Supplementary material

Occurrence and source apportionment of perfluoroalkyl acids (PFAAs) in the atmosphere in China

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CONTENT

TWENTY-ONE pages: SIX tables and FIVE figures, TWO sections,.

Table S1. Physical and chemical properties of target PFAAs compounds

Component	Abbreviation	Molecular structure	Molecular weight	Bio–concentration factor [a]	$\log K_{OW}$ [b]	P^{o}_{L} (mmHg) [c]
Perfluoroalkane carboxylic acids (PFCAs)						
Perfluoropentanoic acid	PFPeA	C4F9COOH	263.98	1.00	5.29	7.9±0.4
Perfluorohexanoic acid	PFHxA	C5F11COOH	313.98	1.00	5.97	3.1±0.5
Perfluoroheptanoic acid	PFHpA	C6F13COOH	363.97	1.00	6.86	0.5±0.6
Perfluorooctanoic acid	PFOA	C7F15COOH	413.97	1.90	7.75	0.3±0.7
Perfluorononanoic acid	PFNA	C8F17COOH	463.97	11.26	8.64	0.2±0.8
Perfluorodecanoic acid	PFDA	C9F19COOH	513.96	44.30	9.53	0.0±0.9
Perfluoroundecanoic acid	PFUdA	C10F21COOH	563.96	128.19	10.42	0.0±0.9
Perfluorododecanoic acid	PFDoA	C11F23COOH	613.95	235.68	11.31	0.0±1.0
Perfluorotridecanoic acid	PFTrDA	C12F25COOH	663.95	474.19	12.19	0.0±1.1
Perfluorotetradecanoic acid	PFTeDA	C13F27COOH	713.95	1903.40	13.08	0.0±1.2
Perfluoroalkane sulfonic acids (PFSAs)						
Perfluorobutane sulfonic acid	PFBS	C4F9SO3H	299.98	1.00	3.68	/ [d]
Perfluorohexane sulfonic acid	PFHxS	C6F13SO3H	399.97	1.00	5.25	/
Perfluorooctane sulfonic acid	PFOS	C8F17SO3H	499.97	1.00	7.03	/

[a]: Predicted data are generated using the Advanced Chemistry Development, Inc. (Canada), cited from (Yu, Liu et al. 2018);

[b]: Predicted octanol–water partitioning coefficients from individual PFAAs structure, cited from (Buck, Franklin et al. 2011, Yu, Liu et al. 2018);

[c]: Predicted pure compound vapor pressure, unit of mmHg at 298 K, cited from (Buck, Franklin et al. 2011, Yu, Liu et al. 2018);

[d]: "/" means lack of related data.

Table S2. The geographic information and annual temperature in different sampling sites of atmospheric PFAAs

I.D.	Region	Province	Type	Location	Elevation (m)	Monthly mean temperature (°C) [a]	Gross Domestic Product (10^8 RMB) [b]	Resident population (10^4) [b]	Crude plastic (10^4 tons) [b]
1	Northern of	Beijing	Urban	Haidian District	31	–5 – 24	127.75	2171	28014.94
2	China, NC	Tianjin	Urban	Jinnan District	3.3	–4 – 25	332.42	1557	18549.19
3		Shanxi	Rural	Linshui County, Jincheng city	376	–11 – 17	79.47	3702	15528.42
4	Eastern of	Shanghai	Urban	Minhang District	4.5	5 – 28	364.04	2418	30632.99
5	China, EC	Zhejiang	Rural	Yinzhou District, Ningbo City	4	4 – 23	896.29	5657	51768.26
6		Jiangsu	Urban	Changzhou City	5	2 – 26	1175.39	8209	85869.76
7		Anhui	Urban	Yinquan District, Fuyang City	30	2 – 27	137.35	6225	27018
8		Fujian	Urban	Huian Country, Quanzhou City	30	12 – 26	235.74	3911	32182.09
10		Jiangxi	Urban	Jiujiang City	32.2	4 – 26	25.46	4622	20006.31
9		Shandong	Urban	Laishan District, Yantai City	47	–1 – 24	710.42	10006	72634.15
11	Southern of	Guangdogn	Urban	Nanshan District, Shenzhen City	7	15 – 26	695.31	11169	89705.26

12	China, SC	Hainan	Urban	Meilan District, Haikou City	12	18 – 26	19.67	926	4462.54
13	Central of	Hubei	Urban	Yunxi District, Shiyan City	437	1 – 24	191.86	5902	35478.09
14	China , CC	Henan	Urban	Gaoxin District, Zhenzhou City	110	–2 – 26	232.47	9559	44552.83
15		Hunan	Urban	Huaxin District, Hengyang City	103	7 – 27	48.4	6860	33902.96
16	Northwestern of	Xinjiang	Urban	Tacheng City	427	–14 – 18	621.72	2445	10881.96
17	China, NW	Shaanxi	Urban	Beilin District, Xi'an City	397	–1 – 24	478.63	3835	21898.81
18		Gansu	Urban	Chengguang District, Lanzhou City	1517	–7 – 19	121.57	2626	7459.9
19	Southwestern of	Sichuan	Urban	Shuangliu District, Chengdu City	506	4 – 23	214.94	3789	15901.68
20	China, SW	Yunnan	Urban	Lanchang Country, Puer City	1950	3 – 19	319.76	4369	23409.24
21		Guizhou	Urban	Xinren Country, Qiandongnan City	1379	6 – 22	127.75	2171	28014.94
22	Northeastern of	Heilongjiang	Urban	Beilin District, Suihua City	172	–22 – 19	332.42	1557	18549.19
23	China, NE	Liaoning	Rural	Neizhou Country,Huludao City	118	–12 – 21	79.47	3702	15528.42

[a]: Meteorological data originated from China Meteorological Administration, http://www.cma.gov.cn/;

[b]: Data originated from China Statistic Yearbook 2018 (National Bureau of Statistics China, http://www.stats.gov.cn/tjsj/ndsj/);

Table S3. MS parameters, MDLs, LODs, LOQs values and recovery rates for individual compounds of PFAAs

Analogues	Parent ions (m/z)	Daughter ion (m/z)	Declustering potential (V)	Collision energy (eV)	Retention time (s)	MDLs (pg/m^3)	LODs (pg/m^3)	LOQs (pg/m^3)	Recovery rate (%)	Internal Standards
PFCAs										
PFPeA	263	219	6	–34	3.16	0.41	0.31	1.05	96±17	1,2–$^{13}C_2$–PFHxA
PFHxA	313	269	10	–36	3.42	0.18	0.14	0.47	108±22	1,2–$^{13}C_2$–PFHxA
PFHpA	363	319→169	11	–28	3.70	0.22	0.16	0.55	93±16	1,2,3,4–$^{13}C_4$–PFOA
PFOA	413	369→169	8	–39	3.99	0.33	0.26	0.87	91±13	1,2,3,4–$^{13}C_4$–PFOA
PFNA	463	419→219	13	–44	4.32	0.61	0.46	1.53	89±17	1,2,3,4,5–$^{13}C_5$–PFNA
PFDA	513	469→219	13	–47	4.67	0.56	0.42	1.39	93±11	1,2–$^{13}C_2$–PFDA
PFUdA	563	519→269	12	–61	5.02	0.28	0.21	0.70	88±16	1,2–$^{13}C_2$–PFUdA
PFDoA	613	569→169	14	–65	5.35	0.28	0.21	0.70	94±18	1,2–$^{13}C_2$–PFDoA
PFTrDA	663	619→169	14	–59	5.64	0.34	0.26	0.87	102±17	1,2–$^{13}C_2$–PFDoA
PFTeDA	713	669→169	15	–57	5.94	0.41	0.31	1.03	97±21	1,2–$^{13}C_2$–PFDoA

PFSAs										
PFBS	299	80→99	38	–64	3.19	0.25	0.20	0.66	81±25	$^{18}O_2$–PFHxS
PFHxS	399	80→99	42	–87	3.70	0.16	0.12	0.40	86±13	$^{18}O_2$–PFHxS
PFOS	499	80→99	38	–98	4.31	0.24	0.19	0.63	95±15	1,2,3,4–$^{13}C_4$–PFOS
Internal standards										
1,2–$^{13}C_2$–PFHxA	315	270	11	–41	3.40	/	/	/	/	/
1,2,3,4–$^{13}C_4$–PFOA	417	372	13	–41	3.99	/	/	/	/	/
1,2,3,4,5–$^{13}C_5$–PFNA	468	423	13	–52	4.34	/	/	/	/	/
1,2–$^{13}C_2$–PFDA	515	470	13	–51	4.69	/	/	/	/	/
1,2–$^{13}C_2$–PFUdA	565	520	13	–61	5.02	/	/	/	/	/
1,2–$^{13}C_2$–PFDoA	615	570	13	–55	5.35	/	/	/	/	/
$^{18}O_2$–PFHxS	403	103	41	97	3.72	/	/	/	/	/
1,2,3,4–$^{13}C_4$–PFOS	503	80	66	97	4.31	/	/	/	/	/

Table S4. The measured abundances of PFAAs in this study（n=268）

Analogues	Detection frequency (%)	Average value (pg/m^3)	Standard deviation (pg/m^3)	Minimum value (pg/m^3)	Maximum value (pg/m^3)	Median value (pg/m^3)
PFCAs						
PFPeA	84.8	4.96	4.77	0.00	35.2	3.55
PFHxA	92.1	5.36	7.17	0.02	79.7	3.73
PFHpA	94.7	3.42	3.71	0.03	28.9	2.39
PFOA	100	8.19	8.03	0.36	70.4	6.24
PFNA	96.6	3.07	2.77	0.00	22.7	2.52
PFDA	96.2	4.13	3.74	0.00	30.5	3.36
PFUdA	75.6	1.24	1.32	0.00	6.72	0.86
PFDoA	63.5	0.56	0.50	0.00	3.18	0.45
PFTrDA	37.3	0.58	0.56	0.00	3.57	0.47
PFTeDA	41.7	0.19	0.25	0.00	2.25	0.11
PFSAs						
PFBS	62.2	1.96	1.85	0.00	9.39	1.37
PFHxS	71.6	0.99	1.38	0.00	13.2	0.56
PFOS	100	5.20	4.30	0.34	25.5	3.87
Total PFAAs						
	/	39.8	28.1	6.19	292.6	35.4

Table S5. **The amount of precipitation in the main city in China in 2017 (mm)** [a]

Province	City	Distance [b]	Winter		Spring			Summer			Autumn			Winter	Annual
			Jan.	Feb.	Mar.	Apr.	May	Jun.	Jul.	Aug.	Sep.	Oct.	Nov.	Dec.	
Beijing			0.2	5.4	12.5		31.2	119.5	97.4	233.9	2 .8	73.3			576.2
Tianjin			1.3	5.5	16.1	0.3	17.5	49.1	159.6	160.4	1.2	101.9			512.9
Shanxi	Taiyuan	260	2 .0	10.1	4.8	25.0	14.8	41.6	142 .5	125.8	1.2	151.9		1.5	521.2
Liaoning	Dalian	210	9.9	3.1		21.8	24.5	18.4	36.2	315.7	4.1	81.4	5.5	1.1	521.7
Heilongjiang	Haerbin	99	4.4	5.3	4 .2	2.9	51.8	92.2	50.2	215.2	37.0	5.2	7.1	5.3	480.8
Shanghai			62.9	20 .6	75.7	93.3	72 .8	158.3	37.6	319.5	351.5	96.9	79.5	20 .2	1388.8
Jiangsu	Nanjing	128	59.3	38.8	55.1	117.9	83.9	309.1	99.6	217.1	176.4	81.0	7.4	9.5	1255.1
Zhejiang	Hangzhou	136	71.7	27.2	189.0	142.9	87.8	307.2	77.5	95.3	168.3	89.2	139.1	46.8	1442.0
Anhui	Hefei		60.9	40 .6	68.4	64.2	123.6	54 .0	63.5	243.2	111.6	97.5	10.8	13.6	951.9
Fujian	Fuzhou	146	32.5	55.8	180.3	197.7	61.1	477.6	136.3	205.0	45.8	14.2	64.3	7.4	1478.0
Jiangxi	Nanchang	113	26.3	42.5	324.1	156.7	69.3	514.4	120.2	206.7	55.1	66.0	85.9	31.6	1698.8

Province	City	Distance	Jan.	Feb.	Mar.	Apr.	May	Jun.	Jul.	Aug.	Sep.	Oct.	Nov.	Dec.	Annual
Shandong	Qingdao	175	44.6	11.1	9.7	12.6	35.6	40 .7	228.7	204.4	82.5	58.8	0.4		729.1
Henan	Zhengzhou		17.7	8.5	15.7	26.0	54.5	46.7	79.4	196.0	76.0	76.8	0.6	0.9	598.8
Hubei	Wuhan	398	48.2	66.4	108.8	222.4	85.4	148.6	52.3	168.7	107.4	72.9	17.5	8.7	1107.3
Hunan	Changsha	154	46.4	61.2	239.8	105.2	121.4	526.5	246.5	152.9	65.4	31.2	61.0	26.7	1684.2
Guangdong	Guangzhou	98	13.4	26.4	174.1	117.7	421.4	405.8	312 .0	244.8	269.4	44.3	36.7	1.4	2067.4
Shaanxi	Xian		3.6	11.1	50.6	55.9	63.6	72.2	82.5	64 .6	98.6	140 .0	6.4		649.1
Gansu	Lanzhou			6.5	5.0	11.7	34.9	63.4	35.6	87.2	16.3	42.4			303.0

[a]: Data originated from China Statistic Yearbook 2018 (National Bureau of Statistics China, http://www.stats.gov.cn/tjsj/ndsj/);

[b]: represent the distance from the city to the sampling site, km.

Table S6. Correlation analysis of PFAAs in the atmosphere in China

	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTrDA	PFTeDA	PFBS	PFHxS
PFHxA	**0.70****											
PFHpA	0.12	0.31*										
PFOA	**0.69****	**0.77****	**0.68****									
PFNA	**0.66****	**0.66****	**0.65****	**0.70****								
PFDA	**0.54****	**0.67****	**0.72****	**0.84****	**0.61****							
PFUdA	0.16	0.32	0.15	0.2	0.14	0.23						
PFDoA	0.39*	0.33	0.27	0.38	0.31	0.32	**0.61****					
PFTrDA	0.53*	**0.48****	0.3	0.42	0.44	**0.51****	**0.65****	**0.62****				
PFTeDA	0.21	0.4	0.39*	0.36*	0.27	0.39	**0.72****	**0.59****	**0.79****			
PFBS	**0.68****	0.26	0.15	0.26	0.15	0.39	0.14	0.23	0.28	0.18		
PFHxS	0.57*	**0.69****	0.27	0.42*	**0.57****	**0.64****	0.3	0.43*	0.54*	0.38*	0.28	
PFOS	**0.69****	0.42*	0.32	0.33	0.36	0.37*	0.25	0.41*	0.46*	0.38	**0.63****	0.40*

*: represent $p < 0.05$;

**: represent $p < 0.01$.

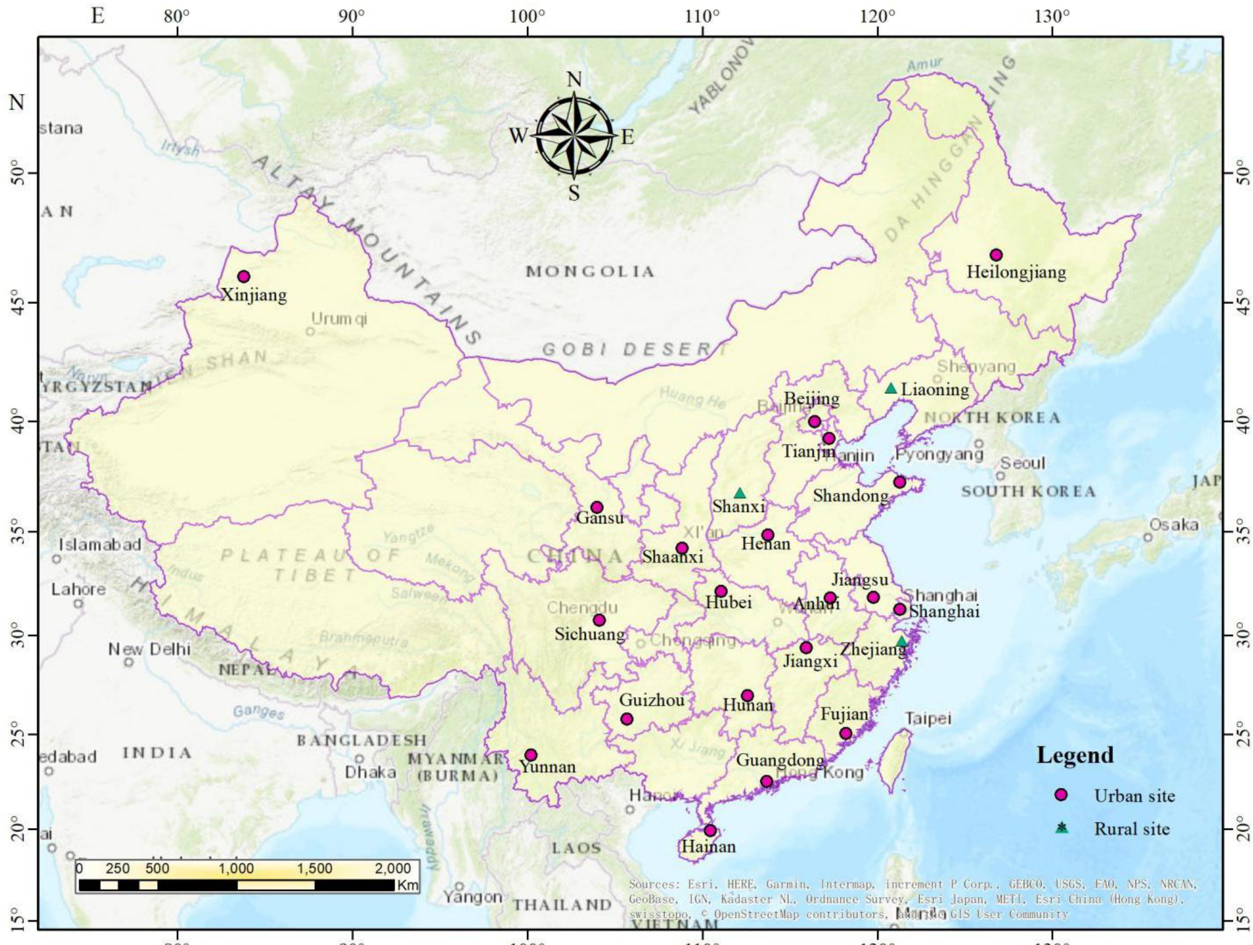


Figure S1. **Spatial distributions of 23 sampling sites of atmospheric PFAAs in China (including 20 urban sites, red circles; and three rural site, green triangles) (created by ArcGIS 10.4 with ArcGIS Online map)**

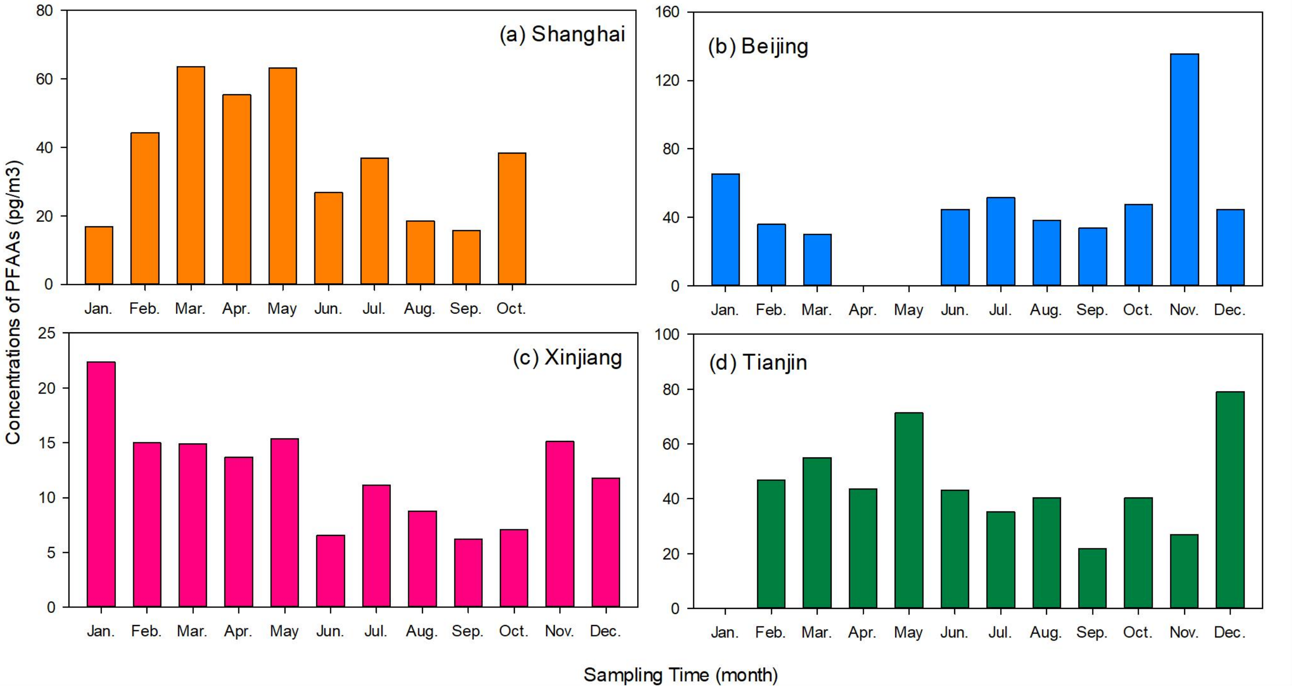


Figure S2. Temporal variations of PFAAs concentrations in selected four typical sites: Shanghai, Beijing, Xinjiang and Tianjin (created by SigmaPlot 14.0, Systat Software, US)

Figure S3. The spatial distributions of fluoride related products manufacturers in China (note that part of industries were not included in this figure) and the different geographical conditions (created by ArcGIS 10.4 with ArcGIS Online map).

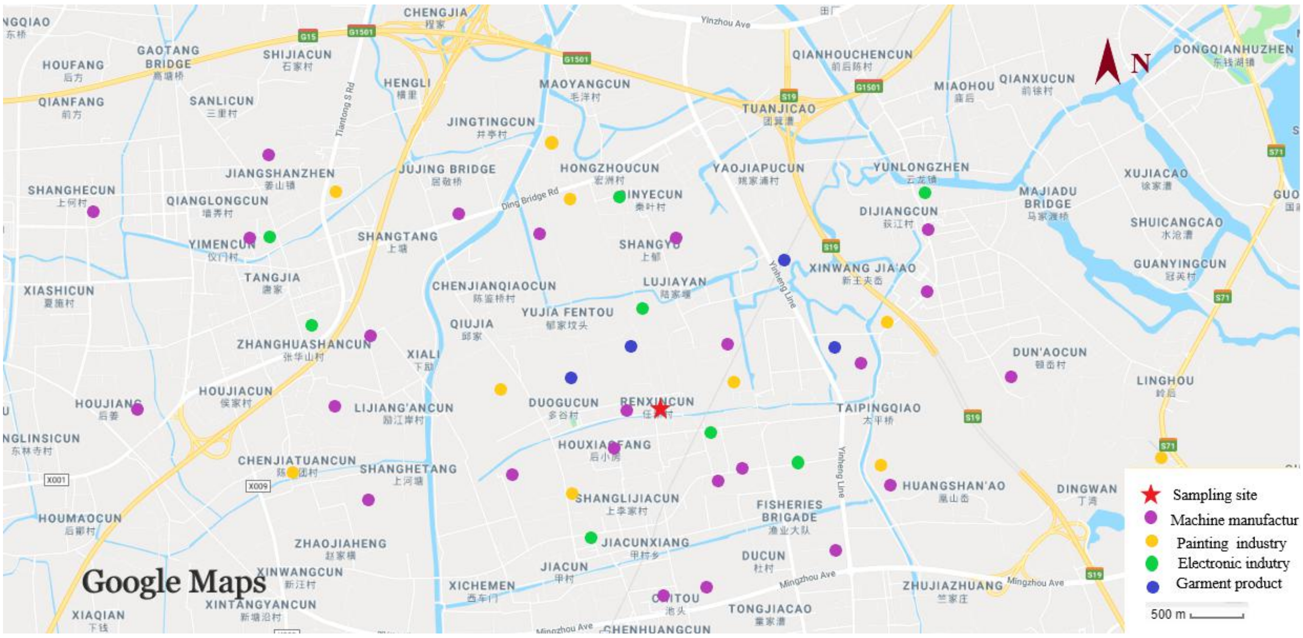
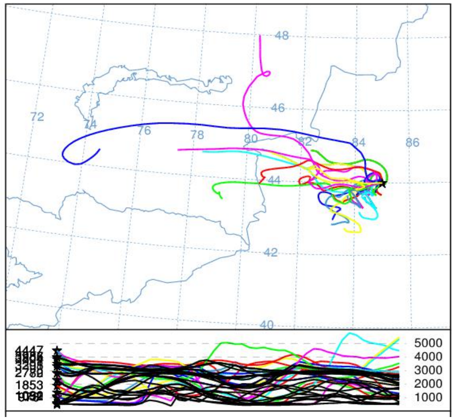
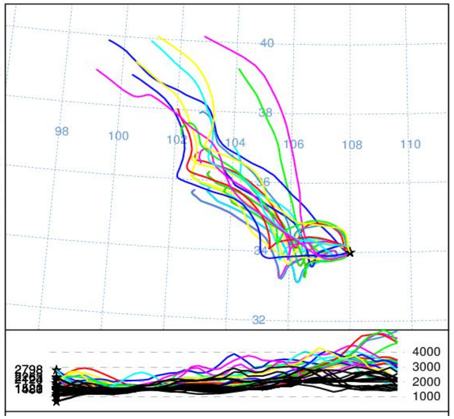


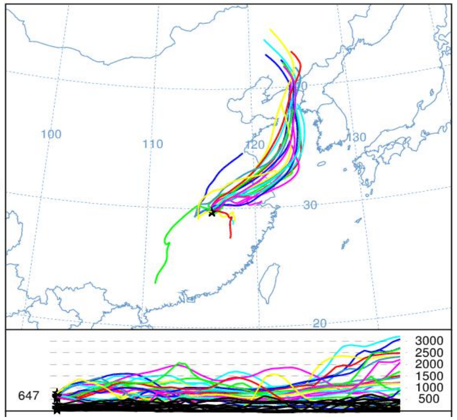
Figure S4. **The spatial distributions of fluoride related products manufacturers in Zhejiang site (a small village in Ningbo City) (created by © Google map).**



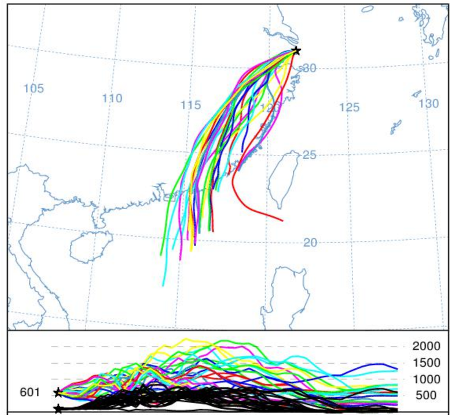
Tacheng, Xinjiang; Summer



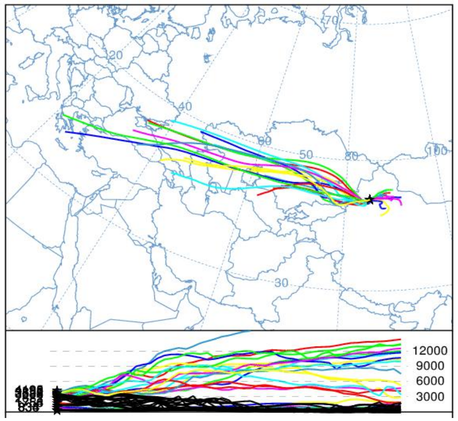
Xi'an, Shaanxi; Summer



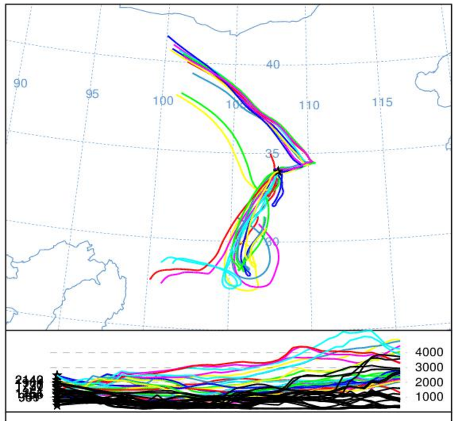
Jiujiang, Jiangxi; Summer



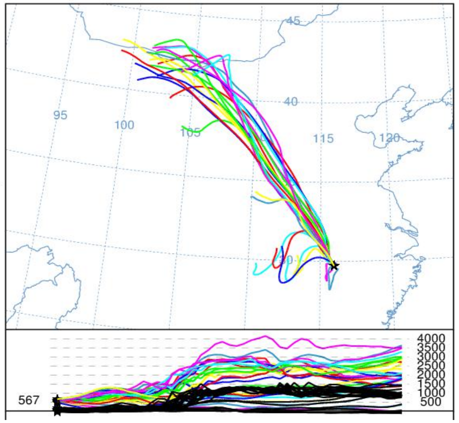
Shanghai; Summer



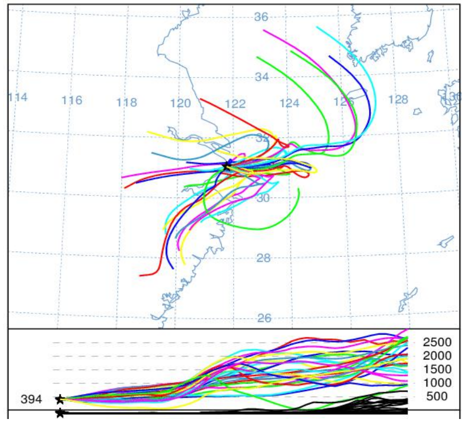
Tacheng, Xinjiang; Winter



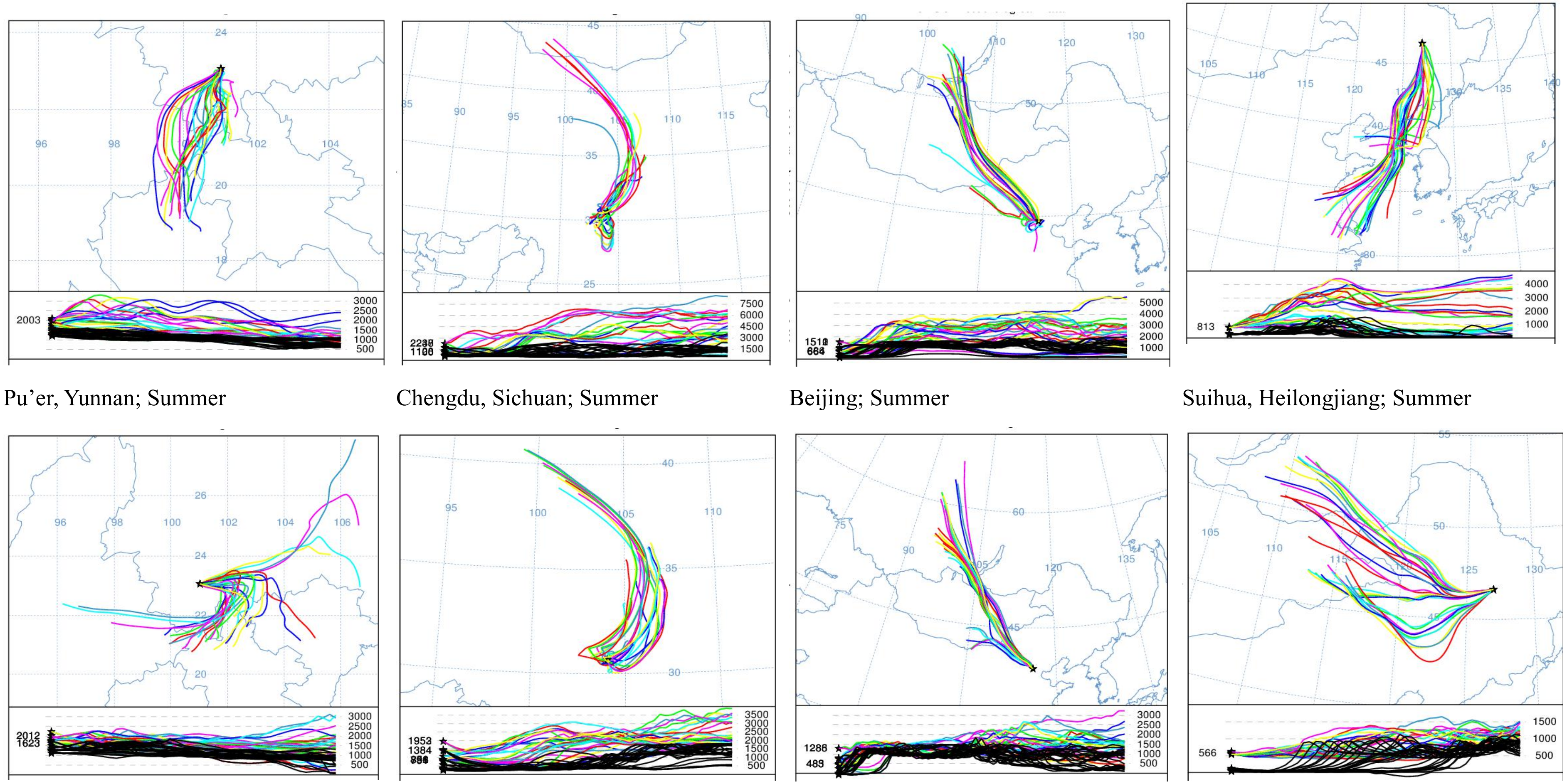
Xi'an, Shaanxi; Winter



Jiujiang, Jiangxi; Winter



Shanghai; Winter



Pu’er, Yunnan; Summer | Chengdu, Sichuan; Summer | Beijing; Summer | Suihua, Heilongjiang; Summer

Pu’er, Yunnan; Winter | Chengdu, Sichuan; Winter | Beijing; Winter | Suihua, Heilongjiang; Winter

Figure S5. **The backward trajectories of air mass extracted by Hysplit trajectory model**

Section S1. **Sampling rate of XAD–PAS in this investigation**

Sampling rate of XAD–PAS is a crucial factor to derive air concentrations from the amounts of chemicals accumulated in the XAD resin. Previous literature suggested the sampling rate of XAD–PAS of 3.5–4.5 m^3/d for PFASs (Li, Vento et al. 2011, Liu, Zhang et al. 2015, Tian, Yao et al. 2018). However, the actual sampling rate is dynamically variable, and affected by several factors. In this study, a standard solution containing mass labeled 1,2,3,4–$^{13}C_4$–PFOA and 1,2,3,4–$^{13}C_4$–PFOS (20 ng/mL) was spiked directly onto the upper XAD resin in the Shanghai sampling site (Floor of 5–story building of School of Environmental Science and Engineering in Shanghai Jiao Tong University) for one month in April 2017, to account for analyte losses during sampling. The sampling rate was calculated as flowing formulas:

$$\mathrm{R} = -\ln(\mathrm{C_t}/\mathrm{C_0}) \times \mathrm{d} \times \mathrm{A} \times (K_{\mathrm{XAD}}/\mathrm{t}) = -\ln(\mathrm{C_t}/\mathrm{C_0}) \times \mathrm{V} \times (K_{\mathrm{XAD}}/\mathrm{t}) \qquad \text{(S1)}$$

$$\log K_{\mathrm{XAD}} = 0.6366 \times \log(K_{\mathrm{OW}} \times \mathrm{S_W}/\mathrm{S_A}) \qquad \text{(S2)}$$

$$\mathrm{S_A} = \mathrm{P_L}/(\mathrm{RT}) \qquad \text{(S3)}$$

where C_t/C_0 represents the measured recoveries of 1,2,3,4–$^{13}C_4$–PFOA and 1,2,3,4–$^{13}C_4$–PFOS; V represents absorbent volume, 207.7(cm^3); K_{XAD} represent $^{13}C_8$–PFOA partition coefficient between air and XAD; t represents sampling time, 30 d; K_{OW}, S_W, and S_A, represent octanol–air partition coefficient (6.3), water solubility, and air solubility, respectively; P_L and R represent liquid vapor pressure and gas constant (8.314 J/(mol·K)), respectively. The $\log P_L$, and $\log S_W$ values was set as 1.3(Pa), and 0.24 (mg/L) in the present study.

The sampling rate of XAD–PAS was calculated as 3.2 m^3/d in the selected geographical site. However, higher temperature and wind speed were suggested to have positive effect on sampler uptake efficiency, while negative effect on the sorption capacity. Although the sampling rate of PFAAs were proposed of site-specific under different meteorological conditions, we have not conduct the depuration compounds loss test in all the 23 sampling sites. Since our calculated XAD–PAS rate value was very close to the recommended rate of 3.5–4.5 m^3/d for PFAAs, the rate value of 3.2 m^3/d was used in the present study.

Section S2. PMF analysis and uncertainty assessment

Positive matrix factorization (PMF) is considered an advanced algorithm among various receptor models, which has been successfully applied for source identification of environmental pollutants (Han, Fu et al. 2018; Han Fu et al. 2019). PMF has the following advantages: each data point is given an uncertainty–weighting; the factors in PMF are not necessarily orthogonal to each other and there is no non–negativity constraint with PMF. In the present study, PMF 5.0 (US EPA) was used to apportion the contributions of different sources to PFAAs in the atmosphere. The matrix X represents an ambient data set in which i represents the number of samples and j the number of chemical species. The goal of multivariate receptor modeling is to identify sources (p), the species profile (f) of each source and the amount of mass (g) contributed by each source to each individual sample as well as the residuals (e_{ij}), as following equation:

$$X_{ij}=\sum_{k=1}^{p} g_{ik} f_{kj}+e_{ij} \tag{S1}$$

The PMF solution minimizes the objective function Q based on these uncertainties (u):

$$Q=\sum_{i=1}^{n}\sum_{j=1}^{m}\left[\frac{X_{ij}-\sum_{k=1}^{p} g_{ik} f_{kj}}{u_{ij}}\right]^2 \tag{S2}$$

The input data files of PMF consist of concentrations and uncertainty matrices, and the uncertainty data were calculated as Equation (S3) as suggested by PMF User Guide. The missing values were represented by average values, while measurements below MDL (method detection limit) were replaced by two times of the corresponding MDL values. The “weak” variables were down–weighted, while “bad” variables were omitted form the analysis process.

$$\begin{cases} \mathrm{Unc}_i=\frac{5}{6}\times \mathrm{MDL}_i & C_i \le \mathrm{MDL}_i \\ \mathrm{Unc}_i=\sqrt{(C_i\times \mathrm{Error\ Fraction})^2+\left(\frac{1}{2}\times \mathrm{MDL}_i\right)^2} & C_i > \mathrm{MDL}_i \end{cases} \tag{S3}$$

The model was run 20 times with 49 random seeds to determine the stability of goodness–of–fit values.If the number of sources is estimated properly, the theoretical Q value should be approximately the number of degrees of freedom or the total number of data points. Three to six factors were examined, and four factors were found to be the most appropriate and most reasonably interpretable. Q (True) is the goodness–of–fit parameter calculated including all points, while Q (Robust) is the goodness–of–fit parameter calculated excluding points not fit by the model, Q (Robust) and Q (True) were 21672.9 and 25935, respectively, with Q(true)/Q_{exp} value of 12.56. Additionally, approximately 97% of the residuals calculated by PMF were within the range of –3 to 3, indicating a good fit of simulated results. The factor did not show oblique edges, suggesting there were little rotation for the solution. All these features implied the model simulation result was acceptable.

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