DEVELOPMENT OF AN AIRBORNE LEAD ANALYSIS KIT AND ITS APPLICATION

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Abstract. We developed a method to analyze airborne lead concentrations in the field. It was a modification of the colorimetric method using the reaction between 4(2-pyridylazo)-resorcinol (PAR) and lead with cyanex302 in an acid medium to reduce interfering metals. The lead concentration was detected with a photometer made in Thailand. The developed method uses an impinger containing 1% nitric acid solution as an absorbing agent to collect airborne lead at a flow rate of less than or equal to one liter/minute. Cyanex302 solution in toluene was used to extract metals from the samples and 0.1M nitric acid was used to extract just lead. The lead solution was reacted in 0.5 ml of 0.03% PAR solution, with 1 ml ammonium chloride buffer; the absorption of this solution was measured by a photometer. The results show the limit of detection (LOD) was 0.01 mg/l. The limit of quantification (LOQ) was 0.03 mg/l. The percent recovery of the lead concentrations of 0.05 - 3.0 mg/l was 94.0 to 103.5%. The precision presented as %CV ranged from 0.65 to 10.27%. Lead concentration in a lead smelting factory detected by this method was not significantly different from that detected by the NIOSH method: 7,303 at a 95% confidence level.

Key words: airborne lead analysis, colorimetric method

INTRODUCTION

In Thailand, lead is used in many industrial processes, such as ore mining, lead smelting, car batteries, the paint industry, ceramic manufacturing, solder work and the ingot and plating industry. Lead is also used in petroleum refining and pesticide manufacturing. These processes result in lead exposure, a health risk for humans. Lead can contaminate the environment, especially in ambient air in the vicinity of the industry. Thirty-five to 50% of lead in the air can be absorbed into the blood during inhalation depending on the particle size and the rate of respiration (Putnam, 1986).

Highly reliable methods of airborne lead analysis are flame atomic absorption spectrometry (AAS) (NIOSH, 1996), graphite furnace atomic absorption spectrometry (GFAAS) (NIOSH, 1996), anodic stripping voltametry (ASV) (Ashley *et al*, 1998), inductively coupled plasma-atomic emission spectroscopy (ICP-AES) (NIOSH, 1996), X-ray fluorescence spectroscopy (XRFS) and isotope dilution mass

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spectrometry (IDMS) (Heumann, 1980). These methods are usually performed in the laboratory. The equipments needed to perform these tests are expensive and need a professional to perform the analysis and maintenance.

Airborne lead analysis in the field is usually semi-quantitative, using a method such as that found in the Merck test kit (NIOSH method 7700) (NIOSH, 1996), or the Lamotte method 7667 (Lamotte Company, 2009). This report describes an inexpensive, simple test developed to provide rapid results. It is essential for test kits to provide precise, accurate, and sensitive results for identifying potential lead exposure.

In this study, a colorimetric method was developed and used for lead analysis. The reaction between 4(2-pyridylazo)-resorcinol (PAR) and lead was used to detect lead in the air because of the stability of the lead complex. 4(2-pyridylazo)-resorcinol (PAR) develops a color in the presence of even small amounts of lead. Cyanex302 is used to extract and separate the lead from the sample under acid conditions. There is a short extraction reaction time and the concentration of the lead can be detected using a photometer. The photometer used in this study was developed in Thailand. We compared this method with the NIOSH Method 7303 for the analysis of airborne lead in a lead smelting factory.

MATERIALS AND METHODS

Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP–OES, Spectro CirosCCD, Spectro, Germany) was used to analyze for lead and other metals. The UV Visible Spectrophotometer (serial no. 6127-006, Hitachi, Japan) and Photometer (serial no.200, 1021-525, Bangkok High Lab, Thailand) were used to analyze for lead.

The standard solutions of lead, copper, cadmium, silver, cobalt, nikel, zinc and mercury were purchased from Merck, Germany. Cyanex302 (diisooctylthiophosphinic acid) and PAR [4-(2-pyridylazo) resorcinol] were purchased from Fluka, Canada and Austria, respectively. The other reagents were of analytical grade.

Ammonium chloride buffer

Thirty-three grams of ammonium chloride was weighed and dissolved in 200 ml distilled water and mixed with 207 ml ammonia solution (d = 0.880 g/ml) (or an equivalent amount of diluted ammonia) which was diluted to a final volume of 500 ml with distilled water.

0.03% 4-(2-pyridylazo) resorcinol (PAR) solution

PAR was weighed (0.03 g) and diluted in 100 ml absolute ethanol.

0.005 M stock solution of cyanex302

Cyanex302 (1.65 ml) was diluted with toluene to a total volume of 1,000 ml.

Nitric acid concentration for airborne lead absorbing solution

Lead monoxide (1.5 mg) was dissolved in 20 ml of 0.1, 0.5, 1.0, 1.5 and 2.0% concentrations of nitric acids. After 15 minutes, the lead monoxide solution was filtered through a No.1Whatman filter. The filtrate was analyzed for lead concentration using the ICP.

Effect of pH on lead extraction by cyanex302

Twenty milliters of 1.0 mg/l lead solution in 1% nitric acid was adjusted with 3% or 15% ammonia solutions until the pH was 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10. The solutions were then transferred to a 100 ml separatory funnel, and 5 ml of 0.005 M cyanex302 solution in toluene was added and the solution was gently shaken for 1 minute. The layers were allowed to separate and the cyanex302 containing lead was carefully removed and extracted twice with 20 ml 0.1 M nitric acid. The resulting lead was extracted in nitric acid, separated and measured by ICP.

Appropriate pH for lead complex formation with PAR

Ten milliters of 1 mg/l lead solution in 1% nitric acid was placed in a 15-ml test tube. The pH of lead solution was adjusted with 3% or 15% ammonia solutions to obtain pH levels of 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and 12. Zero point five milliliter 0.03% PAR solution was added to each tube, mixed and left for 1 minute, then the absorption of the colored lead-complex solution was measured at 525 nm wavelength using an UV visible spectrophotometer.

Stability of PAR solution

Ten milliters of 1 mg/l lead solution was placed in a 15 ml test tube; 1 ml ammonium chloride buffer and 0.5 ml 0.03% PAR solution were added and mixed. After approximately 1 minute, the absorbance of the colored lead complex was measured at 525 nm by UV visible spectrophotometer. The stability of the PAR solution was tested over 10 replications at 0, 30, 60, 90, 120, 150 and 180 days after storage at room temperature.

Stability of lead complex

The experiment was performed by placing 10 ml lead standard solution at 0.05, 0.1, 1, 2 and 3 mg/l into 15 ml test tubes; 1 ml ammonium chloride buffer and 0.5 ml of 0.03% PAR solution were added, mixed and left for 1 minute and the absorbance was measured at 525 nm by UVvisible spectrophotometer. The absorption of the colored lead complex at various lead concentrations was measured after they were kept for 1, 15, 30, 60 and 120 minutes.

Calibration curve of lead solution by UV visible spectrophotometer

The 10 ml lead solution at concentrations of 1.00, 2.00, 3.00, 4.00 and 5.00 mg/l in 1% nitric acid were placed in 15 ml test tubes; 1 ml ammonium chloride buffer and 0.5 ml of 0.03% PAR solution were added, mixed and left for 1 minute and the absorbance was measured at 525 nm by UV visible spectrophotometer. The calibration curve was performed to find the appropriate range of lead concentrations for setting up a calibration curve for standardization of the photometer.

Calibration curve of lead solution by photometer

The lead standard solution was prepared at concentrations of 0.01, 0.02, 0.04, 0.05, 0.06, 0.08, 0.10, 0.20, 0.40, 0.50, 0.60, 0.80, 1.00, 1.20, 1.40, 1.60, 1.80, 2.00, 2.20, 2.40, 2.60, 2.80 and 3.00 mg/l in 1% nitric acid solution; 1 ml ammonium chloride buffer and 0.5 ml of 0.03% PAR solution were added, mixed and left for 1 minute and the absorbance was measured at 525 nm by a photometer. The calibration curve was used to calibrate the photometer.

The sampling of airborne lead

An impinger containing 20 ml 1% nitric acid solution was connected to a personal pump at a flow rate of ≤ 1 liter/minute; the air sample was collected from 120 to 480 minutes.

The airborne lead analysis method

The 20 ml absorbing solution containing lead was then transferred to a 100 ml separatory funnel. The impinger was rinsed with a small amount of 1% nitric acid solution and then put into a 100 ml separatory funnel. The solution was adjusted to pH 5 with 3% or 15% ammonia solution, extracted with 5 ml 0.005 M cyanex302 solution in toluene, then the lead was re-extracted twice with 20 ml 0.1 M nitric acid. Ten milliters of the extracted solution was mixed with 1 ml ammonium chloride buffer and 0.5 ml of 0.03% PAR solution and left for 1 minute; then the absorbance was measured at 525 nm with a photometer.

Reliability of airborne lead analysis method

Specificity. Standard solutions containing 1 mg/l lead with 0.05, 1.0 and 10 mg/l interfering metals, (cadmium, nickel, copper, cobalt, silver, zinc, iron and mercury) were mixed with 1% nitric acid solution. The 20 ml mixed standard solution was transferred to a 100 ml separatory funnel, then airborne lead analysis was carried out using a photometer. The 10 ml lead extracted solution was also determined for the interfering metal by ICP.

Accuracy and precision of the lead analysis method. The lead standard solution was prepared at concentrations of 0.05, 0.06, 0.5, 1, 2 and 3 mg/l in 1% nitric acid solution. Twenty milliters of each of the lead standard solutions was transferred to a 100 ml separatory funnel then analyzed for lead using the photometer. The analysis was performed for 10 replications. The accuracy and precision were calculated in terms of percent recovery and coefficient of variation. Between-day comparison was carried out for 3 days.

Detection limits of the lead analysis method. The lead standard was prepared at lead concentrations of 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09, 0.10, 0.11 and 0.12 in 1% nitric acid solution. Twenty milliters of each lead standard solution was transferred to a 100 ml separatory funnel and the lead was detected using a photometer. The analysis was performed in 10 replications. The detection limit was calculated following NIOSH (1994).

Airborne lead sample collection in lead smelting factory

In this study, a lead smelting factory was selected for a field trial. This factory uses old batteries as raw material for smelting. Airborne lead samples were collected and evaluated with the newly developed method and NIOSH method 7303. Thirty-one sampling points were set up in different areas covering low, medium and high concentrations of airborne lead in the factory. The sampling areas were classified into 3 areas: office building area, maintenance area and production area. The duration of sample collection ranged from 120 to 480 minutes. The filters collected for the NIOSH method 7303 were digested with nitric acid and analyzed by two methods; ICP and the newly developed method using a photometer. The absorbing solution collected by the developed method was also analyzed using the two methods.

Data analysis

The lead concentrations collected by filter cassette and impinger were analyzed with the ICP and photometer. The results were compared with the paired *t*-test at a 95% confidence limit or the Wilcoxon signed-rank test. When comparing the NIOSH method and the developed method, the independent *t*-test was used.

RESULTS

Appropriate nitric acid concentration for airborne lead absorbing solution

The recovery of lead monoxide was 47.2% and 91.4% at 0.1% and 0.5% nitric acid, respectively. The recovery of lead monoxide was 100% when the concentration of nitric acid ranged from 1.0% to 2.0%. Therefore, 1% nitric acid solution was used for dissolving the lead standard and absorbing airborne lead.

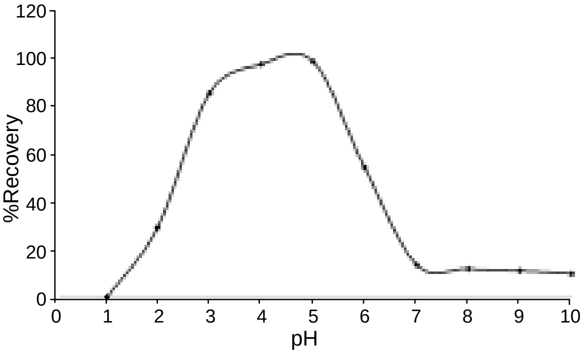

Fig 1–Appropriate pH for lead extraction with Cyanex302.

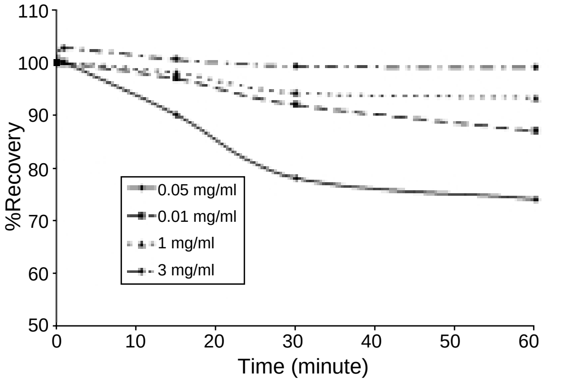

Fig 3–Stability of lead complex formation at lead concentrations of 0.05, 0.1, 1.0 and 3.0 mg/l.

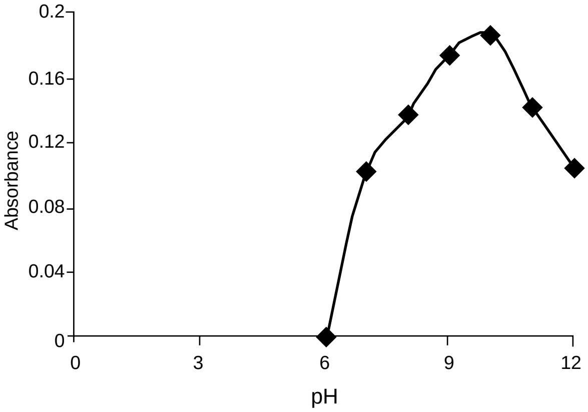

Fig 2–Appropriate pH for lead complex formation.

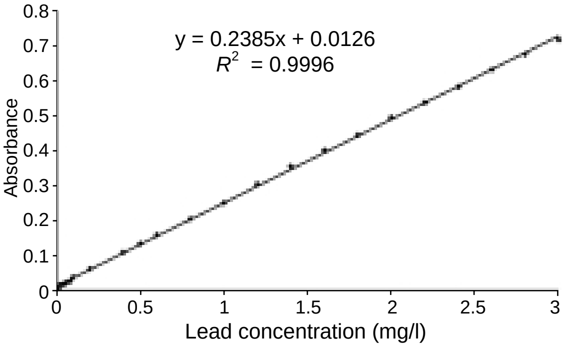

Fig 4–Calibration curve of lead complex using photometer serial 200.

Effect of pH on lead extraction by cyanex302

The lead recovery at pH levels of 3, 4, 5 and 6 were 85.3, 97.5, 98.6 and 54.5% as shown in Fig 1. Therefore, the appropriate pH for lead extraction with cyanex302 was pH 5.

Stability of PAR solution

The recovery of lead analysis after storage as PAR for 180 days was 102%, which was not different from that on the first day. The PAR solution can be stored for up to 180 days at room temperature.

Appropriate pH of the lead complex formation with PAR

The pH of the 1 mg/l lead standard solution in 1% nitric acid was tested from 1 to 12 and the absorbance of the colored lead complex was measured at 525 nm by UV visible spectrophotometer is shown in Fig 2. The absorption of the lead complex increased at a pH of 6 and reach maximum absorption at a pH of 10.

Stability of lead complex

The lead complex was stable at various

Table 1
The specificity of the test showing % recovery of lead by photometer and other metals.

Metal	0.5 mg/l metal added Mean ± SD, (*n*=10)		1.0 mg/l metal added Mean ± SD, (*n*=10)		10.0 mg/l metal added Mean ± SD, (*n*=10)	
	Lead	Other metal	Lead	Other metal	Lead	Other metal
Cd	102.8±0.03	0.18±0.0002	114.4±0.08	0.54±0.0013	132.6±0.03	1.47±0.0025
Ni	103.2±0.03	0.26±0.0003	107.6±0.06	1.35±0.0013	124.9±0.02	1.67±0.0015
Cu	103.4±0.02	0.1±0.0001	97.4±0.05	0.10±0.0002	93.6±0.02	0.17±0.0002
Co	103.9±0.03	0.14±0.0002	101.6±0.05	0.07±0.0001	119.8±0.04	0.14±0.0003
Ag	102.0±0.04	0.27±0.0002	101.0±0.06	0.13±0.0001	103.3±0.02	0.17±0.0002
Zn	103.3±0.03	2.00±0.017	111.0±0.07	2.21±0.0039	127.6±0.02	2.75±0.0032
Fe	102.6±0.02	0.53±0.0003	102.9±0.05	0.31±0.0003	103.4±0.01	0.28±0.0002
Hg	103.5±0.03	0.68±0.0003	101.6±0.05	0.39±0.0003	113.4±0.01	0.81±0.0009

lead concentrations (0.05-3.00 mg/l) for 1 to 15 minutes as the % recovery ranged from 100.0 to 102.7% (Fig 3). The lead complex at lower lead concentrations of 0.05 and 0.1 mg/l had lower recovery than those at 1.0 and 3.0 mg/l lead concentrations. Therefore, lead complex at high lead concentrations was more stable than those at low concentrations.

Calibration curve for setting up the spectrophotometer

Appropriate range of lead calibration curve. The color of the lead complex changed from orange to red when the concentration of lead increased and the absorbance was plotted with the lead concentration. The calibration curve displayed a linear relationship at lead standard concentrations from 0 to 3.0 mg/l. At lead concentrations above 3.0 mg/l, the slope of the calibration curve was almost steady.

Calibration curve of lead for setting up the photometer. The range of lead concentration at 0 - 3.0 mg/l was used to set up the photometer for analysis of lead in the field. To obtain reliable results, the number of calibration points should be as high as possible. Therefore, lead standard concentrations of 0.00, 0.01, 0.02, 0.04, 0.05, 0.06, 0.08, 0.10, 0.20, 0.40, 0.50, 0.60, 0.80, 1.00, 1.20, 1.40, 1.60, 1.80, 2.00, 2.20, 2.40, 2.60, 2.80 and 3.00 mg/l were used to set up the photometer. The calibration curve showed a linear relationship as shown in Fig 4. The equation of the linear relationship was $y = 0.239x + 0.013$; where y was the absorbance at 525 nm and x was lead concentration (mg/l). The correlation coefficient (R^2) was 0.9996.

Reliability of the airborne lead analysis method

Specificity. The heavy metals used for the specificity test were cadmium, nickel, cupper, cobalt, silver, zinc, iron and mercury at the concentrations of 0.05, 1 and 10 mg/l. Zero point five mg/l of cadmium, nickel, copper, cobalt, silver, zinc, iron and mercury and 1 mg/l of cobalt, silver, iron, mercury and 10 mg/l of silver and iron did not interfere with the 1 mg/l lead concentration (Table1).

Accuracy and precision. The between-day accuracy and precision of the method are presented in Table 2. The accuracy and precision of the analysis method in terms of percent recovery ranged from 94.0 to

Table 2
Between-day assay to determine the accuracy and precision of the lead analysis method.

Lead standard mg/l	Lead measured mg/l, (*n*=20) Mean ± SD	% Recovery	%CV
0.05	0.05 ± 0.0048	94.0	10.3
0.06	0.06 ± 0.0052	100.8	8.6
0.5	0.51 ± 0.0081	102.0	1.6
1.0	1.03 ± 0.0216	103.5	2.1
2.0	2.02 ± 0.0167	101.1	0.8
3.0	3.04 ± 0.0198	101.3	0.7

103.5% and the %CV ranged from 0.7 to 10.3% at lead concentration ranging from 0.05 to 3.0 mg/l, respectively. The lead analysis method gave a reliable result at concentrations ranging from 0.05 to 3.0 mg/l.

Detection limit. The analytical LOD and LOQ were estimated from the regression curve to be 0.010 and 0.033 mg/l, respectively. The LOD and LOQ were defined as the atmospheric concentration divided by the sampling volume. Assuming the samples were collected at 1 liter/minute for 8 hours, the air volume was determined to be 480 liters. Therefore, the airborne lead could be detected by this method at the lowest and the reliable concentrations of 0.0004 and 0.0014 mg/m^3, respectively.

Field application

Comparison of airborne lead concentrations using the NIOSH method 7303 and the developed method. The lead concentrations obtained with the two methods were analyzed using the ICP and a photometer (Table 3). The non-detection was assigned to be zero. The Wilcoxon signed-rank test showed there was no significantly different lead concentrations collected by filtered cassette and determined by ICP and photometer among the sampling points in the office building area, the maintenance area and the production area with 95% confidence limit ($p = 0.899$). The lead concentrations collected by impinger and analyzed by ICP and photometer were not significantly different ($p = 0.443$) from each other by paired *t*-test at a 95% confidence limit.

Among the office building area, maintenance area and production area, the results of an independent *t* - test showed the lead concentrations in each area detected with the NIOSH method 7303 and the developed method did not differ from each other significantly (maintenance area, $p = 0.062$; production area, $p = 0.130$). The office building area was not compared due to non-detectable results. The analytical comparison between the NIOSH method 7303 and the developed method revealed no significant differences, the photometer using the developed method detected a range of 0.05 to 3.0 mg/l lead. The photometer gave reliable results, and can be used in the field.

Airborne lead levels in lead smelting factory

Thirty-one sampling points wer e distributed throughout the factory (Fig 5). The production area had the highest airborne lead concentrations (0.006 - 0.139 mg/m^3). The airborne lead concentrations in the

Table 3

Comparison of lead concentration measured (mg/l) between two methods.

Sampling point	NIOSH method with filtered cassette		The developed method with impinger	
	ICP	Photometer	ICP	Photometer
Office area				
1	0.0055	N.D.	0.0090	N.D.
2	0.0050	N.D.	0.0120	N.D.
3	0.0041	N.D.	0.0041	N.D.
4	0.0041	N.D.	0.0041	N.D.
5	0.0041	N.D.	0.0080	N.D.
6	0.0149	N.D.	0.0428	0.05
7	0.0190	N.D.	0.0442	0.05
8	0.0041	N.D.	0.0084	N.D.
9	0.0041	N.D	0.0103	N.D.
Maintenance area				
10	0.0559	0.06	0.0843	0.09
11	0.0139	N.D.	0.0475	0.05
12	0.0389	0.05	0.0523	0.06
13	0.0497	0.05	0.0462	0.05
14	0.0515	0.05	0.1215	0.10
Production area				
15	0.5141	0.53	0.4021	0.38
16	0.2012	0.23	0.2394	0.25
17	0.0593	0.05	0.0598	0.07
18	0.3652	0.35	0.6045	0.55
19	0.2911	0.30	0.4594	0.46
20	1.2522	1.27	1.5715	1.61
21	1.2908	1.32	1.7946	1.78
22	1.0907	1.10	1.3485	1.39
23	0.3661	0.38	0.8010	0.72
24	0.4625	0.48	0.8795	0.86
25	0.6688	0.68	0.5642	0.57
26	0.5906	0.58	0.3841	0.39
27	0.4729	0.46	0.7946	0.82
28	0.4283	0.40	0.6754	0.69
29	0.7716	0.79	1.1044	1.24
30	0.9081	0.88	1.4340	1.45
31	0.9810	0.97	1.5645	1.66

N.D. , Not detected

maintenance area were lower (0.004 - 0.007 mg/m^3). The office building had the lowest airborne lead concentrations (0.0- 0.004 mg/m^3).

DISCUSSION

Lead was soluble in nitric acid (ICPS, 1995). The highest airborne lead concen-

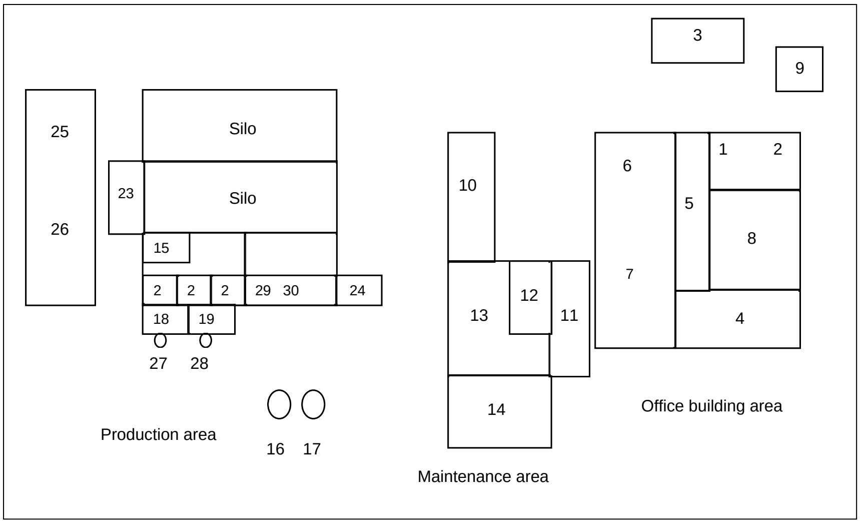

Fig 5–Plant layout showing 31 sampling points.

tration found using the developed method was at an immediately dangerous to life and health (IDLH) concentration of 100 mg/m^3 in 15 minutes. The amount of lead used for testing was 1.5 mg lead monoxide. Nitric acid at a concentration of 1% or higher completely dissolved 1.5 mg of lead. This study used 20 ml 1% nitric acid solution for absorbing solution. The results are consistent with those of Termkeaw (1993).

The duration of air sample collection in the field should be longer than 15 minutes. This gives a longer time for the lead to dissolve in the nitric acid solution. Airborne lead has many forms, but in this study lead monoxide was used because most airborne lead is first formed as an oxide form, then it is combined with carbon dioxide to form lead carbonate, which can also be dissolved in nitric acid.

The lead complex formation was formed at an alkaline pH of 10. We used ammonium chloride buffer at a pH of 10 to control the pH of the lead/PAR complex. Method is similar to that of Merck (2008), LaMotte (2009) and Prohmmee (2003) as shown in Table 4.

The optimum wavelength for measuring the colored lead complex was 515-525 nm. This similar to that of Merck (2008), LaMotte (2009) and Prohmmee (2003) who used 520 nm (Table 4) and Kenichi (Kenichi *et al*, 2003) who used 530 nm. The wavelength of 525 nm was selected in our study due to the specifications of our photometer.

The PAR is solid and is a stable chemical. PAR was dissolved with glacial ethanol and kept in a black tightly closed container at room temperature. The stability of the PAR solution was tested and found to be stable for at least 180 days after preparation.

The PAR-lead complex changes color depending on the concentration of lead. With a low lead concentration, the color is orange. With higher concentrations the color changes to red. The color faded with time and lead concentration, so the color was examined at 1 minute after forming the lead complex.

Table 4
Comparison of lead analysis methods using the PAR colorimetric technique.

Parameter	Merck company method 1.09717.001 (2008)	LaMotte company PAR method Code 4031 (2009)	HACH company PAR method DR- 4000 (2008)	Nopakao Prommee method (2003)	Developed method 2006
Sample	Water	Water	Water	Water	Air
Reading method	Photometer Multi-wavelength	Photometer Multi-wavelength	Photometer Multi-wavelength	Color chart	Photometer Single wavelength
pH	10	10	9	10	10
Wavelength (nm)	520	520	520	419	525
Separation technique	No information	Sodiumdiethyldithio-carbamate for $CaCO_3$	No information	TOPO[a] in sodium salicylate	Cyanex302 in toluene
Range	0.1-5.00 mg/l (10-mm cell) 0.01-1.00 mg/l (50-mm cell)	0-5 mg/l	0-2 mg/l	0.05,1,5 mg/l	0.05-3.0 mg/l
Cell size	10, 50 mm	25 mm	25 mm	-	23 mm

[a] TOPO, Trioctylphosphine oxide

Table 5
Comparison of airborne lead levels by the developed method and other methods.

Parameter	Jariya Termkeaw method (1993)	Lamotte method 7667 (1995)	NIOSH method 7700 (1996)	Developed method (2006)
Technical	Colorimetric	Colorimetric	Spot test kit	Colorimetric
Reagent	Rhodizonate	Dithizone	Rhodizonate	PAR
Reading result	Color chart	Color standard	Color change	Photometer
Solvent-extraction	-	Chloroform	-	Cyanex302 in toluene
Flow rate	-	1 L/min	2 liter/minute	1 liter/minute
Collection method	1% HNO_3 as absorbing solution	Absorbing solution	Filter	1% HNO_3 as absorbing solution
Range	3.6, 5.0 , 6.0 mg/l	0.05-3.0 mg/m^3	Positive ≥ 0.57 mg lead Negative ≤10.2 mg lead	0.05-3.0 mg/l
Sampling time	-	5,10,15,20 minutes	5,120 minutes	Depending on amount of lead

As can be seen in the equation: 2PAR + $Pb^{2+} \rightarrow Pb\text{-}PAR_2$, two molecules of PAR were bound with a lead ion (Snell, 1978). This means that 10 ml of 7.22 mg/l lead concentration was bound completely with 0.5 ml 0.03% PAR solution. The highest lead concentration measured in the studied method was 3 mg/l. Zero point five milliter of 0.03% PAR solution was sufficient to bind 3 mg/l lead concentration.

For setting up the spectrophotometer, the first calibration of the photometer must be carried out by a specialist engineer, but a technician can be trained to calibrate the instrument by himself every 6 months to assure the accuracy of the instrument for measuring lead concentration in air samples. The photometer is efficient in measuring the absorbance of lead in the field. The concentration detected ranged from 0.05 to 3.0 mg/l, which is in the usual range of the instrument. If the concentration of airborne lead is low, the duration of sampling should be longer. The operator can adjust the flow rate and duration of air sampling to obtain a lead concentration within the detection range of the instrument.

This study used cyanex302 as an extraction solvent to extract all metals into cyanex302 organic layers and 0.1 N nitric acid to extract only the lead for analysis. Therefore, cyanex302 was used to eliminate interfering metals from lead analysis; increasing the specificity of the analysis method. The duration of extraction was at 1 minute using the separatory funnel and manual shaking at 100 rounds per minute and left for 5 minutes for complete separation of the aqueous and organic phases. A disadvantage of using cyanex302 is the extraction with the separatory funnel is necessary. This means many separatory funnels must be available at the field site. LaMotte (2009) used sodium diethyldithiocarbamate with the PAR method to remove excess calcium from the water. Merck (2008) and HACH (2008) did not mention how they removed interfering metals before doing lead water for business reasons (Table 5). The disadvantage of using cyanex302 is that toluene is used as a solvent for diluting cyanex302. Toluene has a bad smell and is carcinogenic.

Cadmium, nickel, copper, cobalt, silver, zinc, iron and mercury at concentrations of 0.5 mg/l, did not interfere with lead measurement at 1.0 mg/l. However, Ca, Cr, Al, Mn, Sn, $Cr_2O_7^{2-}$, EDTA, NaCl, Na_2SO_4, $NaNO_3$ can have an effect on measurement of lead concentrations in water. In this study, cadmium, nickel, cobalt, zinc and mercury at a concentration of 10.0 mg/l increased the amount of lead detected but copper at 1.0 mg/l reduced the amount of lead detected (Table 1).

Analytical techniques for detecting lead concentration have continued to increase in sensitivity by using different extraction techniques. In this study, the limit of detection (LOD) was 0.010 mg/l. The limit of quantification (LOQ) was 0.033 mg/l, defined as the limit of detection at the lower limit for precise quantitative measurements.

The lead concentrations using the developed method and the NIOSH 7303 method did not differ significantly. The concentrations of lead collected by the two methods can be influenced by wind speed, wind direction and air flow patterns. At low levels of airborne lead concentration, the average percent differences for the two methods were lower than at the high levels. Therefore, the developed method can be used to assess occupational lead exposure in workplace air.

Lead analysis using the PAR colorimetric method has been used by Merck

(2008) (method 1.09717.001), LaMotte (2009) (method 4031), HACH (2008) (method DR-4000) to analyze lead in water (Table 4). Our developed method is comparable to other methods (Table 5) such as that of Termkeaw (1993), Lamotte (2009) (method 7667) and NIOSH (1996) (method 7700). The photometers of LaMotte Company, HACH Company and Merck Company can be used at several wavelengths, whereas the photometer we developed was designed to be used at a wavelength of 525 nm.

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