



## Technical Note

# Atmospheric Emission of Polybrominated Dibenzo-*p*-dioxins and Dibenzofurans from Converter Steelmaking Processes

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## ABSTRACT

Polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs) and polybrominated diphenyl ethers (PBDEs) were characterized and quantified in stack gas from secondary ventilation systems from five different steelmaking plants. The concentrations of 2,3,7,8-substituted PBDD/Fs (2,3,7,8-PBDD/Fs) and PBDEs were 0.036–0.19 ng/Nm<sup>3</sup> (0.32–4.33 pg toxic equivalency (TEQ)/Nm<sup>3</sup>) and 3.11–40.0 ng/Nm<sup>3</sup>, respectively, in the stack gas. These concentrations were lower than those in other metallurgical processes, such as electric arc furnaces and iron ore sintering processes. The concentrations of PBDEs were 26–94 times higher than those of PBDD/Fs in the stack gas. The PBDD/F and PBDE emission factors in the stack gas from the steelmaking converter processes (per tonne of steel produced) were 0.048–0.59 μg/t (0.66–20.8 ng TEQ/t) and 6.36–76.6 μg/t, respectively. These data are helpful for establishing an emission inventory of PBDD/Fs from the converter steelmaking industry in China.

**Keywords:** PBDD/F; Converter steelmaking; Emission factor; Emission pattern.

## INTRODUCTION

Polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs) have properties and effects similar to those of polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs) (Birbaum *et al.*, 2003). Concern about PBDD/Fs has increased in recent years because of their persistence and wide distribution in the environment and biota (Malmvärn *et al.*, 2005; Ashizuka *et al.*, 2008; Li *et al.*, 2008; Ma *et al.*, 2009; Ren *et al.*, 2011; Zhang *et al.*, 2012; Tue *et al.*, 2013). These compounds have also been found in human blood, lipids, and breast milk (Zober *et al.*, 1992; Ott and Zober, 1996; Choi *et al.*, 2003; Jogsten *et al.*, 2010).

Although lower brominated PBDD/F congeners can naturally form through photochemical and biosynthetic pathways (Haglund, 2010; Arnoldsson *et al.*, 2012), the major sources of PBDD/Fs are thermal industrial activities, such as metallurgical processes, solid waste incineration, and electronic waste dismantling (Du *et al.*, 2010; Gullett *et al.*, 2010; Wang *et al.*, 2010b; Duan *et al.*, 2011; Chang *et al.*, 2013; Chang *et al.*, 2014). Widespread use of brominated flame retardants (BFRs), including polybrominated diphenyl

ethers (PBDEs) and tetrabromobisphenol A, provides an abundance of bromines for PBDD/F formation in industrial processes (Weber and Bertram, 2003; Wang *et al.*, 2010a; Wyrzykowska-Ceradini *et al.*, 2011).

It has been suggested that thermal processes, such as incineration, iron ore sintering (SNT), and electric arc furnace (EAF) processes, are major PBDD/F sources (Wang and Chang-Chien, 2007; Du *et al.*, 2010; Wang *et al.*, 2010b). Du *et al.* (2010) reported that PBDD/Fs could be emitted by various incineration and metallurgical processes (Du *et al.*, 2010; Wang *et al.*, 2010b). These studies revealed that metallurgical facilities are important sources of PCDD/Fs and PBDD/Fs to the environment. In the integrated steelmaking industry, converter steelmaking is a major steelmaking process, and has been identified as an emission source of PCDD/Fs and PCBs (Jager, 1993; Grochowalski *et al.*, 2006; Jackson *et al.*, 2012). However, little is known about PBDD/F emissions from converter steelmaking processes, and it is necessary to further investigate PBDD/F emissions from this source.

The volume of crude steel production in China, where there are more than 600 converters of different types and sizes, is about half of the world's total production. Thus, the investigation of PBDD/F emissions from Chinese iron foundries will likely be important for preliminary estimation of total global PBDD/F emissions. In our previous work, PCDD/Fs, PCBs, and polychlorinated naphthalenes from secondary ventilation systems of five different converters

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in five different steel plants were studied (Li et al., 2014). In this study, we mainly focus on PBDD/Fs formation and emission in stack gas from these plants. The concentrations, fingerprints, and emission factors of PBDD/Fs and PBDEs in stack gases from converter steelmaking processes are compared with other sources. This study provides useful information for source identification and control of PBDD/Fs and PBDEs.

## METHODS

### Basic Information from the Converter Steelmaking Plants

Molten iron, scrap steel, iron alloys, and other materials are used as the primary raw materials of converter steelmaking. Typically, an average of 10–30% of the converter charge is scrap steel. Dust and gas are emitted from converter steelmaking during the charging of scrap and hot metal, oxygen blowing, and tapping from the converter. During charging or tapping operations, the converter is tilted. A secondary ventilation and de-dusting system is often installed to reduce the dust emissions. The secondary ventilation system usually consists of a canopy hood, which is placed just above the converter in a tilted position. During blowing, the secondary system extracts most of the emissions that are not captured by the primary ventilation system. Subsequent treatment of the evacuated gas is usually performed by means of a bag filter or an electrostatic precipitator.

Top- and bottom-blown converters are the most popular converters for steelmaking. Four top- and bottom-blown converters (SG, TG, RC and XJ) and one top-blown converter (XH) from five different steelmaking plants were selected in northern China. Among the five converters, the converter SG uses a more modern steelmaking technique than the others, and this produces higher quality steel, uses less energy, and emits less SO<sub>2</sub> and NO<sub>x</sub>. The converters could process between 50 and 300 t (1 t = 1000 kg) per batch. Details of the selected converter steelmaking plants are shown in Table 1.

### Sampling Procedures

Fifteen stack gas samples were collected from the secondary ventilation systems of the five converters. Bag filters were used to control particulate matter emission in the five converter steelmaking plants. Stack gas samples were collected by an automatic isokinetic sampling system (Isostack, Tecora Italy). Three stack gas samples were collected at each plant to investigate PBDD/F emissions. Each sample was collected for about 2–3 h. Within this sample collection time, the operating process for a single batch was completed.

### Sample Analyses

The stack gas samples were spiked with <sup>13</sup>C<sub>12</sub>-labeled internal standards (EDF-5408 (Cambridge Isotope Laboratories, Inc., MA, USA) and MBDE-MXFS (Wellington Laboratories, Inc., Guelph, Canada). After Soxhlet extraction of each stack gas sample with toluene (pesticide grade, Thermo Fisher Scientific, Inc., MA, USA) for 20 h, the extract was eluted through a multilayer silica

Table 1. Basic information about the investigated converters and concentrations of PBDD/Fs and PBDEs in the stack gas.

	SG		RC		TG		XJ		XH	
	TB <sup>a</sup>	300	TB <sup>a</sup>	120	TB <sup>a</sup>	120	TB <sup>a</sup>	120	T <sup>b</sup>	50
Converter type										
Converter scale (t)										
Average flow rate (Nm <sup>3</sup> /h)		324279		1175206		994669		543129		199229
Stack gas samples										
	Mean	Range	Mean	Range	Mean	Range	Mean	Range	Mean	Range
PBDD/Fs in mass concentrations (pg/Nm <sup>3</sup> )	68.2	43.4–81.0	71.4	51.7–72.3	68.5	35.8–183	59.0	96.4–179	128	90.2–167
PBDD/F TEQs (pg/Nm <sup>3</sup> )	0.74	0.62–0.85	0.51	0.32–0.62	1.33	0.40–2.32	3.35	2.36–4.33	1.16	0.64–1.67
PBDEs (ng/Nm <sup>3</sup> )	37.3	34.6–40.0	5.14	3.11–8.35	6.79	3.52–11.3	4.41	3.96–4.86	6.29	5.93–6.89

<sup>a</sup> Top and bottom combined blown converters.

<sup>b</sup> Top blown converters.

gel column and an activated carbon column. The activated carbon column was sequentially eluted with 80 mL of dichloromethane/hexane (5/95, v/v) for the PBDEs, and an extra elution with 250 mL of toluene was included for the PBDD/Fs. Each extract was then concentrated to a volume of about 20  $\mu\text{L}$ . Before injection,  $^{13}\text{C}$ -labeled injection solutions (EDF-5409 and MBDE-MXFR, Wellington Laboratories Inc., Guelph, Canada) were added for calculating recoveries. Analysis of PBDD/Fs and PBDEs was carried out using a gas chromatograph coupled with a double focusing system mass spectrometer (Thermo Fisher Scientific, Inc., MA, USA). Selected ion monitoring mode was used for data acquisition. A DB-5MS fused-silica column (15 m  $\times$  0.25 mm i.d., 0.10  $\mu\text{m}$ , Agilent Technologies Inc., USA) was used for the separation of congeners of PBDD/Fs and decabromodiphenyl ether with a splitless/surge injection. A DB-5MS fused-silica column (30 m  $\times$  0.25 mm i.d., 0.10  $\mu\text{m}$ , Agilent Technologies Inc., USA) was used for separation of tri to octa PBDE congeners (injector temperature 300°C, transfer glass line temperature 280°C). The MS was tuned and operated at around 10,000 resolution with 45 eV EI energy.

#### Quality Control and Quality Assurance

In this study, thirteen 2,3,7,8-substituted PBDD/F congeners and fourteen typical PBDE congeners (brominated diphenyl ether (BDE): BDE-17, BDE-28, BDE-47, BDE-66, BDE-71, BDE-85, BDE-99, BDE-100, BDE-138, BDE-153, BDE-154, BDE-183, BDE-190 and BDE-209) were analyzed. Peaks of congeners were identified based on comparison of their retention times to available individual standards and from ion ratios. Peaks were quantified if target/qualifier ion ratios were within 15% of theoretical values. The recoveries of the surrogate sampling standards in the stack gas samples were 55–108%. The range for the recoveries of  $^{13}\text{C}$ -labeled standards of tetra- to hepta-BDD/Fs was 40% to 106%, and that for octabromodibenzo-*p*-dioxin (OBDD) and octabromodibenzofuran (OBDF) was 20% to 60%. The range for the recoveries of the  $^{13}\text{C}$ -labeled standards of PBDEs was 58–112%. One blank sample was included in each batch of samples analyzed. The limit of detection (LOD) was defined as a signal-to-noise ratio (S/N, peak to peak definition) greater than three. For congeners with concentrations below the LOD, a value of LOD/2 was used to calculate the total concentrations.

## RESULTS AND DISCUSSION

#### PBDD/F and PBDE Concentrations in Stack Gas from Converter Processes

The PBDD/F and PBDE concentrations measured in the stack gases of the five converters are shown in Table 1. The toxic equivalency (TEQ) values for the PBDD/Fs were calculated using interim toxic equivalency factors similar to the chlorinated analogs, as suggested by the World Health Organization (2005) scheme (Berg *et al.*, 2013). The ranges for mass concentration and TEQ of PBDD/F in the stack gas were 0.036–0.19  $\text{ng}/\text{Nm}^3$  and 0.32–4.33  $\text{pg TEQ}/\text{Nm}^3$ , respectively. These results were lower than those for carbon

steel EAFs (Wang *et al.*, 2010b) but were comparable to those from stainless steel EAFs, SNTs and coal-fired power plants (Wang *et al.*, 2010b; Tu *et al.*, 2011). The PBDD/F concentrations from the converters were lower than those from crematoriums, hazardous waste incinerators, and secondary metal smelters (Du *et al.*, 2010). Fig. 1 shows the concentrations of PBDD/Fs in the stack gases from different size converters. There was no obvious correlation between the size of the converter and the concentrations. Furthermore, although a modern technique was used in the SG converter, the concentrations of PBDD/Fs in its stack gas were not obviously lower than in the stack gases from the other converters.

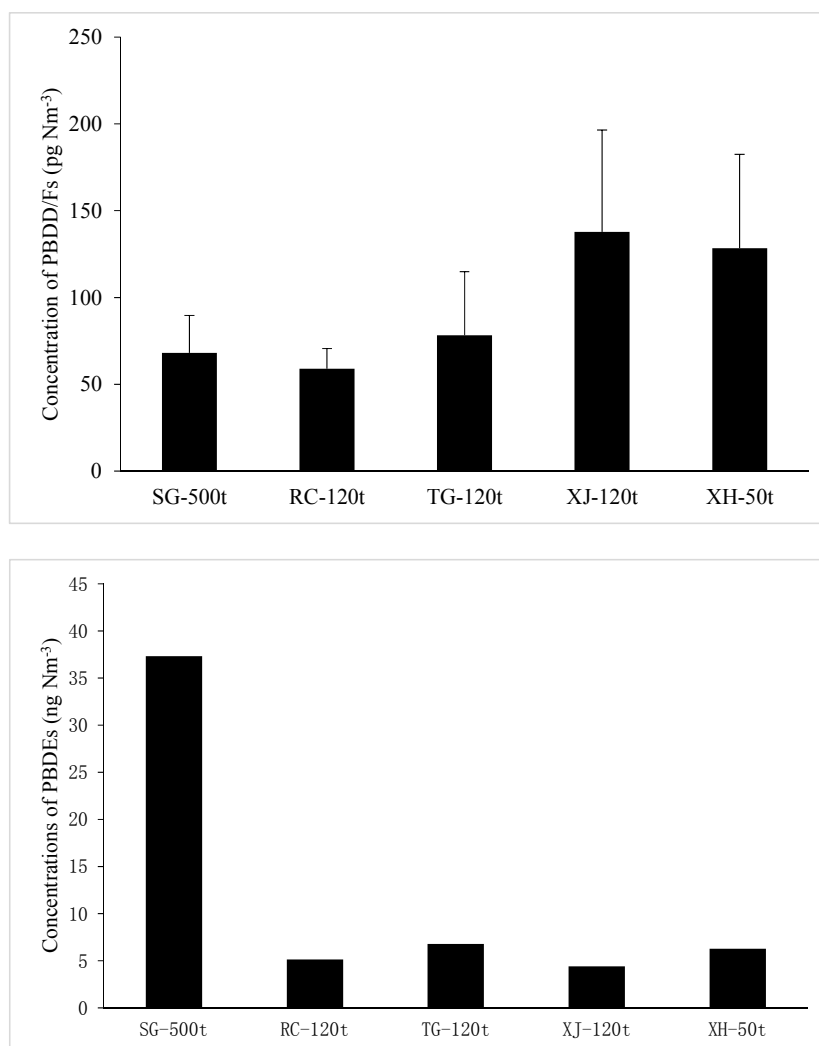
For the PBDD/Fs, the mass concentrations were comparable with those of the PCDD/Fs but the TEQs were slightly lower (Li *et al.* 2014). It has been reported that in several thermal processes, including MSWIs, SNT, EAF steelmaking, and secondary metallurgical processes, the emission levels of PCDD/Fs are higher than those of PBDD/Fs (Du *et al.*, 2010; Wang *et al.*, 2010c).

Because PBDEs are considered to be precursors of PBDD/Fs (Weber and Bertram, 2003), the concentrations of PBDEs were also investigated in this study. The results showed that PBDEs were present in the stack gases from converter steelmaking, and the range for the mean PBDE mass concentrations (14 congeners) in the stack gases of the five converters was 3.11–40.0  $\text{ng}/\text{Nm}^3$ . The PBDE mass concentrations (30 congeners) were comparable to those of EAFs (15.7  $\text{ng}/\text{Nm}^3$ ), fly ash treatment plants (15.7  $\text{ng}/\text{Nm}^3$ ), and SNT plants (35.2  $\text{ng}/\text{Nm}^3$ ) (Wang *et al.*, 2010b). The mass concentrations of PBDEs were about 26–94 times higher than those of PBDD/Fs.

#### Congener Profiles of PBDD/Fs and PBDEs

Congener profiles of the thirteen 2,3,7,8-substituted PBDD/F species are represented as relative concentrations of substituted congeners to the total substituted congeners from each facility in Fig. 2(a). The most abundant congener of the PBDD/Fs was 1,2,3,4,6,7,8- heptabromodibenzofuran (HpBDD), followed by OBDF, 1,2,3,4,6,7,8-heptabromodibenzofuran (HpBDF), and OBDD. The contributions of the other congeners were minimal. The higher brominated congeners, including 1,2,3,4,6,7,8-HpBDF, 1,2,3,4,6,7,8-HpBDD, OBDF and OBDD, accounted for 74–98% of the total mass concentration of the thirteen 2,3,7,8-substituted PBDD/F species. Overall, the concentrations of the PBDFs were generally higher than those of the PBDDs. Similar congener profiles were also found in stack gas from MSWI, SNTs and EAF processes (Wang *et al.*, 2010a, b). These results indicate that higher brominated PBDD/F congeners are likely to form during thermal processes.

For the PBDE congeners, Fig. 2(b) shows that BDE-209 was the most abundant congener and accounted for 38–60% of the total mass concentration of PBDEs. The next most abundant congeners were BDE-99 and BDE-47. These results are similar to those from other thermal processes, including MSWI, SNT and EAF processes (Gullett *et al.*, 2010; Wang *et al.*, 2010a, b). In the converter steelmaking process, the main raw material is molten iron, the



**Fig. 1.** Concentrations of PBDD/Fs and PBDEs in stack gases from converter steelmaking plants.

temperature of which can reach around 1200°C. At such a high temperature, most of the organic compounds in the converter should be destroyed, which might lead to low emission levels for both PBDD/Fs and PCDD/Fs during the cooling processes (Sakai *et al.*, 2001; Hunsinger *et al.*, 2002).

In this study, on average 10–30% of the converter charge was scrap steel. Scrap steel can contain impurities, such as plastic and foam, which can introduce BFRs to the converter (Alcock *et al.*, 2003). Studies on the emission of PCDD/Fs, PBDE and PBDD/Fs from EAF processes, which use scrap steel as the main raw material, have shown that scrap steel can lead to relatively high concentrations of PCDD/Fs, PBDE and PBDD/Fs (Lee *et al.*, 2004; Wang *et al.*, 2010b). Because PBDEs have been widely used as BFRs for decades in China, and because high concentrations of PBDEs were found in the stack gases of the studied converters, scrap steel could be the main source of bromine containing compounds among all the raw materials.

#### **Emission Factors of PBDD/Fs**

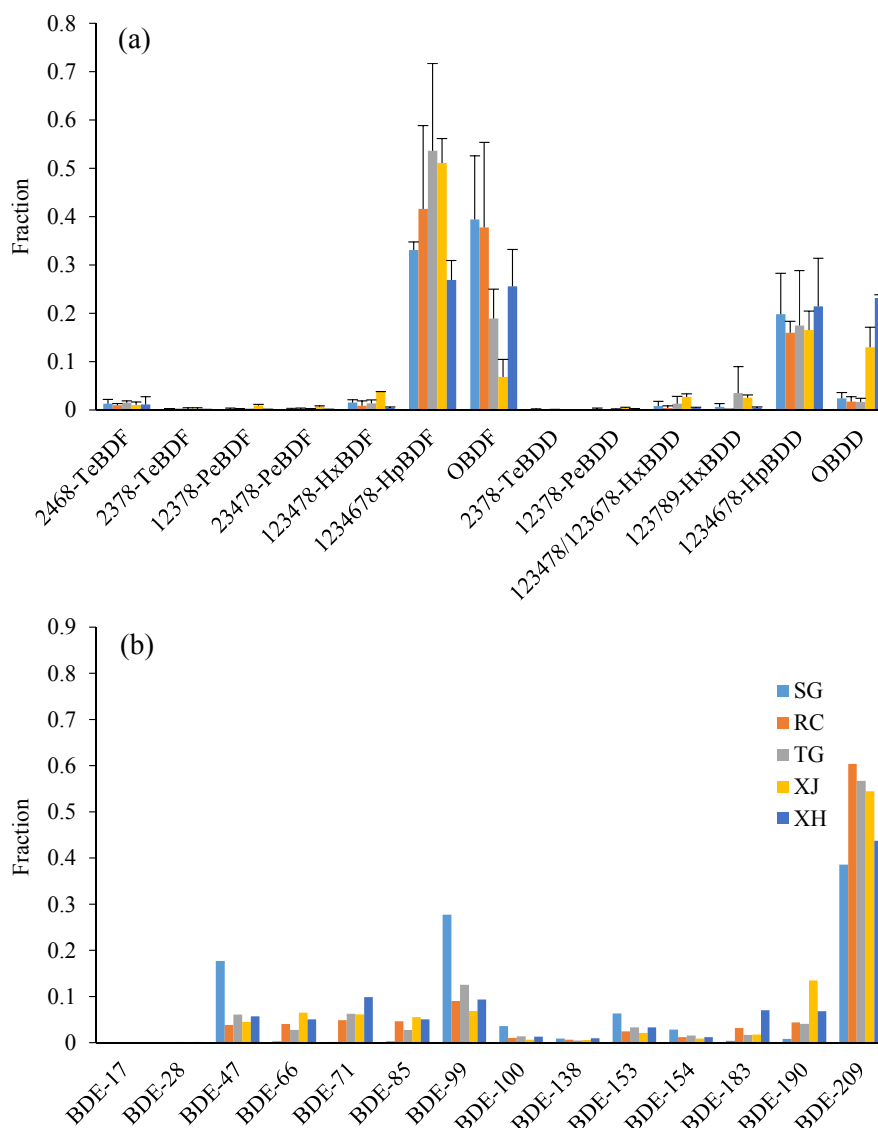
Emission factors are usually used to estimate PBDD/F and PBDE emission inventories. Emission factors and emission

amounts were calculated for PBDD/Fs and PBDE in this study using the following equations:

$$\text{Emission Factor} = (\text{Flow rate} \times \text{Concentration}) / \text{Output rate} \quad (1)$$

$$\text{Emission Amounts} = \text{Activity level} \times \text{Emission factor} \quad (2)$$

The emission factors for PBDD/Fs and PBDE from the stack gas were estimated (per tonne of steel produced) for the converter steelmaking processes. The ranges for the emission factors from the five studied converters were 0.048–0.59 µg/t (0.66–20.8 ng TEQ/t) and 6.36–76.6 µg/t for PBDD/Fs and PBDE, respectively. The emission factors for the PBDD/Fs were lower than those previously reported for EAFs (0.225–22.9 µg/t) and SNTs (0.222–8.15 µg/t). The emission factors of the PBDEs were comparable to those of SNTs with selective catalytic reduction (32.1–45.7 µg/t), and much lower than those of SNTs without selective catalytic reduction (101–169 µg/t) and EAFs (71.8–755 µg/t) (Wang *et al.*, 2010b).



**Fig. 2.** 2,3,7,8-PBDD/F (a) and PBDE (b) fingerprints of the stack gases from converter steelmaking plants.

The average emission factors of the PBDD/Fs and PBDEs in the five converter steelmaking processes were used to estimate the emission masses of these compounds in China. Based on the annual crude steel output of China in 2012 ( $5.67 \times 10^8$  t), preliminary estimates of the annual atmospheric emissions of PBDD/Fs and PBDEs from secondary ventilation systems in converter steelmaking in China were 0.20 kg (3.30 g TEQ) and 17.4 kg, respectively.

## CONCLUSIONS

The emissions of PBDD/Fs from five converter steelmaking plants in China were investigated. Different converter types were studied, and no significant differences in the emissions were found among the converter types. The concentrations of PBDEs were much higher than those of PBDD/Fs in the converter steelmaking process. The high concentrations of PBDEs may provide an abundant source of bromine for PBDD/F formation. The masses of PBDD/Fs

emitted from the converter steelmaking processes were much lower than those emitted from SNT and EAF processes. This suggests that the converter steelmaking process is currently not a priority PBDD/F source for PBDD/F emission control in China.

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