FLOW INJECTION ATOMIC ABSORPTION SPECTROMETRY IN SOLUTION FOR DETERMINATION OF MERCURY

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ABSTRACT

A stopped-flow injection and the merging zone stopped-flow injection technique used for cold atomization with atomic absorption spectrophotometric determination of mercury in solution by reaction with sodium borohydride as reducing agent. All the flow-injection and chemical variables affecting the sensitivity and peak profile are optimized. The atomic absorption signal gave a linear range of 10-80 µgml^{-1} mercury for the first FIA-manifold by injection 50µl sample with a correlation coefficient of 0.994 and a low detection limit (50ng absolute) were observed. The relative standard deviation found was 1.68% with a sample throughput 75 sample h^{-1}.The second FIA- manifold illustrate a determining of mercury in the range 2-50 µgml^{-1} with a correlation coefficient of 0.9992. A low detection limit (5ng absolute), good precision (1.99% relative) and a sampling rate of up to 60 samples h^{-1} was obtained .The method developed is highly selective and relatively simple.

KEYWORDS: *Mercury, Flow injection analysis, Stopped and merging zone technique, Atomic absorption in solution*

INTRODUCTION

Hempel et al.[1995] describe that mercury is widely distributed in nature, circulating among several media and occurring in different chemical forms, which show various degrees of toxicity. In addition Brunetto etal.[1999] recognize that inorganic mercury (In-Hg) compounds are converted into more toxic organo-mercury (Or-Hg) compounds by living organisms during natural cycles. Therefore an average daily intake of 0.02mg mercury per day by adult has been reported by Issag and Zielinski [1974].

Hatch and Ott [1968] in their routine work for measuring the total mercury content of environmental and physiological samples, most commonly water, wastewater, and urine of the numerous analytical techniques thus far used cold vapor atomic absorption spectrometry (CV-AAS), developed three decades ago, which is adequately sensitive for most purpose.

Gallignani et al. [1998] used FIA technique which has been proved to be useful for on–line addition of reagent and for sample pretreatment. In recent year's Dedina and Tsalev [1995] represent on-line sample pretreatment are of particular interests for mercury determination due to the risk of sample contamination, analyte volatilization and adsorption losses during extensive off – line sample digestion procedures. Now a wide variety of different procedures have been carried out by flow-injection (FI) techniques, contamination –free closed sample handling system .

Reduction of metal ions to the metallic state is extensively used as a method for analyte preconcentration in analytical chemistry Mizuike[1983]. The most common, and practically standard procedure utilizes $NaBH_4$ as a reducing agent for the generation of mercury vapor and the volatile hydrides of a number of elements as well as for reductive precipitation of trace metal from solution Schmid[1994].

Mulvaney and Weller [1990] studied elements such as Cu, Ag, Au, pt, pd, and Ni which do not form volatile hydride by reaction with $NaBH_4$ from metallic particles having size ranges from several hundred to several thousand nanometers. Despite the fact that $NaBH_4$ has been extensively used for the preconcentration of metals from solution for some time, little attention has been devoted to the study of the initial stages of particle formation. Reduction of ions most likely proceeds through an intermediate single atom before larger clusters are formed. Growth and reactivity of silver all the studies point the transient existence of reduced atoms of the element in solution following its reduction.

Early attempts to observe the formation of free atoms in solution during electrochemical reduction of metal ions at pt electrode were reported by Tyson and West [1979].Unfortunately, initial conclusions reporting the measurement of atomic absorption by a number of elements while using a broad – band deuterium source were subsequently shown to be erroneous when it was concluded that the spectra were actually due to the presence of hydroxo electrogenerated at the cathode surface .

In contrast of their studies, Panicher and Sturgen [1998] report the first observation of selective absorption of radiation by metal atoms in aqueous solution during chemical reduction of metal ions. High–resolution atomic absorption spectrophotometer with both hollow cathode lamps as a sharp–line sources and deuterium source background –corrected measurements applied to the detection of possible atomic species in solution during the initial stages of metal ion reduction preceding significant cluster formation. Such selective absorption of radiation suggests that free atoms for Ag, Pd and Cu are being observed in aqueous solution and not influenced by the aqueous environment.

The specific advantages of coupling FIA system to various analytical techniques have been widely reported. The present work aimed to study the flow injection manifolds using stopped –flow and merging zones technique developed for determination of mercury by atomic absorption in solution. The method is simple and accurate. It describes a selective absorption of radiation by Hg-atoms in the aqueous solution during chemical reduction of Hg^{2+} ion using sodium borohydride as a reducing agent.

EXPERIMENTAL

Reagents and Solutions

All chemicals used were of analytical grade reagents. Standard solution of 1000 μg $Hg.ml^{-1}$ were prepared from Merck Titrisol solution. Mercury stock solution 100 $\mu g.ml^{-1}$ prepared by dilution the stock reference standard solution with deionized water 0.5% (w/v). Sodium borohydride ($NaBH_4$) stock solution (BDH) was prepared daily by dissolving 0.5g of reagent in 100 ml of deionized water. Sodium hydroxide at concentration of 1% w/v was added as a stabilizer. The solution was filtered before use. Working solutions of $NaBH_4$ was prepared by dilution of the initial stock solution. Triton X-100 surfactant (Aldrich, Milwaukee, WI) 1% V/V stock solution was prepared using deionized water. The stock solution was added in a volume ratio of 1:10 to the working solution. Ammonia – Ammonium chloride buffer solution (pH=10) prepared by adding 142 ml concentrated ammonia solution (sp.gr 0.88 – 0.9) to 17.5 g ammonium chloride and diluted to 250 ml with deionized water. Deionized water (DIW) served as the carrier stream.

Prepared of Working Solution

Working calibration solutions having mercury concentration in the range of 0.01 – 80.00 $\mu g.ml^{-1}$ were prepared daily by taking appropriate aliquots of the stock solution. The pH was adjusted by adding 1ml of buffer solution; a further 1 ml of 0.1 % stock solution of Triton X-100 added, then diluted with DIW to the mark using calibrated 10ml volumetric flask.

A blank solution was prepared in 500ml volumetric flask by mixing 50ml of pH 10 buffer and 50mL of 0.1% Triton (X-100) solution, then diluted to the mark with DIW.

Apparatus and Procedures

The majority of measurements were performed using a model SP9 Flame Atomic Absorption Spectrophotometer (PYE Unicam). The burner was replaced by a special quartz flow cell (Hellma) located in the optical path and supported by the burner surface. Figure(1) provides schematic characterizations for the flow cell which have been used in this work; having a 10-mm path length with internal volume of 750μl.

Mercury hollow cathode lamp at λ253.6nm with a maximum current of 6 mA and a deuterium lamp for background correction used as incident source for atomic absorption measurement. The position incident source beam was optimum, (i. e., maximum atomic absorption signal) at height of about 5mm above the bottom of the cell cavity.

Experiments were conducted under both flow condition and the stopped flow technique. Measurements were undertaken in several modes; two types of the FIA manifolds have been investigated. The manifold shown in Figure(1-A), two channel peristaltic pump (Ismatic Mini-S-840) was employed furnished with suitable tygon pumping tubes used to propel all solutions in a variable speed. All the connectors made of polyethylene tubing (0.5mm i.d.). Under continuous flow condition, the two separate streams of DIW and $NaBH_4$ were merged continuously at a 45° angle when entering the cell by using a Y-connector made from Perspex (Technicon). The flow-rate was varied in five stages down to minimum deliverable, by using continuous flow and stopped-flow technique, without changing pump tubing.

RESULTS AND DISCUSSION

A preliminary investigation indicates that the reaction between mercuric ion (Hg^{2+}) and $NaBH_4$ solution which occurs was spontaneously in an alkaline aqueous medium, is expressed as follows:

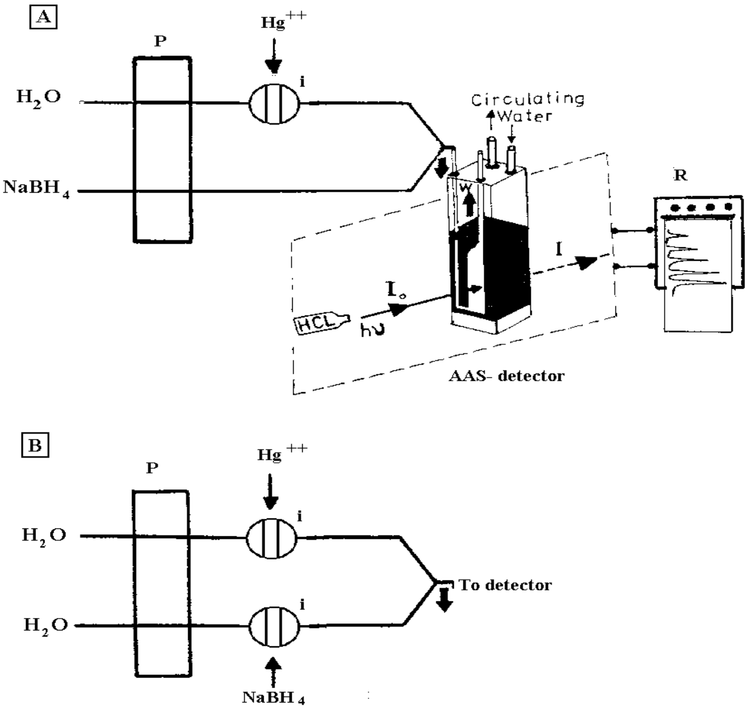
$$8Hg^{2+} + BH_4^- + 8\,OH^- \rightarrow 4Hg_2^{2+} + 6H_2O + BO_2$$

$$4Hg_2^{2+} + 2BH_4^- + 8OH^- \rightarrow 8Hg^o + 4H_2O + 2BO_2 + 4H_2$$

The atomic absorption of initial mercury atoms in solution, before larger clusters are grown, associated with a FIA technique is linked to factors such as the flow injection and chemical variables, which must be fixed and studied in order to obtain sensitive and more accurate and reproducible results.

Optimization of chemical variable using Simple – FIA (Manifold - A)

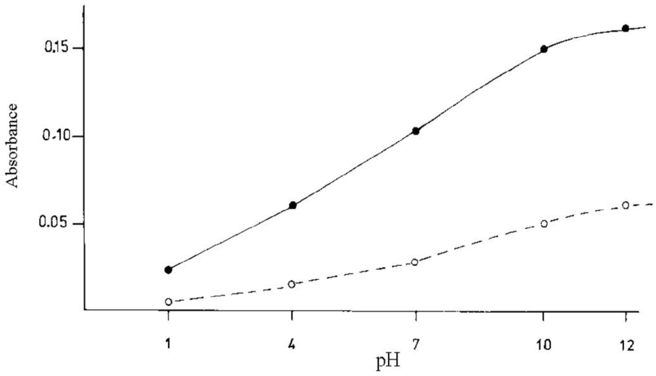
FIA system shown in Figure (1-A) investigated at two modes using continuously flow and stopped –flow techniques. A typical absorption peaks are depicted in all over figures. 50µl volume of 50µg ml^{-1} Hg^{2+} solution (2.5µg absolute) was delivered to the cell in 3 ml.min^{-1} steam of H_2O where it was continuously merged with the reagent stream of $NaBH_4$ solution at 3 mL.min^{-1} and for the stopped flow technique the pump turned off at a preselected delay time and then restarted after a stop time period elapsed = 20 sec.



Figure(1) Schematic diagram of the FIA system. (A) Simplest two line flow manifold for determination of Hg by AAS in solution using stopped – flow technique . (B) Merging zones manifold for determination of Hg by AAS in solution using stopped – flow technique: P, peristaltic pump ; i , Injection valve ; AAS Atomic Absorption spectrophotometer ; HCL , Hg Hollow cathode lamp; Io , incident light ; I, transparent light; w, west ; R, Recorder.

Effect of pH

The effect of pH on the reaction was examined by adjusting the mercury samples to various pH values. The plots of absorbance Vs. pH for Hg^{2+} ion show that the pH has an important effect on the development the analytical signals, the absorbance is constant for pH value between 10 and 12. In subsequent experiments pH 10 was chosen. Solid line in Figure (2) describes the results for stopped –flow technique were compared with that achieved by continues flow (dash line), the best results were obtained and the sensitivity increased by the stopped–flow technique.



Figure(2) Effect of pH of sample on Mercury absorption as obtained by using the system from Figure 3-1 (A). Injection volume was 50 µl of 50 ppm Hg with a flow rate of 6 ml / min , in all experiments contained Triton x-100, using 0.05% $NaBH_4$ for (o---o) continuous flow and (●—●) stopped- flow technique.

Effect of $NaBH_4$ concentration

This reagent was varied from 0.02 up to 1.0%. The absorbance increased until a limiting plateau at about 0.1% $NaBH_4$ solution Figure (3-a). Peak height at higher $NaBH_4$ concentration was identical with that at 0.1 % $NaBH_4$; however, this is not the case for more concentrated $NaBH_4$ reagent. Figure (3-b) shows a splitting in the signals shapes when using a concentration more than 0.5% $NaBH_4$ reagent resulted in immediate and rapid formation of black precipitate and a noise could be achieved within the time domain of the present experiments.

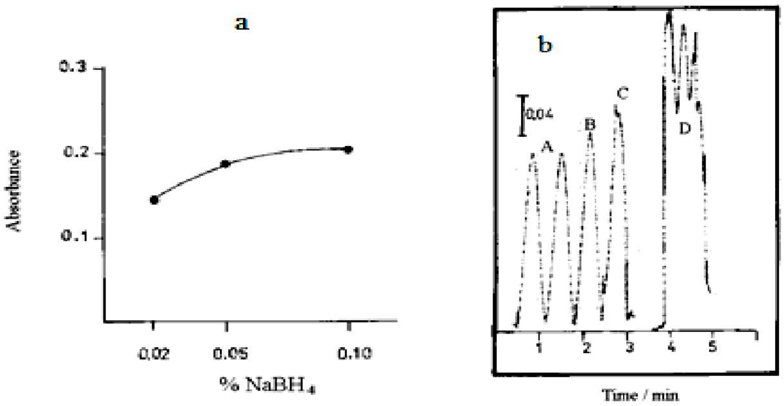
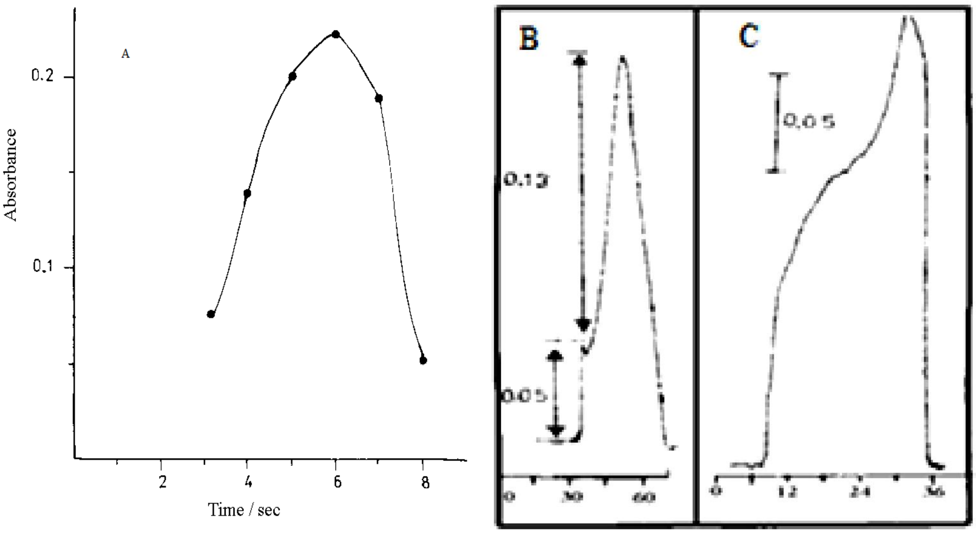


Figure (3)a- Effect of sodium borohydride concentration on the sensitivity using buffer pH=10 solution by stopped – flow technique. b- Responses produced for a series of sodium borohydride concentration 0.02, 0.05 , 0.1 and 0.5% for A,B,C and D respectively showing time consuming for each injection and peak profiles at higher concentration of $NaBH_4$.

Influence of delay time

The influence of delay time, for the stopped–flow technique on the absorbance and the signals shape was studied by changing a pre-selected delay time for each run, while keeping constant both the pH and $NaBH_4$ concentration at optimum values. A plot of absorbance vs. delay time is presented in Figure(4-A). The results show that the absorbance varies largely with increasing delay time. A delay time of 6 sec gave the greatest signal. The reason for this is shown in peaks B & C of Figure (4). The peak profile B and C obtained by stopping the pump when the signal reached its maximum absorption peak height and for the pre-selecting delay time of 6 sec, respectively. For the peak B we could find maximum absorbance of 0.05, this indicates a case of continues flow mode, and an absorbance of 0.13 when the pump stopped for a period elapsed (20 sec). The results indicate that the sensitivity is improved and an increase of approximately 3 fold could be achieved for the stopped –flow technique.

The absorbance decreased in the case of using larger delay time because the mercury sample plug passes the absorbance interval.



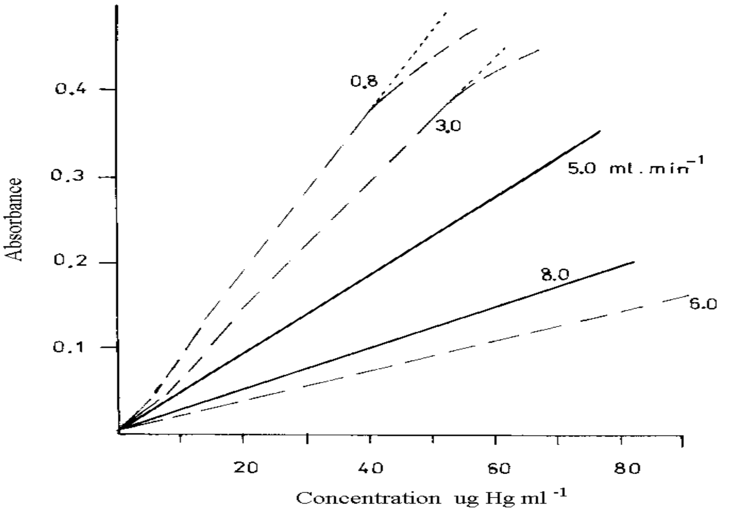
Figure(4) A- Influence of delay time on the sensitivity, injection volume was 50µl of 50ppm Mercury at optimum condition of pH=10 and 0.1% $NaBH_4$. **B-** Peak profile obtained by stopping the pump when the signal reaching its maximal absorption in the case of continuous flow showing the peak maximum as recorded during the delay period. **C-** Peak profile for pre selecting delay time = 6 sec and then re-started after the stopped – flow period elapsed = 20 sec.

Effect of total flow rate

The maximum absorbance signals were found for flow rates below 1.0 $ml.min^{-1}$ using a continuous flow, shown as a dashed line in figure(5) for the range of flow rates investigated, peak height was increased and at the same time a broadening in the signal peak was obtained as the flow rate was decreased. Under these conditions sample through put decreased dramatically.

Lower peak heights were found for flows rates above 3 $ml.min^{-1}$ probably because the reaction between Hg^{2+} and BH_4^- had not gone to completion, owing to very short residence time, with flush the free atoms produced out of the observation interval.

Stopped –flow results were plotted as a solid line also in figure(5). The highest analytical signals were found for flow rate ranging from 5 to 6$ml.min^{-1}$. A decreasing in both peak heights and broadening observed for flow rates above 6$ml.min^{-1}$. However, total flow rate < 5 ml min^{-1} slightly effects the sensitivity as shown in Table (1).



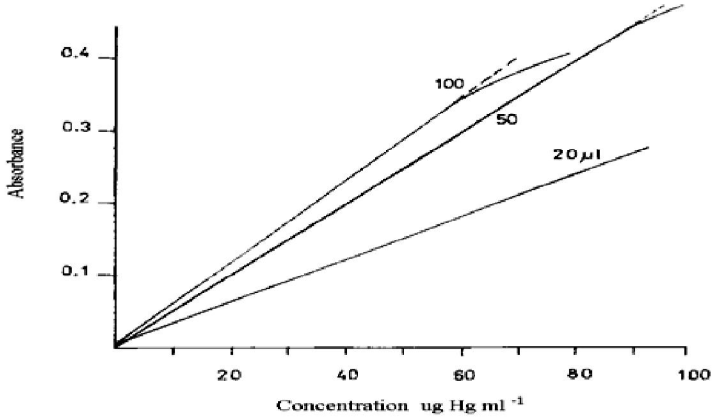
Figure(5) Effect of different flow rates on the sensitivity (slope) of the method for continuous flow method (dashed line) and for stopped-flow technique (solid line).

Table (1): Comparison for the sensitivity resulted from the two manifold

Flow –rate ($ml.min^{-1}$)	Slope (ml . μg^{-1} × 10^{-3})	
	Continuous flow	Stopped –flow
0.8	9.640	-
5	-	4.557
6	1.716	-
8	-	3.406

Influence of injected volume

The influence of sample volume on absorbance peak height was examined at a total flow rate of 5ml min-1 using stopped flow technique. As one would expected, the peak height increases with increasing sample volumes figure (6) this tended to become constant at sample greater than 100µl, the peak width also become broader. The range of linearity of mercury standard concentrations vs. absorbance depends on the number of mercury free atoms produced, the chemical reaction starts at the point where the $NaBH_4$ reagent reaches the center of the sample zone. Table (2) summarizes the analytical data for each injection volume. The result shows that injected volume of 50µl consent to be optimal. Figure (7-A) shows a relationship between dispersion (D) and the injection volumes for $50\mu g.ml^{-1}$ mercury. The dispersion, using 50µl sample injection and flow rate of $5ml.min^{-1}$ was found to be 4.20. A flow rate 5 $mL.min^{-1}$ was selected that gives best compromise between sensitivity and sample through put.



Figure(6): Influence of sample volume on the sensitivity using stopped – flow technique, delay time = 6 sec and stopped flow period = 20 sec.

Table(2) Effect of Mercury volume on the sensitivity and sample throughout put using stopped –flow technique

Volume (µl) of mercury injected	Slope ml.µg ×10^{-3}	Linear range $\mu g.ml^{-1}$	Through put samples h^{-1}
20	2.197	20-90	81
50	4.557	10-80	75
100	5.610	5-60	52

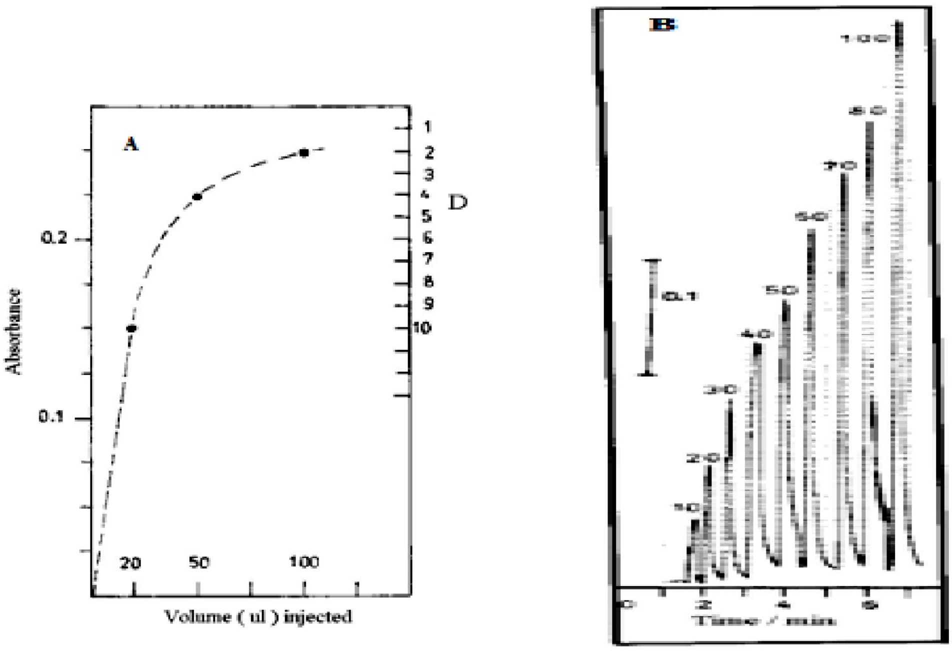
Analytical characteristics of the method

Under optimum chemical and flow conditions, summarized in Table (3), a set of standard mercury solutions was injected to test the linearity of the method. The results are shown in Figure (7-B) The calibration curve was linear over the range 10-80 µg. ml^{-1} using the stopped flow technique with correlation coefficient of 0..997 the sample throughput was about 75h^{-1} without overlapping.

The corresponding equation was **A = 4.557×10^{-3} Conc. + 0.0043**, where A is the peak height in absorbance and conc. is the mercury concentration in µg.ml^{-1}. The relative standard deviation of the slope was 1.5% and that of the intercept was 2.1% (n=3).

Table (3) Results for the optimum chemical and flow injection condition using stopped –flow technique for atomic absorption determination of mercury in solution settings

Parameters	Optimum Value
pH of the injected Mercury solution	10
NaBH$_4$ concentration.	0.1% w/v
Injected volume (µl)	50
Total flow rate (ml.min^{-1})	5
Delay time (Sec)	6
Stopped time (Sec)	20



Figure(7) A- Influence of sample volume on the dispersion (D) by injection of 50 ppm Hg^{2+}solution. B- High-speed recording peaks obtained as a calibration runs for a series of Mercury buffered standards solution (10,20,30,40,50,60,70 and 80 µg Hg^{2+}ml^{-1}) using the system showed in figure(1-A) by the stopped – flow technique.

Precision of the method

To measure the repeatability, ten injections of three different concentrations of standard mercury solutions (3 injections for each of 15 and 55 µg HgmL^{-1} with 5 injections of 90µg Hg ml^{-1} are included in Figure (8-B) were investigated to calculate the relative standard deviation. The results were shown in Table (4).

Table (4) Repeatability of the stopped –flow technique for determination mercury (All results are means of ten injections).

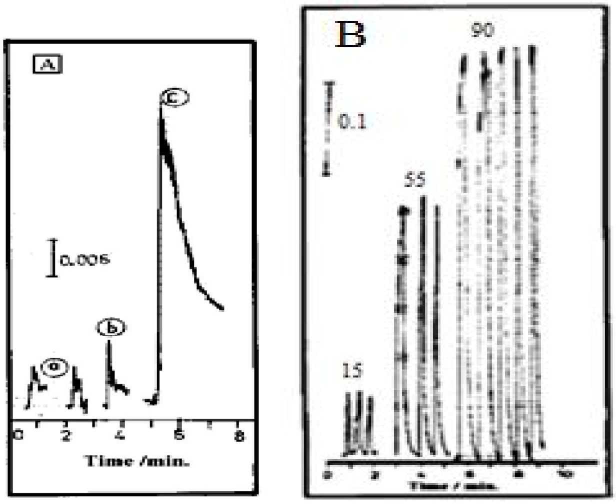
Mercury concentration µg.ml^{-1}	Mean absorbance	Standard deviation (absorbance unit)	RSD%	Analytical time (min)
15	0.069	0.00173	2.51	4.7
55	0.277	0.00470	1.66	6.6
90	0.457	0.00346	0.75	13.3

Detection limit

The detection limit is defined as the concentration that produced an absorption signal equivalent to three time base line noise (absorbance “peak height” > 3Sd, n=11 for background zero absorption). In accordance with this definition, the detection limit of this method is 1.0μg ml^{-1} (50ng mercury absolutes). Figure (8-A) shows a typical peaks to determine the detection limit (a), limit of quantization (b) and the least concentration for the linear range (c).

Optimization of FIA variables using merging zone technique (Manifold B)

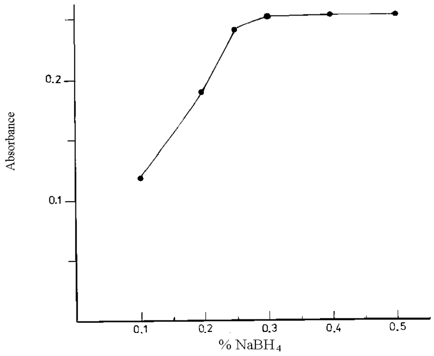
In all continuous flow system, the main disadvantage of the method is the high reagent consumption, in contrast to batch method a continuous flow reagent is necessary, regardless of the presence of sample. FIA system shown in Figure (1-B) (Merging zone mode) investigated using stopped –flow technique. As mentioned previously mercury sample volume (50μl) was chosen to be optimal in order to obtain the required degree of sample dispersion.



Figure(8) A-Recording peaks obtained by injection of 1,2 and 10 μg ml^{-1} Mercury solutions for a, b and c respectively using the system showed in figure(1-A) by stopped – flow technique , with increased amplification to illustrate the limits of quantitation (LOQ) and limits of detection (LOD). B- Results of triplicate injections of Hg standards recording from left to right (15, 55, 90 μg. ml^{-1}).

Influence of $NaBH_4$ concentration :

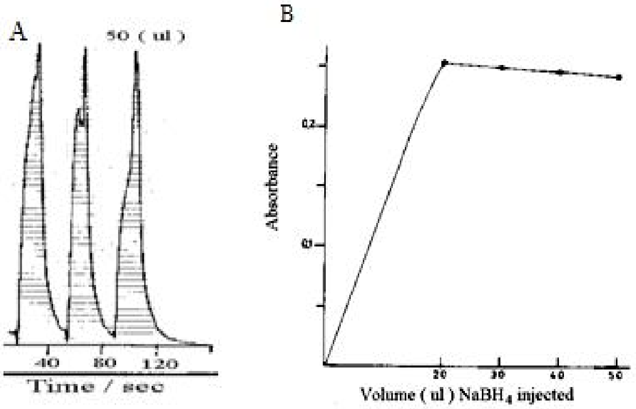
The influence of varies concentration of $NaBH_4$ was tested, by injecting equal volumes (50μl) of each of mercury and $NaBH_4$ simultaneously into the two separate carrier DIW stream (3mL.min^{-1}). The reagent concentration was carried from 0.1 to 0.5%. Figure (9) shows that the absorbance increased with increasing $NaBH_4$ concentration until a limiting plateau at about 0.25% $NaBH_4$ solution and selected to be optimal; at higher concentration (more than 0.3% $NaBH_4$ w/v) a broadening in peak signal was observed. As a compromise between sensitivity and peak profile, a 0.25% $NaBH_4$ was selected to be injected.



Figure(9) Effect of sodium borohydride concentration on Mercury absorption as obtained by using merging zone with stopped – flow technique as showed in figure (1-B). Hg = 50 ppm, injected volume = 50μl, in all experiments the pH adjust using pH buffer =10 contained Triton x-100 and injected volume of 50μl $NaBH_4$ simultaneously.

Influence volume of $NaBH_4$ injected:

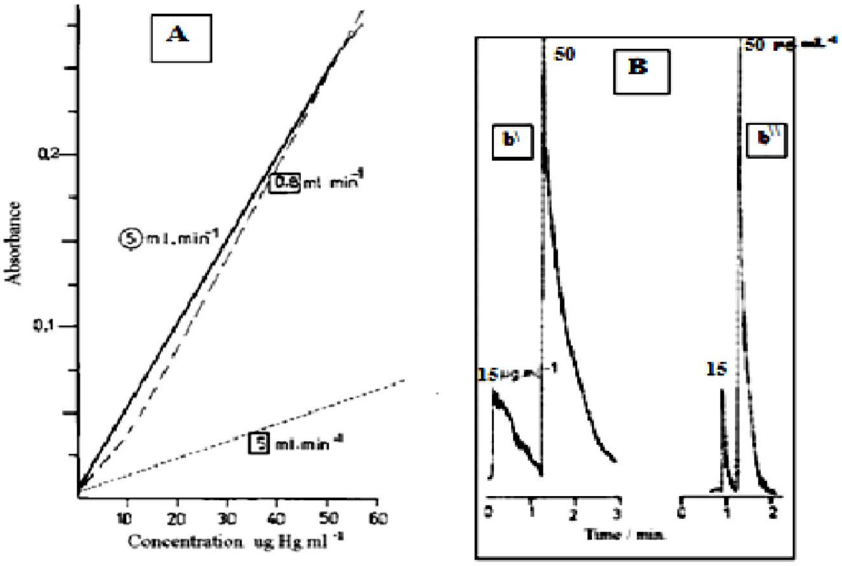
The effect of $NaBH_4$ volume on absorption peak height and profile was examined at total flow rate 5 ml.min^{-1}, the mercury injected volume (50μl) was kept constant using stopped flow technique. As shown in Figure (10-B), the absorbance was not highly affected with increasing the injected volume of 0.25% $NaBH_4$. Typical peaks recorded for a volume of 50μl $NaBH_4$ injected are shown in Figure (10-A); a splitting and a broadening obtained. Therefore a minimum volume of 20μl $NaBH_4$ is required and selected to be the optimum.



Figure(10) A- Signals produced for injecting 50μl of 0.25% sodium borohydride using merging zone manifold with stopped – flow technique. B- Influence volume of sodium borohydride injected using merging zone manifold with stopped – flow technique

Effect of the total flow rate

The effect of the total flow rate of the carrier DIW stream on the absorption peak shape was examined; the injected volumes for both mercury and $NaBH_4$ are kept constant, for each continuous and stopped –flow techniques. The total flow rate 5ml. min^{-1} gave best sensitivity using stopped flow technique as shown in figure (11-A). In the case of continuous flow a decreasing in flow rate increased the sensitivity while the peak width increased also. The overall variable in peak shape is shown in figure (11-B). Flow rates at 5ml.min^{-1} using stopped –flow technique indicated as (b'') while 0.8 ml.min^{-1} using continuous flow indicated as (b′).



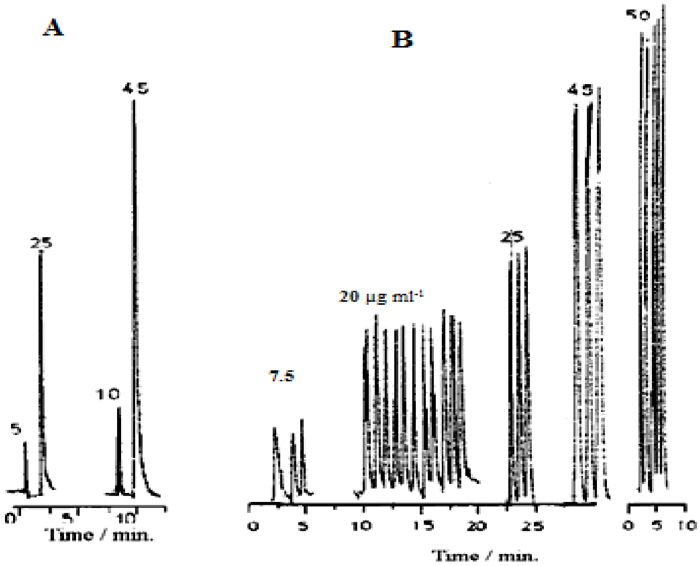
Figure(11) A- Effect of flow rates on the sensitivity using merging zone manifold at continuous flow (dashed line) and for stopped – flow technique (solid line). B- Recorded peaks for two standard Mercury solutions (15 and 50 μg mL^{-1} obtained using merging zone with continuous flow [b$^{\backslash}$] (0.8 mL.min^{-1}) and stopped flow[b$^{\backslash\backslash}$] (5 mL.min^{-1}).

Analytical characteristics of the method

Under optimum chemical and flow-injection conditions, a series of standard mercury solutions was injected to test the linearity of the method. The results of typical recorded peaks are shown in Figure (12) using merging zone with stopped flow technique. The corresponding equation obtained was as follows: $\mathbf{A = 4.743 \times 10^{-3}Conc. + 0.0068}$ with correlation coefficient of 0.9992 and the sample through put was about $60h^{-1}$.

Precision and detection limit

To measure the repeatability, eleven injections of the 20 µg.mL-1 mercury are investigated. The peaks obtained shown in Figure (12-B) The relative standard deviation (RSD %) = 1.99 % , and the absolute mass of 5ng recorded as a detection limit



Figure(12) Typical recorded peaks obtained using merging zone with stopped flow technique for (A) series of Mercury buffered standard solution (5, 10, 25, and 45µg ml^{-1}) injected randomly. (B) High-speed recording peaks obtained as a calibration runs for series Mercury buffered standard (7.5, 20, 25, 45 and 50µg ml^{-1}).

Comparative study

The figures of merit for determination of mercury and the comparative study shown in table (5). The two manifolds at optimum parameter studied for kinetic behavior of the signal by injected of 50µl volume of $25\mu gm^{-1}$ mercury. Figure (13- a) shows two peaks recorded at room temperature by using merging zone technique (Manifold B). The left peak recorded under stopped and continuous flow, required 42sec as analytical time. The profile of the right peak, recorded (80 sec) by leaving the pump stopped without regeneration so as to observe signal development under static conditions. This permitted the recording of the growth of the background absorption following the formation and disappearance of free mercury atoms in solution, leads to cluster formation after relative long period of time.

The other two peaks shown in figure (13- b) recorded by using the manifold A. The same manner used for left and the right peaks profile and it seems to be required an analytical time of 30sec and 57sec, respectively. The reason for this difference in the results is directly related to amount of the $NaBH_4$ in the flow cell.

Excesses amount of $NaHB_4$ reagent or more concentrates solution decreased the time for particle formation or increased the removal time for the free Hg° atoms in the solution. Therefore, flow –injection with merging zone technique must be selected for the kinetic study so as the free Hg° atoms stay relatively for long period of time.

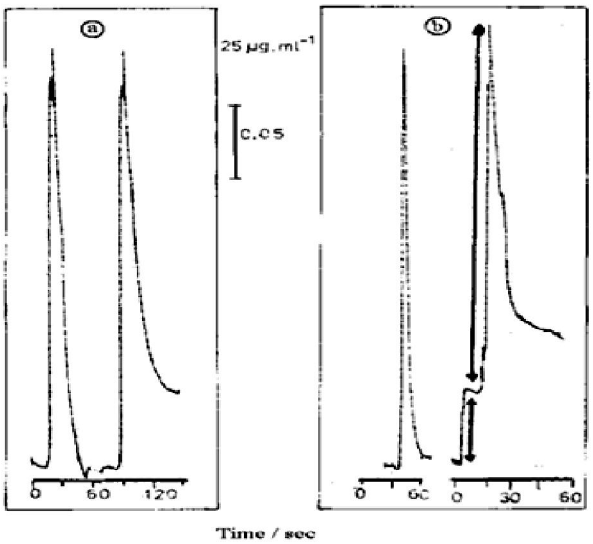
Excellent correlation (r = 0.9990) was obtained between the results for the series standard mercury solution by the stopped-flow technique as a comparative study between the simple - FIA (manifold A) and the merging zone - FIA (manifold B).

Effect of divers ions

A number of cations were examined for interference in the procedure. The specificity of the signals were studied by injection of 50µl volume of 50µg.mL^{-1} ions solution to the system when the atomic Absorption spectrophotometer was operated with background –correction at the mercury resonance line 253.6.nm shows no response by using stopped –flow technique for the two manifold.

Table (5) The comparative study

Parameter	Stopped -flow	
	Simple – FIA Manifold(A)	Merging zone – FIA Manifold(B)
Sensitivity (ml. µg^{-1})	4.557×10^{-3}	4.743×10^{-3}
Repeatability (RSD%)	1.68%	1.99%
Correlation coefficient	0.9970	0.9992
Detection limit	50ng	5ng
Through put (sample h^{-1})	75	60
Relative error (E%) at 20 µg.ml^{-1}	4.1%	4.4%
Linear rang (µg.ml^{-1})	80 – 10	2 - 50



Figure(13) Typical peaks recorded showing time consuming for injection of 25 ppm Hg^{2+} at optimum condition. (a) using manifold -A shown in figure (1-b) and using merging zone manifold -B shown in the same figure.

Conclusion

A stopped-flow injection and the merging zone stopped-flow injection technique can be used for cold atomization with atomic absorption spectrometric determination of mercury in solution by reaction with sodium borohydride as reducing agent which considered as a relatively fast method up to 60 samples h^{-1}.The method developed is highly selective and relatively simple and can be can applied for determination of mercury in highly polluted industrial waste water sample .

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الملخص

منظومتان لطريقة الجريان المتوقف وطريقة المناطق المتلاقية استخدمت لتحليل الزئبق بطريقة مطيافية الامتصاص الذري ذي التذرية الباردة (داخل المحلول)حيث أن الزئبق يتفاعل مع هيدروبورونات الصوديوم فيتحول الزئبق إلى ذرات حرة طليقة داخل المحلول فيمكن قياس الامتصاصيه لها باستخدام شعاع الرنين المنبعث من مصباح الكاثود المجوف للزئبق. تم دراسة الظروف المثلى الكيميائية والفيزياويه للحصول على أفضل حساسية مع الحصول على إشارة واضحة وثابتة . منحني المعايرة بمدى خطية 80 – 10 مايكروغرام لكل ملي لتر وقيمة معامل الارتباط 0.994 تم الحصول عليها بالنسبة للمنظومة الأولى وبمعدل سرعة تحليل 75نموذج في الساعة . وايضاً تم الحصول على منحني معايرة باستخدام المنظومة الثانية وكانت حدود الخطية 2 – 50 مايكروغرام لكل ملي لتر وقيمة معامل الارتباط تساوي 0.9992 وبمعدل سرعة تحليل بحدود 60 نموذج في الساعة.

پوختە

لەم توێژینەوەیەدا دوو سیستەمي رۆیشتوو یەکگرتوودا وەستاو بەکار هات بۆ شیکردنەوەي جیوە بە ڕێگای هەڵمژیني گەردیلەیي لەناو گیراوەدا. جیوە لەگەڵ بۆروهایدریدي سۆدیۆم کارلێك دەکات وە ئایۆني جیوە دەگۆڕێت بۆ گەردیلەي ئازاد کە دەتوانرێت پێوانەي هەلمژیني تیشکي لەرینەوە بۆ بکرێت لە سەرچاوەي هالۆکاسۆدي تایبەت بە جیوەوە دەردەچێت, بۆ ئەم مەبەستە چەندین لێکۆلینەوە کرا بۆ دیاۆیکردني گونجاوترین بارودۆخي کیمیاوي و فیزیاوي بە مەبەستي بەدەستهێناني باشترین هەستیاري و باشترین سیگناڵ. لە سیستەمي یەکەمدا مەوداي ڕاستەهێلي خەملاندنێ جیوە لە نێوان 10-80 مایکروگرام/ملیلتر و نرخي هاو کۆلگەي بەستنەوەي ڕاستەهێلەکە یەکسان بوو بە 0.994 ، وە بە ڕێژەي 75 نموونە لە یەك کاتژمێردا. لە سیستەمي دووەمدا مەوداي ڕاستەهێلي خەملاندنێ جیوە لە نێوان 2-50 مایکروگرام/ملیلتر و نرخي هاو کۆلگەي بەستنەوەي ڕاستەهێلەکە یەکسان بوو بە 0.992 ، وە بە ڕێژەي 60 نموونە لە یەك کاتژمێردا.