

Research Articles

Determination of PCDDs, PCDFs, PCBs and HCB emissions from the metallurgical sector in Poland

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Abstract

Background. The aim of the project was to measure the actual dioxins, furans, PCB and HCB emissions from the 20 selected metallurgical installations in Poland for updating the national inventory of dioxin emission from the metallurgical industry. The goal of the project is as well the strategy development for dioxins and furans emission abatement from the subject facilities (UNEP 2005).

Methods. Sampling methodology used in this work was developed at Cracow University of Technology because of the complexity of PCDFs, PCDDs, PCB and HCB simultaneously sampling and determination. For the determination GC-MS/MS system was used.

Results and discussion. Results from the work indicated, that the highest dioxins and PCB concentrations were recorded for sintering of iron ore plants at 1.10 – 1.32 ng total TEQ/Nm³ as well as for aluminium scrap melting at 0.03 – 0.66 ng total TEQ/Nm³. The highest HCB concentrations at 613 – 1491 ng/Nm³ were also recorded for iron ore sintering plants, however, for aluminium plants the HCB concentrations were in the range of 10.1 – 22.7 ng/Nm³.

Conclusions. It indicates that secondary aluminium production is the most significant dioxins source if calculated as an emission factor value, however, iron ore sintering plants are operated in much higher product mass efficiency, caused this process to be the most important for dioxins, PCB and HCB pollution to the atmosphere in Poland.

Recommendation and Perspective. Basing on the performed tests and the environmental reviews of selected plants recommendations were formulated for the reduction of generation or emission of these substances from iron ore sintering plants, electric arc furnace steel production processes, hot-blast furnace operations, secondary aluminium smelting and primary zinc production from zinc cathodes.

Keywords: dioxin sources; dioxins; emission inventory; furans; HCB; metal industry; PCB; PCDD; PCDF; POPs;

Introduction

Until 2003 the emission inventory data for metallurgical sector in Poland was estimated on the basis of default emission factors taken mostly from UNEP toolkit (Quaß et al. 2000). Because the dioxins, furans, PCB and HCB formation is process specific, which is especially applicable for thermal processes realised in metal industry (US EPA, 2002), the use of default emission have to be considered as highly uncertain (Quaß et al. 1997, Lassen et al. 2003). The aim of the project was to measure the actual dioxins, furans, PCB and HCB emissions from the selected metallurgical installations in Poland for updating the national inventory of dioxin emission from the metallurgical industry.

The goal of the project is as well the strategy development for dioxins and furans emission abatement from the subject facilities. For this purposes there were selected installations in 20 different facilities of the metallurgical sector in Poland. They were chosen from among those, which replied positively to the invitation to participate. Emission of selected chlorinated POPs from these installations was measured. Participation of all the industries in the project was voluntary, the results obtained, the names or the facilities, and their addresses are confidential.

In selection of plants and installations, an effort was made to include into investigations each of the production process and ensure that the tested installation was typical emission standard for the given industrial process. The technological process at time of measurement had to be run with maintenance of typical, nominal load of production standards. The measurements were performed by Cracow University of Technology within the Polish-Danish "Reduction of Dioxin

Emission from the Metallurgical Sector in Poland” project, financed by the Danish Environment Protection Agency (from DANCEE funds). Project activities were co-ordinated by COWI A/S.

1. Plant selection and the description of plant profile.

Measuring was conducted in the 20 metal industry installations in Poland as it is presented in **Table 1**, which were selected and accepted by Polish Ministry of Environment for this work. (for confidential reasons, only sector and profile of the plant is given)

Table 1: List of selected and examined installations

Installation examined and its allocated identity number	
01 Foundry of spheroid and cast iron	11 Aluminium scrap melting-natural gas furnace
02 Iron ore sintering plant	12 Aluminium scrap and cans melting-electric furnace
03 Enrichment of copper ore	13 Zinc ore roasting (sphalerite)- H ₂ SO ₄ production from stack gases
04 Copper smelting (production of sulphuric acid)	14 Smelting waste from production of zinc cathodes
05 Copper smelting (de-coppering of slag)	15 Steel scrap melting-steel production in an electric arc furnace **
06 Copper smelting (production of sulphuric acid)	16 Production of primary iron in a blast furnace
07 Foundry of cast iron *	17 Steel production in a basic oxygen furnace
08 Foundry of cast steel-electric arc furnace	18 Grey and spheroid graphite iron production -gas rotary furnace
09 Aluminium scrap melting-electric furnace	19 Iron ore sintering plant
10 Aluminium scrap melting-rotary furnace	20 Steel scrap melting-steel production in an electric arc furnace **

* - measurements were performed on raw gas. Sampling of cleaned gas was not possible for this plant because of technical reasons.

** For these plants measurements were performed only on secondary off-gas. However, the secondary off-gas represents only a very small part of the total PCDD/PCDF/PCB/HCB emission, because of technical reasons, measuring of primary off-gas for these plants were not possible.

1.1. Steel and iron metallurgy

There are four different processes performed in this sector:

Sintering of iron ore. This process is commonly known as generating high concentrations of dioxins and PCB. As can be drawn from technical literature 2–4 g of TEQ of these substances are released annually into air by the European countries.

Steel production: blast furnace and converter process. In both of these processes, a relatively low dioxin concentration was measured, which may be the result in the presence of a high SO₂ concentration causing blockage of the chlorination of aromatic compounds. This phenomena (however still disputable – Buekens et al. 2001) results from the mechanism of the, so called, de-novo synthesis, in which dioxins are created during the reaction of atomic chlorine with molecules of atomic carbon. In most cases the dioxins concentration is less than 0.1 ng I-TEQ/m³ in stack gases from these processes.

Melting of iron and steel scraps. Because of the high content of plastics, coatings, paintings and sealing as well as other nonferrous materials in the charge during preliminary thermal operations concerned with heating up the iron and steel scrap a strong emission of pollutants, including dioxins and PCBs takes place.

1.2 Copper metallurgy

Two basic processes are being implemented in the Polish copper metallurgy described below.

Sintering and enrichment of copper ore is connected with high emission of SO₂, which is used directly for sulphuric acid production (SOLINOX plant). Because of the extremely high of SO₂ concentration, reaching up to 250 g/m³, the dioxins formation in this process is negligible. Moreover, the process of H₂SO₄ production requires multi-step scrubbing of process gases leading to a very effective removal of dioxins and PCBs traces. The investigations performed had to confirm this theory.

Recovery of copper from slag of thermal processes requires application of various additives and represents a high temperature process. The copper oxide is a known catalyser for the formation of dioxins in thermal processes.

In both cases the concentration values for dioxins, PCBs and HCBs from these processes are unknown. Reactions of dioxins formation under such circumstances are not recognised and only measurements may confirm the actual values of their concentrations.

1.3 Aluminium metallurgy

In all cases (four installations) the thermal process was concerned with melting aluminium scrap using different technologies: electric furnace, gas chamber furnace, gas and oil fired rotary furnace and electric furnace with preheating to remove plastics and decoration coatings (aluminium beverage tins).

1.4 Zinc metallurgy

Two processes taking place in two technological operations in zinc metallurgy were subject of examination. Enrichment and sintering zinc ore connected release of a high SO_2 concentration. Similarly to copper ore the process gases in this case contain a high concentration of SO_2 , which is used also for production of sulphuric acid. Low concentration of dioxins and PCBs confirms the theory of dioxin formation inhibition in presence of high SO_2 concentrations and that the process of sulphuric acid production reduces dioxins from process gases to trace values, below 0.1 ng I-TEQ/m^3 . Remelting zinc from waste obtained from production of zinc electrodes used in electrolytic processes. In this process, an additive of ammonium chloride (NH_4Cl) is applied, which presents a potential source of dioxin formation because of chlorine content.

2. Sampling and analysis methodology

However, there is established an EU standard for the determination of PCDD and PCDF concentration in stack gas from incineration of wastes (European Standard EN-1948:1966), the standard is not actually valid for the determination of these compounds in flue gases from metal industry. Moreover, the EN-1948 standard is not validated for the determination of PCB and HCB yet. Therefore, sampling methodology used in this work was developed at Cracow University of Technology because of the complexity of PCDFs, PCDDs, PCB and HCB simultaneously sampling and determination.

2.1. Stack gas sampling

The sampling train is designed for filter/condenser method as it is described in European Standard EN-1948:1996 part 1. In this work large volumes of flue gas were sampled in average of 10 Nm^3 * samples because it was assumed that concentrations of PCDFs and PCDDs would be at ng/m^3 or even pg/m^3 level. Samples of stack of off-gas were taken using the EMIOTEST 9265 sampling apparatus (Wrocław, Poland). The schematic presentation of the apparatus is presented in **Fig.1**. This instrument was specially designed for isokinetic sampling of industrial gas samples for the determination of trace organic compounds and is validated for the determination of PCDD, PCDF, PCB and HCB. Sampling nozzles (1) and probe (2) were made of titanium (1). Fly ash (if present) were taken using glass fibre filters of pore size of $0,2 \mu\text{m}$ which were placed in the heated compartment (3). For stack gas or off-gas of high humidity an aqueous condensate containing dissolved compounds was collected. PCDD, PCDF, PCB and HCB present in vapour form in cooled flue gas stream were adsorbed on PU-foam plug placed in a cartridge (4). In this work the product No: 20038 obtained from "Supelco", Bellefonte, USA was used.

Samples gas was dried with silica (5) and gas volume, humidity, temperature and pressure were recorded in central unit (6). Pulse damper (7) is used for stabilizing of sampling gas pump operation (8).

All compartments before sampling were spiked with appropriate ^{13}C sampling standards for sampling recovery control, accordingly EN-1948 part 1. No ^{13}C -PCB or ^{13}C -HCB were used for sampling recovery control.

Samples of fly ash collected on glass fibre filters, PU-foam and water condensate were immediately delivered to the laboratory. After standard addition samples were extracted with toluene according to procedures recommended by EN-1948.

This procedure guarantees 90-95% collection efficiency of PCDDs, PCDFs, PCBs and HCB from flue gas. Limit of Detection (LD) for PCDFs/PCDDs, calculated for stack gas samples was $0,002 - 0,005 \text{ ng-TEQ/Nm}^3$ in gas sample and depended on the plant which was measured. LD for HCB was in the range of $0,05 - 0,1 \text{ ng/Nm}^3$

The validation tests performed on iron ore sinter plant (Plant No:02) have shown that the average break-thru mass of PCDD, PCDFs, PCBs and HCB is not higher than 10% of total mass of measured congeners in the measured stream of sampling gas. Validation was performed using two PU-foam plugs connected in series and the break-thru masses of individual congeners were determined in the second adsorbent (Grochowalski et al. 2001).

* Nm^3 is one cubic meter of stack or off-gas in standard conditions (so called: "Normal cubic meter")

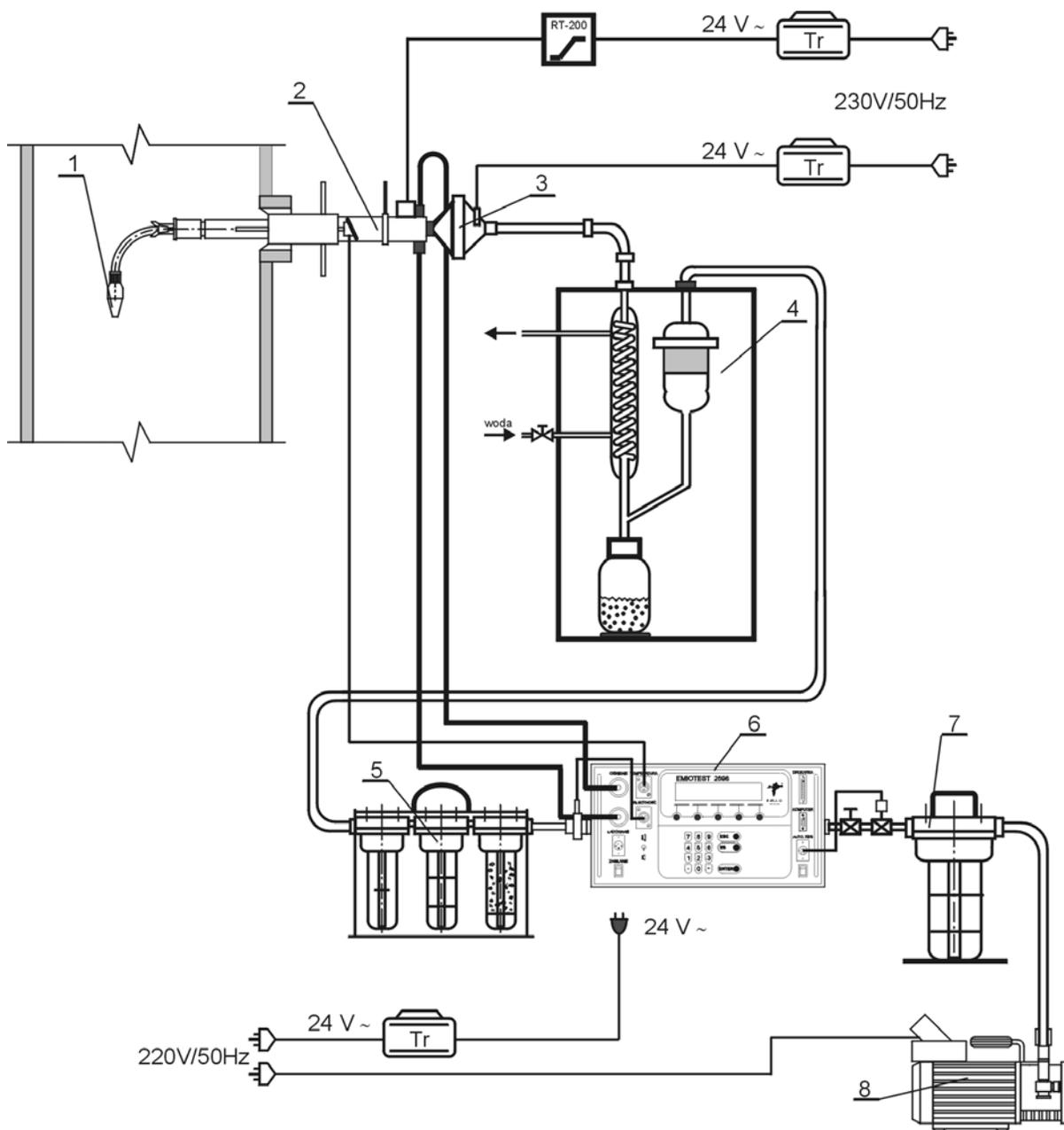


Fig. 1: Sampling apparatus used in the determination of PCDD, PCDF, PCB and HCB in stack and off-gases in metal industry in Poland in 2004.

2.2 Sample extraction, clean-up and analysis

Samples extraction and clean-up have been performed using the standard procedures based on active carbon column, acidic/base silicagel and Alumina columns as it is indicated in EN-1948 p.2, however, in the determination of indicator PCBs and HCB some modifications were made with respect to the collected fractions from carbon column and Alumina column (Grochowalski et al, 1996,1997,1998).

A ThermoQuest GCQplus GC-MS/MS system was used for analysis. The system consists of a Trace 2000CE gas chromatograph equipped with split/splitless injector coupled with ion trap mass selective detector working in MS/MS mode. Two different columns were used for chromatographic separation of PCDD/F congeners: DB-5MS (60m, 0,25 mm) as routinely used, low polarity capillary column and DB-17 (30m., 0,25 mm).

3. Methods of calculation

The list of PCDDs/PCDFs/PCBs congeners used in this work for the determination of I-TEQ for PCDD/F calculation is presented in **Table 2**. Moreover, in Table 2 there is presented 12 WHO-PCB congeners which were used in the determination of WHO-PCB-TEQ.

Calculation of the T-TEQ is a sum of I-TEQ and WHO-PCB-TEQ based on the, so-called, NATO (or International) toxic equivalent factor of 2,3,7,8-TCDD (I-TEF) for PCDDs/PCDFs (**Table 3**) and WHO-PCB-TEQ based on WHO-PCB-TEF factors (**Table 4**).

Table 2: List of measured congeners for: polychlorinated dibenzodioxins - column I, polychlorinated dibenzofurans - column II, coplanar non-ortho and mono-ortho WHO-PCB – column III and so called Indicator I-PCB – column IV

PCDD congener	PCDF congener	WHO-PCB congener	I- PCB congener
2,3,7,8-TCDD	2,3,7,8-TCDF	PCB 77	PCB 28
1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDF	PCB 81	PCB 52
1,2,3,4,7,8-HxCDD	2,3,4,7,8-PeCDF	PCB 105	PCB 101
1,2,3,6,7,8-HxCDD	1,2,3,4,7,8-HxCDF	PCB 114	PCB 118
1,2,3,7,8,9-HxCDD	1,2,3,6,7,8-HxCDF	PCB 118	PCB 138
1,2,3,4,6,7,8-HpCDD	2,3,4,6,7,8-HxCDF	PCB 123	PCB 153
OCDD	1,2,3,7,8,9-HxCDF	PCB 126	PCB 180
	1,2,3,4,6,7,8-HpCDF	PCB 156	
	1,2,3,4,7,8,9-HpCDF	PCB 157	
	OCDF	PCB 167	
		PCB 169	
		PCB 189	

Table 3: List of individual PCDD/F congeners and their respective TEF_i values (as recommended with EN-1948 EU standard)

PCDD i-congener	Value of the TEF _i	PCDF i-congener	Value of the TEF _i
2,3,7,8-TCDD	1	2,3,7,8-TCDF	0,1
1,2,3,7,8-P5CDD	0,5	2,3,4,7,8-P5CDF	0,5
1,2,3,4,7,8-H6CDD	0,1	1,2,3,7,8-P5CDF	0,05
1,2,3,6,7,8-H6CDD	0,1	1,2,3,4,7,8-H6CDF	0,1
1,2,3,7,8,9-H6CDD	0,1	1,2,3,6,7,8-H6CDF	0,1
1,2,3,4,6,7,8-H7CDD	0,01	1,2,3,7,8,9-H6CDF	0,1
OCDD	0,001	2,3,4,6,7,8-H6CDF	0,1
		1,2,3,4,6,7,8-H7CDF	0,01
		1,2,3,4,6,7,8-H7CDF	0,01
		OCDF	0,001

Table 4: List of individual WHO-PCB congeners and their respective TEF_j values, however, expressed mostly for food analysis

WHO-PCB j-congener	Value of the TEF _j	WHO-PCB j-congener	Value of the TEF _j
PCB77	0,0001	PCB126	0,1
PCB81	0,00001	PCB156	0,0005
PCB105	0,0001	PCB157	0,0005
PCB114	0,0005	PCB167	0,00001
PCB118	0,0001	PCB169	0,01
PCB123	0,0001	PCB189	0,001

Calculation of the I-TEQ mass value was performed in accordance with the **formula (1)**:

$$I - TEQ = \sum_{i=17}^{i=1} (m_i \times TEF_i) \quad (1)$$

where:

m_j - mass of individual i-congener of PCDD and PCDF (according to **Table 3**) [ng],

Calculation of the WHO-PCB-TEQ mass value was performed in accordance with the **formula (2)**:

$$WHO-PCB-TEQ = \sum_{i=12}^{i=1} (m_j \times TEF_j) \quad (2)$$

where:

m_j - mass of individual WHO-PCB j -congener (according to **Table 4**) [ng],

Total T-TEQ is calculated from the **formula (3)** as a sum of I-TEQ obtained from the formula (1) and WHO-PCB-TEQ obtained from the formula (2):

$$T - TEQ = I - TEQ +_{WHO-PCB} TEQ \quad (3)$$

4. Results and discussion:

Results obtained for 20 selected plants are presented in the **Table 5**.

Table 5: PCDD/Fs, PCBs and HCBs concentrations in flue gases from 20 metallurgical plants investigated in Poland during the period June–September 2004. Values are given in ng/Nm³ for flue gases under standard conditions and for the actual O₂ concentration

Plant No	Concentrations in flue gas					
	PCDD/F [ng I-TEQ/Nm ³]	PCB [ng WHO-PCB TEQ/Nm ³]	Total [ng T-TEQ/Nm ³]*	Total PCB [ng/Nm ³]**	HCB [ng/Nm ³]	O ₂ [volume %]
1	0,0240	0,0004	0,0244	2,935	12	19,5
2	1,2714	0,0481	1,3195	75,16	1491	13,1
3	0,0774	0,0043	0,0817	2,806	1,391	18,2
4	0,0101	0,0009	0,011	0,269	17,05	9
5	0,0025	0,0004	0,0029	12,74	3,691	19,2
6	0,0028	0,0035	0,0063	0,211	1,57	10,3
7	1,1757	0,1614	1,3371	947,8	140,2	18,8
8	0,0042	0,0025	0,0067	34,19	4,608	20,9
9	0,5809	0,0546	0,6355	0,417	17,51	20,7
10	0,0314	0,0034	0,0348	4,169	10,87	17,4
11	0,1259	0,0115	0,1374	10,09	12,25	20
12	0,1185	0,0086	0,1271	112,1	22,69	20,3
13	0,0175	0,0002	0,0177	1,257	4,415	8,8
14	0,006	0,0003	0,0063	36,15	7,814	20,9
15	0,0035	0,0019	0,0054	1208	7,822	19,8
16	0,0029	0,0002	0,0031	1,328	0,304	18
17	0,0177	0,0008	0,0185	5,311	1,941	17,9
18	0,0024	0,0011	0,0035	1,904	0,472	13,6
19	1,0539	0,0464	1,1003	278,8	613,1	17,4
20	0,0539	0,0272	0,0811	108,4	51,58	20,4

* - Values T-TEQ are calculated from the formula (3) as a sum of respective data from column II and III.

** - Total PCB was calculated as a sum of the masses of 7 I-PCB congeners as it is given in the Table 2

Measurements were carried-out during typical operational conditions, nominal capacity of the plant to obtain the most representative data for the process, which is realised in the installation.

However, in some cases (for example, steel converter plants – 16,17) measurements were realised in shorter time, because of safety reasons or specific operation conditions for those plants.

In the plant 7 the measuring was performed on the gas stream which was before the cleaning system. The reason was, because after cleaning the gas is released directly to the atmosphere, without any installation (stack, duct), where sampling port might be installed. There were no possibility to measure the real concentrations in the gas stream directed to the atmosphere, so, the real emission may only be estimated from the cleaning gas installation efficiency.

Also for plants 15 and 20 the secondary off-gases were taken. Mostly from technical limitations and personal safety measurements on the primary off-gas were not possible in this work.

The highest dioxins concentration was recorded for sintering of iron ore plants as well as for aluminium scrap melting. For casting of iron and steel (melting of iron and steel scraps) also in some cases (07, 08) relatively high concentration of dioxins was obtained. The reason is in most cases, that beside the metal melting process, waste sealing, paintings, coatings are also burning and fume gases are not completely after burned. The only exception in this case is the plant 10, where afterburning of gases from aluminium cans preheating and melting is realised.

In most of the measured plants neither thermal nor catalytic/sorption processes are used for dioxin formation decreasing. The main goal in those processes is to obtain final product and not to emit so high concentration of dust. Mostly, installations are equipped in electro filters or fabric filter bughouses. Only for copper and zinc industry where sulphuric acid

is produced, scrubbing towers are used for SO₂ removal and H₂SO₄ obtaining. Simultaneously, dioxins concentration is decreased because of their very low volatility in low temperatures and very effective dust removal in scrubbing process. The highest concentration of PCDDs, PCDFs and PCBs were found in iron ore sintering plant. Also in one case, for foundry for cast iron

(plant No: 7) relatively high concentrations of PCDD/F was found.

It should be emphasized that, however, the iron ore sintering plant is the most significant dioxins in stack gas concentration values (for operational O₂ value), the highest emission factor value is calculated for plants: 7, 9 and 12 - foundry of cast iron, secondary aluminium production - aluminium scrap melting - Electric Furnace and secondary aluminium production - aluminium scrap and cans melting.

It indicates that secondary aluminium production is the most significant dioxins source if calculated as an emission factor value, however, iron ore sintering plants are operated in much higher product mass efficiency, caused this process to be the most important for dioxins, PCB and HCB pollution to the atmosphere.

In **Table 6** the emission factors calculated from the measurements are presented.

Table 6: Emission factors estimated during the measurement program

Type of activity	Emission factor(s) µg I-TEQ/Mg of product
Iron and steel metallurgy plants	
Iron ore sintering plant	1.47; 1.10
Primary Iron production-Blast Furnace (BF)	0.01
Primary and secondary steel production-Basic Oxygen Furnace (BOF)	0.02
Iron casting-hot air cupola (good APCs)	0.06; 4.11
Iron casting-gas rotary kiln	0.02
Secondary steel production; steel scrap melting-electric arc furnaces	0.62; 0.02*
Steel casting-electric arc furnace	0.03
Non-ferrous metallurgy plants	
Primary copper production from concentrate with H ₂ SO ₄ production; copper slag recycling	0.005; 0.004; 0.002
Copper scrap melting	0.007
Secondary aluminium production; aluminium scrap and cans melting	8.65; 3.05; 1.69; 0.34
Primary zinc production	0.012
Zinc casting	0.02

* only secondary off gas was measured in the plants

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