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By Universitas Muhammadiyah Sidoarjo

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## Flow injection system design for the determination of brown bismarck dye

Desain sistem injeksi aliran untuk penentuan pewarna bismarck coklat

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#### Abstract

This study presents a low-cost flow injection system for estimating Bismarck brown dye, addressing the need for economical and efficient dye concentration measurement methods. The system's design and optimization process are detailed, focusing on parameters like carrier solution speed, dye model size, frequency, and dilution coefficient. Results show optimal conditions for accurate estimations, with the system outperforming traditional color methods in terms of sensitivity and measurement range. This novel approach offers a practical solution for various analytical applications requiring precise dye concentration assessments, with implications for cost-effective chemical analysis in diverse fields.

Highlight:

Low-cost flow system for Bismarck brown dye. Optimized for accuracy in dye concentration measurement. PBactical and efficient for various analytical applications.

Keyword: flow injection system, Bismarck brown dye, low-cost, optimization, dye concentration measurement

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## Introduction

The technique of flow injection analysis discovered in (1975) by (Hanssen & Ruzicka) in Denmark and (Stewart) in America of the most advanced technologies <sup>(3-1)</sup>, which have received acclaim and acceptance in various Analytical areas <sup>(4)</sup>. The technique of flow injection analysis (FIA) has come an important step, it is characterized by simplicity Speed and low cost, as it is based on the use of very small amounts of reagents and high frequency in the analysis process Automatic or semi-automatic <sup>(5)</sup>. The flow injection technique has important analytical capabilities in terms of volumes. Very small of the model and reagents and large modeling number and low detection limits and wide focus range, composed The flow injection system of a peristaltic pump that pushes the carrier current solution and reagents through The system has stable constant flow, the carrier solution, the injection unit for the model, the reagents, the reaction coil, the detector and the recorder To respond  $^{(6-9)}$ . Flow injection analyzes are of two types: segmented flow analysis <sup>(10)</sup> The technique of intermittent flow injection analysis was invented by the scientist Skeyys<sup>(10)</sup> and the goal of fragmentation is to obtain redundancy in which the model is injected into the solution of the reaction materials and the flow is fragmented by the process of leaking air bubbles. These bubbles disturb each section of the pattern for the purpose of completing the blending process. Within a small size to prevent contamination of the model and dilute it, air bubbles must be removed before the carrier solution passes through the detector Using the air bubbler branch<sup>(11)</sup>. This type is not preferred for its disadvantages, which are irregular flow due to air pressure and difficulty controlling the size of air bubbles, as well as air bubbles must be emptied before they reach cell <sup>(12)</sup>, as well as the vacuum device Be large in size and complex, the second type is continuous flow injection analysis<sup>(6)</sup> This type is based on continuous pumping for the carrier solution, the model and other reaction materials without the use of air bubbles, and with this technique many can be analyzed of models with the lowest volume of reagent and sample and within a very short period of time, sometimes reaching 360 models Hourly (5)



Figure (1) Intermittent flow injection system

#### Figure 1.

The analysis of continuous flow injection goes through four basic stages <sup>(13)</sup>, including the first injection stage, during which Introducing the model into the stream of the carrier current, secondly, the propagation stage, in which the model spreads through the carrier current where it took place Loosening the model, III detection phase at this stage the product of the interaction leaves the forked unit towards the detector that In turn, it measures absorption, electrode voltage or any physical factor, fourth, the washing phase is carried out after each A measurement process where it is represented by passing the carrier current alone through the flow injection system in order to remove the remnants of all reaction materials This is because their survival causes inaccurate results when the analysis is performed again and Figure (2) illustrates these The four stages <sup>(14)</sup>.



Figure (2) Injection stages

#### Figure 2.

Bismarck brown R dye is classified among the azo dyes and is considered azo dyes One of the most important types and varieties of dyes at all. They make up more than half of the dyes  $^{(16,15)}$  used in Nowadays for its widespread applications it has been used in different fields depending on its chemical composition and method of application  $^{(18,17)}$ . Azo dyes were discovered a long time ago and the first patent for the production of insoluble azo dyes was

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granted By Thomas and Robert Holliday Robert in 1880 <sup>(19)</sup>, it is characterized by containing a chromophore group -N = N- a charge carrier group so it gives absorption beams in the visible and ultraviolet region (U.V-vis) depending on the electron donor and drawing groups in the dye. Brown bismarck is a chemical compound with the formula  $C_{21}H_{24}N_8$ .2HCl that dissolves in water forming a colloidal solution with a dark color, but it dissolves better in ethanol, and the formula of the brown bismarck dye is shown in the following figure



Figure (3) Formula Composition of Bismarck brown dye

#### Figure 3.

parameter	Value
1- Molecular formula	C21H24N8.2HCl
2- Molecular weigth	461.40 g / mole
3- Adsorption maxima	460
4- Nature	Anionic azo dye

Table 1. Physical properties of brown bismarck dye

Bismarck brown dye is used in the fields of histology and biochemistry to dye microscopic models. and used This dye in the clinical diagnosis of diseases, including forgetfulness using X-rays and electron microscopy, as well as used in dyeing leather, wool, cotton and synthetic fabrics  $^{(2)}$ , The harms of this formula are toxic pigment and have carcinogenic properties  $^{(20)}$ , cause skin irritation  $^{(21)}$  and have many problems and acute or chronic effects on exposed organisms, and affect water systems when they are in the waterway  $^{(22)}$ .

#### Chemicals and devices used

Chemicals used - Bismarck brown R dye, carrier solution (distilled water).

Used Devices and Design - Peristaltic Pump Made by Germany Ismatic Model and Spectrometer Photovoltaic made by Labomed.inG and Simons model 1032 Kompensog Gaph C and cell Standard flow with two holes for the entry and exit of the carrier solution company (HILMA) and loading pipes made of alkalon As carrier connections for reactant materials locally made, scale made by Denver Instrument and UV-visible device spectrophotometer shimadzu, Japan and locally made plastic injection valve in flux injection laboratory Advanced College of Science -Department of Chemistry as shown in the following figure. That the design of an integrated unit for dye estimation Brown bismarck is done in certain stages, including, first, pumping water into the system and second, injecting brown bismarck into a junction The model and then the dye is pumped to the unit with the carrier current represented by distilled water.

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Figure 4. The first stage is the process of pumping the carrier solution (water) through the injection system



Figure 5. Second stage Brown Bismarck dye loading process

Figure 6. The third stage is the process of pumping dye to the unit through the carrier current (water)

## Working methods

3-1 The amount of volume that the connections used to inject the model were tudied and calculated, as this was done through the application of the equation for calculating the volume of the cylinder ( $V = r^2 L$ ) as in Table (2), noting that the pipes used and made of Teflon have a diameter of 1 mm.

3-2 The pump speed was calibrated differently and the volume per minute was calculated using a stopwatch as in Table (3) and the pump accuracy was with an accuracy factor of  $R^{2}0.997=$ .

3-3 The effect of flow speed was studied and the optimal speed of the system reaction was selected at a concentration of 7pmm and a volume of  $\mu$ L 78.50, where the speed was studied and the results were obtained as in Table (4).

3-4 The effect of model size at 5 ppm concentration and at 2.6 ml/sec velocity was studied where volumes from  $\mu$ 178.50 to size  $\mu$ L 196.25 were studied and the results were obtained as in Table (5).

3-5 The frequency was studied at the volume of  $117.00 \ \mu$ l and the speed of  $2.6 \ m$ /sec where the study was carried out at two different concentrations, the first 5ppm and the second 7ppm and the results were obtained as in Table (6)

3-6 The dilution coefficient D of the system used to estimate the dye of brown bismarck was estimated, and at the speed of pumping ml/sec 2.6 and the size of  $\mu$ l 117.00 was first calculated by injecting 5ppm of brown bismarck dye into the system through the injection valve and secondly the value of H<sub>max</sub> (response without dilution) was calculated by mixing the reaction materials in a cup and then injected into the system and this method was repeated to the concentration of 7ppm The results were as in Table (7).

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3-7Dead volume was studied at pumping speed ml/sec 2.6 and volume 117.00  $\mu L$  and the responses shown in Fig. (11)

3-8 The number of models that can be completed per hour was calculated.

3-9 It was done using the flow injection technique at the optimal speed ml/sec 2.6 and a special link to fill the dye with a size of  $117.00 \ \mu$ l and at different concentrations the titration trend was found and the results were as in the table (8).

3-10 The titration curve was found for different concentrations of brown bismarck dye by photometric method and the results were as in Table (9).

## **Results and discussion**

#### Calculate the amount of volume for each form links

A study of the effect of junction length and knowledge of the size that Widening as in the results shown in Table (2) it was noted that By increasing the length of the reaction coil from 10, 15 to 25 cm give Volume of 117,00  $\mu$ l, 78.50  $\mu$ l, 157.00  $\mu$ l, 196.25  $\mu$ l respectively.

(cm) Model link length	Size (µL)
10	78.50
15	117.00
20	157.00
25	196.25

**Table 2.** Amount of size per link (Loop) Calibration of pump speed

The pump speed was calibrated as the results in Table (3) show the calibration of the pump speed and convert the amount of speed to mL/min units as in Figure (7).

Speed (ml/min)	Speed number in the pump
0.500	2
1.200	3
1.500	4
2.100	5
2.500	6
3.000	7
3.700	8

Table 3. Effect of pump speed per ml /min

#### Figure 7. The graphical relationship between speed and volume per minute

It is one of the factors that affect the process of dilution of the model, where Table (4) is the effect of the speed of the carrier solution with the response Less attenuation at slow speeds as this leads to increased retention time The model before moving to the reagent, where the model after injection suffers from The process of longitudinal propagation towards the carrier current as well as transversely due to my process Osmosis and load  $^{(24,25)}$ , and by studying the effect of flow velocity at the length of Wave( 460 nm) and a concentration of 7 ppm and a volume of 78.50 µL Done Obtaining the results of Table (4) as it is noted that the response decreases with increasing speed Pump from speed 2.500 to speed 3.700 ml / min, observed Speed increase from 0.500 to 2.100 per ml / min increases Response from 1.9 to 6.2 cm.

The fifth velocity (2.100) ml / min with a response of 2.6 cm is preferred to sixth, seventh and eighth speeds with a response of 1.5, 1.4, 1.3 cm respectively for being the highest, where the response in Low speed is low because it provides a longer time and causes a loosening process. for the size of the dye through the carrier solution so it is low value opposite to what It occurs at high speeds where the response increases and then the speed increases Lead to a small drop in height down to 2.500 ml/min as in Figure 8.

respones	pump speend (ml/min)

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1.9	0.500
2.1	1.200
2.3	1.500
2.6	2.100
1.5	2.500
1.4	3000
1.3	3.700

**Table 4.** Effect of Velocity of Carrier Solution with Response

2.000 mining as in rigaro o.



Figure 8 Graphical relationship between response (cm) and speed (ml/min)

#### Figure 8. Graphical relationship between model size and response

The effect of model size is one of the factors affecting the response in the Flow injection In general, the response increases with the increase in the size of the model But with high sizes a wide or distorted top appears or other uniform <sup>(4)</sup>, at a wavelength of 460 nm and a velocity of 2.1 And the concentration of 5 ppm was studied the effect of model size or pipe length On the response as Table (5), it was observed that the volume of 78.50  $\mu$ L to 196.25  $\mu$ L Increased response from 1.3 cm to 2.1 cm and after increasing the size it was noted that the response remains constant A little or a little lower so the volume was adopted 157.00  $\mu$ L As the best size of the model and as in Figure (9)

Recurrence means repeating the values of the readings, when re-injecting For the same concentration several times where by measuring Repetitiveness can know the accuracy of the method and the efficiency of the unit Optimal conditions . The effect of the re-injection process was studied, where it was found Results listed in Table (6) Frequency values and response measurement at two concentrations Different (7pprn, 5pprn), and S.D values Standard deviation and R.S.D that have been found at these two concentrations according to the equations below, if Repetitiveness and for ten times where the response appeared A good frequency of readings is shown in

Frequency values	Response when focused ppm7	Response when focused 5 ppm
1	3.6	2
2	3.6	2
3	3.6	2
4	3.6	1.9
5	3.6	2.1
6	3.6	2
7	3.7	2
8	3.6	2
9	3.6	2
10	3.6	2
S.D	0.0316	0.0471
R.S.D	0.8759	2.3550



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Figure 9.

#### Figure 10.

**Dispersion coefficient** Dispersion coefficient <sup>(23)</sup> means the ratio between the concentration before the occurrence of stealth After the occurrence and denoted by the symbol (D), the results listed in Table (7) show the amount of response values per absorbance with dilution and without dilution to obtain through it the so-called dilution coefficient which is as in Figure (11) which gives the graphic relationship between the response with and without mitigation presence. It is noted through these results that the absorption decreases with the presence of mitigation and the response is also reduced.

Concentration	Respond without dilution (H°) cm	Response with dilution ( Hmax) cm
5ppm	3.5	2.0
7ppm	5.7	3.6

#### Table 6. Response Values for Absorption with Dilution and without Dilution

The mitigation factor was calculated in terms of the response (peak height) through the following law:

 $D = H^{\circ}/H_{max}$ .....(3)

1- At 5ppm

D= 3.5 / 2.0 = 1.75

2- At 7ppm

D= 5.7 / 3.6 = 1.58



Figure (11- A) Dilution coefficient at concentration (5 ppm).

Figure 11.

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#### Figure 12.

#### **Dead size**

Dead size is the remaining volume in the sample valve During the injection of the form or reagents and it leads to the appearance of False, double, distorted or signal peak in line foundation, in which case the valve is not perfect but If the dead volume is equal to zero, then the valve is perfect, Through the responses obtained, This valve has a dead zero volume.

Whereas, the responses measured in the form of peak heights in which a single response starts from the baseline zero at The analysis then returns to the zero baseline after the completion of the analysis and other peaks are not launched except with another injection process and this case Ideally made the LED has a dead zero volume as in Figure )12(.



#### Figure (12.) Dead Size

#### Figure 13.

#### Number of models

Studied at models that can be completed per hour at the previously installed optimal letters where the reaction time is calculated ,It was found that the time from the moment the system is operated until the summit is reached and the speed of analyzing models per hour and using a stopwatch to the highest is 28 seconds, which is the reaction time, and by dividing it by 3600 seconds. (1 hour) on 28 seconds is 128 models per hour, which is the speed good analysis and this is one of the advantages of the flow injection technique.

The results listed in Table (8) study of the effect of dye concentration Brown bismarck (titration approach) where it was observed that the response increases From 0.5 cm to 17.1 cm with increasing concentration from 0.05ppm to 35 ppm and Figure (13) gives the shape of the graphical relationship between Concentration (ppm) and response (cm ) titration orientation to dye Brown bismarck by flow injection method, either the extent of concentration by the method of The injection is from 0.05ppm to 35ppm with a value of Linear  $R^2$ =0.9806 which is a very wide range, and the detection limit was 0.05 ppm

Results listed in table (9) absorbency values for brown bismarck dye in the color way about certain concentrations, as the absorbency increases from 0.004 to 0.996 by increasing the concentration from 0.05 ppm to 25 ppm.

Figure (14) gives the form of the graphic relationship between concentration )ppm (and absorption It is a calibration approach to the brown bismarck dye In the color way .

**Calibration curve by flow injection method** The results listed in Table (8) study of the effect of dye concentration Brown bismarck (titration approach) where it was observed that the response increases From 0.5 cm to 17.1 cm with increasing concentration from 0.05ppm to 35 ppm and Figure (13) gives the shape of the graphical relationship between Concentration (ppm) and response (cm ) titration orientation to dye Brown bismarck by flow injection method, either the extent of concentration by the method of The injection is from 0.05ppm to 35ppm with a value of Linear  $R^2$ =0.9806 which is a very wide range, and the detection limit was 0.05 ppm.

#### Figure 14.

## Conclussion

The determination of the brown bismarck dye by hand-designed unit and locally is characterized by speed in analysis, high sensitivity in estimating low concentrations and wide range of concentration, as well as the system

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designed to estimate the brown bismarck dye is characterized by non- Consumption of chemicals in large quantities, as they are characterized by the use of very low volumes and low concentrations for each of the form of the solution vector

In addition, the dead size is zero, which indicates that the manufactured valve does not leave any errors and its efficiency is high.

The brown bismarck dye can be appreciated by the color method, but it needs more time as well as larger quantities of the model

The low concentrations cannot be measured, which indicates the efficiency of the flow injection method that has been used.

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