



## Characterization and Source Identification of PM<sub>10</sub> bound Polycyclic Aromatic Hydrocarbon (PAHs) in Semi-Arid Region of India

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Available online at: [www.isca.in](http://www.isca.in), [www.isca.me](http://www.isca.me)

Received 16<sup>th</sup> February 2015, revised 17<sup>th</sup> March 2015, accepted 16<sup>th</sup> April 2015

### Abstract

Respirable particulate (PM<sub>10</sub>) and particulate phase polycyclic aromatic hydrocarbons (PAHs) in ambient air were measured by collecting samples during winter, summer and post-monsoon season at rural environment in north-western part of India. The concentration of 8 selected PAHs, Acenaphthene (Ace), Fluorene (Flu), Phenanthrene (Phen), Anthracene (Anth), Fluoranthene (Flt), Pyrene (Pyr), Chrysene (Chr), Benzo[a]pyrene (BaP) were quantified and characterized for different seasons. The ratio of average total PAHs concentration of winter to summer and winter to post-monsoon were assessed for rural environment was 5.7 and 6.4 for summer and post-monsoon season respectively which is in agreement with the general trend of 1.5-10. The four membered ring PAHs were predominant and contributed to about 62% of total PAHs during winter season. A correlation study was performed among selected PAHs for source identification for all the three seasons. The results are in agreement with the traditional method of burning biomass, wood, coal and agricultural waste for cooking, a practice still followed in rural environment.

**Keywords:** Akkalkuwa, aerosols, co-relation, fluorescence, PAHs.

### Introduction

Polycyclic aromatic hydrocarbons (PAHs) have well established carcinogenic and mutagenic properties and hence are of major health concerns<sup>1</sup>. High molecular weights PAHs are mostly associated with particulates as compared to low molecular weights which are concentrated more in vapor phase. It is therefore imperative to establish the presence, distribution and potential sources of PAHs in ambient air in aerosols, so that the pollution due to PAHs could be controlled efficiently. The main sources of PAHs in cities are road traffic and certain industrial processes. In order to enhance the possible emission source, statistical tools were used in many studies<sup>2</sup>. Since, most of the PAHs are associated with particulates may vary significantly with respect to their emission sources, some PAHs concentration ratio have been used to indicate traffic emission sources. The amount and type of PAHs indicate seasonal fluctuations with mostly higher concentration in winter<sup>3</sup>.

In the present study, the concentration of PAHs in particulate phase has been evaluated in Akkalkuwa, Nandurbar, Maharashtra representing rural environment with the application of fast analytical techniques. Samples were collected from the ambient air in particulate phase and variation in PAHs concentration were assessed during winter, summer and post-monsoon (2009-10). Statistical analysis was also carried out to assess the possible sources of PAHs in a rural environment.

### Material and Methods

**Study Area:** The Akkalkuwa, Nandurbar, Maharashtra is

located in Satpuda range hills. The river Narmada is on the northern side of the village. This is typical Indian rural area of a semi arid region in North western side of the State (figure-1). The area was hilly terrain and falls in forest area. The area of Akkalkuwa spread about 878 sq. km. with population of 17737. Traditional agriculture is the main occupation of farmers and have unique crop diversity which includes maize, sorghum, minor millets and pulses. The city of Nandurbar District is generally hot and dry temperatures can be as high as 45°C during the peak of Summer and 11°C in winter season. The average rainfall in the village is 859 mm. The rapid urbanization has resulted in the increased utilization of fuels for transportation and wood for domestic purposes and type of fuel used for cooking in homes. The types of fuel used in vehicles are all important parameters that influences the PAH concentration in any area.

**Sampling Methodology: Aerosol Sampling:** Aerosols sample was collected from Akkalkuwa Station on 24 hrs basis using pre-weighted quartz fibres during November 2009-October 2010 representing winter (December-January-February), summer (March-April-May) and post-monsoon (September-October-November) season. Samples were collected at a flow rate (1.1-1.3 m<sup>3</sup>/min) using particulate samplers (APM-460 Envirotech India) at 3 m above the ground level. The procedure followed for preparation of the blank filters were 2 h pre-heating at 550±C, 48-h equilibration of the filters in an air-conditioned weighing room at 20±C and 40% relative humidity. After particle collection, the filters were kept in desiccators for about 48 hrs and subsequently measured the weight of the aerosols. The exposed filters were immediately frozen to minimize losses

volatile compounds after re-weighing. After re-weighing, the exposed filters were stored in a freezer to limit losses of volatile components. From the difference of the initial and final weights and volume of air sampled, the mass concentration of fine particulate matter was calculated by dividing mass deposited by volume of air.

**Sample Preparation: Aerosols:** The fraction of filters trapped with particulate matter was cleaned by sonication applying 5 ml dichloromethane (DCM) in a 15 ml glass vial. The sonication was done for 15 min in ultra-sonication. The process of ultra-sonication was treated using 5 ml methanol for the same used filter paper in the same vials. This process was done for all samples. The vial was left overnight. The white crystalline content obtained in the glass vial was dissolved by adding 5 ml acetonitrile and agitated in water bath for 3 hours at 70 rpm, later the glass vial was centrifuged and supernatant was collected and analysed. The supernatant collected was put for analysis by fluorescence spectrophotometer.

For PAHs extraction from air particulates, sonication was applied in the study because of its easy procedure, no need of large volumes of organic solvents and lesser extraction time. This is inexpensive, yielded high recoveries and fast method for sample extraction, when applied to aerosol samples. Also the ultrasonic method is well suited for samples with lower PAH concentration, such as rural environment and suitable for airborne particulate samples.

**Sample Analysis: Fluorescence Spectrometry Analysis of PAHs:** Model F-4500, Hitachi Japan. Fluorescence spectrometry was used for PAHs quantification. The system has optimized for software and hardware in the widest range of fluorescence applications. The principle of fluorescence emission is a type of photoluminescence in which a molecule is promoted to an electronically excited state by adsorption of ultraviolet visible near infrared radiation. Then, the excited light source is commonly a xenon arc lamp. This lamp has an intense emission spectrum from 200 – 900 nm, which is capable to excite molecules using both ultraviolet and visible wavelength ranges. The location of the peak indicates the type of fluorophore. (a molecule to fluorescence) emitting the fluorescence. Peak intensities represent concentrations of targeted molecule in the sample. The quantification of trace concentration of fluorescence respective compounds is difficult to accomplish due to the interference of matrix with other compounds which quenches fluorescence yield.

The emission fluorescence is detected by a photomultiplier tube (PMT) positioned at a 90° angle from the incident light path. The analytical and technical detail of fluorescence spectrophotometer used in the study is given in table-1. For peak measurement, the scan speed of 2400 nm/s and PMT voltage 700 V was maintained. Excitation and emission slit width were 5nm. Excitation source was 100 W xenon lamp. A fluorescence wavelength was set between 300 and 500 nm wavelength, respectively, for excitation and emission and adjusted for maximum selectivity.

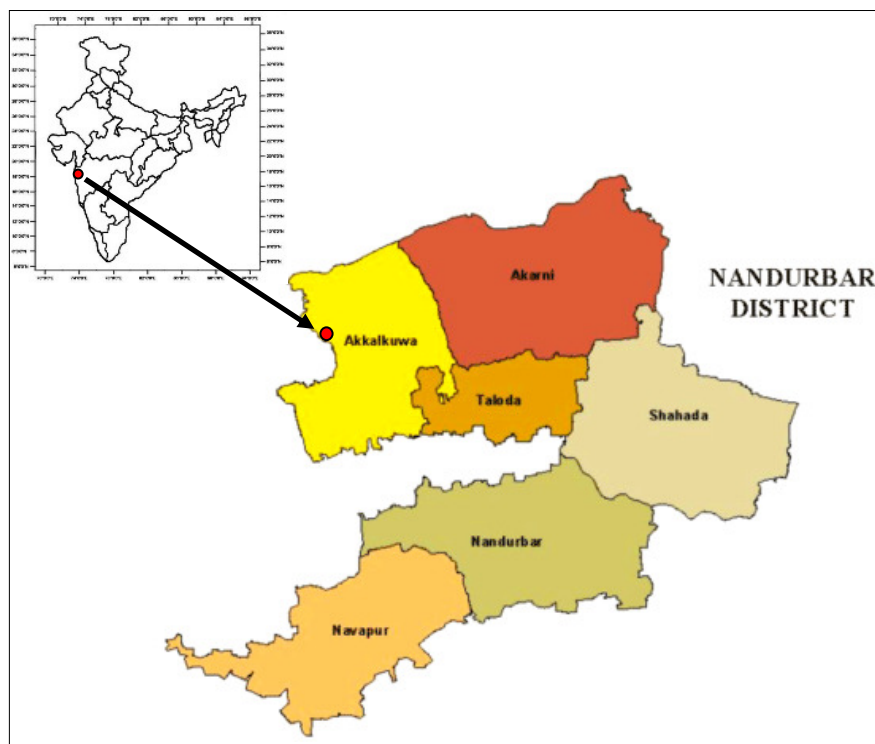


Figure-1  
Sampling Location of Akkalkuwa

**PAHs Concentration in Standard Solution:** The fluorescence spectrophotometer was calibrated by using PAHs mix standards (Accu Standard, AE-00025, New Heaven, CT). It contained 8 analytes in acetonitrile. Acenaphthene (Ace) Fluorene (Flu), Phenanthrene (Phen), Anthracene (Anth), Fluoranthene (Flt), Pyrene (Pyr), Chrysene (Chy), Benzo[a]pyrene (BaP). Three levels of concentrations were made from the standard PAHs mix in the concentration range 2.5, 5 and 10 parts per billion (ppb). Identification of peak was done on the basis of wavelength. Each level of PAHs standard was analyzed and intensity was obtained from the axes of wavelength versus intensity.

Peak intensity was calculated at the experimental wavelength versus intensity peak. The concentration of all fluorescent PAHs was determined in air particulate samples using following expression.

$$\text{Conc. of PAHs} = \frac{\text{Peak intensity of individual Sample}}{\text{Peak intensity of std}} \times \text{conc. of calc. PAHs} \quad (1)$$

**Quality Control and Data Analysis:** During the analysis, known amount of standard mixtures were spiked onto blank filters to ensure Quality Control (QC). In all 8 unknown samples, two filter blanks and 2 filter QC samples were taken for analysis. Also an external recovery standard and calibration standards were also analyzed in parallel along with sample extracts. The external recovery standard included all the eight PAHs. In addition the following steps were followed to ensure complete QC during the course of analysis. These include the measurement of the target analytes in the QC sample measurements which may not fall above or below 2 standards.

Further quality assurance was ensured by conductivity and drift check. Fluorescence intensities were recorded for de-ionized, distilled water at 277 nm EX/303 nm EM to assure consistent measurements between analyses. Similarly, wavelength accuracy checks were also be made four times to assure consistent emission from the xenon lamp. The parameters were

set (according to software guidelines) to analyze a standard diffusion parameters.

## Results and Discussion

**Seasonal Variation in PAHs concentration profile:** The concentration of PAHs in different seasons is given in table-2. The concentration of total PAHs ranged between 2.67 and 17 ng m<sup>-3</sup> while the concentration of total PAHs in winter showed highest concentration 17 ng m<sup>-3</sup>. The fluorescence responsive PAHs found in PM<sub>10</sub> are (Ace), (Flu), (Phen), (Pyr), (Chr), (Anth), (BaP) and (Flt). The total PAHs concentration measured was found in the range of 1.1-4.9 ng m<sup>-3</sup> for Flt, 1.06-6.4 ng m<sup>-3</sup> for Pyr, 1.13-4.21 ng m<sup>-3</sup> for Phen, 0.40-3.91 ng m<sup>-3</sup> for Ace, 1.04-4.34 ng m<sup>-3</sup> for Chr, 0.46-3.81 ng m<sup>-3</sup> for Anth, 0.42-0.92 ng m<sup>-3</sup> for BaP and 1.05-6.29 ng m<sup>-3</sup> for Flu. The increasing order of concentration of individual PAHs in the aerosols follows the decreasing order as Pyr>Flt>Phen>Flu>Chr>Anth>Ace>BaP. The 4 membered ring PAHs were more dominant (50%) followed by 3 membered ring PAHs (46%) and 5 membered ring PAHs (4%). The high concentration of 4 membered ring PAHs could be attributed to low temperature pyrolysis of biomass burning. Pyr is the most dominant 4 membered ring at the sampling site whereas the Chr and BaP and others PAHs found in the rural environment. The major sources of PAHs are biomass burning and refuse combustion. The mean concentration of BaP was 0.42 ng m<sup>-3</sup> and concentration range found was between 0.52-0.98 ng m<sup>-3</sup>, respectively.

During summer, the PAHs concentration was ranged between 2.51-3.79 ng m<sup>-3</sup> with an average 2.96 ng m<sup>-3</sup>. The PAHs compounds in PM<sub>10</sub> found were Ace, Flu, Phen, Pyr, Chr, Anth, Bap and Flt following increasing order of concentration follows Chr> Pyr> Anth> BaP> Phen> Flu> Ace. In summer, the concentration contribution of 4 membered ring PAHs was 63%. The 3 membered ring PAHs contributed 28% and 5 membered ring PAHs contributed 9%. The mean concentration and the range of BaP in aerosols were 0.15 and 0.12-0.22 ng m<sup>-3</sup> respectively.

**Table-1**  
**Wavelength and Peak Intensity of PAHs Standard Solutions**

Sr. No.	Fluorescent Compound	PAHs	Theoretical Wavelength range (nm)	Experimental Wavelength (nm)	Peak Intensity of Standard Sample (a.u)	Calculated Conc. (ppb)
1	Acenaphthene		320 -370	306.0	5696	0.78
2	Fluorene		330 -340	313.0	5154	1.25
3	Phenanthrene		340 -420	364.2	1853	0.93
4	Pyrene		360 -420	385.0	2090	1.24
5	Chrysene		370 -398	393.6	1649	0.62
6	Anthracene		394 -408	407.0	3247	0.78
7	Benzo[a] pyrene		400 -460	431.4	2018	0.62
8	Fluorene		400 -500	454.6	972.9	1.26

In post-monsoon season, the concentration of total PAHs and average ranged between 1.63-3.59 and 2.74 ng m<sup>-3</sup>. The increasing order of concentration of PAHs in the aerosols followed: Chr>Pyr>Anth>Phen = Flu>BaP>Flt>Ace. The 4 membered ring PAHs dominated (63%) while 3 membered ring (30%) and 5 membered ring (7%) compounds contribution was lower as compared with the 3-membered ring. Chr was the most dominant 4 ring compound like summer. The mean concentration and the range of BaP in ambient air were 0.18 and 0.13-0.29 ng m<sup>-3</sup> respectively.

Among the three seasons, relatively higher concentrations of PAHs were found in winter as compared with summer and post-monsoon seasons. Source emissions and meteorological conditions as well as gas particle partitioning may result in winter, summer and post-monsoon difference of PAHs concentrations. As emission increased is due to the biomass, wood and coal burning may be the reason for the higher concentrations in winter. Lower temperatures and atmospheric mixing heights as well as decreased photochemical oxidation intensity may also be another reason of the higher PAHs concentrations in winter.

Ratio of winter to summer and winter to post-monsoon were assessed for total PAHs in rural environment. It was observed that the average ratio was 5.7 and 6.4 for summer and post-monsoon with respect to winter season respectively. The ratios of PAHs in winter were generally higher by a factor of 1.5-10

than that in summer. In summer lower PAHs levels were likely to be attributed to the combination of easier atmospheric dispersion of pollutants, wash-out effects, and to a lesser extent, photo-degradation and higher percentage in the air in vapor phase. The reported ratios of winter to summer total PAHs was 8.6 at Hung Hom (PU) site and 7.5 at Kwun Tong (KT) site of Hong Kong, respectively.

In all the seasons, the low molecular weight PAHs compounds concentrations was observed to be low which may be due to higher tendency to evaporate. These PAHs compounds have a high vapor pressure and also have tendency to exist in the gas phase, thus easily evaporated as compared to higher PAHs. The low molecular weight PAHs which were lighter tend to remain in the gaseous phase than the particulate phase aerosols.

**Comparison of PAHs Concentration with Other Cities:**

Comparison of PAH concentrations in different areas of Indian cities provided some insight on the spatial distribution as well as local factors influencing PAH profiles. The total PAHs concentration at Akkalkuwa ranged between 2.67-17ng m<sup>-3</sup> which is less as compared to other sites in India and other cities (table-3). The concentration of PAHs was 53 ng m<sup>-3</sup> at Delhi while Vishakapatnam and Agra showed total PAHs concentration 57 and 120 ng m<sup>-3</sup> respectively<sup>8-9</sup>. The highest total PAHs concentration reported at Agra was 154 ng m<sup>-3</sup><sup>10</sup>. Taiwan urban sampling station reported highest total PAHs concentration of 831 ng m<sup>-3</sup>.

**Tables-2**  
**Seasonal Variation in PAHs**

	PM <sub>10</sub>	Flt	Pyr	Phen	Ace	Chr	Anth	BaP	Flu	Total PAHs
	Mol. Wt.	202	202	178	154	228	178	252	166	
	ring	3	4	3	3	4	3	5	4	
	µg m <sup>-3</sup>	ng m <sup>-3</sup>								
<b>Winter</b>										
Min	56	1.11	1.06	1.13	0.4	1.01	0.46	0.42	1.05	11.6
Max	95	4.9	6.54	4.21	3.91	4.34	3.81	0.92	6.29	23.4
Med	78	2.73	3.52	2.25	1.76	1.87	1.62	0.58	2.14	16.9
Avg	77	2.63	3.57	2.36	1.74	2.01	1.83	0.61	2.25	17
SD	12	0.8	1.32	0.87	0.77	0.86	0.87	0.12	1.07	2.9
%		15	21	14	10	12	11	4	13	
<b>Summer</b>										
Min	65	0.09	0.1	0.14	0.02	0.68	0.08	0.29	0.73	3.79
Max	110	0.4	0.63	0.6	0.16	2.54	0.64	0.26	0.12	2.51
Med	90	0.22	0.34	0.26	0.07	1.36	0.27	0.28	0.22	2.69
Avg	89	0.22	0.35	0.28	0.07	1.42	0.31	0.33	0.15	2.96
SD	14	0.07	0.13	0.12	0.03	0.53	0.15	0.35	0.29	3.22
%		7	11	9	2	44	10	9	8	
<b>Post-monsoon</b>										
Min	44	0.06	0.08	0.12	0.01	0.56	0.09	0.13	0.11	1.63
Max	74	0.27	0.47	0.51	0.14	2.09	0.75	0.29	0.68	3.59
Med	61	0.15	0.25	0.22	0.06	1.12	0.32	0.18	0.23	2.74
Avg	60	0.15	0.25	0.24	0.06	1.17	0.36	0.19	0.24	2.67
SD	10	0.04	0.09	0.1	0.03	0.44	0.17	0.04	9.15	0.5
%		5	10	9	2	44	13	7	9	

Studies reported across the world showed high concentration of PAHs as Seoul Korea (89 ng m<sup>-3</sup>). Chicago, USA (13-1869 ng m<sup>-3</sup>). Compo Grade, Brazil (8.9-62.5 ng m<sup>-3</sup>). Brisbane, Australia (0.4-19.73 ng m<sup>-3</sup>). Taichung Urban, Rural and industrial (220, 830, 1620 ng m<sup>-3</sup>). Hong Kong (ng m<sup>-3</sup>), Hung Hom (15.9 ng m<sup>-3</sup>), Kwun Tong (23.56 ng m<sup>-3</sup>)<sup>5,7,11-13</sup> respectively.

**Table-3**  
**Comparison of Total PAHs (ng m<sup>-3</sup>) with other Cities**

Cities	Total PAHs Conc.	References
Delhi ITO (January)	52.96	15
Paravada, Vishakapatnam	57	8
Amritsar	154	10
Agra	120	9
Seoul, Korea	89	11
Chicago, USA	13-1865	5
Campo Grande, Brazil	8.94-62.5	12
Brisbane, Australia	0.4 - 19.73	13
Tai Chung, Taiwan urban	220 ± 520	2
Tai Chung, Taiwan rural	831± 427	
Tai Chung, Taiwan industrial	1650 ±1240	
Hong Kong	5.30	16
Akkalkuwa (winter)	17	Present Study
Akkalkuwa (summer)	2.96	
Akkalkuwa (Post-monsoon)	2.67	

**Correlation of Particulate Phase PAHs:** Major sources of PAH pollutants in the study are attributed to be biomass, wood and coal burning for domestic purpose. A co-relation was performed among selected PAHs to estimate the type of PAHs most common in the ambient air. In the present study, the statistical package for Social Scientist (SPSS version 10.0) computer software was used to perform analysis<sup>14</sup>. The concentrations of selected PAHs compounds were used to indicate towards the corresponding emission sources, such as Anth, Phen, Flt, Pyr, and Chr for coal combustion and Anth, Phen, Flt and Pyr for combustion of wood, Flt and Pyr for oil burning. Flt and Pyr for petrol/diesel powered vehicles. These markers indicate some degree of similarity and overlap between the profile from different emission source categories. During winter season, significant correlation was observed for Ace vs Flu (r=0.80), Ace vs Pyr (r=0.68) and Ace vs Flt (r=0.65) indicating that PM<sub>10</sub>, particulate phase PAHs in rural environment was generated mainly through in house combustion of coal and biofuel (table-4). In addition coal fire wood and agricultural waste straw are the main fuels for cooking winter season. Further traditional biomass combustion for cooking is still popular in the rural environment. The correlation of Flt vs Chr (r=0.92), Chr vs Anth (r= 0.88). Phen vs Pyr (r=0.78) and Pyr vs Chr (r=0.58) indicated burning of coal

for cooking medium, however, atmospheric concentrations of PAHs were higher during winter season and likely caused by biomass, wood and coal burning for domestic use<sup>9</sup>. During summer and post-monsoon season, significant correlation was obtained among PAHs like Ace, Pyr, Chr, Anth, BaP and Flt. Significant correlation among PAH such as Pyr and Chry point out the wood combustion sources biomass burning like wood, dung cake burning activities<sup>8</sup>.

## Conclusion

The particle phase PAH concentration were characterized at Akkalkuwa, Nandurbar, Maharashtra during winter, summer and post-monsoon seasons. The concentration of total PAHs as well as the individual compounds was higher in winter as compared to summer and post-monsoon seasons which may be due to wood, coal burning for cooking and other household purposes. The average total PAHs concentration was about 5.7 and 6.4 times higher than that of summer and post-monsoon season. The total PAHs with comparative ring sizes showed significant correlation with each other due to similarity both in their sources and in physic-chemical properties. Based on the co-relation analysis it is concluded that biomass, wood and coal burning were the predominant source of PAHs in rural environment.

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**Table-4**  
**Correlation Studies of PAHs**

	Ace	Flu	Phen	Pyr	Chr	Anth	BaP	Flt
<b>Winter</b>								
Ace	1.0							
Flu	<b>0.81</b>	1.0						
Phen	0.12	0.08	1.0					
Pyr	<b>0.68</b>	-0.34	<b>0.78</b>	1.0				
Chr	<b>0.51</b>	0.49	0.42	<b>0.58</b>	1.0			
Anth	<b>0.62</b>	<b>0.68</b>	-0.09	-0.19	<b>0.88</b>	1.0		
BaP	<b>0.55</b>	<b>0.58</b>	-0.08	-0.31	0.42	-0.28	1.0	
Flt	<b>0.65</b>	<b>0.67</b>	0.11	-0.28	<b>0.92</b>	<b>0.92</b>	-0.28	1.0
<b>Summer</b>								
Ace	1.0							
Flu	<b>0.52</b>	1.0						
Phen	0.08	-0.14	1.0					
Pyr	<b>0.52</b>	-0.52	<b>0.69</b>	1.0				
Cht	<b>0.56</b>	0.42	0.12	0.48	1.0			
Anth	<b>0.53</b>	<b>0.52</b>	-0.28	-0.41	<b>0.72</b>	1.0		
BaP	<b>0.61</b>	<b>0.51</b>	-0.35	-0.52	0.21	-0.52	1.0	
Flt	<b>0.59</b>	<b>0.53</b>	0.41	-0.48	<b>0.71</b>	<b>0.71</b>	-0.38	1.0
<b>Post-monsoon</b>								
Ace	1.0							
Flu	0.48	1.0						
Phen	0.02	-0.22	1.0					
Pyr	0.42	-0.41	<b>0.52</b>	1.0				
Cht	<b>0.51</b>	0.32	0.24	<b>0.51</b>	1.0			
Anth	<b>0.59</b>	<b>0.56</b>	-0.61	-0.22	<b>0.58</b>	1.0		
BaP	<b>0.64</b>	0.48	-0.49	-0.61	0.35	-0.12	1.0	
Flt	<b>0.55</b>	<b>0.61</b>	0.30	-0.62	<b>0.54</b>	<b>0.52</b>	-0.51	1.0

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