



Indoor and Outdoor PM₁₀-Bound PAHs in an Urban Environment. Similarity of Mixtures and Source Attribution

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Abstract

Given that the European Union lays down air quality objectives associated with outdoor environments, indoor air mixtures' study acquires a remarkable relevance. This work aims to submit a stepwise methodological framework for assessing similarities between indoor and outdoor air mixtures and apportioning potential emission sources. For reaching this goal, PM₁₀ particles were systematically and simultaneously collected at an indoor (dominant emission sources free) and outdoor environment during a year to determine the PAH content in both air mixtures. Broadly, outdoor PAHs levels were higher than at the indoor location, supporting a strong association between both mixtures ($r=0.968$, $p>0.001$), mainly during the cold period ($r=0.896$, $p>0.001$). The light molecular weight PAHs were highlighted at the indoor site, in particular to naphthalene and anthracene. Outdoor emission sources influenced the indoor PAH levels, especially high and medium molecular weight PAHs. The local-traffic load was identified as a dominant pollution source responsible for more than half PAHs determined at both environments. Therefore, the control of outdoor emission sources would be translated into an improvement of indoor air quality.

Keywords Air quality · Indoor vs outdoor · Airborne PAH · Mixture likeness · Apportionment of sources

Polycyclic aromatic hydrocarbons (PAHs) are semivolatile compounds comprising two or more fused aromatic rings made up of carbon and hydrogen atoms. They are generated coming from the incomplete combustion of organic material and are widely distributed into the atmosphere. Their sources are usually associated with anthropogenic activities in an urban environment, such as industrial activities, vehicle emissions, and gas/coal heating systems. PAHs have received special consideration as they have been identified as carcinogenic, teratogenic, and genotoxic (Ross 2005). Given that they can be inhaled by human beings (Mesquita et al. 2014), their assessment is mandatory in the European Member States. In this context, current European legislation

lays down air quality standards for the PAH group. Directive 2004/107/EC set a target value of 1 ng/m³ for benzo(a)pyrene associated with PM₁₀ in ambient air, expressed as an annual average. This air quality objective legislates outdoor environments, although people spend a large part of their time indoor environment (Goel et al. 2019).

Given that the occurrence of pollutants in the air matrix depends on emission sources, studies for identifying potential sources responsible for pollution levels both in indoor and outdoor environments acquire remarkable importance. Many researchers have broadly developed studies for determining potential emission air pollutant sources at indoor and outdoor, such as nitrogen dioxide, nitrogen monoxide, carbon monoxide, ozone, ammonia, volatile organic compounds, and atmospheric particles (Ayyildiz and Esen 2020). Nevertheless, the number of studies that simultaneously assessed indoor-outdoor PM₁₀-bound PAHs is highly limited.

In this sense, this study aims to (i) systematically and simultaneously monitor PM₁₀ particles at both urban indoor and outdoor location along a year (ii) discern potential similarities between the indoor and outdoor PAH mixture (iii)

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identify potential indoor and outdoor emission sources and estimate the weight of each emission source.

Materials and Methods

This study was conducted in Madrid City (Spain). It is located in the center of the Iberian Peninsula. It consists of 21 districts and 128 neighborhoods, and it has a population slightly higher than 3 million inhabitants and a surface area of approximately 600 km². The sampling point was the International Vaccination Center (Montesa Street, 22, Salamanca district, Madrid, Spain). This building is located near to roads with residential traffic (between 20 and 150 m away and daily traffic intensity < 1000 vehicles) and with punctual traffic jams (between 150 and 250 m away and > 25,000 vehicles/day). It consists of several medical consultation offices equipped with electronic devices and an opening in the corridor. The ventilation system is mechanical. The windows remain closed.

One outdoor point (40° 25′ 43.08″ N-3° 40′ 18.37″ W) located on the building's courtyard, and one indoor point (40° 25′ 43.75″ N-3° 40′ 18.92″ W) sited on the building's first floor (3 m above the building's courtyard level) was monitored along the studied time (Fig. 1). The outdoor site was cataloged as an urban background location (non-industrial); therefore, the vehicular emission and combustion processes from house-heating were considered the principal emission PAHs sources. The indoor sampling point was a smoke-free place, and it was in the corridor of the first floor, far from the center's kitchen facility and at a distance > 20 m away with regards to closer windows and < 10 m away from

the closer doors, remaining these open. Potential emission PAHs sources at the indoor point aim to electronic devices placed at medical consultation offices.

Based on previously mentioned, the building's walls' infiltration processes are the primary way of interchange of air pollutants between the target outdoor and indoor environment.

The sampling period for collecting PM₁₀ particles encompassed a civil year between May 2017 and April 2018, always at the previously mentioned two locations. Reference high-volume samplers were used, according to the EN 12,341:2014 standard for collecting PM₁₀ particles. They were equipped with quartz fiber filters (150 mm in diameter), and previously cleaned by thermal treatment for 12 h. The flow rate and sampling time were set at 30 m³/h and 24 h, respectively, and the sampling frequency was once every 3 days; therefore, 10 monthly individual samples were collected, except for May 2017 and October 2018 (N = 11), sampling a total of 122 particulate samples. Finally, the set of monthly samples were combined in a composite sample in terms of location.

Following the sampling of PM₁₀ particles, the sampled filters were stored in the dark at 4°C. Before the extraction process, each sampled filter was spiked with the internal standards benzo(a)pyrene-D12 (m/z ratio = 264) and benzo(a)anthracene-D12 (m/z = 240), then they were extracted by accelerated solvent extraction, concentrated using a nitrogen flow and cleaned up using BakerBond extraction cartridges. Finally, they were analyzed and quantified by gas chromatography (capillary column DB – 5MS, 30 m × 0.25 mm × 0.25 μm id) coupled with a mass spectrometer detector. Calibration curves with coefficients of determination better than 0.99 were accepted and relative uncertainty values between 20% and 40% were reached, according to other studies (Olmedo et al. 2012). Limit of quantification ranged between 0.13 and 0.18 ng/m³ (for PAHs with m/z ratio ≤ 153) and 0.01–0.07 ng/m³ (m/z ratio ≥ 166). Details of the extraction procedures and analysis are provided by EN 15,549:2008 standard. The laboratory has accredited the determination of PAHs in ambient air, according to the EN ISO/IEC 17,025:2017 standard, by the National Entity of Accreditation (ENAC 223/LE 460), which guarantees the technical competence of the analytical test.

PM₁₀-bound PAHs collected and analyzed were: naphthalene (in abbreviation, Nap, m/z = 128), acenaphthylene (Acy, 152), acenaphthene (Acp, 153), fluorene (Flr, 166), phenanthrene (Phen, 178), anthracene (Ant, 178), fluoranthene (Flt, 202), pyrene (Pyr, 202), benzo(a)anthracene (BaA, 228), chrysene (Chr, 228), benzo(b)fluoranthene + benzo(j)fluoranthene (BbjF, 252), benzo(k)fluoranthene (BkF, 252), benzo(a)pyrene (BaP, 252), indeno(1,2,3-c,d)pyrene (IcdP, 276), dibenzo(a,h)anthracene (DahA, 278) and benzo(g,h,i)perylene (BghiP, 276). PAHs were grouped in

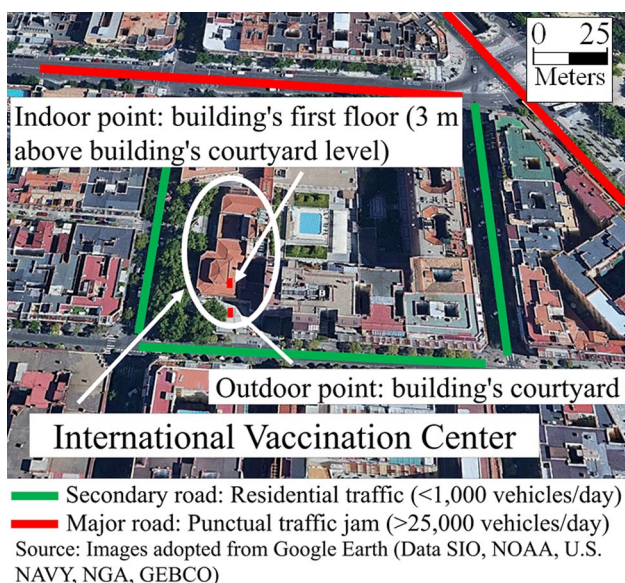


Fig. 1 Location of indoor and outdoor sampling point in Madrid City

terms of number of aromatic rings, named: light weight molecular PAHs (2 and 3 rings, from here on afterwards LMWPAHs, $m/z \leq 178$), medium weight molecular PAHs (4 rings, MMWPAHs, m/z 202 and 228) and high weight molecular PAHs (5 and 6 rings, HMWPAHs, m/z between 252 and 278).

In order to provide an overview concerning the affinity between the indoor and outdoor mixture over the investigated time, the total and monthly PAHs concentrations were examined.

Several tests were executed to detect possible emission focuses responsible for the PAHs levels monitored at the indoor and outdoor locations.

Test (1) Given that the vehicular emission is a polluting source highlighted in urban environments (Kumar and Elumalai 2018), the relationship between the traffic-related pollutants (independent variables) and the outdoor PAHs amount (dependent one) could or not confirm the vehicular emission as a possible origin of outdoor PAHs. Traffic-related pollutants were daily recorded by a fixed monitoring station belonging to the Municipality of Madrid's air quality monitoring network, named: Escuelas Aguirre (urban traffic station, 670 meters a.s.l., $40^{\circ} 25' 17.63''$ N $3^{\circ} 40' 56.35''$ W). Data was acquired from the Municipality of Madrid's open data portal (<http://www.madrid.es>). This station is the nearest to the outdoor air point selected for monitoring PM_{10} particles (~1 km away).

Test (2) Given that both the indoor and outdoor mixture is composed of several PAH congeners, the identification of the most representative compounds within each mixture would link those compounds to significant emission sources, according to the scientific literature. An artificial neural network (k-means clustering analysis) was conducted to detect the most representative 3 PAHs for reaching this end. Govender and Sivakumar (2020) widely described this type of analysis. In this study, Euclidean distance was handled as a spatial indicator to form the k-means cluster (Galán Madruga et al. 2018).

The weight of each emission source determines its contribution to the indoor and outdoor PAHs. Firstly, a principal component analysis (PCA) was carried out for setting the apportionment of each source (Hamid et al. 2018). PCA is a multivariate numerical tool used to transform uncorrelated variables into a dataset of correlated variables (Asbahi et al. 2019). Varimax method and cumulative variance > 90% were adopted to select the principal components (PCs), which explains most of the cumulative variance as regards the original dataset. A combined PCA-MLR analysis (multiple linear regression) was then applied to the results reached by the PCA technique.

Statistical analysis of the dataset was performed using the software IBM SPSS Statistics v22.0 (IBM Corp Armonk, NY, USA).

Results and Discussion

The total concentration of each PAH congener in both environments is shown in Fig. 2a. While the mixtures showed remarkable quantitative differences concerning the concentration (outdoor > indoor), the cumulated levels over the study for each PAH evidenced a strong connection (Fig. 2b). This relationship was broadly sustained monthly, except for September, with Pearson's coefficients higher than 0.6 (see Fig. 2c). In terms of the seasonal period, higher correlation in the cold (from October 2017 to March 2018, average indoor and outdoor temperature: $25.18 \pm 0.65^{\circ}\text{C}$ and $9.92 \pm 4.69^{\circ}\text{C}$, respectively) than warm period (from May to September 2017, average indoor and outdoor temperature: $25.38 \pm 0.07^{\circ}\text{C}$ and $24.61 \pm 3.15^{\circ}\text{C}$, respectively) was observed (cold: $r = 0.896$, $p > 0.001$ and warm: $r = 0.741$, $p > 0.001$). A detailed seasonal analysis found the highest correlation on Winter > Autumn > Spring > Summer ($r = 0.900$, 0.892 , 0.831 and 0.681 , respectively). Hu et al. 2017 found higher PAHs concentrations in Winter than Summer. This evidence is in line with the physic-chemical PAHs features, given that atmospheric PAHs mixtures prove more stable to lower temperatures, thereby favoring their condensation onto particulate matter. On the other hand, the typical meteorological conditions during the cold period avoid outdoor PAHs from chemically decomposing due to photochemical reactions (Li et al. 2005). Similarly, other studies reported good correlations for atmospheric pollutants between indoor and outdoor locations (Chamseddine et al. 2019; Yu et al. 2017).

It is relevant to indicate that Acy and Flu were not detected during the studied period. The ratio of indoor-to-outdoor Σ PAHs concentration for each PAH compound was < 1.0, except for naphthalene and anthracene. The PAH released from printers, photocopiers and computers could explain this fact (Mullins et al. 2013), as the International Vaccination Center consists of several medical consultation offices equipped with electronic devices and opening in the corridor. In the same argumentation, Destailats et al. (2008) reported that the amount of dust released into the atmosphere during normal computer operation could reach 4.0–6.3 mg dust per day. In particular, naphthalene and anthracene in non-smoking offices were ranged between 0.06 and 0.52 $\mu\text{g/g}$ and 0.16–0.94 $\mu\text{g/g}$, respectively.

The outcomes reached by applying a simple linear regression analysis between the monthly average concentrations of Σ PAHs and traffic-related pollutants are shown in Table 1. Moderate correlations were observed, being slightly higher at the indoor than outdoor site, thereby confirming a likely influence of outdoor pollution focuses

Fig. 2 **a** Total concentration of each PAH congener. **b** Correlation between indoor vs. outdoor accumulated level over the study in terms of individual PAH. **c** Monthly Pearson's coefficient of correlation

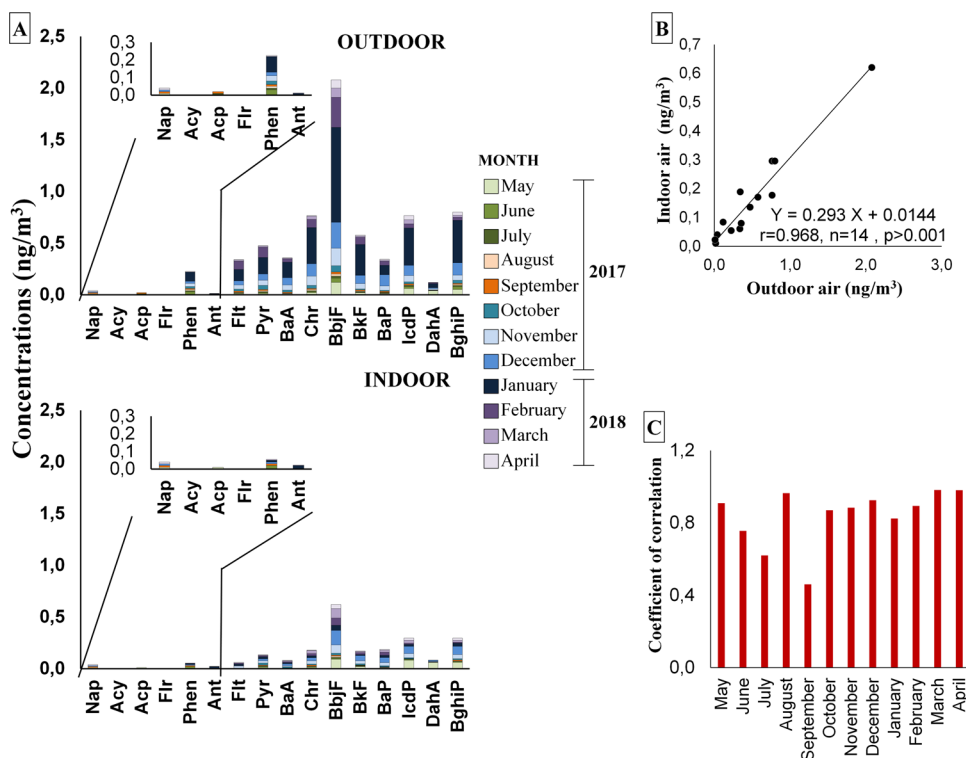


Table 1 Pearson's coefficients of correlation: monthly average concentrations of ΣPAHs vs. traffic-related air pollutants

Pollutant	Outdoor (PAHs)		Indoor (PAHs)	
	r^a	p^b	r	p
Nitrogen monoxide	0.534	<0.05	0.590	<0.05
Carbon monoxide	0.527	<0.05	0.680	<0.01
Nitrogen oxides	0.503	<0.05	0.560	<0.05
Benzene	0.430	<0.1	0.556	<0.05
Nitrogen dioxide	0.387	<0.1	0.434	<0.1
Ozone	-0.660	<0.05	-0.559	<0.05

^aPearson's correlation coefficient

^bSignificance level (one-tailed test)

on indoor air. In the case of nitrogen dioxide, secondary pollutant yielded by reactions between nitrogen monoxide and tropospheric ozone (Lin et al. 2016), a weak association was sustained between both environments because nitrogen dioxide needs a determined time for its formation in the ambient air. On the other hand, given that the formation of tropospheric ozone depends on the nitrogen oxides/volatile organic compounds ratio (Deng et al. 2019) under certain synoptic meteorological conditions with the presence of solar radiation, a negative correlation was estimated. This pollutant plays a paramount role in the photodegradation processes of particle-bound PAHs. A more detailed analysis in terms of BaP, given that it is

the main congener within the PAH mixtures (Directive 2004/107/EC) drove to significant correlations regarding traffic-related air pollutants (Pearson's coefficient of correlation = 0.743, 0.687, 0.707, 0.643, 0.566 and -0.823 for nitrogen monoxide, carbon monoxide, nitrogen oxides, benzene, nitrogen dioxide, and ozone, respectively).

As indicative data, given that the particles released into the atmosphere from vehicle emissions can have PAHs into their composition, the monthly average PM₁₀ particle concentrations monitored by the selected fixed station over the study ranged between 6.97 μg/m³ (March 2018) and 26.83 μg/m³ (June 2017).

Figure 3 pictures each PAH congener's annual representativeness defining this parameter as the total number of times each PAH was identified as the most representative monthly compound in applying k-means clustering analysis (k = 3). The more representative PAH compounds at both locations fell into the HMWPAHs group, followed by MMWPAHs and LMWPAHs. At individual PAH level, the more representative 3 PAH compounds were BbJF, Chr and BaP together to BghiP at the outdoor site as BbJF, Chr and IcdP at the indoor site. Given that Chr is a tracer compound to estimate the infiltration rate between indoor and outdoor sites (Choi and Spengler 2014), an indoor and outdoor interplay between the monitored environments is supported. Numerous studies associate individual PAHs with emission sources, given that those chemicals are considered fingerprints for those sources. In this sense, BbJF is taken as a tracer of fossil

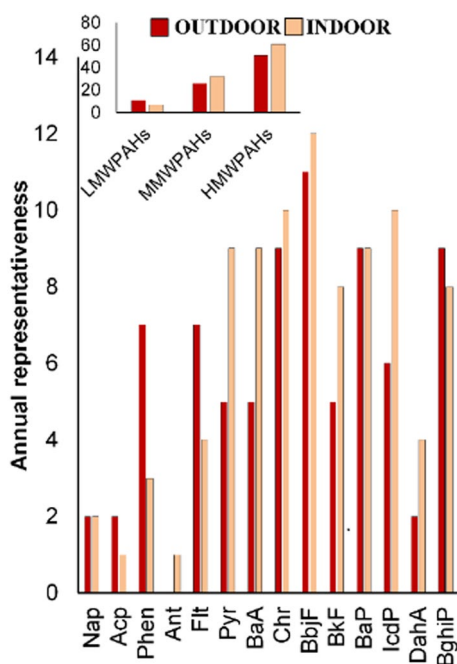


Fig. 3 Annual representativeness of each PAH congener

fuel combustion, Chr is a marker for combustion of biomass (Khalili et al. 1995), and finally, BaP, BghiP and IcdP are a good indicator of vehicular emission (Liu et al. 2010).

The combined PCA-MLR analysis transforms the data into significant linear dimensions, and quantitatively estimates the source (Hamid et al. 2018). Those PAH congeners with monthly data coverage lower than 75% were not included in the combined analysis. The 1st step was to execute a PCA technique. Table 2 presents PCA resulting

Table 2 Factor loadings of PCA analysis

PAH congener	Indoor		Outdoor	
	PC1	PC2	PC1	PC2
Phen	–	–	0.959	0.274
Flt	–	–	0.883	0.459
Pyr	0.800	0.042	0.880	0.456
BaA	0.965	0.048	0.811	0.579
Chr	0.927	0.161	0.879	0.477
BbjF	0.705	0.684	0.909	0.415
BkF	0.820	0.562	0.907	0.413
BaP	0.936	0.260	0.362	0.923
IcdP	0.339	0.933	0.904	0.421
DahA	– 0.297	0.930	0.916	0.393
BghiP	0.536	0.042	0.897	0.426
Total variance (%)	68.39	22.76	94.71	4.59

PC principal component

Higher factor loadings are marked in bold

factor loadings. According to the criteria laydowns in the Material and Methods section for the implementation of PCA analysis, two components (PC1 + PC2) were extracted from the original dataset at both sites. In the case of the indoor location, both components explained 91.15% of the initial dataset variance. A more detailed analysis reported PC1 accounted for 68.39% and was highly loaded with Pyr, BaA, Chr, BkF and BaP, while PC2 accounted for 22.76%. For the outdoor point, the sum of the two components contained 99.30% of the total information, mainly included in the PC1 (94.71%), loaded with medium and high molecular weight PAHs. As individual tracers, the previously mentioned compounds are good indicators of vehicular emission and biomass processes, thereby corroborating the PAHs origin in executing clustering analysis.

The 2nd step was to apply an MLR analysis on PCA outcomes. Table 3 shows the individual contribution of each indoor and outdoor PAH compound on the total variance. Given that the vehicular emission and combustion processes coming from house-heating were considered as the most significant emission sources at the outdoor point, each PAH congener was included into one group of polluting source: (i) emission vehicular: Flt, Pyr, BkF, BaP, IcdP, DahA and BghiP and, (ii) biomass-related processes: Phen, BaA, Chr and BbjF. In this context, the PAHs considered as good indicators of the vehicular emissions and biomass processes exhibited a higher outdoor contribution than at the indoor site (61.04% and 56.17%, vehicular emission: outdoor and indoor, respectively and, 38.26% and 34.98%, biomass processes: outdoor and indoor, respectively).

Although few studies have systematically and simultaneously examined the particulate PAHs at indoor-outdoor sites, authors as Elorduy et al. 2016 addressed the indoor–outdoor PAH interplay in Bilbao (Spain), as well as Delgado-Saborit et al. 2011 collected PM₁₀-bound PAHs samples at an urban indoor and outdoor location in the United Kingdom. While

Table 3 Outcomes reached by using combined PCA-MLR analysis

PAH congener	Indoor	Outdoor
Phen	–	10.81
Flt	–	9.41
Pyr	8.82	9.36
BaA	12.85	8.21
Chr	12.00	9.36
BbjF	10.13	9.89
BkF	11.21	9.84
BaP	12.55	2.96
IcdP	7.71	9.80
DahA	7.30	10.02
BghiP	8.59	9.66
Total variance (%)	91.15	99.30

in the first case, they used binary diagnostic ratios (I/O ratio) and principal component analysis to identify possible emission sources, in the second case, they used BaP as a marker for different environmental source mixtures. Since these analysis methods provide qualitative outcomes, the present study exhibits quantitative results concerning the apportionment of the emission sources, highlighting those most relevant ones regarding their influence on the determined target pollutants.

As conclusion remarks, this work exhibits a methodological framework to evaluate PM₁₀-bound PAHs mixtures at indoor and outdoor locations in terms of similarity and potential emission sources. The developed study has no limitations because the collection of PM₁₀ particles and the PAHs analysis associated with those are based on European Standards. Its implementation would allow developing air quality plans in specific sectors, pertinent or applicable within a frame of limited resources. Those plans should mainly focus on vehicular emissions in urban environments, which would improve outdoor and indoor air quality; therefore, those strategies should be interpreted as a preventive action within the public health area.

According to the developed work, the reached findings sustain a high correlation between indoor and outdoor PAHs concentrations, which clearly reveals that ambient air pollutants can significantly pollute the indoor air quality. Therefore, actions controlling outdoor pollutant emissions may significantly improve indoor air quality.

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Author Contributions DGM and JGC were responsible for the designing study. DGM validated the analytical method for the PM₁₀-bound PAHs determination, performed the data treatment, and wrote the manuscript. RMU and JMT analyzed ambient samples and, with SGS, collected PM₁₀ particles.

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Compliance with Ethical Standards

Conflict of interest The authors declare that there are no conflicts of interest.

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