Determination of lead, naphthalene, phenanthrene, anthracene and pyrene in street dust

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ABSTRACT: Street dust is a potential source of lead exposure to humans, however scarce information about the pollution levels with lead and polyromatic hydrocarbons exists in Venezuela, limiting the appropriate evaluation of the levels of risk of the people. This work was aimed in the determination of the concentrations of lead, naphtalene, anthracene, phenanthrene and pyrene in the street dust of the most transited avenues and streets of Maracay city. Thirty street dust samples were collected at the streets and avenues, troll and bus main station. Lead was determined by atomic absorption spectrometry after acid digestion (Pb-total), also the fractions of lead soluble in 1 M $MgCl_2$ and 0.5 M ammonium acetate (pH = 7) were quantified. The polyaromatic hydrocarbons concentrations were determined by capillary gas chromatography equipped with a flame ionization detector. The Pb-total ranged between 734 and 11.439 µg/g with the higher values at the most transited streets and avenues. About 60 % of samples exhibited concentrations between 1.000 and 2.500 µg/g, similar to the values reported in the literature for soils of urban areas. The fraction of lead soluble in magnesium accounted for less than 3 % of Pb-Total, while the 0.5 M ammonium acetate solution represented more than the 75 % of the total loads of the pollutant. The most contaminated samples were those taken at the toll with concentrations of 695.5 and 252.1 µg/g phenanthrene and anthracene were the most abundant, while at the bus station all compounds were detected.

Keywords: *Environment; Polyaromatic hydrocarbons determination; Street dust; Urban pollution*

INTRODUCTION

The street dust is the product of the interaction of solid, liquid and gaseous materials produced from different sources on the roads and may contain pollutants such as heavy metals and dangerous organic compounds, becoming a growing concern in recent years because it is in a continuous contact with the habitants of modern cities (Xiandong *et al.,* 2001). A considerable number of studies on the concentration of harmful pollutants in the urban environment have been done in developed countries with long history of industrialization; however, scarce information exist in developing countries limiting the appropriate evaluation of the levels of risk of the people who lives in the most important cities (Shinggu *et al.,* 2007). Street dust has been identified as a potential source of lead exposure to humans, both through direct inhalation and from ingestion, particularly to children (Sharaf *et al.,* 2008); this heavy metal is released from the combustion of leaded gasoline, by the 1970's lead fall out from

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combustion represented around the 90 % of the atmospheric lead in the urban environment (Farmer, 1987). The heavy metal and other contaminants present in the soil surrounding the roads can reach the road via rain water, also the dry and wet atmospheric deposition, degradation of road paint, vehicle wear and tear, vehicular fluids and particulate emissions all add to the levels of these pollutants (Karbassi, *et al.,* 2008; Malakootian *et al.,* 2009). Lead is one of the most important heavy metals in the Venezuelan environment because of the extensive use of lead tetraethyl as a gasoline additive, since the early years of the 20 century until 2005 when this compound was prohibited and the unleaded petroleum introduced. According to Fernandez and Ramirez (2005), the concentration of lead in solid particles from roads of the city of Caracas ranged between 5.500 to 13.000 µg/g. Carrasquero (2006) reported levels from 2.000 to 4.000 µg/g in urban soils of Maracay City, with the higher concentrations in soils near of the most transited streets and avenues. The bioavailability of the released lead had been identified

by Diez *et al.,* (1996), who reported levels of this heavy metal of 15 μg/g, in the wood of pines located close to the roads of Merida City. In a study with humans, Nazila *et al.,* (1998) determined concentrations of lead up to 15 μg dL in the blood of 180 Venezuelan children. On the other hand, according to Yuan *et al.,* (1999), there are linkages between the presence of lead in gasoline and the formation of organic contaminants such as polyaromatic hydrocarbons (PAHs), which are compounds that contain more than one fused ring and have a considerable interest of study because of their toxic, mutagenic and carcinogenic characteristics, several types of cancer are directly related with the exposure to PAHs, among them lung, bladder, skin and internal organ cancer (Mastandrea *et al.,* 2005). Maracay, the fifth most important city of Venezuela, possesses a population of more than 500.000 habitants and has experienced an important urban and industrial development in the last 30 years. This development has occurred with an exponential growth of the use of gasoline and diesel in vehicles for public and commercial transport. The emissions of polluted compounds along this time have not been well evaluated and little information exists about the contamination with PAHs in the environment (Gueu *et al.,* 2007; Okafor and Opuene, 2007). This paper is aimed in the determination of the concentrations of lead, naphtalene (Nap), anthracene (A), phenanthrene (Phe) and pyrene (Py) (Fig. 1) in the street dust of the most transited avenues and streets as indicators of the impact produced by the emissions of the vehicles, in order to obtain valuable information to evaluate he potential risk of the habitants of this Venezuelan City.

MATERIALS AND METHODS
Location of the study area and sampling sites
Maracay is the capital of the Aragua State, located in the central north of Venezuela at the coordinates 10º 15' 41" N and 67º 37' 09" W, surrounded by the mountains of the Caribbean coast and its climate is typical of the tropical zone with wet and dry seasons. The sampling sites were selected as follows (Fig. 1): Three samples were taken at the toll of the highway that connects the city with Caracas, the country's capital (S1 to S3). Four samples were collected at the bus station (S4 to S7), other four samples were collected from two main avenues (S8 to S11) and three more samples were from the centre of the city (S12 to S15). For the determination of lead 14 additional samples were taken along the most transited avenues. The samples were taken during the months of June and July, 2006.

Sampling procedure
Once the sampling point was identified, the dust on a surface of 1 m² was swept using a brush and a little stainless steel shovel, the powder was put into a sealed plastic bag. In the laboratory, the samples were extended on paper sheets, air dried for three days, sieved to particle size < 75 μM and stored in capped glass bottles.

Lead determination
The total concentration of lead (Pb-Total) was determined following the procedure described by Sillanpa and Janson (1992). Briefly, 200 mg of the sieved street dust was digested using 20 mL of *aqua regia* in a digestion block programmed at 120 °C for 2 h. Once digestion vessel was cold, 20 mL of distilled water were added, the solutions were filtered using Wahtman filter paper N 42 into 50 mL volumetric flasks and the volume was made up with distilled water. This same procedure was applied to the Certified Reference Materials. Calibration curves were made using lead nitrate (analytical grade) standard solutions. Additionally the lead soluble in 1 M $MgCl_2$ and 0.5 M ammonium acetate (pH = 7) were determined weighing 500 mg of the sample in a 50 mL centrifuge tube containing 5 mL of the extracting solution. The suspension was mechanically shaken during four hours at ambient temperature for the magnesium chloride extraction or 80 °C for the ammonium acetate solution. The supernatants were separated by centrifugation at 3.000 rpm for 15 min and the lead determined. Standards for calibration curves were prepared in the same extracting solutions. Due to the high levels of lead in the samples the quantification was made by air-acetylene flame atomic absorption
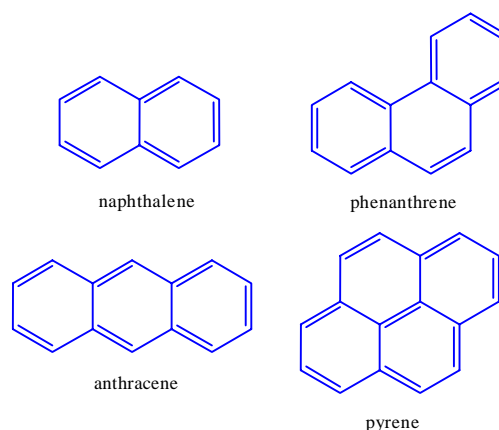

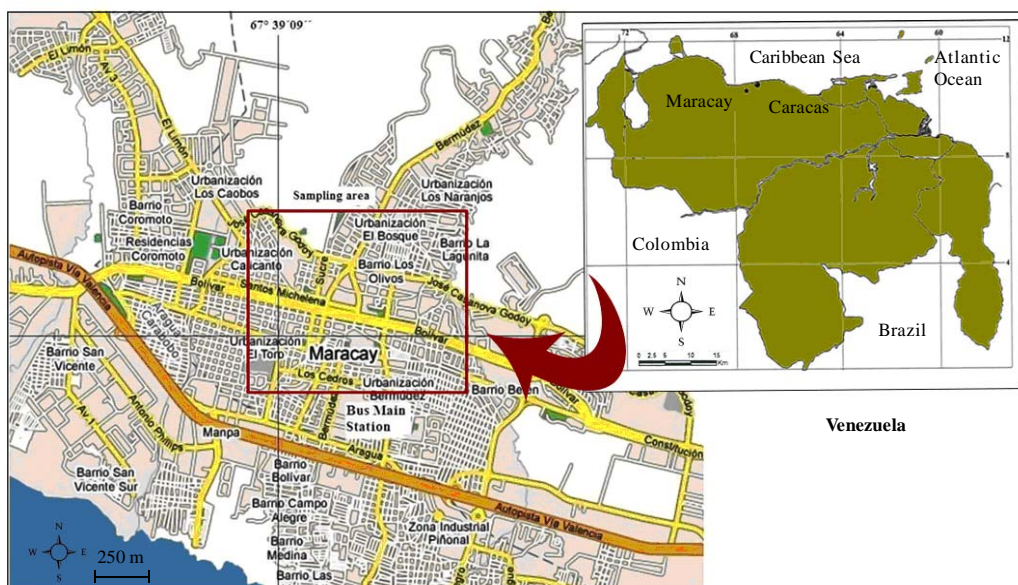Fig. 1: Molecular structure of the PAHs of interest in this study

Fig. 2: Maracay city and sampling sites

spectrometry (AAS) using a Perkin Elmer Analyst 100 and a hollow cathode lamp for lead. The quality control of Pb-Total determinations was assessed by the traceability to the follow Certified Reference Materials: SDC-1 and G-2 of the United States Geological Survey, with certified values of 25.1 ± 0.5 and 49.5 ± 0.3 µg/g and values found of 24.6 ± 1.3 and 51.3 ± 2.5 µg/g, respectively. Also, a highly contaminated soil CRM 020-050 (RT-Corp, USA) with a Pb content of 5.111 ± 622 µg/g and a found value of 4.907 ± 215 µg/g was used.

PAHs determination

The extraction of the PAHs of interest was made weighing 500 mg of the street dust sample into 10 mL glass vials. Then, 10 mL of cyclohexane (NIOSH, 1994, Gfrerer *et al.,* 2002) were added and the suspensions were mechanically shaken for 24 h and after centrifuged at 3.000 g for 15 min. The yellow coloured extracts were clean up on silica columns and the solvent was evaporated with a flow of nitrogen. The residue was taken with 1 mL of cyclo hexane and transferred to sealed vials and stored at -4 °C before analysis. Two microlitres of this solution were injected to a gas chromatograph GC Agilent 4890 provided with a HP 5 capillary column and a flame ionization detector (FID). The chromatographic conditions were: Injector temperature 250 ° C, detector temperature 300 °C. Oven program: 60 °C for 1 min, increase to 160°C (40 °C/min) and finally increase to 250 °C (10 °C/min). The software Agilent Cerity was used to register the chromatographic signals. Tetramethylbenzene was used as the internal standard (ISTD) to obtain the relative retention times (t_{rr}). Five street dust samples with non quantifiable levels of the PHAs of interest were used for the recovery evaluation of the analytical method. In such way, samples of 500 mg previously weighed in glass vials were treated with a solution containing the four PAHs to make a final concentration of 10 µg/g. The suspensions were shaken for 1h and the vials were left uncapped for one week. Finally, the organic compounds were extracted and quantified by the method described. Calibration curves were made using pure compounds, naphthalene and phenanthrene (Synchemica, UK), anthracene (Fluka Chemicka, Switzerland), pyrene and cyclo hexane (Riedel de Haen, Germany). Standard solutions between 5 and 50 µg mL of each compound in cyclo hexane were injected to the GC-FID system under the same conditions employed for samples. The limits of detection (LOD) and quantification (LOQ) were calculated.

RESULTS AND DISCUSSION

Lead concentration in street dust

Samples were divided into three groups in function of the density of transit. Those samples taken at the major roads and streets with a heavy transit (around 100/h vehicles) belonged to the group A, while those

with a vehicles transit between 100 and 50 corresponded to the group B, the streets with light transit (< 50 cars/h) were in the group C. Table 1 shows measured lead levels between 734 and 11.439 µg/g among the three groups, with the highest levels in the group A, which is consistent with the intense traffic of vehicles. The maximum lead concentrations were found at the crossroads where come together the vehicles that go to the northern and southern sides of the city. The major percentage of the samples, about 60 %, exhibited concentrations between 1.000 and 2.500 µg/g. Similar values were reported by Madrid and Diaz-Barrientos (2002) for soils of urban parks in Sevilla, Spain, impacted by the surrounding roads. On the other hand, the fraction of lead extracted with the solution of $MgCl_2$, representing the amount of the heavy metal under very soluble and reversible adsorbed forms also called the mobile fraction (Kashem *et al.,* 2007), accounted for less than 3 % of Pb-Total, while the 0.5 M ammonium acetate solution dissolves the insoluble lead, as for example $PbSO_4$ (Vogel, 1974), representing more than the 75 % of the total loads of the pollutant. This suggested that most of the lead present in the dust samples comes from the gasoline and the oxidation of the sulphur containing compounds favoured precipitation of lead sulphate. In future studies this extractive dissolution would be used instead of the more harmful *aqua regia* to dissolve the contaminant is similar matrices.

PHAs concentrations in street dust
Identification of the chromatographic peaks and recoveries

Solutions containing each single aromatic compound, including the ISTD were injected five times to the GC-FID system to locate the chromatographic peaks. The t_{rr} were calculated using the Eq. 1 giving the results shown in Table 2.

$$t_{rr} = \frac{t_r^{compound}}{t_r^{ISTD}} \qquad (1)$$

These results were contrasted with the t_{rr} determined from the street dust samples treated with the PAHs for the recovery evaluation, observing good agreements between both sets of results, allowing an adequate identification of the polycyclic aromatic compounds with typical chromatograms as those shown in Fig. 4. The recovery evaluation showed results between 89 and 95 %, similar to he values reported by Gao and Zhu (2004) of 95.6 and 91.3 % for Phe and Py extracted from soil samples using ultrasound and dichloromethane. De Lucca *et al.,* (2005) used a mixture of chloroform and nhexane using the Soxhlet method to dissolve the Nap, Phe, A and Py from polluted sediments obtaining recoveries of 57.8; 88.7; 67.6 and 7.1 %, respectively. With a similar Soxhlet system Nadal *et al.* (2004) dissolved the 90 % of the Py

Table 1: Measurement of lead in Maracay street dust, average concentrations ± standard deviation (µg/g) according to the type of locality

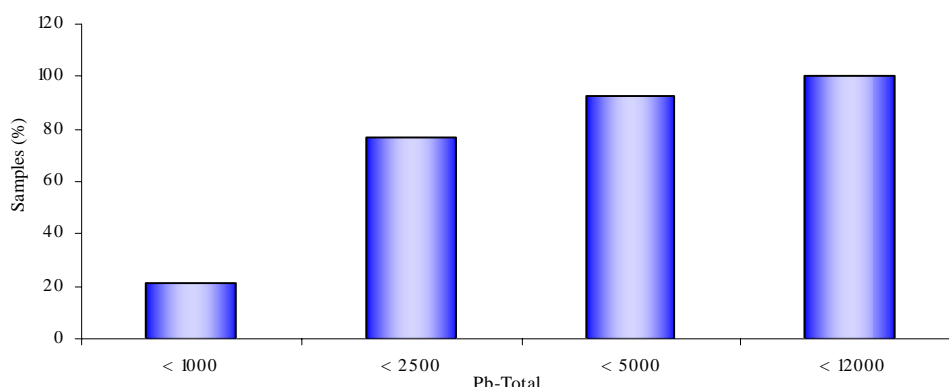| Category	Pb-Total (µg/g)	Pb-MgCl$_2$ (µg/g)	Pb-CH$_3$CO$_2$NH$_4$(µg/g)
A	6,156.4 ± 3,647.0	160.1 ± 75.6	4,625.5 ± 2,600.1
B	3,275.0 ± 1,249.9	62.2 ± 31.2	3,105,5 ± 1037.4
C	1,874.0 ± 780,4	56.2 ± 21.0	1,698.7 ± 702.3

Fig. 3: Distribution of street dust samples as function of Pb-Total concentrations

accumulated in urban soils. These results indicate that the procedure employed in the present study is adequate for the quantification of the PHAs present in the samples (Song *et al.,* 2002).

Quantification of Nap, Phe, A and Py in the street dust samples

The parameters of the regression equations employed in the quantification of the PAH are showed in table 2. The linear responses of the FID detector to the concentrations of each compound are evidenced by the significant determination coefficients (r^2). The detection limits were between 0.97 and 3.20 µg/g, with quantification limits above 3.24 µg/g. The total concentration of the PAHs of interest ranged between 9.93 and 695.5 µg/g (Table 3). The most contaminated samples were those taken at the toll (S1 and S2) with concentrations of 695.5 and 252.1 µg/g, the four aromatic compounds were detected with Phe and A as the most abundant. A similar situation was observed in the samples of the bus station, where the four PHAs were detected with concentrations from 28.54 to 69.84 µg/g, being the four rings aromatic compound the dominant component. A similar to the result was reported by Byung-Cheol *et al.,* (2005), who found that pyrene was the most abundant PAH in deposited road particles from Japan. On the other hand, the dust samples taken on the streets and avenues of the central part of the city exhibited low values for total PAHs and in many cases the naphthalene, anthracene and phenanthrene were absent. Only the S10, which corresponded to the exit of the bus Main station, had a total concentration of 108.5 µg/g, with quantifiable amounts the four PAHs. However, the concentrations lowered to 57.32 µg/g for S11 which was taken 50 m

away. In all cases, the street dust samples exhibited Pb levels significantly higher than the values reported by Shinggu *et al.,* (2007) for dust samples from Mubi, Nigeria, which represents a significant risk of contamination for the habitants of the city. In the case of urban soils, Mellor and Bevan (1999) indicated that the maximum level allowed by the British legislation is 500 µg/g while the Dutch regulation is stricter, with a lead concentration of only 150 µg/g for uncontaminated soils. Although street dust is not considered to be a soil strictly speaking, these values give an idea of the degree of lead contamination of Maracay City. Similar values have been reported by Imperato *et al.,* (2003) for urban areas in Naples City; also Lu *et al.,* (2008) found considerable pollution in street dust collected from Baoji, China, which mainly comes from traffic and industrial activities. In a study of lead in dust for Manchester streets, Nageotte and Day (1998) show that lead concentrations increased from an average of 941 µg/g in 1975 to 569 µg/g in 1997, showing the impact of the introduction of unleaded gasoline in 1985. On the other hand, the same study detected lead levels in street dust taken from Paris streets much higher and lead isotopes ratios measurements revealed that lead present in France and UK come from different sources being the vehicles emissions one of the most important. According to the above results, most of the lead quantify in this study comes from the gasoline and considering that it was not but until the year 2005 that in Venezuela the leaded petrol was replaced with unleaded gasoline, it would be necessary to wait several years for detecting a decrease of the current levels of pollution. In general, the concentrations of the four PAHs are much higher than other values reported in the literature. For instance,

Table 2: Relative retention times and recoveries for the GC-FID determination of PAHs in Maracay street dust

Parameter	Naphthalene	Phenanthrene	Anthracene	Pyrene
Determination coefficient (r^2)	0.9993	0.9995	0.9957	0.9992
Intercept on the *y* axis (*a*)	9.30	6.14	3.46	2.41
Standard deviation of *a* (S_a)	16.11	11.23	29.49	9.71
Slope (*m*)	29.19	27.77	27.63	29.98
Limit of detection (LOD)[*]	1.66	1.21	3.20	0.97
Limit of quantification (LOQ)[**]	5.52	4.04	10.7	3.24

*LOD $3xS_a$ (µg/g); **LOQ $10xS_a$ (µg/g)

Table 3: Calibration curve and statistical parameters for the GC-FID determination of PAHs

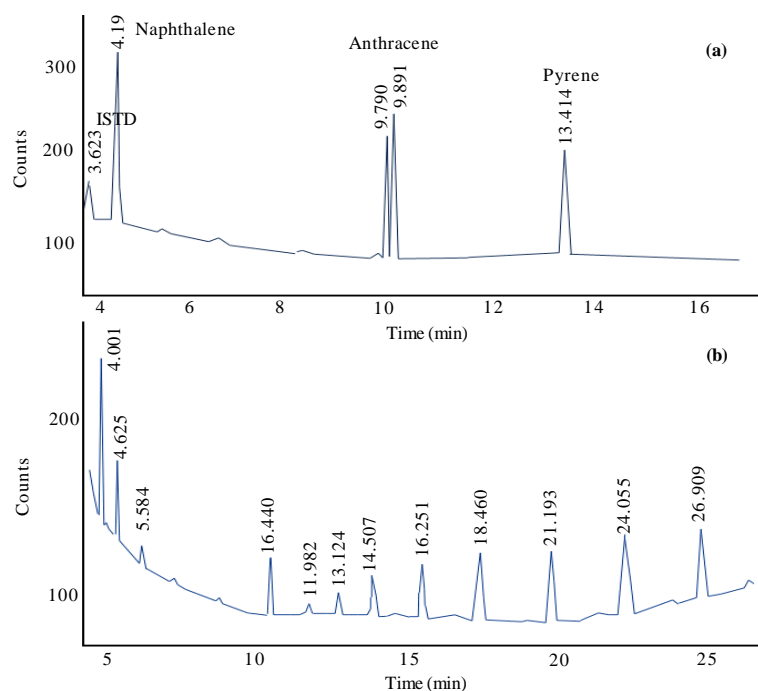| Compound	t_{rr} Pure Solutions (min)	t_{rr} Dust sample (min)	Added (µg/g)	Found (µg/g)	Recovery (%)
ISTD	1.000	1.000	-	-	-
Naphthalene	1.158 ± 0.002	1.076 ± 0.030	10.0	9.5	95
Phenanthrene	2.671 ± 0.041	2.791 ± 0.047	10.0	8.9	89
Anthracene	2.699 ± 0.041	2.819 ± 0.047	10.0	9.0	90
Pyrene	3.688 ± 0.095	3.870 ± 0.088	10.0	9.4	94

Fig. 4: Typical GC-FID chromatogram for A: Mixture of pure PAHs. B: Street dust sample

Table 4: Concentration of PAHs in street dust samples

| Sample | Concentration (µg/g) | | | | | |
	Naphthalene	Phenanthrene	Anthracene	Pyrene	Sum	Phe/A (Origin)
S1	19.49	111.14	547.36	17.49	695.5	0.20 (Pyrogenic)
S2	5.99	34.15	168.2	43.7	252.0	0.20 (Pyrogenic)
S3	< LOD	2.58*	22.53	12.08	37.19	0.11(Pyrogenic)
S4	3.36	12.48	9.06	59.57	84.47	1.38 (Pyrogenic)
S5	< LOD	< LOD	27.56	42.28	69.84	-
S6	< LOD	12.85	11.85	33.89	58.59	1.08 (Pyrogenic)
S7	< LOD	12.71	8.48*	7.35	28.54	1.50 (Pyrogenic)
S8	6.8	13.47	5.11*	83.37	108.8	2.64(Pyrogenic)
S9	< LOD	< LOD	< LOD	57.32	57.32	-
S10	< LOD	< LOD	< LOD	23.30	23.30	-
S11	< LOD	< LOD	< LOD	17.96	17.96	-
S12	< LOD	< LOD	< LOD	19.62	19.62	-
S13	< LOD	< LOD	< LOD	34.61	34.61	-
S14	< LOD	< LOD	< LOD	9.93	9.93	-
S15	4.45*	< LOD	< LOD	10.22	14.67	-

< LOD below the detection limit

* only for reference

in sediments from Taylor Creek in Southern Nigeria, Okafor and Opuene (2007) reported concentrations of phenanthrene between 0.146 and 1.61 µg/g and the levels of anthracene were from 0.552 µg/g to 1.270 µg/g, which were considered elevated when compared with typical levels of PAHs found in pristine areas. Working in an urban environment, Tang *et al.,* (2005) found total concentrations for 16 PHAs between 0.37 and 27.85 µg/g in urban soils from Beijing; while at the city of Bangkok, the range was 12 to 380 µg/g (Wilcke *et al.,* 1999). Urban soils from New Orleans presented concentrations of Nap, Phe, A and Py of 10; 36; 4 and 56 µg/g, respectively (Mielcke *et al.,* 2001). However, it is necessary to consider that the street dust concentrates the solid particles released by the engines of the vehicles with very high amounts of these pollutants and when the dust is mixed with the soils a dilution occurs that lower the concentration of the aromatic compounds. Combustion processes and the release of uncombusted petroleum products have been identified as the two main

causes of anthropogenic PAHs. To distinguish between the natural and anthropogenic PAHs inputs in soils some indexes are used as for example the ratio Phe/A within the 3-ring group, as well as in other isomers such as perylene isomers (Opuene *et al.,* 2007). The catagenesis of the organic matter to produce petroleum is a process that occurs at low temperature and the synthesis of polyaromatic hydrocarbons is governed by thermodynamic properties. On the contrary, during the pyrolisis of the organic matter the distribution of these compounds is determined by kinetics factors (Colombo *et al.,*1989). Usually, Phe/A ratios > 15 indicate the petrogenic origin because phenanthrene is more thermodynamically stable and at low temperature and its molar fraction tends to be higher than that of anthracene. The opposite occurs when the organic matter is burned and the pyrolitic origin of the PAHs is evidenced by a ratio < 10 (Baumard *et al.,* 1998, Yunker *et al.,*1996). Results of this study evidence the pyrogenic origin of the PAHs in the street dust with Phe/A ratios between 0.1 and 2.6 that supports this idea that the combustion of fossil fuels is one of the most important reasons of the presence of these pollutants in the street dust samples. Traffic- and urban influenced areas are prone to enhanced pollution with products of incomplete combustion of fossil fuels and biomass such as the PAHs among others. This contamination levels may be originated not only from automobile exhausts, here primarily diesel, but also it is necessary to take in account from tire abrasion and tailpipe soot which may contributed to the traffic-caused PAH contamination (Glaser *et al.,* 2005). The levels of pollution of Maracay are characteristic of a growing city and may be used as indicator of the situation of many other cities with similar characteristics. For example, actually lead concentrations in street dust are very high, requiring future studies on the impact of the elimination of the leaded petrol. Also, evaluations of the impact on the people who live in this Venezuelan city must be carried out by the sanitarian authorities. On the other hand, a more extensive evaluation of the concentration of PAHs is necessary that includes a major number of sampling points and other compounds with incidence on the human health. The next step must involve the design of remediation strategies for the most polluted places.

CONCLUSION

The most important findings of this study may be summarized as:

- The street dust samples of Maracay City showed elevated pollution with lead, the most polluted sites coincided with the heavily transited main avenues and streets which may the consequence of a prolonged accumulation of the heavy metal released from the lead treated gasoline.

- The Pb of dust samples was present in form soluble in ammonium acetate suggesting the presence of lead sulphate, which is insoluble in water, but may be dissolved at the acid pH of the stomach facilitating its absorption when the dust is ingested.

- PAHs represented other important source of environmental pollution associated with the combustion of fossil fuels to which the inhabitants of the city are exposed. Mainly, workers and users of the bus main station.

ACKNOWLEDGMENTS

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