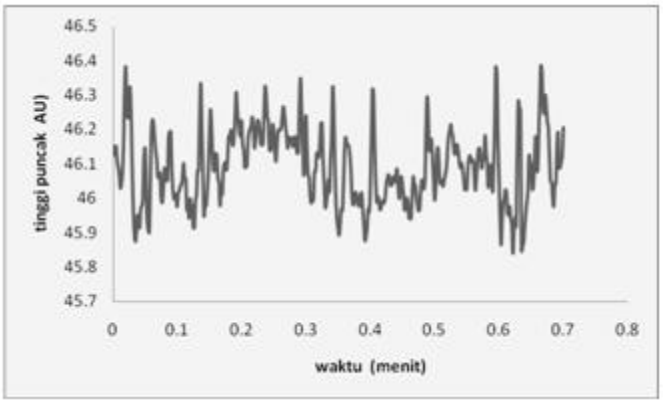

Figure 7. The influence of NaCl 1% addition

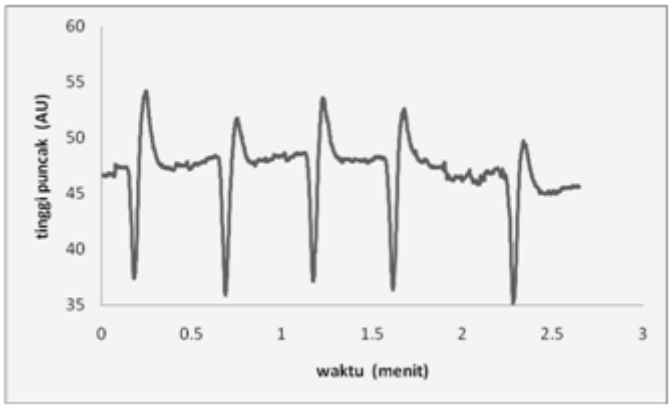

Figure 8. The influence of NaCl 10% addition

In the addition of 1% NaCl did not significantly affect of the reaction. The addition of 10% NaCl, found a high peaks that can disturb the measurement and data processing. The peak profile is caused of the schileren effect caused of the difference refractive index between the standard or sample with carrier contains NaCl. The addition of the same NaCl concentration in standard and carrier is a technique for reducing the effects of schileren.

The Influence of Sodium Chloride Addition

Variations of % NaCl addition do to see the influence of sodium chloride to high peak (AU) and the concentration iodate in salt sample.

Standard solution 0.1-1.0 ppm and carrier designed contain NaCl 1%. The results of the measurement, the addition of NaCl 1% influence the high peak of iodate standard and salt sample 1%. The high peak decrease twice, if compared with sample without the addition of sodium chloride. The decrease is due to the presence of iodine bound by chloride ion to form ICl or I_2Cl^-, so that I_2 reacts with I^- will be decreased.

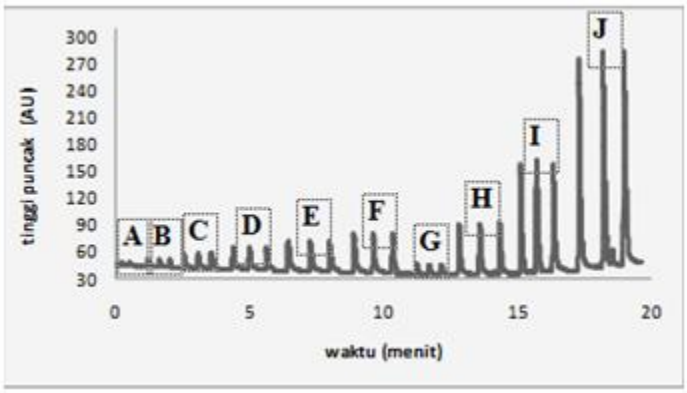

Figure 9. Signal profile of standard solution 0.1-1.0 ppm

G = salt1% ; H = 5% ; I = 10% ; J = 20%
The result of the optimization then created the calibration curve of standard KIO_3 between the high peak (AU) to KIO_3 concentration.

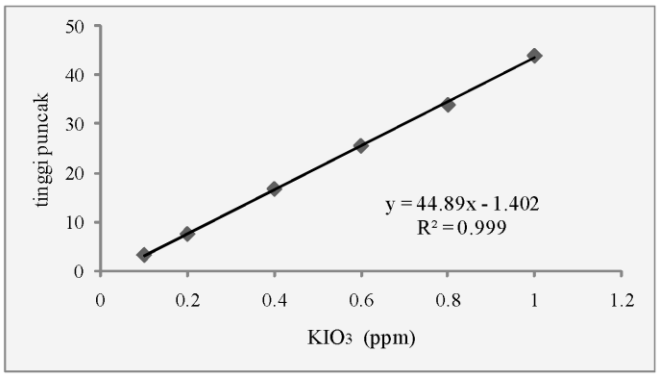

Figure 10. Calibration curve of KIO_3 standard

Based on the equation y = 44.89x - 1.402 so the concentration iodate in each salt sample can be calculated. The concentration of iodate in each salt concentration can be seen in table below

Tabel 1. Iodate (iodine species) concentration in salt

No.	Salt concentration (%)	High peak (AU)	KIO_3 in salt sample (ppm)
1.	1	10.41	26.0
2.	5	55.27	25.2
3.	10	121.96	27.6
4.	20	239.76	26.9

From the table it is known that the addition of NaCl 1% in standard solution and carrier will increase the concentration of iodate in salt sample, it indicates that the concentration of iodate will be decreased if the standard solutions and the carrier does not contain of sodium chloride. Variation of the salt concentration (%) will not increase the content of iodate in sample.

IV. Conclusion

From the results obtained that the species can be determined using flow injection, when the addition of NaCl can influence the reaction. This method can be used for determination iodine species selectively and sensitive.

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