



# Spatial distribution and sources of pesticidal persistent organic pollutants in the Hooghly riverine sediment

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## Abstract

Given the extensive indiscriminate usage in the past and limited ongoing use, organochlorine pesticides (OCPs) have been widely reported in the Hooghly riverine environment. Hence, surface riverine sediment samples were collected along the urban and suburban transects of the Hooghly River and OCPs were quantified in gas chromatography mass spectrometry (GC-MS). Mean concentration of HCH, DDT, and endosulfan was  $5 \text{ ng g}^{-1}$ ,  $10 \text{ ng g}^{-1}$ , and  $4 \text{ ng g}^{-1}$  respectively. DDT was dominant among all the OCPs and contributed nearly 40% to the total OCPs possibly due to the ongoing use of DDT for vector control programs. Diagnostic ratios suggest recent source of lindane, DDT, and endosulfan. Using OCP concentration from previously published data in surface water during the same time frame, sediment-water partitioning of OCPs was estimated. Excluding  $\alpha$ -HCH and  $\gamma$ -HCH in few pockets, majority of the OCPs tend to partition more on to sediment. Comparing the sediment concentration with the sediment quality guideline values, risk on ecological integrities was indicated due to DDT and HCH contamination. Suburban sites indicated higher risk than urban sites according to the calculated sediment quality guideline quotient (SQGQ). A brief review on the approach to pesticidal POP pollution management in India revealed that older management approaches should be replaced with a unique, integrated, and holistic system.

**Keywords** Organochlorine pesticides · Sediment–water partitioning · Hooghly riverine sediment

## Introduction

Organic pollutants like organochlorine pesticides (OCPs) are important due to their chemical characteristics and toxic impacts on human and environment. OCPs like dichlorodiphenyltrichloroethane (DDT), aldrin, heptachlor, and hexachlorobenzene (HCB) are classified under the legacy persistent organic pollutants (POPs). POPs are chemicals of global concern due to their characteristics like long-range transport, persistence, bioaccumulation, and biomagnifications in the

environment, as well as their adverse impact on human health and the environment (Jones and De Voogt 1999). OCPs are known carcinogens and endocrine disruptors (Jayaraj et al. 2016). OCPs are important in Indian context due to its extensive production and usage. India has been identified as a hotspot for contamination of OCPs like dichlorodiphenyltrichloroethane (DDT) and hexachlorocyclohexane (HCH) and there is no evidence of decline in OCP residue in environment in recent years (Sharma et al. 2014).

The lower stretch of Ganga River also known as the Hooghly River (HR) adds up to 4% of the Ganga River's annual sediment release (Mukhopadhyay et al. 2006). During the monsoon, a sediment-laden stream of the river interacts with the salinity and tidal flow in the Hooghly estuary and reaches the Bay of Bengal (Das 2015b). Tropical climate enhances atmospheric dissipation of OCPs in this region (Chakraborty et al. 2010). Due to extensive usage of OCPs in the past, soil is acting as secondary source through re-emission from soil (Chakraborty et al. 2015). Past and ongoing sources of OCPs in the surface water of HR seem to be evident (Chakraborty et al. 2016). Evidence of atmospheric deposition and direct

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discharge points are potential sources of OCPs in HR (Khuman and Chakraborty 2019).

Sediment acts both as a pollutant sink and as a carrier from direct discharges, surface run-off, atmospheric fall out, and secondary source of pollutants (Lu et al. 2012). Pollutants from sediment can impact benthic organisms via flux through the water phase and can be accumulated in sediment-dwelling organisms and edible fish (Chau 2005; Licata et al. 2003). Riverine sediment can act as temporary or abiding repositories for micro-organic pollutants coming from point and non-point sources (Chakraborty et al. 2019). Thus, sedimentation is an important aspect considering the fate of pollutants in freshwater ecosystems. Sediment studies are important in context of organic pollutant contamination because organic pollutants with high  $K_{ow}$  coefficient tend to sorb to sediment and are most likely retained in sediment (Doong et al. 2002b; Xu et al. 2007; Zakaria et al. 2002). Therefore, distribution of POPs in sediment and biota can serve as a helpful index for pollution and future environmental risks (Barakat et al. 2013). However, there is limited study on current status of OCPs in the Hooghly riverine sediment (HRS). Hence, this study was undertaken with the following objectives:

(i) study the spatial distribution of OCP contamination in HRS along the urban and suburban transects, (ii) identify the potential sources using diagnostic ratios and compositional profiles, (iii) understand the sediment-water partitioning, and finally (iv) estimate the possible risk to the ambient ecology.

## Materials and methods

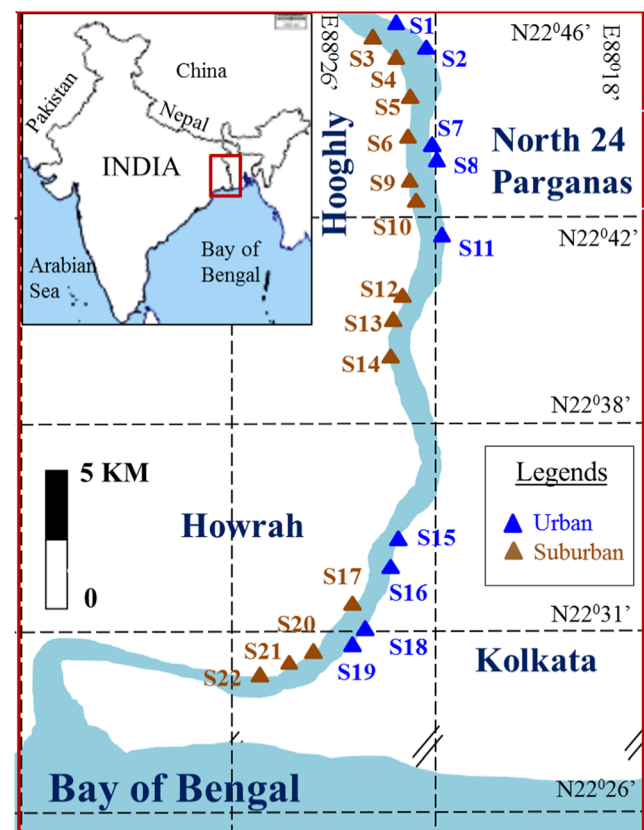
### Sampling

Hooghly River (HR) forms the lower stretch of River Ganga which has a basin area of 72,618 km<sup>2</sup> in West Bengal. HR is approximately 260 km long. A notable feature of HR is the formation of “bore-tides” characterized by a sudden and almost vertical rise of water level immediately after low water. The study area covered nearly 50 km stretch of the Hooghly River over urban and suburban transects covering four districts along the bank of the river viz., North 24 Parganas, Howrah, Hooghly, and Kolkata. North 24 Parganas and Kolkata are urban districts while Hooghly and Howrah are dominated with suburban settlements. In these four districts, the municipal solid waste generation is more than 15,000 MT/day (Chakraborty et al. 2019). Surface riverine sediment samples (0–10 cm) were collected using a clean stainless steel scoop along both the sides of Hooghly River during March 2015. Five sub-samples were collected over an area of 1 m<sup>2</sup> in a zig-zag manner to form a representative sample for each site. Details of sampling sites are given in SI Table S1 and

presented in Fig. 1. Sediment samples were packed in polyethylene-ziploc bags and stored in ice box, and finally transported to the laboratory. Sediment samples were stored at – 20 °C until further analysis.

### Extraction

All sediment samples were air-dried in room temperature and homogenized using mortar and pestle. About 20 g of sample from each site was extracted using USEPA method 3546 in a microwave extractor (CEM Mars 6) with 25 mL acetone/hexane mixture (1:1 v/v) at 110 °C (15 min ramping, 15 min extraction, 15 min cooling). A known concentration of surrogate standard 2,4,5,6-tetrachlorom-xylene (TCmX) was added to each sample prior to extraction process. The extract was concentrated, solvent-exchanged to hexane and made upto 1 mL. The extract was purified using alumina-silica gel column conditioned with 20 mL hexane and eluted with 10 mL of hexane/dichloromethane mix (1:1 v/v) as mentioned in details elsewhere (Khuman and Chakraborty 2019). Briefly, neutral alumina, neutral silica, and anhydrous sodium sulfate were baked for 12 h at 250 °C, 180 °C, and 450 °C, respectively, before use. The elute was concentrated to 0.2 mL under a gentle steam of nitrogen. A known quantity of pentachloronitrobenzene (PCNB) was added as an internal



**Fig. 1** Details of sampling locations along urban and suburban transect of Hooghly riverine sediment

standard prior to gas chromatography-mass spectrometry (GC-MS) analysis.

### Instrumental analysis

OCPs were analyzed using an Agilent-7890B GC-5977A MSD system with a HP 5MS capillary column (30 m, 0.25 mm i.d, 0.25  $\mu\text{m}$ ), operating under single-ion monitoring (SIM) mode. Helium was used as the carrier gas. Flow rate of carrier gas was 1.2 mL  $\text{min}^{-1}$ . Oven temperature was set at 60  $^{\circ}\text{C}$  for 1 min and increased to 290  $^{\circ}\text{C}$  (10 min hold time) at a rate of 4  $^{\circ}\text{C} \text{ min}^{-1}$ . Splitless injection of a 1- $\mu\text{L}$  sample was performed with a 6-min solvent delay time. Injector temperature was 250  $^{\circ}\text{C}$ . OCPs viz.  $\alpha$ -HCH,  $\beta$ -HCH,  $\gamma$ -HCH,  $\delta$ -HCH,  $p,p'$ -DDT,  $p,p'$ -DDE,  $\alpha$ -endosulfan,  $\beta$ -endosulfan, heptachlor, heptachlor epoxide, aldrin, and methoxychlor were quantified.

### Quality assurance and quality control

Chemical standards were purchased from Accustandard Co. U.S. Laboratory. For every set of five samples, one procedural blank was run. Limits of detection (LODs) were determined as the concentration of each analyte in a sample gave rise to a peak with a signal to noise ratio (S/N) of 3:1. Limit of quantification (LOQ) for OCPs was based on GC-MS performance and on laboratory background levels, which were determined by analyzing procedural blanks. Limit of quantification (LOQ) was established at three times the standard deviation of the procedural blank level, resulting in a certainty of more than 95% for results given to the samples. Concentrations lower than method detection limits (MDLs) were considered non-detectable for quantified OCPs. Surrogate recovery for TCmX was 75–110%, and the values were satisfactory; therefore, no correction was applied to the results reported in this study.

### Total organic carbon

Total organic carbon was determined using the loss of ignition method as mentioned elsewhere (Chakraborty et al. 2019). Briefly, sediment samples were dried at 105  $^{\circ}\text{C}$  overnight to remove moisture content and weighed before they were combusted at 360  $^{\circ}\text{C}$  for 2 h in a muffle furnace. After combustion, samples were weighed again to calculate the percentage of total organic carbon (TOC).

### Ecological risk assessment

Potential risk on the ecological integrities due to OCP residues in sediment has been reported by comparing the mean concentrations with the threshold effect level (TEL) and probable effect level (PEL) of Canadian Council of Ministry of the Environment Guidelines (2001) and effect range of low (ERL) from other studies (Long et al. 1998; Yang et al. 2013).

Further, potential biological effects caused by the contaminant mixtures were calculated in terms of sediment quality guideline quotient (SQGQ) as mentioned elsewhere (Long and MacDonald 1998; Wang et al. 2017).

$$\text{SQGQ} = \frac{\sum_{i=1}^n \text{PELQ}_i}{N} \quad (1)$$

where

$$\text{PELQ}_i = \frac{C_i}{\text{PEL}} \quad (2)$$

where PEL is the guideline value for contaminant  $i$  and  $C_i$  is the measured concentration of the same contaminant.  $n$  is the number of analyzed contaminants for which sediment guidelines can be acquired. SQGQ values  $< 0.1$  indicate no effects;  $0.1 \leq \text{SQGQ} < 1$  indicates moderate effects and  $\text{SQGQ} \geq 1$ , high adverse biological effects (Costa et al. 2011).

### Statistical analysis

All statistical analyses including box plots,  $t$  test, ANOVA etc. were performed using IBM SPSS 19.0 windows version.

## Results and discussion

### General discussion

Range of individual OCPs are given in Table 1.  $\Sigma_{13}$ OCPs ranged between 16 and 69  $\text{ng g}^{-1}$ . DDT was dominant contributing around 38% to the total OCP concentration. HCH, endosulfan, and other OCPs (heptachlor, heptachlor epoxide, aldrin, and methoxychlor) contributed 18%, 16%, and 28% of total OCPs, respectively. HCH, DDT, heptachlor, heptachlor epoxide, and aldrin correlated well with the total OCP concentration ( $r^2 > 0.7$ ,  $p < 0.05$ ). Highest concentration of total OCPs was observed in a suburban site in the Hooghly district (site S3, 20  $\text{ng g}^{-1}$ ). Geomean of total OCPs in suburban transect was nearly two folds higher than urban transect. Suburban transect contributed nearly three-fourth of the total OCP concentration. Also, mean concentration of HCH, DDT, endosulfan, heptachlor, heptachlor epoxide aldrin, and methoxychlor was higher in the suburban transect (Table 1). Other organic micropollutants like pharmaceutical compounds and plasticizers seem to be dominant in the suburban transect over urban transect in the Hooghly riverine sediment (HRS) (Chakraborty et al. 2019). Total OCPs in urban and suburban transects were significantly different ( $p < 0.05$ ). Comparing with a previous study in Hooghly riverine sediment (Guzzella et al. 2005), OCP concentration has increased over time possibly due to ongoing use of OCPs in this region (Khuman and Chakraborty 2019). The compounded effect of

**Table 1** Concentration of individual OCPs in ng g<sup>-1</sup> in Hooghly riverine sediment along urban and suburban transects

Compounds	Urban		Suburban	
	Range	Avg ± SD	Range	Avg ± SD
α-HCH	0.08–2.93	1.52 ± 1.02	0–3.98	1.71 ± 1.11
β-HCH	0.11–2.58	1.07 ± 0.89	0.01–3.4	1.21 ± 1.02
γ-HCH	0.02–2.75	1.12 ± 0.84	0.01–3.97	1.56 ± 1.13
δ-HCH	0.02–1	0.4 ± 0.29	0.01–1.77	0.71 ± 0.6
ΣHCH	0.22–8.49	4.11 ± 2.83	0.03–12.07	5.19 ± 3.39
<i>p,p'</i> -DDT	0.38–37.87	6.07 ± 12.03	0.35–55.2	9.96 ± 16.46
<i>p,p'</i> -DDE	0.04–3.33	1.72 ± 1.16	0.21–4.89	1.71 ± 1.4
ΣDDT	0.46–41.2	7.79 ± 12.76	0.63–56.57	11.67 ± 16.81
α-Endosulfan	1.02–3.68	2.26 ± 1.04	0.58–6.17	2.28 ± 1.42
β-Endosulfan	0.43–1.34	0.84 ± 0.37	0.29–1.8	0.9 ± 0.47
Endosulfan sulfate	0.01–7.81	1.13 ± 2.53	0.04–10.27	1.12 ± 2.77
ΣEndosulfan	1.46–12.02	4.24 ± 3.15	1.67–12.66	4.29 ± 2.97
Heptachlor	0.4–1.86	1.12 ± 0.49	0.46–12.81	2.26 ± 3.32
Heptachlor epoxide	0.06–7.85	2.31 ± 2.36	0.02–16.57	4.3 ± 4.48
Aldrin	0.44–4.94	2.47 ± 1.32	0.02–4.71	2.19 ± 1.45
Methoxychlor	0–0.06	0.02 ± 0.02	0–0.14	0.03 ± 0.04

past and current usage of the technical HCH might have increased the load of HCH on the sediment. Low vapor pressure and high resistance to microbial degradation make β-HCH the most persistent among HCH isomers (El-Kady et al. 2017). Higher accumulation of β-HCH over time could be due to the fact that β-HCH is metabolically inactive by nature (Pandit et al. 2006). Increase in the *p,p'*-DDE could be due to its affinity to accumulate in sediment. DDT has an extensive past as well as ongoing usage. Comparison of OCPs in HRS with other studies across the globe is given in SI Table S2. Mean concentration of ΣHCH and ΣDDT are higher than the mean concentration in sediment from Bizerte lagoon in Tunisia (Barhoumi et al. 2014), Wu-shi River in Taiwan (Doong et al. 2002a), Bohai Bay in China (Hu et al. 2009), Danube River in Serbia (Škrbić et al. 2007) but lower than Chenab River in Pakistan (Mahmood et al. 2014) and other Indian rivers like Brahmaputra in the north-east (Mishra et al. 2013) and Yamuna (Kumar et al. 2012), and Gomti (Malik et al. 2009) in northern India. Mean concentration of Σendosulfan is comparable with sediment from Wushi river, Taiwan (Doong et al. 2002a) and lake Qarun (Barakat et al. 2013) but higher than sediment from Bohai Bay in China (Hu et al. 2009), Gomti River in India (Malik et al. 2009) and Chenab River in Pakistan (Mahmood et al. 2014) but lower than Zha long wetland in China (Liu et al. 2012).

Mean concentration of aldrin, heptachlor, and heptachlor epoxide are higher than sediment from Beijing Guanting reservoir (Xue et al. 2006). Mean aldrin in HRS is also higher than surface riverine sediment of Yamuna (Parween et al. 2014) and Gomti (Malik et al. 2009) in

northern India but is comparable with lake Qarun (Barakat et al. 2013) in Egypt and lower than East lake (Yun et al. 2014) and Zha Long Wetland (Liu et al. 2012) in China and Naroibi River (Ndunda et al. 2018) in Kenya. Mean heptachlor in HRS is lower than surface sediment of Gomti (Malik et al. 2009). Heptachlor epoxide in HRS is higher than surface sediment of Naroibi River (Ndunda et al. 2018), Yamuna River (Parween et al. 2014), and Gomti River (Malik et al. 2009) but lower than East lake (Yun et al. 2014). Methoxychlor in HRS is lower than surface sediment of lake Qarun (Barakat et al. 2013) and Gomti (Malik et al. 2009).

## Overall spatial distribution and sources

### HCH

Spatial distribution of HCH in HRS is presented in Fig. 2(a). It can be observed that HCH residue in HRS is fairly uniformly distributed. Highest concentration of total HCH has been observed at site S4 (12 ng g<sup>-1</sup>) in suburban transect of the Hooghly district. ΣHCH (sum of α, β, γ, and δ-HCH) ranged between 0.03 to 12 ng g<sup>-1</sup> with an average of 5 ng g<sup>-1</sup>. Mean concentration of ΣHCH is in the order: Howrah (5.5 ng g<sup>-1</sup>) > Hooghly (5 ng g<sup>-1</sup>) > North 24 Parganas (4.4 ng g<sup>-1</sup>) > Kolkata (3.7 ng g<sup>-1</sup>). ΣHCH contributed around 15% of the total OCP concentration. In HRS, we observed the dominance of α-HCH with more than 33% contribution to the total HCH concentration. Dominance of α-HCH is in line with past observation in HRS (Guzzella et al. 2005). Nearly 30% of the



total HCH concentration is composed of  $\gamma$ -HCH. Significant correlation was observed between  $\alpha$ - and  $\gamma$ -HCH with  $\Sigma$ HCH ( $r^2 > 0.8, p < 0.05$ ) in sediment (SI Fig. S1). Mean concentration of  $\alpha$ -HCH,  $\gamma$ -HCH, and  $\delta$ -HCH was higher in suburban transect. But  $\beta$ -HCH which is most persistent isomer in sediment (Pandit et al. 2006) was higher in urban transect and is similar to the observation in the surface water of HR (Khuman and Chakraborty 2019). Technical grade HCH constitutes of 67–70%  $\alpha$ -HCH, 5–6%  $\beta$ -HCH, 13–15%  $\gamma$ -HCH, and 6%  $\delta$ -HCH. Higher percentage of  $\beta$ -HCH (~30%) in urban areas indicates only past usage of HCH possibly for vector control programs.  $\alpha/\gamma$  HCH in technical HCH ranges from 4 to 7 and  $\alpha/\gamma$  HCH less than 1 indicates lindane usage (Yun et al. 2014). Overall range of  $\alpha/\gamma$  HCH was between 0.6–5 with an average of 1 which indicates technical HCH usage (Fig. 3). Considering urban and suburban transects,  $\alpha/\gamma$  HCH was less than 1 in nearly 10% and 30% sites in urban and suburban transects respectively indicating lindane usage. But  $\beta/(\alpha + \gamma)$  HCH ratio was less than 1 in all suburban and urban sites (except S15) strongly indicating past usage of technical HCH. Our result indicates use of both technical HCH and lindane which is in line with the past observation in air (Chakraborty et al. 2010), soil (Chakraborty et al. 2015), and sediment (Guzzella et al. 2005) in the Hooghly riverine environment. In India, technical HCH was banned in 1997 while lindane was banned in 2013 (Chakraborty et al. 2017). Since lindane was banned recently, availability of stockpiles might have resulted in dominance of lindane. However, under subtropical climate where there is lindane use, transformation of  $\gamma$ -HCH to  $\alpha$ -HCH under anaerobic conditions might take place in sediment (Mahmood et al. 2014). Similar dominance of  $\alpha$ -HCH was observed in sediment of Haihe River, China, where technical HCH mixture was extensively used (Yang et al. 2005).

## DDT

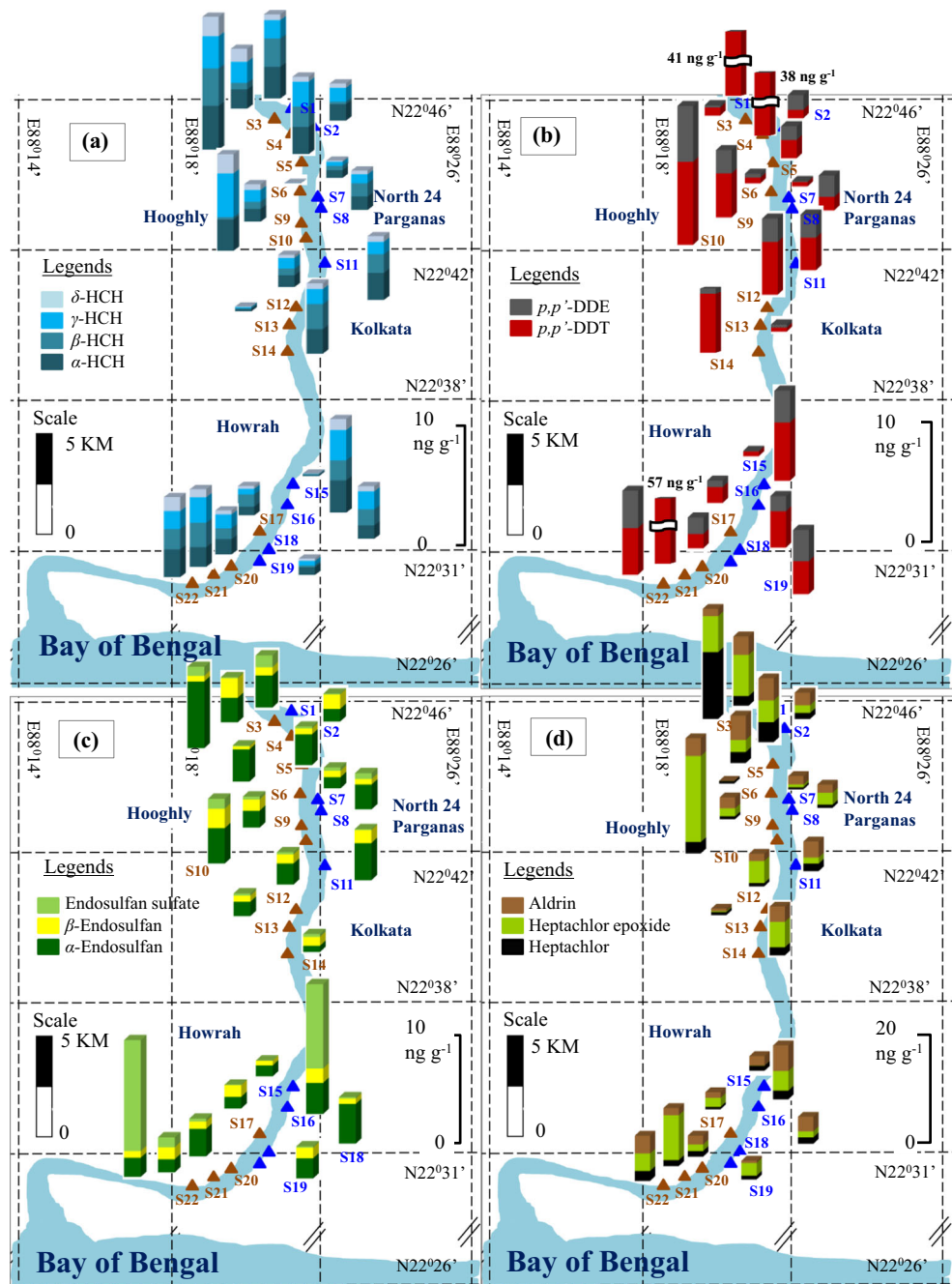
Spatial distribution of DDT is shown in Fig. 2(b).  $\Sigma$ DDT (sum of  $p,p'$  DDE and  $p,p'$  DDT) varied between 0.5–57 ng g<sup>-1</sup> with an average of 10 ng g<sup>-1</sup>. Highest concentration of  $\Sigma$ DDT was observed in suburban site of Howrah district (S21, 57 ng g<sup>-1</sup>). Mean concentration of  $\Sigma$ DDT was in the order: Howrah (17 ng g<sup>-1</sup>) > North 24 Parganas (10 ng g<sup>-1</sup>) > Hooghly (8 ng g<sup>-1</sup>), and Kolkata (5 ng g<sup>-1</sup>). Concentration of  $p,p'$  DDT was higher than  $p,p'$  DDE and contributed more than 80% of the total DDT concentration thereby indicating fresh input (Khuman and Chakraborty 2019). Dominance of  $p,p'$  DDT compared with other metabolites in sediment is consistent with past observation of riverine sediment of Hooghly River (Guzzella et al. 2005) and Tamiraparani River (Kumarasamy et al. 2012) in

India. Both  $p,p'$ -DDT and  $p,p'$ -DDE were higher in the suburban transect over urban transect. The suburban sites contributed more than 70% of the total  $p,p'$ -DDT concentration and nearly 60% of the total  $p,p'$ -DDE concentration. The average load of  $p,p'$ -DDE is greater than the parent isomers showing soil-related DDT contamination from past use in agricultural and vector-control programs (Chakraborty et al. 2015). Diagnostic ratio of  $p,p'$ -DDT/ $p,p'$ -DDE was more than 1 in 90% sites indicating ongoing usage of DDT in the study area (Fig. 3). Overall mean  $p,p'$ -DDT/ $p,p'$ -DDE ratio in HRS was 5 thereby indicating ongoing use of DDT. It can be noted here that DDT is still in restricted use in India for vector control under Stockholm Convention. West Bengal contributes 11% of the malaria cases in India (Kumar et al. 2007). DDT use for controlling vector-borne diseases might be the prime reason for high DDT residues in HRS. However, the extensive past use of DDT for agricultural purpose cannot be ruled out. Comparing the mean value of both  $p,p'$  DDT and  $p,p'$  DDE in Hooghly sediment with other studies, the mean value in HRS is lower than riverine sediment of Yamuna (Parween et al. 2014) in India but higher than riverine sediment of Arc in France (Kanzari et al. 2012), Haihe in China (Yang et al. 2005).

## Endosulfan

Spatial distribution of endosulfan in HRS is given in Fig. 2(c). Concentration of  $\Sigma$ endosulfan (sum of  $\alpha$ -endosulfan,  $\beta$ -endosulfan, and endosulfan sulfate) is in the range 1.5–13 ng g<sup>-1</sup> with an average of 4 ng g<sup>-1</sup>. Highest concentration of endosulfan has been observed in a suburban site of Howrah district (S22, 13 ng g<sup>-1</sup>). High levels of endosulfan in suburban areas of Howrah district was observed earlier in soil (Chakraborty et al. 2015) and air (Chakraborty et al. 2010) due to historic usage of endosulfan. Mean concentration of  $\Sigma$ endosulfan is in the order: Kolkata (5 ng g<sup>-1</sup>) > Howrah (4.7 ng g<sup>-1</sup>) > Hooghly (2.9 ng g<sup>-1</sup>) > North 24 Parganas (3.5 ng g<sup>-1</sup>).  $\alpha$ -endosulfan is dominant in both urban and suburban transects (Fig. 2c) and contributed more than half of the total endosulfan.  $\beta$ -endosulfan and endosulfan sulfate contributed 23% and 30% of the total endosulfan concentration, respectively. Mean concentration of  $\beta$ -endosulfan and endosulfan sulfate are higher in suburban transect. The ratio of  $\alpha/\beta$ -endosulfan in technical grade endosulfan is 2.33. Nearly half of the urban and suburban sites showed  $\alpha/\beta$ -endosulfan values more than 2.33 indicating ongoing technical endosulfan usage (Fig. 3). Dominance of  $\alpha$ -endosulfan suggests ongoing use of technical endosulfan associated with fishing activities in the Hooghly river (Das 2015a) and similar dominance has been observed in the

**Fig. 2** Spatial distribution of (a) HCH, (b) DDT, (c) Endosulfan, and (d) other OCPs in Hooghly riverine sediment

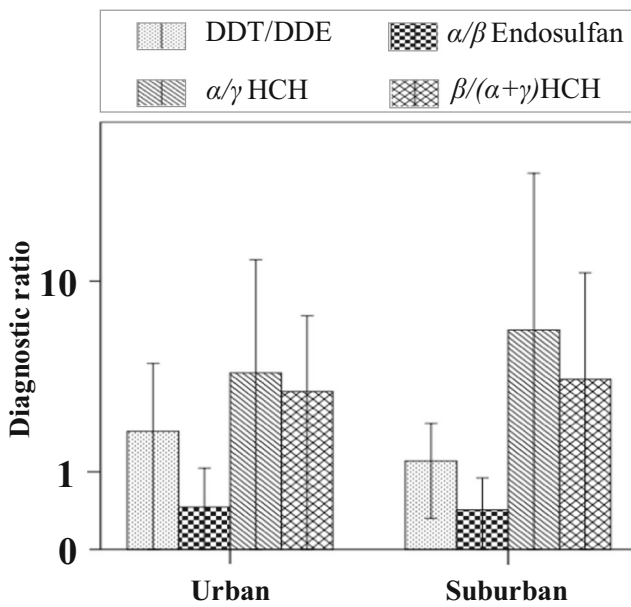


surface water of the Hooghly River (Khuman and Chakraborty 2019).  $\beta$ -endosulfan is more persistent isomer with higher half lives of 104 to 265 days compared to  $\alpha$ -endosulfan (35 to 65 days) in soil as it is more bound to soil particles (ATSDR 2000). Presence of endosulfan sulfate can be useful indicator of past endosulfan usage. Mean concentration of  $\alpha$ -endosulfan and  $\beta$ -endosulfan is higher than riverine sediment viz., Gomti (Malik et al. 2009) and Yamuna (Parween et al. 2014) in India, Beijing Guanting reservoir in China (Xue et al. 2006), Chenab River in Pakistan (Mahmood et al. 2014), Naroibi in Kenya (Ndunda et al.

2018) and Arc in France (Kanzari et al. 2012). Mean value of endosulfan sulfate in our study is lower than surface sediment of Yamuna in India (Parween et al. 2014), Zha long wetland in China (Liu et al. 2012), and Naroibi in Kenya (Ndunda et al. 2018).

#### Others

Heptachlor, heptachlor epoxide, aldrin, and methoxychlor together contribute 23% of the total OCP concentration. Spatial distribution of heptachlor, heptachlor epoxide, and



**Fig. 3** Error plots showing diagnostic ratios of HCH, DDT, and endosulfan for source apportionment

aldrin are shown in Fig. 2(d). Heptachlor and heptachlor epoxide are in the order: Hooghly > Howrah > Kolkata > North 24 Parganas. Methoxychlor contributed less than 0.5% of the total OCP concentration and hence not included in the spatial map. Heptachlor epoxide is dominant in sediment compared with heptachlor and is in line with the surface water of the Hooghly River (Khuman and Chakraborty 2019).

**Relationship with total organic carbon**

Sediment is affected by the kinetic behavior of hydrophobic organic pollutants (Yang et al. 2005). OCPs are hydrophobic in nature and are more likely to bind with organic matter. Organic matter can also provide more carbon sources to support microbial degradation (Wu et al. 1997). In general, organic matters are assumed to have around 60% organic carbon (Bianchi et al. 2008); however, the percentage may vary. In our study, total organic carbon (TOC) ranged between 3 and 39%. Relationship between TOC and OCPs has been explored in terms of correlation coefficients. Strong correlation of individual OCPs with TOC can imply that organic matter can enhance the absorption of OCPs (Gong et al. 2004; Jiang et al. 2009). Excluding Hooghly district, there was no significant correlation between concentration of OCPs and TOC in other districts and is in line with Lake Chaohu in China (Liu et al. 2013). Interestingly in Hooghly district, TOC showed strong correlation with *p,p'*-DDT, *p,p'*-DDE, total DDT, heptachlor epoxide, and total OCPs ( $r^2 > 0.9, p < 0.01$ ) and good correlation for total HCH and HCH isomers and total endosulfan ( $r^2 > 0.7, p < 0.01$ ). But low correlation was observed for  $\alpha$ - and  $\beta$ -endosulfan, endosulfan sulfate, heptachlor, and

aldrin ( $r^2 < 0.5, p < 0.05$ ). Strong correlation of OCPs with the sediment TOC implies strong binding of the organic pollutants to the organic carbon of the sediment possibly from the extensive past usage. Significant correlation of OCPs in sediment with TOC was also observed in riverine sediment of Gomti in India (Malik et al. 2009) and Haihe in China (Yang et al. 2005).

**Sediment water partitioning**

Under steady-state equilibrium conditions, partition coefficient  $K_{oc}$  can be predicted by the given formula as mentioned elsewhere (Kumata et al. 2002; Tan et al. 2009).

$$\log K_{oc} = \log K_{ow} - 0.61 \tag{3}$$

Organic carbon-normalized partition coefficient ( $K_{oc'}$ ) can be calculated using the formula mentioned elsewhere (Hu et al. 2014).

$$K_{oc'} = K_{sw} / f_{oc} \tag{4}$$

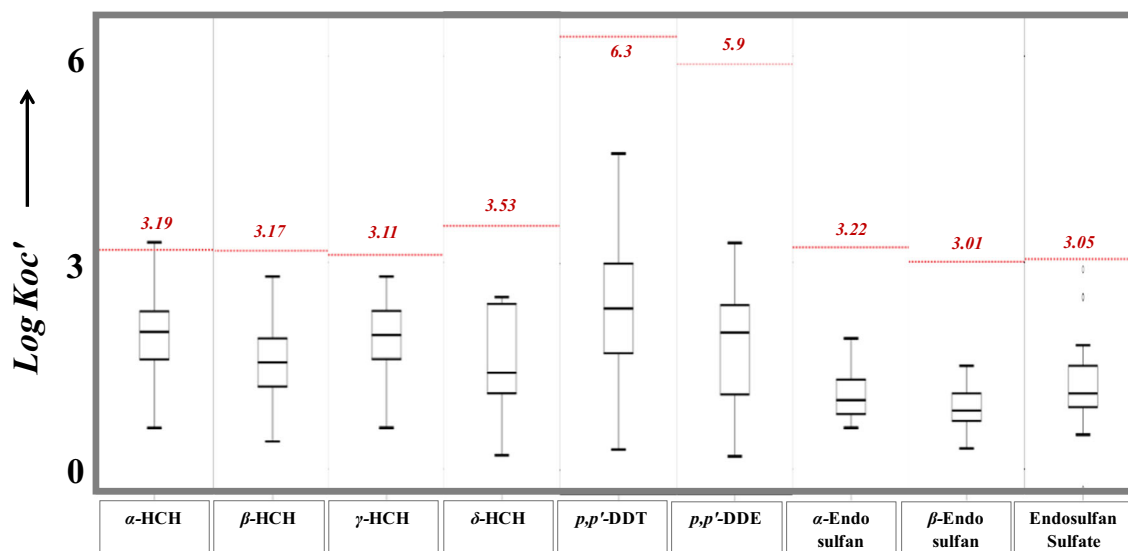
where

$$K_{sw} = C_s / C_w \tag{5}$$

$C_s$  is the concentration in sediment ( $\text{ng kg}^{-1}$ ) and  $C_w$  is the concentration in water ( $\text{ng L}^{-1}$ ),  $K_{ow}$  is the octanol water partition coefficient,  $f_{oc}$  is the organic carbon fraction.  $K_{sw}$  is the sediment–water partition coefficient (liter per kilogram). Concentration in water from corresponding sites were taken from previously published work (Khuman and Chakraborty 2019) sampled during same time frame. Sediment–water partitioning of OCPs can be predicted by comparing  $\log K_{oc}$  (equilibrium conditions) and  $\log K_{oc'}$  (natural conditions). When  $\log K_{oc}$  is higher than  $\log K_{oc'}$  it can be suggested that the sediment and water are not in equilibrium condition (Hu et al. 2014).

SI Table S3 presents the mean  $K_{sw}$  values for individual OCPs. Mean  $K_{sw}$  value ranged between 664 and 3201 for HCHs, 1545 and 8284 for DDTs, 72 and 768 for endosulfans, and 308 and 1232 for heptachlors. While  $K_{sw}$  range was lower for DDTs, HCHs were higher than those reported from Yangchaihu Lake in China (Hu et al. 2014).

Sediment receive OCPs from other environmental matrices and is the ultimate sink for OCPs in the environment (Barakat et al. 2013). Calculated  $\log K_{oc}$  was less than predicted  $\log K_{oc}$  in equilibrium condition in all sites for  $\beta$ -HCH,  $\delta$ -HCH, *p,p'*-DDT, *p,p'*-DDE,  $\alpha$ -endosulfan,  $\beta$ -endosulfan and endosulfan sulfate, aldrin, heptachlor, and heptachlor epoxide (Fig. 4 and SI Table S3). This indicates that OCPs are not in equilibrium condition between water and sediment and these OCPs might have higher affinity towards sediment (Hu et al. 2014). Unlike HCH and endosulfan isomers, *p,p'*-DDT, *p,p'*



**Fig. 4** Box and whisker plot showing range of calculated  $\log K_{oc'}$  in natural condition for individual OCPs with red dotted line indicating predicted  $\log K_{oc}$  in steady-state equilibrium condition

'-DDE, aldrin, heptachlor, and heptachlor epoxide have higher  $\log K_{ow}$  (SI Table S3). Higher  $\log K_{ow}$  compounds are more likely to deposit in sediment compared with compounds with lower  $\log K_{ow}$  value (Van Drooge et al. 2004). There was no significant correlation between  $\log K_{ow}$  and  $\log K_{oc'}$  ( $R^2 = 0.001$ ) as observed in Chaohu Lake in China (Liu et al. 2013). Due to ongoing usage of DDT in vector control programs and from past usage in nearby agricultural soil, *p,p'*-DDT and *p,p'*-DDE contamination have been reported in surface water of the HR via surface runoff and direct discharge points (Khuman and Chakraborty 2019). DDT from the surface water is finally getting accumulated in sediment possibly due to their higher  $\log K_{ow}$  value. Even though endosulfan isomers and metabolite have lower  $\log K_{ow}$  values, but after direct application of endosulfan in surface water for fishing purpose seems to have impacted the loading of endosulfan in water (Khuman and Chakraborty 2019). It has been anticipated that endosulfan transported to water will eventually get partition into sediment (ATSDR 2000). Hence, HRS is acting as sink for OCPs.

$\log K_{oc'}$  was slightly higher than  $\log K_{oc}$  at one urban site (S1) and specific sites in suburban transect (S9, S12) and urban transect (S19) were nearing equilibrium condition for  $\alpha$ -HCH. Similarly for  $\gamma$ -HCH,  $\log K_{oc'}$  was slightly higher than  $\log K_{oc}$  at one suburban site (S4) and few urban (S1) and suburban sites (S3, S5, S9, S12) showed  $\log K_{oc'}$  value nearing equilibrium condition. Majority of these sites are located in the Hooghly district. Interestingly, in Hooghly district, significant correlation was observed between TOC and  $\gamma$ -HCH and  $\alpha$ -HCH ( $r^2 > 0.7$ ). Correlation with TOC in sediment indicates stronger binding capacity of these compounds with sediment resulting from extensive usage in the past. Unlike  $\alpha$ -HCH and  $\gamma$ -HCH,  $\beta$ -HCH and  $\delta$ -HCH showed higher  $\log K_{oc}$  than  $\log K_{oc'}$  in all the sites

indicating higher affinity to sediments for these HCH isomers. Among HCH isomers,  $\beta$ -HCH has the highest half-life in sediment (12.6 years) (Dorsey 2005) and  $\delta$ -HCH has low mobility and is expected to get absorbed in sediment and suspended solid in aquatic environment (HSDB 2017).

### Risk assessment

Table S4 presents comparison of the sediment quality guideline values with concentration observed in sediment in the present study. The percentage of sites indicating risk by TEL was higher than those indicated by PEL for  $\gamma$ -HCH, *p,p'*-DDT. Concentration of heptachlor epoxide was higher than the PEL in 40% of the total sites. Suburban sites had more sites having value above PEL for  $\gamma$ -HCH, *p,p'*-DDT, *p,p'*-DDE, and heptachlor epoxide compared with urban sites. However, urban sites showed higher vulnerability for  $\gamma$ -HCH and *p,p'*-DDE when compared with the PEL values. Risk indicated by  $\gamma$ -HCH in suburban (> 80% sites) and urban (56–88% sites) transects can be a significant concern. This suggests that OCP contamination in sediments may cause adverse biological and ecological risks. Unlike sediments in Chenab river in Pakistan (Mahmood et al. 2014), our study showed lesser risk with less than 27% sites affected by *p,p'*-DDE, 64% by *p,p'*-DDT, and 81% by  $\gamma$ -HCH in overall sites.

Table 2 present the calculated PELQs and SQGQs for individual sampling sites. The SQGQs ranged between 0.03 and 4.36 with an average of 1.22. Overall, 14% sites showed SQGQ values less than 0.1 indicating no effects, 45% sites showed values between 0.1 and 1 indicating moderate effects and 41% sites showed values greater than 1 indicating high biological effects. Among transects, majority of the urban sites



(nearly 70%) sites showed moderate effects. In suburban transects, nearly 40% sites showed high adverse biological effects and nearly 50% sites showed moderate effects. Average value of SQGQ in suburban sites (1.4) was higher than urban sites (0.9) suggesting that suburban sites have higher adverse biological effects.

### Regulatory framework of pesticidal POPs management in India

The Government of India ratified the Stockholm Convention (SC) on 13th January 2006. As a party to the convention, India is developing and promoting non-POP alternatives to reduce intentional and unintentional emissions of POPs. The manufacture, use, and import into India of most of the pesticides that are listed as POPs in the SC are banned under the Insecticide Act 1968 and Insecticide Rules 1971. In 2011, India submitted its National Implementation Plan (NIP) on POPs, which gives information on the inventories of 12 legacy POPs. However, the NIP is yet to be updated to include

the POPs newly added to the SC. India regularly participates in various treaties and conventions aimed at protecting environmental and human health from the risk from chemicals (MoEF 2011). In India, most of the pesticidal POPs have been banned for intentional use since 1983. However, India is authorized to produce on average 5000 t of DDT annually for the control of vector-borne disease such as Malaria. Similarly, although banned for agricultural use, lindane ( $\gamma$ -HCH) was also allowed, specifically for termite control, up to 24th March 2013 (CIBRC 2014). The retrospective approach to pesticidal POPs pollution management in India needs to be replaced with that based on risk management, with policies formulated in the light of scientific knowledge, and the large but fragmented network of acts, laws, rules, etc. should be replaced with a unique, integrated, and holistic system supported by clear mandates for a limited number of designated authorities.

### Conclusion

Various environmental matrices around Hooghly River being contaminated with OCPs, the riverine sediment seems to be acting as sink for OCPs. Suburban transects which are mostly closer to the agricultural areas are showing higher OCP concentrations compared with urban transects. The concentration of OCPs in sediment has increased over time in Hooghly riverine sediment possibly due to continuous loading from water and atmosphere. Diagnostic ratios showed that ongoing usages of banned OCPs are still prevalent which is a matter of concern. Sediment–water partitioning indicates that OCPs are not in equilibrium and might move towards sediment. Even though sediment is an ultimate sink for OCPs, with continuous loading of OCPs from various matrices, sediment may have saturation point wherein the OCPs might contaminate the nearby water via sediment reflux. With strong tidal influence in this region, OCPs can deposit from water to sediment during low tide and vice versa during high tide conditions. Risk on ecological integrities seems to be more on the suburban sites than urban sites. But with the continuous use and loading from other environmental matrices, the risk on ecological integrities can increase in future. Available policies for pesticidal POPs can be strengthened with focus on holistic approach to control illegal availability and include risk management.

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**Table 2** Estimated probable effect level quotients (PELQs) and sediment quality guideline quotients (SQGQs) of selected OCPs in sediments

Site	PELQ				SQGQ
	$\gamma$ -HCH	<i>p,p'</i> -DDE	<i>p,p'</i> -DDT	Heptachlor epoxide	
H <sub>s</sub> 1	1.7	0.9	7.9	2.9	3.3
H <sub>s</sub> 2	1.3	0.3	0.2	0.5	0.6
H <sub>s</sub> 3	1.9	0.1	0.1	2.5	1.2
H <sub>s</sub> 4	3.0	0.9	7.2	1.5	3.1
H <sub>s</sub> 5	2.3	0.3	0.3	0.8	0.9
H <sub>s</sub> 6	0.0	0.1	0.1	0.0	0.1
H <sub>s</sub> 7	0.4	0.0	0.1	0.2	0.2
H <sub>s</sub> 8	0.8	0.5	0.2	0.8	0.6
H <sub>s</sub> 9	1.1	0.5	0.8	0.6	0.7
H <sub>s</sub> 10	4.0	1.3	1.5	6.0	3.2
H <sub>s</sub> 11	1.1	0.5	0.6	0.4	0.7
H <sub>s</sub> 12	1.0	0.5	1.0	1.5	1.0
H <sub>s</sub> 13	0.1	0.1	0.1	0.1	0.1
H <sub>s</sub> 14	1.4	0.3	3.0	1.8	1.6
H <sub>s</sub> 15	0.0	0.0	0.1	0.0	0.0
H <sub>s</sub> 16	2.8	0.7	1.1	1.4	1.5
H <sub>s</sub> 17	0.5	0.2	0.3	0.6	0.4
H <sub>s</sub> 18	1.7	0.4	0.7	0.4	0.8
H <sub>s</sub> 19	0.4	0.7	0.6	0.9	0.7
H <sub>s</sub> 20	1.3	0.4	0.3	0.5	0.6
H <sub>s</sub> 21	2.3	0.4	11.6	3.2	4.4
H <sub>s</sub> 22	1.7	0.9	0.9	1.2	1.2

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