Sampling of atmospheric Hg^o using home-made gold-coated sand sorbent prior to analysis by atomic absorption spectrometry

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Abstract

Gold-coated sand for amalgamation was synthesized and applied for the determination of mercury in ambient air using a home-made dual gold trap unit coupled to atomic absorption spectrometer. Gold-coated sand is prepared by chemical reduction of Au(III) solution with hydroxylamine depositing elemental gold on acid-etched sand. A home-made dual gold trap unit which focused time-resolved mercury trapped from the sampling/first trap provided an increase in sensitivity and reliability for the analysis of ultra-trace mercury in air was designed and tested. Instrumental detection and quantitation limits (IDL and IQL) of system were 3.9 and 13pg Hg, respectively. Method detection and quantitation limits (MLOD and MLOQ) of system were 0.04 and $0.13ngHg.m^{-3}$ for sampling flow rate of $200mL.min^{-1}$ and sampling time of 8 hours. Sampling system for gaseous elemental mercury was set up and cooperated with home-made desorption system were preliminarily applied for analysis of atmospheric mercury in samples collected at Hochiminh city University of Science. The atmospheric mercury concentrations were in range of 2.7 – 8.1ng Hg. m^{-3} which were comparable to Hg concentration found in other cities in the world.

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Keyword
gaseous element mercury (GEM), air sampling, dual gold trap, atomic absorption spectrometry

1 Introduction

Mercury is one of the most toxic heavy metals. Mercury exposure, through digestion of Hg contaminated food and/or water and breath of Hg polluted air, may pose risk to permanent damage to the nervous system, as well as renal toxicity, myocardial infarction, immune malfunction, and irregular blood pressure. Nowadays, acute poisoning incidents of mercury such as Minamata or Iraq pollution is unlikely occurred but chronic exposure at low concentrations of Hg are in danger for any living creatures.

Generally, atmospheric mercury exists as gaseous elemental Hg (Hg^o, GEM), reactive gaseous Hg (RGM), and particle-bound Hg (Hg(P)). Hg^o is the predominant form in the atmosphere (>95%) and the rest accounts for RGM (≈3%) and Hg(P) (≈1%)[1]. The analysis of atmospheric mercury is not straightforward task due to its low existing concentrations and high risk of contamination. The determination of atmospheric mercury has been carried out by automated and manual approaches. Automated system has been mainly used for online monitoring of mercury that based on commercialized systems from Tekran, PS Analytical, Gardis … and provides high time-resolved data for a long period of monitoring time[2]. However, the equipment for online monitoring is costly and requires skillful person to operate and maintenance. Manual monitoring approach including gas sampling then analyzing mercury in laboratory provides discrete data which is useful only for short-term evaluation purpose. The equipment is simple, versatile, easy to work and inexpensive. The most commonly used of sorbent for elemental mercury has been noble metals- based materials such as coiled gold wire, gold nano-structures, or gold coated onto a high surface-area substrate[3]. Elemental mercury collected on solid sorbents was either digested by concentrated nitric acid followed by concentrated hydrochloric acid and determined with cold vapor (CV) atomic absorption spectrometry (AAS) (according to NIOSH method 6009 or OSHA Method ID-140)[4] or thermally desorbed to an atomic absorption spectrometer or atomic fluorescence spectrometer for

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measurement (ISO 20552 – 2007)[5]. The gold - based sorbents were quite highly cost that was unaffordable for our research.

In this study we, for the first time, developed an analytical method for atmospheric mercury based on the home-made gold coated sand as sorbent, a dual amalgamation for thermal desorption following atomic spectrometer for detection. The synthesis, activation and usage of gold coated sand for mercury sampling, the sampling and analysis of atmospheric mercury in our laboratory were discussed.

2 Materials and methods

2.1 Instrumentation and chemicals

All measurements for mercury was performed a Mercury Analysis System, FIMS 100 (Perkin Elmer, USA) equipped with a mercury incandescent lamp, a 17-cm long borosilicate cell fitted with two quartz windows at both ends and Winlab AA32 for signal acquisition and integration. The cell input is connected with the home-made dual stage amalgamation. The cell output is connected to tube packed with activated charcoal to recover mercury vapor generated from the analytical system. Hg-free argon 99.999% (Singapore Industrial Gas) was used as carrier gas at a flow rate of 120mL min^{-1}.

All reagents used in the studied were analytical grade: Hg, $NH_2OH.HCl$, $HAuCl_4$, NaOH, HCl (37%), methanol from Merck, H_2SO_4 (98%), H_2O_2 (30%), acetone from Merck; 3-aminopropyl-trimethoxysilane (APTMS) from Sigma Aldrich.

Gaseous mercury standard was prepared by placing high purity Hg^o into glass flask, fitting with septum. The flask was thermo-stated and the temperature was monitored with an accuracy of 0.1^0C.

Glassware and Teflon containers were cleaned by soaking in BrCl 0.02N, then with HCl solution and rinsed with distilled water prior to use.

2.2 Dual amalgamation system

The design of dual gold trap and the schematic diagram of the analytical system is shown *Figure 1*. Two gold traps named sample trap and analytical trap, each packed with 0.17g gold coated sand fitted with two quartz wool plugs at both ends, were interconnected in series by a short Teflon tubing as a connector. The gold traps were wounded by 0.3Ω Cr-Ni resistors heated by a 11V power supply. The sample trap, which could be replaceable, was used to collect Hg^o from the emission source. The analytical trap was used to focus time-resolved released from the sample trap. An injection port, used for input of gaseous mercury standard, made by a glass T with a GC septum on the side arm and the main arms were connected in-line between the carrier gas supply and the dual gold trap. A charcoal trap and a gold trap were used to purify the Ar carrier gas from contaminated mercury. The two purified traps were replaced twice for every 6-m^3 Ar cylinder. The vent of the detector was connected with a charcoal trap (not shown in *Figure 1*) to retain discharged mercury. Teflon tubing was used throughout the system to minimize contamination and memory effect of mercury.

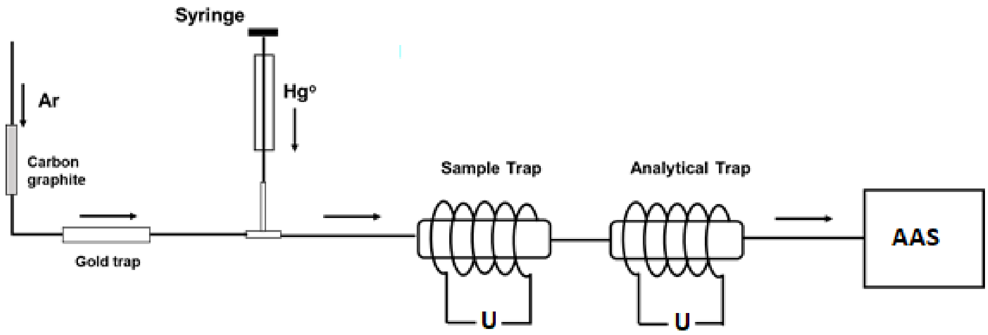

Fig. 1 Dual gold-coated sand trap coupled AAS

2.3 Preparation of gold coated sand

Gold-coated sand was prepared as described elsewhere with some modification[6,7].

Surface activation

The sand with grain size between 500 – 1000μm was first sieved through corresponding sieves, then underwent preliminary cleansing with H_2SO_4 1:1 (v/v) solution in an ultrasonic bath until the washing solution was clean. The sand was subsequently ultra-sonicated with acetone, MeOH, MeOH:HCl=1:1 (v/v), H_2SO_4 then rinsed with distilled water and finally with isobutanol. The sand was then dried and activated in piranha solution (H_2SO_4:H_2O_2=7:3 (v/v)) in 6 hours. To facilitate surface-coating with nano-Au particles (AuNPs), the –OH silanol group was functionalized by amine groups. This was carried out by mixing 30mL (3-aminopropyl) trimethoxysilane (ATPMS) 1 % in methanol per 6-gram sand batch and mixed on an orbital shaker in 16 hours. Note that pH check by litmus paper is essential in every step before another chemical is added.

Nano-gold coating

The pH of a 200mL $HAuCl_4$ 10mg L^{-1} was firstly adjusted to 7 ± 1 using NaOH 1% solution. Next up, the solution was heated until initially boiled, then 5mL of sodium citrate 1% was slowly added for chemical reduction. The AuNPs would form as the solution is heated, and when the suspension became dark red and cooled, the modified sand was added, kept mixing on the orbital shaker for another 6 hours. This step was expected to form a single even layer of gold atoms on the amine-functionalized sand surface.

Additional coating

To ensure a solid gold coating, three further Au layers were added on the nano-coated sand, one layer using 20mL Au(III) 500mg L^{-1} with the other two of 20mL Au(III) 250mg L^{-1}. After pH adjustment of the Au(III) solution to 6.8 – 7.2 using HCl and NaOH, the solution became colorless and the modified sand was added. 0.5mL of $NH_2OH.HCl$ 0.22M solution was quickly added for chemical reduction to Au(0), while the mixture was vigorously and manually shaken for the first 5 minutes, following up by another 25 minutes shaken at 60rpm on the orbital shaker. The coating procedure is completed when the Au solution became colorless, then the coated sand was rinsed with bi-distilled water (≥3times) following by heating at 260^0C/4 hours. The procedure is then performed similarly for two other Au layers, using the Au (III) 250mg L^{-1} solution.

Conditioning of gold-coated trap

The newly-prepared gold trap was first conditioned by at least five cycles of a three-step procedure: (i) a volume of Hg^0 saturated air (19.85 ng Hg^0 mL^{-1} at 25.0^0C) was passed over a gold trap, following by (ii) subsequent heating at ~500 ^{0}C to release mercury vapor, and (iii) the sensitivity check of gold trap between cycles was carried out until the slope value remained stable.

For each activation cycle, an increasing sequence of Hg^0 saturated air volume at 0.5 – 2 – 4 – 6 – 8 – 10mL was injected into the gold trap, following by subsequent thermal desorption at 500 – 600^0C until all quantitatively removed. The activation cycles are conducted at both ends of the gold trap until a stable slope value of the sensitivity check is recorded and specified for that particular gold trap. After activation, the trap is sealed at both ends by Parafilm® membrane and double-bagged until use.

2.4 Atmospheric mercury sampling

Sampling sites

In this study, mercury was sampled at two locations, one was outside of the laboratory at the second floor (Building B) surrounded by higher building around and the other was on the top of one of the highest buildings of the university (Building E). The former place allows to evaluate local mercury emission from the activities of laboratories around in the campus while the latter can provide an indication of atmospheric mercury in Hochiminh city.

Sampling procedure

Gaseous elemental mercury (GEM) was sampled using home-made two-stage gold trap following Method IO-05 (Sampling and Analysis for Atmospheric Mercury – USEPA 1999). A set of sampling trap consisted two gold traps: main sample trap and the breakthrough trap acting as a backup. The breakthrough trap was made of in the same manner as the sample trap and used to recover Hg^o unretained from the sample trap. The sampling flow rate was maintained around 0.2L min^{-1} for each trap set. A soda-lime trap was placed in front of the sampling traps to remove water vapor, acidic gases and other interfering chemicals, that might hamper the amalgamation efficiency of the gold layer, from the sampled air. The granular soda-lime trap was packed in Teflon tube and kept between quartz wool plugs at both ends. Particulate-bound Mercury (PBM) was removed from the sampled air by the glass filter and quartz wool plugs in the soda-lime trap. Reactive gaseous mercury (RGM) is sticky and can be retained by the soda-lime trap (*Figure 2*). The sampling system was set up and checked to ensure for its tightness. The sampling parameters (sampling time, flow rate) and environmental conditions (temperature, weather, wind flow and direction) were recorded. GEM was collected for 8-10 hours during daytime (8am – 5pm) and nighttime (9pm – 7am). Site blanks were also made to control the sampling quality.

After sampling, two gold traps were sequentially analyzed in AAS system.

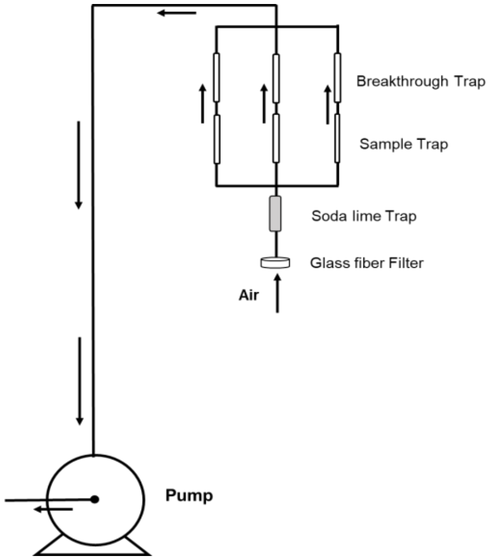

Fig. 2 Atmospheric mercury sampling system in lab

2.5 Analysis

Calibration

Certain volumes of elemental gaseous mercury (corresponding to 0.15 – 3ng) were taken by a 100µL gas tight syringe (SGE, Australia) from a 4L thermo-stated glass bottle containing 5g acid-purified liquid mercury and injected into an injection port in front of the gold trap (*Figure 1*). The amounts of mercury were calculated based on the temperature of the mercury saturated air which was accurately measured to 0.1 ^{0}C [8, 9].

Sample measurement

After the injection of gaseous mercury, the first gold trap was kept standstill for 25 seconds followed by heating until 600 ^{o}C for 15 seconds then cooled. Waited for another 40 seconds, the second trap was then heated to 600 ^{o}C to release mercury vapor to the AAS for measurement. Analytical signal was recorded and integrated as peak height and peak area.

For the analysis of atmospheric mercury, the analytical (main) trap and the breakthrough (recover) trap were separately measured then the corresponding amounts of mercury were combined.

3 Results and discussion

3.1 Activated gold surface

It is well known that amalgamation is the sorption mechanism of Hg^o vapor onto gold surface. When amalgamation occurs, elemental Hg atoms replace with Au atoms in the Au crystalline lattice to form a thin layer of amalgam on the surface. If the number of Hg atoms increase, Au atoms deeply penetrate under the surface. When the amalgam is heated to elevated temperatures, the “alloy” is destroyed to release mercury atoms leaving tiny holes on the gold surface. This results an increase in surface area of gold i.e. increase in the amalgamation efficiency as well as retention capacity of the sorbent. The surface area was raised which mercury capture efficiency increased[10]. In this study, the best capacity was achieved around the amounts of mercury of 260 ng which was used for gold trap activation (*Figure 3*).

The maximum amount of Hg required to saturate the gold trap containing of 0.17g gold coated sand was about 260 ng corresponding to 1.5µg Hg/1 g sorbent. This capacity was much exceeding the usual amounts of mercury sampled from atmosphere ensuring the use of this sorbent in sampling of mercury in ambient air with low risk of sample loss. The amount of 0.17 g gold-coated sand in a 3-mm id quartz tube to form a 2.5cm bed length was relevant to the thermal desorption system.

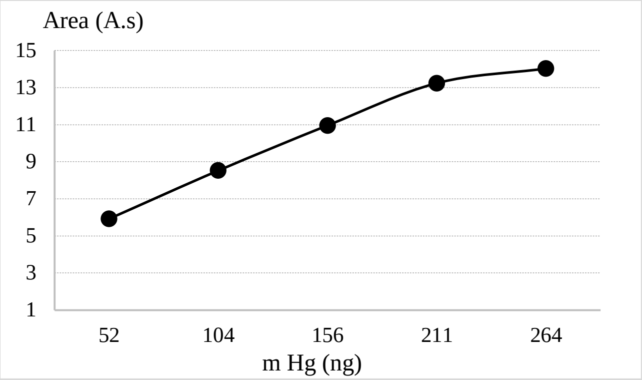

Fig. 3 Conditioning gold trap (12cm long, 0.35mm id packed with 0.17g gold coated sand), with various amounts of mercury

3.2 The performance of gold coated sand as sorbent for sampling of atmospheric mercury

The synthesized gold coated sand showed excellent adsorbent for elemental mercury in standard conditions e.g. short time sampling for gaseous mercury in clean air. This material has been used in our lab as analytical trap to enrich ultra -trace mercury sample prior to analysis. A trap packed with this material could be used for more than one thousand sorption – desorption cycles with any noticeable degradation in sensitivity. However, the actual sampling conditions onsite were far from the ideal ones because numerous reactive chemicals that hamper the amalgamation efficiency are existing in sampling medium[11]. In consequence, the life time of the trap can be reduced significantly indicated by the breakthrough of mercury from the sampling trap. The goal coated sand prepared in this study showed very reasonable sampling efficiency even after 20 sampling cycles during six months usage (*Figure 4*). The breakthrough of the analytical (main) trap of around 10 % can be considered acceptable for an accurate analysis since all mercury retained on the two traps (main and recover traps) were accounted for.

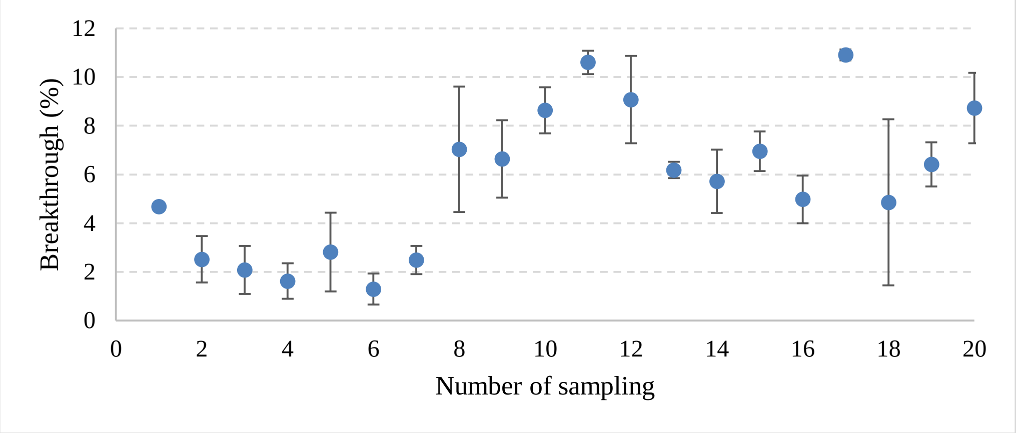

Fig 4 Breakthrough of mercury (%) on sampling trap packed with goal coated sand. Trap: 3.5 mm id, 12 cm long quartz tube containing 0.17 g sorbent, sampling at 200 mL.min^{-1}for 8 hours. Error bars represents the standard deviations of breakthrough from three parallel sampling traps.

3.3 Dual-amalgamation coupled AAS

The gold trap should efficiently focus mercury vapor that is kinetically released from the sample and form a symmetric, smooth absorption profile for highly accurate integration. This could be possible for samples that generate non-reactive substances interfering the amalgamation process on the sorbent surface. It can be seen from *Figure 5A* that absorption peak profile obtained by single trap was symmetric and smooth indicating the excellent role of single gold trap as focusing device for standard samples. The use of dual trap was not necessary. However, for the air sample collected at Nhieu loc river side, the absorption profile obtained from the single trap desorption system was broad with distortion making the integration difficult and inaccurate. It could be explained that the air at the sampling place consisted numerous chemicals that might compete with mercury atom and partly occupy the surface of the gold trap. The amalgamation band was therefore defocused to the whole surface area of sorbent in the trap during long sampling time (120 minutes in this case). When the trap was heated, the amalgam closed to the wall of the trap was heated, decomposed and release mercury before the amalgam at the center. This resulted in peak profile broad and distorted. In the dual trap desorption system, the second trap efficiently collected all mercury from the first trap (sampling trap) in quite a short time (40 seconds). The amalgam band was therefore focused and once this amalgam is heated, a sharp, symmetric and smooth absorption profile was achieved (*Figure 5B*).

3.4 Analytical performances

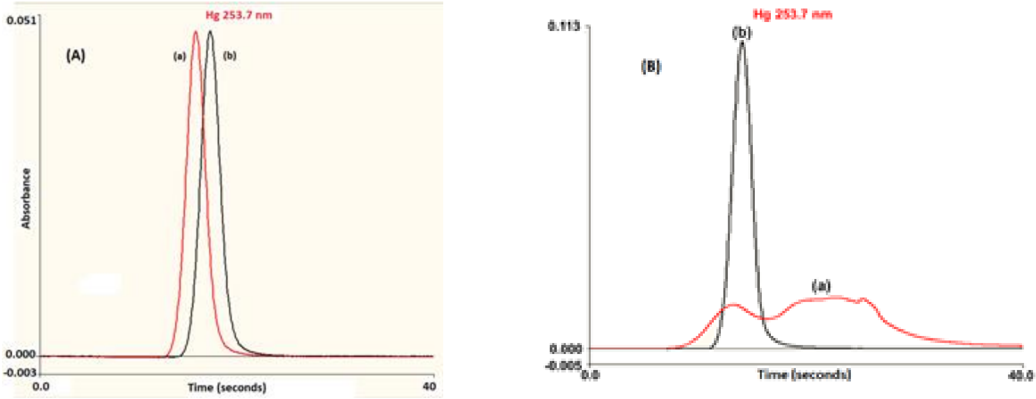

Fig. 5 Absorption profiles of Hg obtained from (A) standard solution and (B) a typical sample collected at Nhieu Loc canal at 300 mL.min^{-1} for 120 minutes, absorption trap was desorbed as (a) single gold trap, (b) dual gold trap for AAS measurement. The appearance time of peak was shifted for clarity.

Calibration curve was made by gaseous Hg standard in air instead of aqueous Hg standard as usual. Accurate amounts of mercury were quantitively and rapidly transported to the gold trap. All problem related to the use of aqueous Hg standard including inaccurate Hg concentration due to adsorption or volatility, non-quantitative chemical generation of elemental mercury, purging efficiency, volatile reactive vapors interferences and time consuming were avoided. The calibration was therefore quite straightforward to made with R^2 value close to 1 and the intercept close to zero. ($A=0.0916m_{Hg} - 0.0026$; $R^2=0.9995$). The control chart was performed every working day to ensure analytical system operated properly and stable (*Figure 6*).

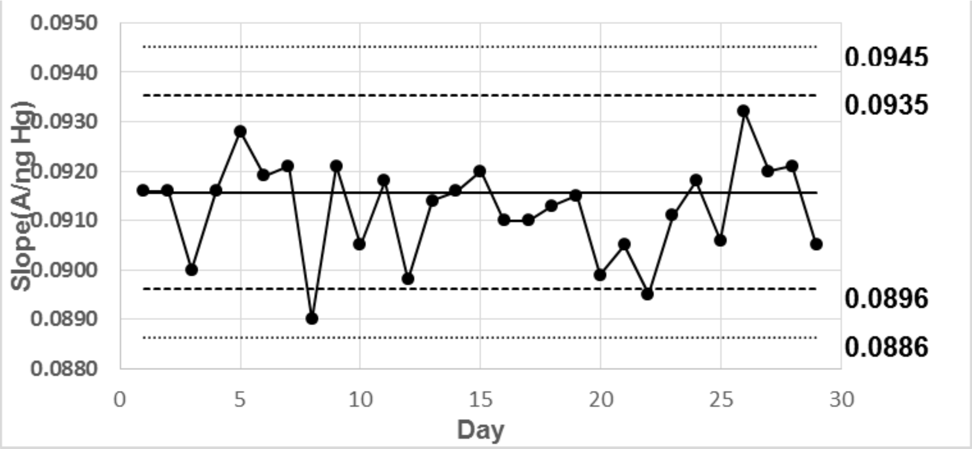

Fig. 6 Control chart for home-made dual gold trap coupled AAS

The instrument detection limit (IDL) and instrument quantification limit (IQL) were 3.9 and 13 pg Hg, respectively. For a sample with sampling time of 8 hours at 200 mL min^{-1}, an estimated method detection limit (MLOD) and method quantitation limit (MLOQ) of 0.04 ng m^{-3} and 0.14 ng m^{-3}, respectively, could be achieved. The IDL achieved in this study were comparable with those obtained from well-known commercial systems with AAS detection such as Tekran, Lumex or Gardis[12]. Comparing to the background level of mercury in atmosphere of 1.1 – 1.7ng m^{-3}[13], this method could therefore be applied for the analysis of atmospheric mercury.

The analytical procedure including the dual gold trap and the detector was proved to be stable during a long-term usage (*Figure 6*).

3.5 Analysis gaseous mercury in air

Mercury in ambient air at the building B corridor just outside our laboratory was considered more influenced by the laboratory activities than the transportation of mercury by wind from other part of the city. The concentrations collected at this place were relatively low when no sample treatment occurred the laboratory (*Table 1*). We noticed that high levels of Hg measured on 05/03, 07/03 and 09/03 were coincident with the time that digestion of soil samples for heavy metal analysis was performed.

Table 1 Mercury concentration at B lobby

Day	C (ng m^{-3})	SD	Day	C (ng m^{-3})	SD
05/03/2018	**8.1**	0.63	15/03/2018	**4.6**	0.35
06/03/2018	**4.3**	0.12	16/03/2018	**3.5**	0.22
07/03/2018	**5.2**	0.45	17/03/2018	**4.4**	0.27
08/03/2018	**4.2**	0.25	18/03/2018	**3.3**	0.28
09/03/2018	**6.5**	0.55	19/03/2018	**3.7**	0.27
14/03/2018	**4.2**	0.31	21/03/2018	**2.7**	0.11

Relatively low concentrations of GEM were obtained at the top of Building E where the no laboratory occurred nearby (*Table 2*).

Table 2 Mercury concentration at E terrace

Day	Day (8 am – 5pm)		Night (9pm – 7am)	
	C (ng m^{-3})	SD	C (ng m^{-3})	SD
24/05/2018	**2.6**	0.09	**2.0**	0.41
28/05/2018	**3.1**	0.32	**2.4**	0.27
29/05/2018	**2.3**	0.27	**2.8**	0.38
30/05/2018	**2.5**	0.18	**2.0**	0.35
31/05/2018	**3.1**	0.02	**2.7**	0.28

The concentration of GEM at this place is considered to indicate the pollution of mercury in city atmosphere. The atmospheric mercury was likely different between daytime and nighttime. However, more thorough investigation should be needed for more profound interpretation about the environmental characteristics of atmospheric mercury.

(i)After sampling proceed ended, offed pump and two gold-coated sand traps were analyzed with AAS.

Hg concentrations at the building B corridor adjacent of laboratory (4.6 ± 1.5ng m^{-3}) was relatively higher than those measured at open air on top of Building E (2.7 ± 0.4 in daytime and 2.4 ± 0.4 in nighttime). The results indicated that the release of Hg to ambient air due to laboratory activities occurred and appropriate action should be taken to eliminate the discharge.

4 Conclusions

In this study, laboratory made gold-coated sand was proved to be successfully applied as sorbent for the sampling of atmospheric mercury. The dual gold trap was fabricated and efficiently worked to improve the accuracy of the measurement of Hg in ambient air. The results of this study provided the local environmental and analytical researchers a useful tool to expand their researches in atmospheric mercury.

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Nghiên cứu phương pháp lấy mẫu bằng vật liệu cát phủ vàng và phân tích thủy ngân nguyên tố trong không khí bằng hệ thống hai bẫy vàng ghép nối đầu dò hấp thu nguyên tử

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Tóm tắt Trong nghiên cứu này, vàng được phủ lên bề mặt cát sau xử lí qua phản ứng khử Au(III) bằng dung dịch NH_2OH và tính chất của vật liệu đã được kiểm tra trước khi sử dụng. Hệ thống hai bẫy cát phủ vàng ghép nối với đầu dò AAS được thiết kế và tối ưu nhằm hỗ trợ cho quá trình phân tích thủy ngân trong các mẫu thực tế có hàm lượng siêu vết (IDL và IQL của hệ thống lần lượt là 3.9 và 13 pg Hg). Hg^o trong không khí được bắt giữ trên hệ thống lấy mẫu hai bẫy vàng liên tục trong 8h với tốc độ khí 200 $mL.phút^{-1}$ sau đó hàm lượng Hg được phân tích trên hệ thống phân tích tại phòng thí nghiệm với MDL và MQL của phương pháp là 0.04 và 0.13 $ngHg.m^{-3}$. Hệ lấy mẫu và phân tích tự thiết kế được sử dụng để xác định hàm lượng thủy ngân trong không khí tại hai điểm của trường Đại học Khoa học tự nhiên với hàm lượng đo được nằm trong khoảng 2.7 – 8.1 $ngHg.m^{-3}$.

Keywords thủy ngân nguyên tố, không khí, hệ hai bẫy vàng, phổ hấp thu nguyên tử