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1.43 THE ROLE OF NAA IN THE ENVIRONMENTAL STUDIES: QUANTITATIVE DETERMINATION OF HEAVY METALS POLLUTANT ON ENVIRONMENTAL SAMPLES*

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ABSTRACT

THE ROLE OF THE NAA IN ENVIRONMENTAL STUDIES: QUANTITATIVE DETERMINATION OF HEAVY METALS POLLUTANT ON ENVIRONMENTAL SAMPLES: The neutron activation analysis technique was applied in the elemental analysis of environmental samples to solve an environmental pollution problem. We focused our study in the analysis of heavy metal which has potentially become a pollutant. The environmental samples analyzed were some water, sediment and an air particulate matter. The tap water sample was collected from five samplings points located at region of Serpong and Muria. Meanwhile the river water samples were taken from five samplings points of Ciliwung River. Eight samplings points of Cisadane river estuary located at Tanjung Burung were selected to collect sediment samples. Air particulate samples were collected from Jakarta Metropolitan and Serpong using high volume air sampler. Trace elements analyses of water samples were done using a combination of INAA and pre-concentration stage prior irradiation. All samples were irradiated at GA. Siwabessy reactor located at Serpong using a thermal neutron flux of about 10^{12}.n.cm^{-2}.sec^{-1}. After cooling time, the samples irradiated were counted by a high resolution HPGe detector coupled to a multichannel analyzer. The quantitative analyses have been done using a comparative method to a fresh laboratory standard and we used some standard references materials to validate our analytical result. The obtained result from the tap water analysis show that the elements of As, Cr, Co, Cd, Mn, Sb and Zn could be determine quantitatively and they have a concentration range from about 0.02 μg/L to 103.9 μg/L. The analysis result of Ciliwung river water samples show that elements of Ag, As, Co, Cu, Fe, Hg, Mn, V and Zn are present in the range of 2.4 μg/L to about 1365.8 μg/L. Meanwhile some important elements were obtained in the sediments samples taken from Cisadane River estuary such as Ce (40.4 – 63.6 mg/kg), Co (15.2 – 40.2 mg/kg), Cr (21.6 – 57.8 mg/kg), Eu (1.2 – 1.8 mg/kg), Fe (7.0 – 16.8 mg/kg), Mn (887 – 1810 mg/kg) and V (160 - 558 mg/kg). Finally the heavy metals of Ag, As, Cd, Ce, Co, Cr, Cs, Fe and Zn were detected at air particulate matter taken from Jakarta Metropolitan and Serpong area which have a range concentration of about 0.04 ng/m^3 (Cd) to 24000 ng/m^3 (Fe). From the above result we conclude that some elements have exceeded the concentration permitted by the Indonesian Government regulation. The NAA technique has a high capability in the trace elements analysis and potentially can be used in the monitoring of environmental pollutant.

Key words: NAA. Pollutant. Heavy metals. Trace elements. Pre-concentration. Environmental samples.

INTRODUCTION

Industrial development has a global effect to environmental, not only in the improvement in the social economic, but also a negative impact of an environmental pollution. These impacts are pollution to the ecosystem by industrial which have release a waste that contain many toxic compound and elements. Many toxic elements and compounds have a pathogenic effect to human and this can cause many diseases, such as cancer and nervous disorder [1]. Some heavy metal that should be seriously notice are Pb, Hg, Cr, Sb, As, Cd, Ag,

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Zn, Se and Mn, and also Al, Co and V. If the toxic substance entered to the ecosystem and it could not be neutralized by the ecosystem itself, because the capability of the ecosystem was over, we say that the ecosystem was polluted. In this condition, the water, air and soil are the ecosystem that shall receive those pollutants. As part of the ecosystem, the human shall receive all consequences caused by this environmental pollution.

Trace element of heavy metal is required by human body as an essential micronutrient. It involves in the activity of large a number of enzyme, synthesis and degradation of carbohydrate, lipids, proteins and nucleic acid. But, in the others, if the quantity of the element is very high, they can cause diseases and poison. Heavy metals are widely used in the process of industry, for instance in the industry of automotive, textile, polymer, electroplating, paint and accu. In the environmental, quantity of the heavy metal is very low and they exist in the order of nanogram level.

Elemental quantification of the environmental sample becomes important because it could be used to monitor a level of environmental pollutant. The obtained data is valuable information for the government or the environmental management center.

Many methods of trace elemental analysis have been developed to evaluate quantitatively the pollutant in the water, soil and air particulate matter, but only a few methods can be used to determine the trace elements, for instance Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and Neutron Activation Analysis (NAA) [2, 3]. The neutron activation technique is one of the methods that have a good capability in the trace elements analysis for wide range samples type, include of environmental samples.

Meanwhile, trace elements analysis of tap water samples involved of a pre-concentration stage prior to irradiate. This stage applied to remove a high content ot Na, Cl and Br, and also to improve a sensitivity [4, 5]. Utilization of Chelex-100 [6] and ammonium pyrolidine dithiocarbamate [7, 8, 9] have been established to separate some metal from their matrix. The Chelex-100 will remove most of the cation and it will retain in the resin; meanwhile an anion will pass through the resin. The cation retained in the resin then can be elute or irradiate directly by a neutron thermal.

In accordance with the government program of "clean river", the Cisadane and Ciliwung River have been studied to determine a pollution level. Those rivers have been use as water source for community drinking water installation at Jakarta and Tangerang region respectively. The Cisadane River pass an industrial area such as automotive and textile industry, as well as urban area. Previous research show that the total Hg contained in the

Cisadane River has a concentration of about 2 μg/L [10].

The elemental quantification of river water sample taken from Ciliwung River has been determined quantitatively using INAA combined with an ion exchange resin. These elements absorbed in Chelex-100 ion exchange resin at pH of about 5, than after that the elements retained were determined quantitatively. Meanwhile the tap water sample taken from urbane area, the concentration and separation processes were done by liquid-liquid extraction technique involved a complex formation of metal with a dithiocarbamate compounds [11]. A quantitative analysis carried out by the instrumental neutron activation analysis (INAA) using a comparative method to a fresh laboratory standard.

The elemental analyses in the sediment sample taken from Cisadane River estuary were done without a chemical treatment. The estuary is place where all elements and compounds contained in the river water will accumulate and contact with the ecosystem of the sea, such as plankton and fish. The toxic element in the fish contaminated will transfer to human via a food pathway.

In the case of the air pollution, the Indonesian government has a program of Blue Sky in the aim to reduce Pb content in the air. This program was introduced by the Environmental Impact Management Agency (Bapedal) of Indonesia who responsible in the monitoring and management of the environmental. The intensive study of air pollution was carried out to support those by Bapedal and Japan International Cooperation Agency (JICA) for the study of the air quality at Jabotabek region in the period of November 1994 – March 1997 [12, 13]. The parameters observed are limited in the determination of SO_x, NO_x, O_x, SPM and HC. Meanwhile, the application of INAA in the analysis of particulate matter has been done since 1992 under Workshop on the Utilization of Research Reactors program.

In the present papers, we report the result of NAA application in the field of environmental study. The samples analyzed consist of water, sediments and air particulate matter, and it was focused to the elemental determination using instrumental neutron activation analysis technique.

METHODS

Sampling: The river water samples were taken from five sampling point at Ciliwung River using polyethylene bottle which has been cleaned before. After removing any particle by filter, the river water samples were acidic by pure HNO_3 and pH condition was set to 2. The same methods have been applied for sampling of tap water from six sampling point of a

public drinking water sources located at Serpong and Muria. All water samples collected than were stored at a free dust location. The sediments samples were collected from eight sampling point at Cisadane River estuary Tangerang. Meanwhile, two sampling point were selected to collect the air particulate matter using high volume sampler that equipped by a filter polyacetate. Two sampling points are the Jakarta and Serpong region that represent an urban and rural area respectively.

Samples preparation: Tap water samples were concentrated by liquid-liquid extraction technique involved a complex formation of metal with derivate of dithiocarbamat compounds at range pH of about 5 to 6 in ammonium acetate buffer. The Ciliwung River water samples were treated by the Chelex-100 resin at range pH of about 5.2 to 5.7. Meanwhile, a non-destructive method was used in the analysis of sediment and air particulate matter.

Irradiation and data acquisition: Irradiation carried out at GA. Siwabessy reactor located at Serpong at a thermal neutron flux of about 10^{12} $n.cm^{-2}.sec^{-1}$. Normally we use an irradiation time schema of 40 second, 5 minutes and 30 minutes irradiation for analysis of short half live (5 minutes < $t_{1/2}$ < 2 hours), medium half life (2 hours < $t_{1/2}$ < 1 week) and long half life ($t_{1/2}$ > 1 week). After cooling time, each irradiated sample than counted using high resolution HPGe detector (Canberra, resolution of 1.9 keV at 1332 keV, efficiency of 18.5 and ratio peak to Compton of 44) coupled to Multichannel Analyzer.

Data analysis: Gamma rays analysis has been done using commercial software of GENIE from Canberra Inc. Identification and quantitative determination has been done using comparative method to laboratories standard. We use a NIST 1643c to validate our analytical result of water. Meanwhile polluted grade compare to the government regulation of water pollution. We also used NIST SRM 1646 Estuary Sediment, IAEA Urban Dust and NIST SRM 1648 Urban Particulate to control our work.

RESULT AND DISCUSSION

Tap Water Analysis

We select three sampling point at Serpong region and three others at Muria region. The first location represents a community area around the Puspiptek which have a high activity in the research and development. The second sampling point represents an ambient region that has a rare community and we choice it as a non-contaminated region. Figure 1 below show the distribution of some metal obtained by INAA combined with liquid-liquid

extraction using APDC-$CHCl_3$. We obtained some heavy metal such as Mn, Sb, Zn, Cr, Sc, Cd, As and Co.

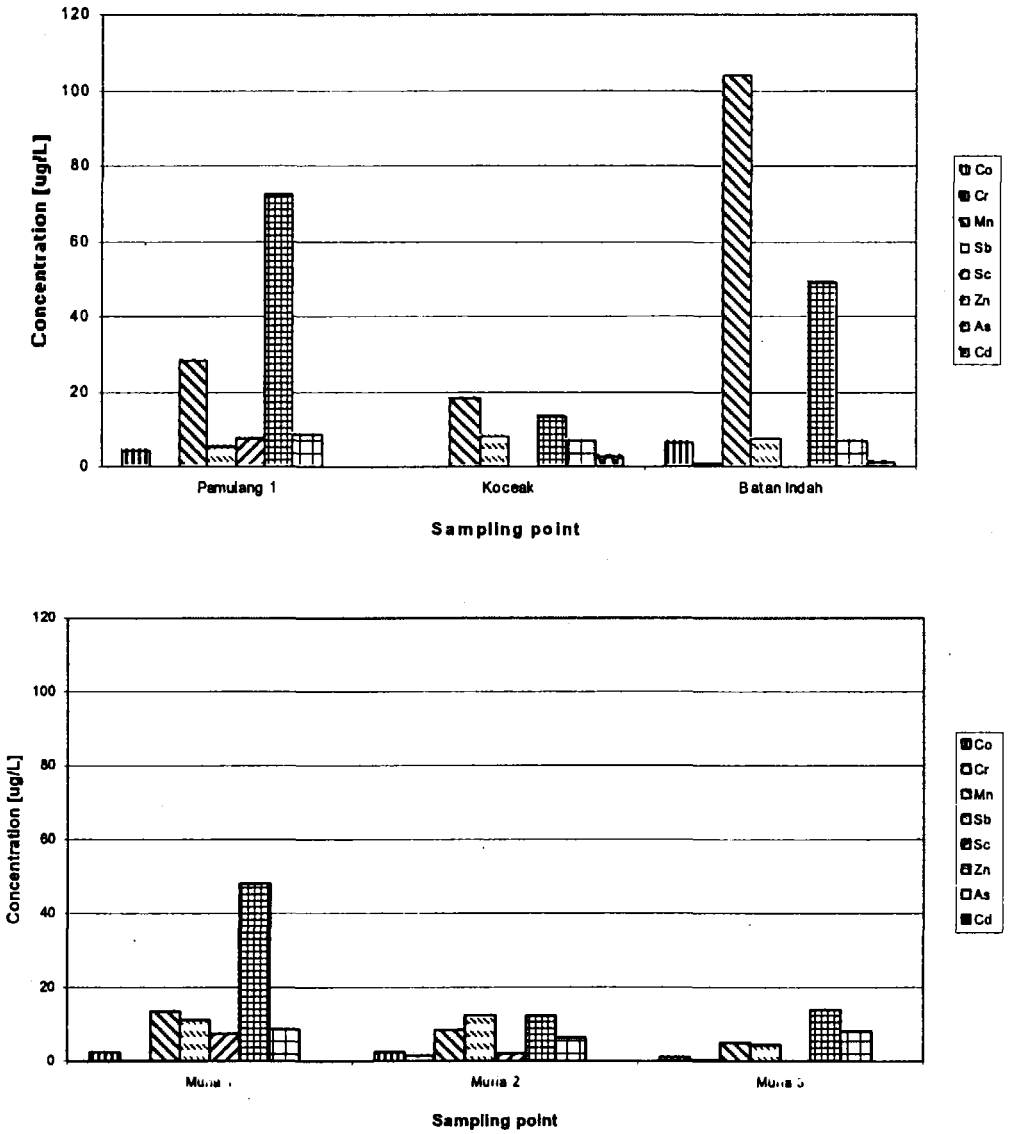

Figure 1: Distribution of heavy metals in five sampling point for Serpong (top) and Muria (below) [14].

As we presented in the Figure 1, the tap water sample contain a high content of Mn (of about 20 μg/L to 104 μg/L) and Zn (of about 14 μg/L to 72 μg/L) at Serpong sampling point. All elements concentration obtained for the sample taken from Muria location are lower than the value permitted by the Indonesian government regulation (Class A). These facts indicate that the water at Serpong and Muria region have not polluted.

River water analysis

As previous mention, we have been select five sampling points and each sampling

point have different characteristic activity and region. Some industries used this river as media to release its waste; meanwhile some people used it to support their activity. In this work, the Chelex-100 resin was used either to concentrate the trace element and to remove some interfere elements, such as alkali metals especially Na, alkaline-earth metals and the halogens, especially Br and Cl. Table 1 show the result obtained for Ag, As, Co, Cu, Fe, Mn, V and Zn contain in the NIST SRM 1643c Water that have been used to control and to validate our result.

Table 1. Analysis result of some elements in NIST SRM 1643c after treated by Chelex-100 resin. Value in the parentheses shows a Relative Standard Deviation [14].

Elements	Concentration [ng/ml]	Certificate value of NIST [ng/ml]	
		Mean	Range
Ag	2.0 (8%)	2.2	1.9 -2.5
As	77.9 (5 %)	82.1	80.9 -83.3
Co	20.1 (2 %)	23.5	22.7 - 24.3
Cu	19.6 (10 %)	22.3	19.5 -25.1
Fe	101.5 (2 %)	106.9	103.9 – 109.9
Mn	32.2 (3 %)	35.1	32.9 – 37.3
V	27.4 (5 %)	31.4	28.6 - 34.2
Zn	70.9 (2%)	73.9	73.0 - 74.8

Table 2: Elemental concentration of river water sample taken from five sampling point. Unit concentration is μg/L [14].

Elements interest	Sampling point				
	Kedung Badak	Pancoran Mas	Tanjung Barat	Kampung Melayu	Pantai Marina
Ag	14.9 – 21.8	19.7 – 37.3	1.9 – 8.9	22.3 – 52.4	15.2 – 55.5
As	11.4 – 128.9	13.8 – 30.3	11.7 – 18.2	12.6 – 26.5	17.1 – 25.5
Co	1.3 – 1.7	1.4 – 2.3	1.4 – 1.6	1.1 – 2.2	1.5 – 1.7
Cu	2.4 – 26.2	0 – 15	0 – 17.0	0.5 – 3.7	0 – 34.5
Fe	404.6 – 472.0	394.9 – 428.8	369.3 – 413.2	336.1 – 425.3	129.2 – 375.4
Hg	5.9 – 9.5	6.9 – 16.4	9.5 – 13.3	7.8 – 11.5	5.9 – 12.7
Mn	33.4 - 84.8	21.0 – 147.6	23.1 – 185.1	39 – 372.9	235.0 – 1953.6
V	5.6 – 10.6	2.4 – 10.4	4.4 – 6.7	5.1 – 7.6	4.1 – 6.5
Zn	13.7 – 30.4	8.6 – 27.8	8.4 – 30.0	11.4 – 33.0	17.6 – 36.8

In general our results are quite lower compare to the certificate value given by NIST. They have a sistimatic error, and we used a correction factor to correct the analytical result. Table 2 shows the concentration range of Ag, As, Co, Cu, Fe, Hg, Mn, V and Zn elements for five sampling point location which have various value for each location. According to the Indonesia Government regulation No. 20:1990, for the Class B category. The quantities of Hg at all location has exceeded the value permitted, meanwhile the Fe element have a critical value for all location of the sampling point. We obtained also Mn concentration at Pantai Marina relatively height compare to other location and this value also was exceeded the value permitted for Class B category. This data show that many activity along the Ciliwung River have used product contain of Hg, Fe and Mn.

Sediment

Many industries and human activities use the Cisadane River that pass the Tangerang city. Some industries use this river to release their waste without pre treatment before. Identification and characterization of the sediment become very important to know the quantity of the toxic element contained in this sediment. The sediments samples were collected from eight sampling point located at the Cisadane River estuary, Tanjung Burung, Tangerang (Figure 2).

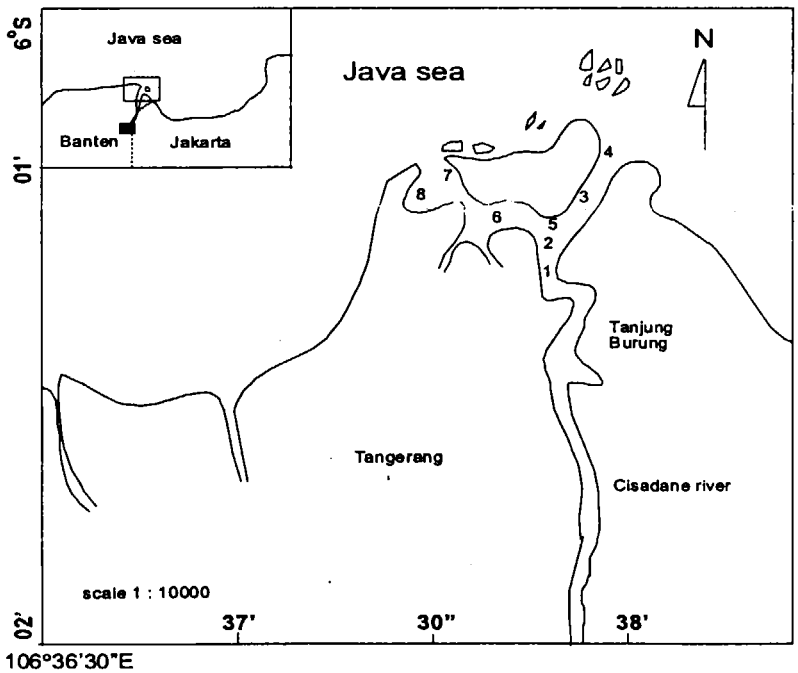

Figure 2: Mapping of eight sampling point at Cisadane River estuary located at Tanjung Burung, Tangerang. The numbers represent a location of the sampling [15].

Table 3 : Elemental analysis result of NIST SRM 1646 Estuary Sediment by INAA [15]

Elements [Concentration]	BATAN	NIST
Al [%]	7.3 ±0.1	6.25 ± 0.2
Ce [μg/g]	82.9 ± 6.0	(80)
Co [μg/g]	11.8 ± 1.5	10.5 ± 1.3
Cr [μg/g]	76.9 ± 9	76 ± 3
Eu [μg/g]	1.7 ± 0.1	(1.5)
Fe [%]	3.4 ± 0.4	3.35 ± 0.1
Mn [μg/g]	383 ± 28	375 ± 20
Th [μg/g]	10.1 ± 0.8	(2)
Ti [%]	0.5 ± 0.1	(0.51)
V [μg/g]	85 ± 84	94 ± 1

We used the NIST SRM 1645 Estuary Sediment to verify our method, and the results are given in the Table 3. As show in the table, all result obtained are good enough compare to the certificate value given by NIST. Figure 3 show the distribution of element obtained from each sampling point, SP No. 1 to SP No.8. We have analyzed a nine element for each sampling point, and each location show a similar distribution. The sediment samples have a mayor component of Fe (7.1 % to 16.8 %), Mn (0.1 % - 0.2 %), Ti (0.5 % - 1.7 %) and V (0.02 % - 0.05 %), meanwhile the minor component consist of Ce (40.4 μg/g – 63.6 μg/g), Co (15.2 μg/g - 40.2 μg/g), Cr (21.6 μg/g – 57.8 μg/g), Eu (1.2 μg/g – 1.8 μg/g) and Th (5.5 μg/g – 9.1 μg/g).

The enrichment factor (EF) was calculated as recommended by Vinogradov [16]. This factor was calculated using Al as reference elements and Manson crust rock average concentration are used as reference values. Figure 4 indicated that the EF values for elements detected are in the range of about 0.1 to 6.3. This show that the sediment at Tanjung Burung is not contaminated.

Urban particulate matter

Some result of trace element analysis by INAA technique have been reported in the last workshop and other publication [17, 18, 19, 20]. The utilization of INAA in the environmental study involved the Environmental Management Center (EMC) and Jakarta Urban Assessment and Environmental Office (JUAEO) who responsible in the sampling air

particulate matter.

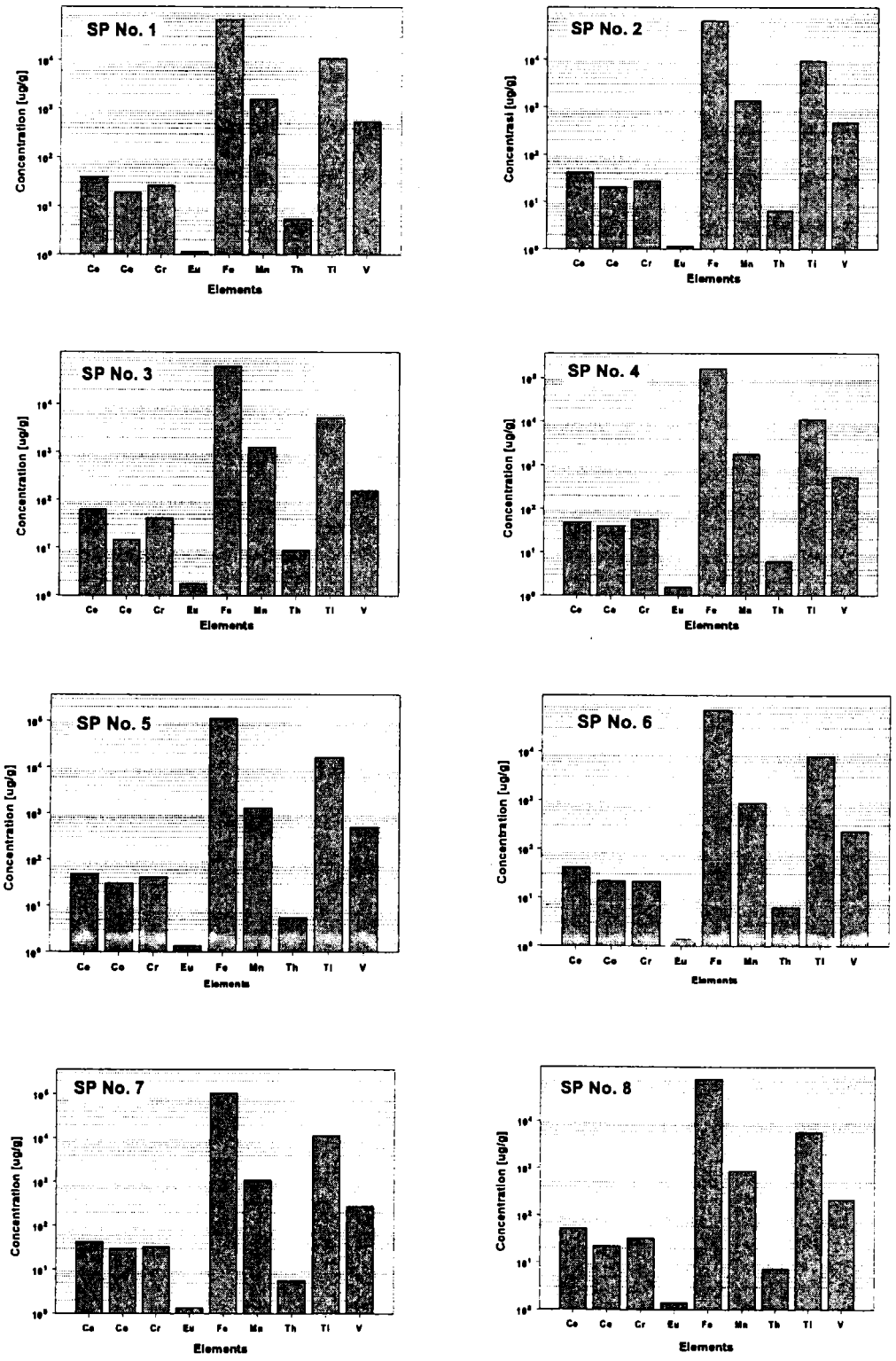

Figure 3: Distribution of the elements obtained in the Cisadane River estuary. SP No.1 to SP No.8 indicates the sampling point located at Tanjung Burung, Tangerang [15].

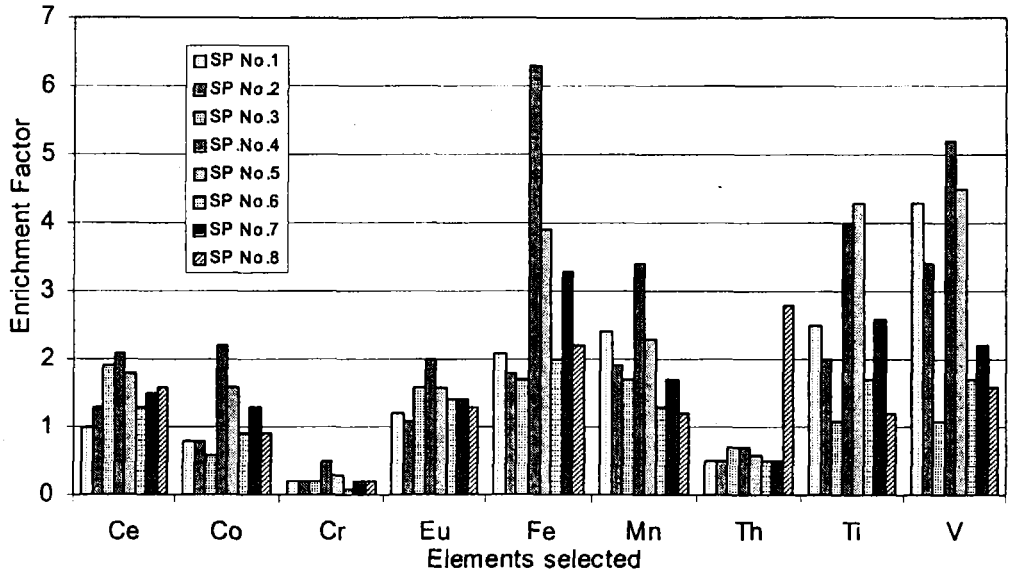

Figure 4 : Enrichment factor of the elements based on the Al [15]

Table 5: Quantitative determination result of the elemental analysis in NIST SRM 1648 and IAEA-0395 Urban Dust by INAA method. Concentration unit in μg/g, and value in parentheses show Relative Standard Deviation in %.

Elements interest	NIST SRM 1648		IAEA-0395 Urban Dust	
	NIST	BATAN [19]	IAEA	BATAN [21]
Al	34200	-	24000 - 33200	28300 (3)
As	115 ± 10	-	0.09 – 27.9	12.7 (10)
Cr	403 ± 12	368.5 (8.6)	23.2 - 237	-
Cd	75 ± 7	82.8 (10.4)	5.34 – 33.0	45.8 (>10)
Co	18	18.47 ± 2.6	13.3 – 16.6	-
Fe	39100	-	46000 - 88900	70800 (10)
Mn	860	936.5 (8.9)	687 - 1420	1260 (6)
Ni	82	-	15 - 128	75.2 (3)
Sb	45	49.32 (9.6)	14.7 – 18.9	9.4 (>10)
Se	27 ± 1	24.5 (9.3)	0.08 – 5.3	2.4 (>10)
V	140 ± 3	151.5 (8.2)	76.3 - 102	101 (5)
Zn	0.476 ± 0.14	0.566 (9)	1710 - 4240	3120 (>10)

Table 5 show the elemental concentration in SRM NIST 1648 Urban Particulate and IAEA-0395 Urban Dust as determined by INAA. The concentrations of many elements are close to the certificate value given for NIST SRM 1648, and in the range of IAEA accepted values for IAEA-0385 Urban Dust sample

The elemental distribution of air particulate matter sample obtained from two

sampling point are shown in the Figure 5 below. It was found that the concentrations of all elements interest at Jakarta site are higher than that at Serpong site. This fact shows that the Jakarta region is more polluted than the Serpong region.

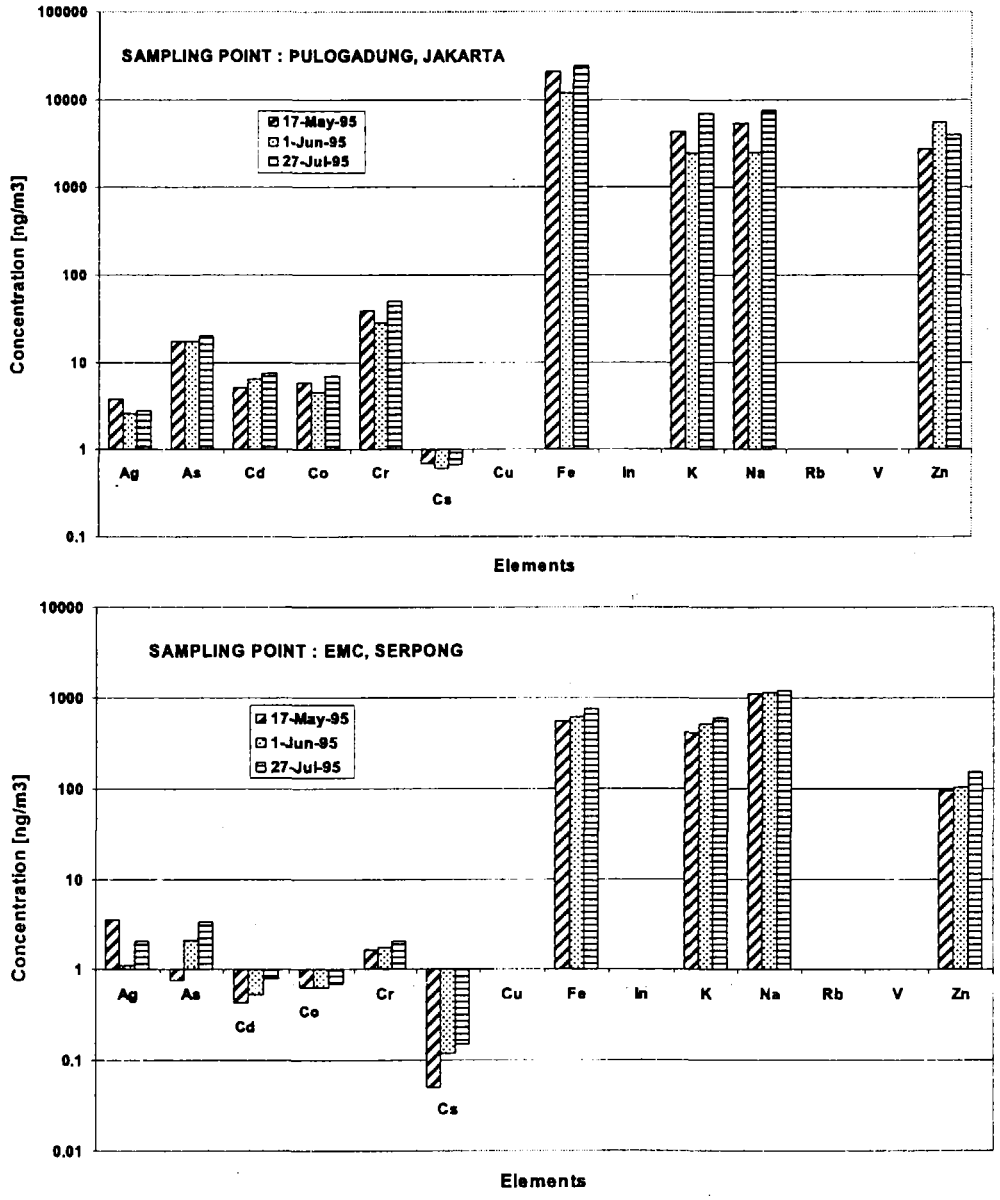

Figure 5 : Elemental concentration of air particulate matter measured by INAA for Pulogadung, Jakarta (urban) and Serpong (rural) [19]

CONCLUSION

From the above result we conclude that some elements in the Ciliwung River have exceeded the concentration permitted by Indonesian Government regulation No. 20:1999, especially Hg, Fe and Mn. The Jakarta Metropolitan city more polluted than the Serpong area. The NAA has a high capability in the trace elements analysis and become useful tool in the

monitoring of environmental pollutant. The NAA is useful technique and can be used for many type of environmental sample.

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