



# Measurement of Benzo(a)pyrene in PM<sub>10</sub> Collected in New Delhi

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**Abstract:** Polyaromatic hydrocarbons (PAH) are the compound which consists of multiple benzene rings bonded in straight, groups or angular forms. They are also found in atmospheric aerosols. In the atmosphere, they can be emitted primarily as a result of incomplete combustion of natural sources (fossil fuels, forest fires, smoke etc.) or anthropogenic sources (coal burning, vehicular emissions, smoke, etc.) or secondarily by atmospheric processes. Depending on the anthropogenic sources, PAHs may occur in significant concentration in urban and industrial ambient air, i.e., bounded with particulate matter (PM). A particle whose aerodynamic diameter is  $\leq 10 \mu\text{m}$  is called PM<sub>10</sub>. Benzo(a)pyrene (BaP) is among the most toxic and carcinogenic PAHs. Both PM<sub>10</sub> and BaP are among the 12 criteria pollutants listed in Indian National Ambient Air Quality Standards (NAAQS). In this paper, BaP concentration in PM<sub>10</sub> collected in a representative site of New Delhi was studied during the year 2014–2015. The average concentration of BaP is varied from 0.04 to 25.7 ng m<sup>-3</sup>. The uncertainty components in measurements were also estimated along with statistical analysis. The most significant uncertainty component is the purity of the BaP standard which has the highest uncertainty contribution as 77%.

**Keywords:** NAAQS; PM<sub>10</sub>; PAHs; Benzo(a)pyrene (BaP)

## 1. Introduction

The primary source of polyaromatic hydrocarbons in the atmosphere is mainly due to incomplete combustion of fossil fuels emitted from suburban, natural, industrialized and vehicular sources. PAHs having small molecular weight exist in a gaseous phase in the atmosphere and also in particulate phase by the condensation after emission. PAHs having high molecular weight are mainly found as particulate in the atmosphere [1]. Thus, PAHs are ubiquitous as they are present in air, soil, and water in our environment [1, 2].

Chemically, the term “PAH” is used for compounds having carbon and hydrogen atoms. Their structure consists of multiple benzene rings bonded in straight, groups, or angular forms [3, 4]. They have several sources as shown in Table 1.

There is an urgent need for monitoring the toxic compounds present in the atmosphere as they are hazardous for human health and the environment [6] International

Agency for Research on Cancer (IARC) has selected few PAHs (e.g., Benzo[a]anthracene (BaA) and Benzo[a]pyrene (BaP)) as distinguished carcinogens for human [7]. Among them, BaP is referred to as a “carcinogenic substance- level 1” [8–10]. A lot of studies have been done regarding the increasing concentrations of BaP in the atmosphere all over the globe. The highest concentration of BaP ( $> 0.4 \text{ ng m}^{-3}$ ) was found in central and eastern part of Europe [11]. Exposure to BaP concentration beyond the permissible value was major and extensive in eastern and central part of Europe. From 2009 to 2011, 22–31% of the urban residents were exposed to BaP concentrations beyond the permissible limit of  $1 \text{ ng m}^{-3}$  [12]. In the latest report, it was observed that in summer, concentrations of BaP in the atmosphere in urban areas was averaged  $3.6 \pm 4.11 \text{ ng m}^{-3}$ . In winter, concentrations are higher (average  $7.1 \pm 5.1 \text{ ng m}^{-3}$ ) mainly because of the emission from domestic and industrial heating sources using fossil fuels.

The snow covers in the Eastern region of Moscow showed a high concentration of BaP due to its everyday fallout into the atmosphere. In 2010, the average mass of the BaP in the snow was found as  $1942 \text{ ng g}^{-1}$  while its mean values of intensity of fallout was  $7.13 \text{ ng m}^{-2}$  per

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**Table 1** Main sources of emission in China, India and the USA for the USEPA [5]

Sources of emission	Global (%)	China (%)	India (%)	USA (%)
Biofuel	56.7	66.4	92.5	9.1
Wildfire	17.0	0	0	3.3
Consumer product wise	6.9	0.9	0.6	35.1
Traffic oil	4.8	2.0	IS	23.0
Coke production	3.7	10.7	1.3	IS
Domestic coal	3.6	14.4	IS	IS
Petroleum refining	2.4	1.0	IS	8.7
Waste incineration	1.9	IS	IS	9.5
Aluminum electrolysis	1.4	IS	IS	1.9
Open straw burning	IS	2.0	3.2	IS
Gasoline distribution	IS	IS	IS	3.0
Aerospace industry	IS	IS	IS	2.5
Other	1.5	0	2.7	3.9
Tonnes in thousand	530	114	90	32

IS Insignificant

day. Throughout the country, during winter BaP fallout varied between 0.3 and 1100 ng m<sup>-2</sup> per day [13].

PM<sub>10</sub>-bound BaP emitted from domestic heating sources was responsible for the lung cancer cases in various parts of cities of Poland. In rural areas, the average concentration of BaP was 1.39–4.86 ng m<sup>-3</sup> [14]. PAH levels in the atmosphere are very low, i.e., ≤ ng m<sup>-3</sup>; therefore, the measurement methods for their recording, collection and analysis study are very complex and have high uncertainties values due to low recovery in spite of large sampling volume [15]. This paper deals with the concentration of BaP in the PM<sub>10</sub> samples of New Delhi collected in the year 2014–2015. The uncertainty components during sampling extraction and analysis of BaP are also taken into consideration.

## 2. Materials and Methods

The sampling was carried out for PM<sub>10</sub> at NPL (National Physical Laboratory) in New Delhi for a year from January 2014 to January 2015 using a low-volume sampler (Envirotech APM 550) [16]. This measurement site was selected as it is a good representative site of urban aerosols of New Delhi with the mixed effect of industrial, vehicular, residential and agricultural emissions [17]. A total of 104 samples collected during year-round sampling as per NAAQS.

BaP was extracted from the exposed PM<sub>10</sub> sample. The sample filter (quartz) was cut into a 14-mm-diameter disk.

**Table 2** Gradient flow of mobile phase with composition and time

Time (min)	Water (%)	Acetonitrile (%)	Flow (mL/min)
0.01	50	50	0.5
20	0	100	0.5
35	0	100	0.5
40	50	50	0.5
45	50	50	0.5

The filter papers were then extracted with toluene using an ultrasonic bath. The toluene extracts was then passed through a silica gel column for the cleanup process using cyclohexane (HPLC grade) [18]. Finally, before analysis, the extract was evaporated on a rotary evaporator till dried up and was dissolved in 1 mL acetonitrile (HPLC grade). The extract obtained was then analyzed for BaP through a high-performance liquid chromatography (HPLC) system (Shimadzu Prominence Model LC-20AD) with programmed fluorescence detection (Shimadzu RX-10AXL).

Analysis of the BaP was carried out using a HPLC reversed phase C18 column (dimension 250 mm × 4.6 mm, make Shim-pack), with a elution of binary gradient from 50% acetonitrile (HPLC grade) and water mixture to 100% acetonitrile in 45 min (Table 2). BaP was detected in PM<sub>10</sub> samples by means of a fluorescence detector with respect to its retention time. The fluorescence detection was done at 361 nm (excitation wavelength) and at 405 nm (emission wavelength).

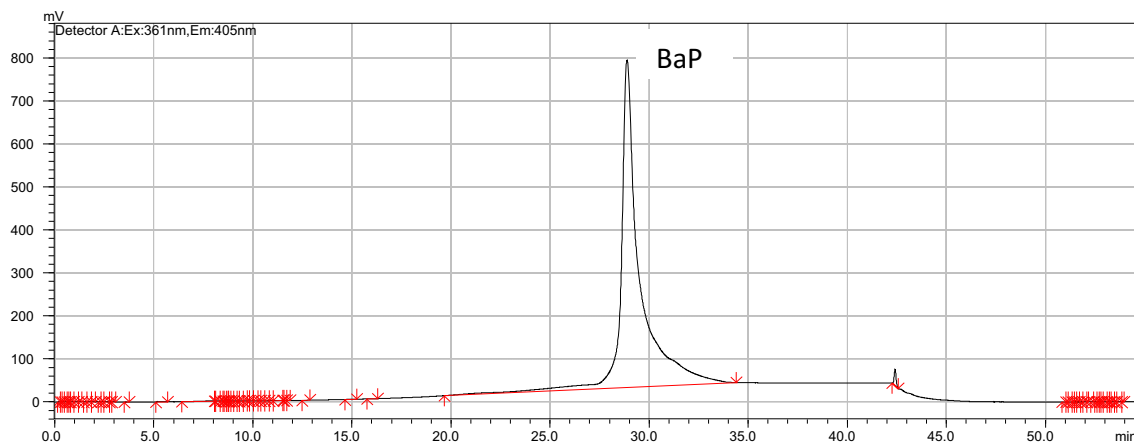
The method was tested with quality assurance (verification of instrument calibration and repeatability test of the instrument). Two replicates of blank samples were used as real samples to check any contaminations of the analytes. The stock solution of standard was prepared by diluting aliquots of the BaP standard (Sigma-Aldrich, B1760). For calibration, BaP standard solution was prepared by successive dilution of stock solutions with acetonitrile (by weight). Calibration standard solutions were stored at 4 °C in the dark.

In India, the regulatory limit of BaP in National Ambient Air Quality Standards is 1 ng m<sup>-3</sup> (annual). The PM<sub>10</sub> sample filters were analyzed for BaP according to the ISO 16362: 2005. The data was calculated as follows.

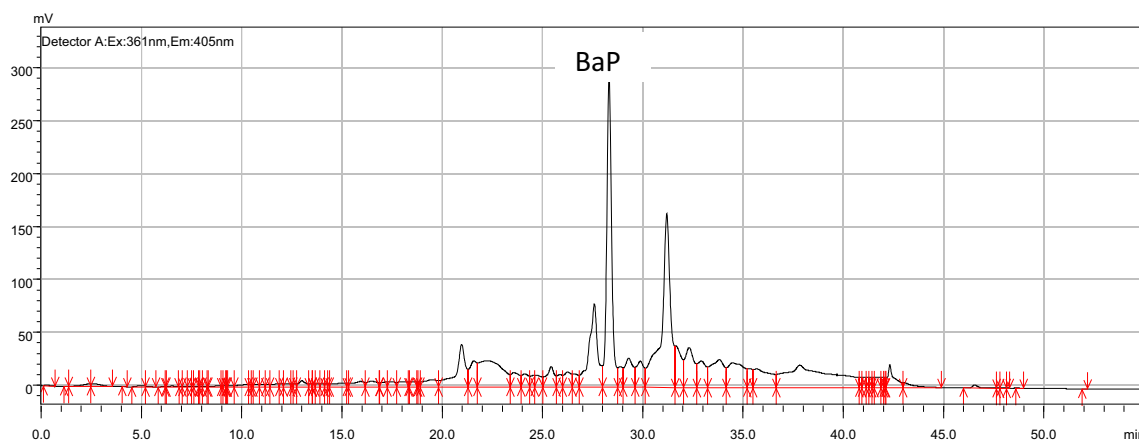
The BaP concentration in PM<sub>10</sub> samples (in ng m<sup>-3</sup>) is calculated from the Eq. (1) given as:

$$\begin{aligned} \text{Concentration of BaP (ng m}^{-3}\text{)} \\ = \frac{C(\text{ng m}^{-2}) \times A_{\text{sample}}(\text{m}^2)}{V_{\text{air}}(\text{m}^3)} \end{aligned} \quad (1)$$

where  $C$  is the BaP concentration calculated from the analytical data and area of filter taken (ng m<sup>-2</sup>),  $A_{\text{sample}}$  is



**Fig. 1** HPLC chromatogram of Benzo(a)pyrene standard



**Fig. 2** HPLC chromatogram of Benzo(a)pyrene (BaP) for PM<sub>10</sub> sample

the total area of the PM<sub>10</sub> filter exposed ( $m^2$ ),  $V_{\text{air}}$  is the volume of the sampled air ( $m^3$ ).

Calibration of the instrument was done by injecting three different concentrations of BaP standards as a function of peak area using a linear curve. The peak identification of BaP in PM<sub>10</sub> samples was done by comparing with the retention time of standard (Figs. 1 and 2). The instrument was calibrated after every 10 analysis of the sample. Other statistical data for instrument calibration, i.e., correlation coefficients ( $R^2$ ), standard deviation (SD) etc., along with a limit of detection (LOD) and limit of quantification (LOQ) were also calculated and are listed in Table 3.

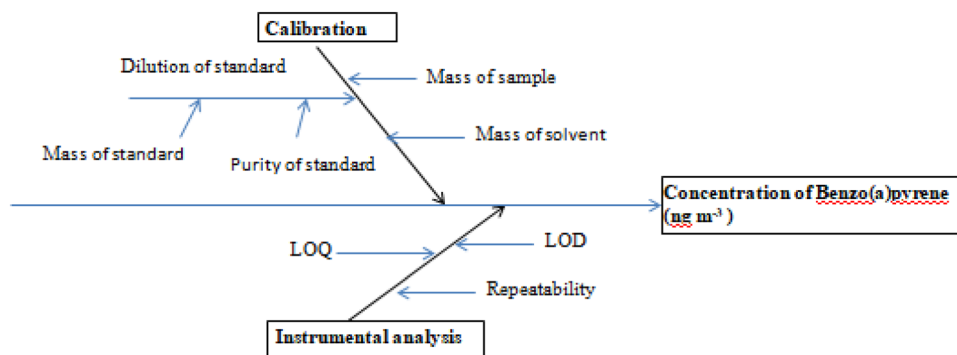
### 2.1. Calibration Range and Linearity

In this method, the five-point calibration curves were prepared with different concentration levels of BaP standard (Table 3). The calibration range for the method is the interval between the lower to the higher levels of BaP that have been used to determine precision, accuracy and linearity. Linearity is determined by a series of 5–6 injections of five or more BaP standards whose concentrations are 80–120% of the probable concentration range [19]. Linearity and matrix effects were calculated by a plot of the linear regression equation for BaP (Table 3).

**Table 3** Statistical data for HPLC calibration

Compound name	Retention time (min)	Calibration range (ppm)	Precision (%)	Correlation coefficients ( $R^2$ )	LOD (ppm)	LOQ (ppm)
BaP	28.8	0.025–1.00	0.89	0.9928	0.011	0.021

**Fig. 3** Cause-and-effect diagram showing uncertainty components for the analysis of Benzo(a)pyrene



## 2.2. Limit of Detection (LOD) and Limit of Quantification (LOQ)

LOD was calculated to check the sensitivity of the instrument [19, 20]. LOD and LOQ were obtained by analyzing ten aliquots of PM<sub>10</sub> sample with the smallest concentration of the BaP standard that will generate a valid quantifiable peak at the signal-to-noise ratio > 3:1 and 10:1, respectively, for a 20 µL of injection.

Thus, LOD was calculated according to the EUR-ACHEM method as follows:

$$\text{LOD} = 3.3 * \text{Standard Deviation}$$

The LOQ was calculated as:

$$\text{LOQ} = 10 * \text{Standard Deviation}$$

The calculated values of LOD and LOQ were 0.01 ppm and 0.02 ppm, respectively (Table 3).

## 2.3. Precision

Precision is the nearness of conformity involving individual analysis results obtained under validated conditions. It is generally expressed in the form of standard deviation or relative standard deviation. The precision of method used in the analysis was obtained by the repeatability of measurements (intraday) and intermediate precision (interday) of both BaP sample and standard solutions. Precision was determined by analyzing six replicates of both BaP standard and sample solution on the intraday, i.e., same day and six times in the interday, i.e., each day for 1 week. The results obtained were expressed in terms of % RSD of the analysis. (Table 3).

## 2.4. Uncertainty Associated with Standard and Sample Preparation

The main sources of uncertainty in the preparations of standard stock solutions in the HPLC method as shown in Fig. 3 were as follows:

Mass of standard (Mst) and Purity of the standard (P)

Uncertainty in mass weighing was calculated for standard and sample according to Equation:

$$u(\text{Mst}) = \frac{S}{\sqrt{n}} \quad (2)$$

where  $s$  represents the standard deviation (as per the balance certificate), and  $n$  is the number of measurements of mass weighing of 20 g weight (in case of standard) and 1 g weight in case of sample), respectively.

The purity of BaP standard is given in the form  $\geq y\%$  and the range  $(100 - y)\%$  is described as the rectangular distribution. The standard uncertainty is therefore calculated as:

$$u_p = \frac{100 - y}{2\sqrt{3}} \quad (3)$$

## 3. Results and Discussion

### 3.1. Uncertainty Calculation Involved in the Measurement of BaP Concentrations

The uncertainty components involved in the measurement of concentration of BaP as discussed are calculated in Table 4. The major contribution of uncertainty components was of the purity of the standard (77.9%). The other parameters like mass of standard and sample contributed to 8.5% and 13.5%.

The combined uncertainty is calculated by summing up the square of all the uncertainty components calculated.

**Table 4** Uncertainty components and their standard and relative uncertainties associated with the preparation of standard and sample of BaP by HPLC

Symbol	<i>U</i> component	<i>x</i>	<i>U</i> ( <i>x</i> )	<i>U</i> ( <i>x</i> )/ <i>x</i>
Mst	Standard mass	20 g/g	4/√10 g/g = 1.265	0.06
Up	Standard purity	2 mg L <sup>-1</sup>	2/√3 mg L <sup>-1</sup> = 1.155	0.57
<i>M</i> <sub>sample</sub>	Sample mass	1 g/g	0.2/√4 g/g = 0.1	0.10

The input quantities are those that are responsible for the uncertainty of the analytical method.

$$u_c = \sqrt{(Mst)^2 + (up)^2 + (uM \text{ sample})^2} \quad (4)$$

The combined uncertainty (*u<sub>c</sub>*) is calculated as 0.35.

Calculation of the expanded uncertainty (*U*) is done by multiplying the combined standard uncertainty (*U<sub>c</sub>*) of the input quantity with *k* (coverage factor). The value of *k* = 2.

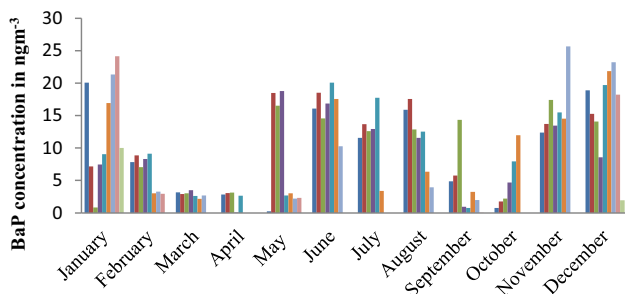
$$u = u_c \times k \quad (5)$$

### 3.2. Seasonal Variation of BaP in PM<sub>10</sub> Samples

The total year was classified into four seasons viz., winter, summer, monsoon and post-monsoon. The winter months are from December to February. March to May are the summer months. Monsoon starts from June to last in the month of August. Post-monsoon months are from September to November. The temperature of Delhi varies from lowest in winter to highest in summer. The average rainfall during monsoon season in Delhi is normally of the order of ~ 800 mm. Delhi has a subtropical climate and, due to emission of trace gases from various sources of emission, the ambient air quality is considerably affected [21].

Figure 4 shows the monthly concentration (ng m<sup>-3</sup>) of BaP calculated for the year 2014–2015 in NPL, New Delhi. The seasonal variation in concentrations of B(a)P was found to be 0.04–25.7 ng m<sup>-3</sup> in PM<sub>10</sub> samples collected at the sampling site from January 2014 to January 2015.

A general trend in the monthly distribution of BaP is observed. Referring to the trend graphs, we observe that

**Fig. 4** Monthly BaP concentration in PM<sub>10</sub> samples in the year 2014–2015

BaP concentrations decrease during the monsoon and post-monsoon season, i.e., June–September. This is possible due to the scrubbing effect of precipitation. During the rains, the suspended particulates in the air settle down, and therefore, the concentration of pollutants in the ambient air is reduced [22]. Winter months experience a great increase in BaP levels (November–January) possibly because of the additional fuel combustion, biomass burning, increase in vehicular traffic etc., [17, 23]. Major vehicular emission include both diesel and natural gas combustion, while the emissions from vehicles using coal, coke, and gasoline as fuels have a minor contribution [24]. The high BaP concentration in the winter months was due to the stable inversion layer in which the pollutants remain suspended in the air for long and have greatly increased residence times. This makes winter months particularly harmful for asthmatics and people suffering from other respiratory disorders. As the inversion layer starts to break in the summer months, the pollution level also starts decreasing.

In Delhi, there has been increase in number of vehicles over the years which affect the entire consumption of fuel. This also increases the idling time of vehicles leading to traffic congestion which finally results in increased emission of hydrocarbons, CO and NO<sub>x</sub> [25]. As given by CPCB (Central Pollution Control Board), the average mean mass concentration of PM<sub>1</sub> PM<sub>2.5</sub> and PM<sub>10</sub> was 3–4 times higher than the permitted values given in NAAQS due to emission from the crop residue burning [26].

Similar work was done on by collection of PM<sub>10</sub> samples using a low-volume sampler on quartz filters for 24 h each day for a period of 15 days between October 2012 and July 2013 in Naples. The PM<sub>10</sub> mass concentration was determined gravimetrically. It was observed that concentration of BaP varied from 0.065 ng m<sup>-3</sup> during autumn season to 0.872 ng m<sup>-3</sup> in spring season and from 0.120 ng m<sup>-3</sup> during autumn season to 1.48 ng m<sup>-3</sup> of winter season. The major sources observed for the increase in levels of BaP were from the traffic emission [27].

In North India, there is a high variation in all the seasons as compared to South India. In the western part of India, there are deserts and western disturbances. Apart from the natural influences, increase in the urbanization also result in the changes in the atmospheric conditions [28].

Therefore, in recent years, the concentration of BaP reported in the ambient air of India is much higher than developed countries like Germany, Italy, Greece, USA, and UK. The concentration of BaP in India was also reported higher than Asian countries like Japan, Korea, Hong Kong, and Taiwan but was comparable with China [29].

Any type of small error in the measurement of aerosols can have a strong impact on its understanding. Therefore, to understand the physical and chemical properties of aerosols, their measurements should be accurate [30]. National metrology institute (NMI) should provide the calibration requirements of industries involved in the gas and aerosol metrology. They should also work with the regulatory authorities for traceable aerosol measurement data [31]. NMI should collaborate with each other and also with other institutes for calibration and standardization of measurements which will reduce the uncertainties [32].

#### 4. Conclusions

From the study, it can be concluded that risk associated with high PAHs concentrations still persists in New Delhi. The average annual levels are 10–20 times higher than the permitted value of  $1 \text{ ng m}^{-3}$ . The measurement of uncertainty of various parameters involved in the measurement of BaP is also calculated which is an important aspect for quality assurance and quality control studies.

BaP level is very high in winters (November–January) as compared to summers (March–May). The main reason behind the high PAHs concentration in New Delhi is the vehicular exhaust due to increase in number of vehicles over the years.

Measures should be taken to control the vehicular exhausts especially diesel exhaust by implementing strict emission norms. Also the use of solid fuels should also be reduced which will help to lower the BaP concentrations in the atmosphere.

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