Subject Area 6.3: Monitoring and fate of persistent chemicals

Research Article

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 emissions of PCDD/F, PCBs and HCB from 20 selected metallurgical installations in Poland, in order to update the national inventory of dioxin emission from metallurgical industry for developing a strategy for dioxins and furans emission abatement from the subject facilities (UNEP 2005).

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

Results and Discussion. Results from the work indicate that the highest dioxins and PCB concentrations were recorded for iron ore sintering plants at 1.10–1.32 ng total[1] TEQ/Nm^3 followed by aluminium scrap melting at 0.03–0.66 ng total TEQ/Nm^3. The highest HCB concentrations at 613–1491 ng/Nm^3 were also recorded for iron ore sintering plants, whereas at aluminium plants the HCB concentrations were in the range of only 10.1–22.7 ng/Nm^3.

Conclusions. The above investigations indicate that secondary aluminium production is the most significant dioxins source, if calculated as emission factor values. However, iron ore sintering plants are operating at much higher production capacity, causing this process to become the major source of dioxins, PCB and HCB pollution to the atmosphere in Poland.

Recommendations and Perspectives. Based on the performed tests and the environmental reviews of selected plants several recommendations were formulated for the reduction of generation or of emission of these pollutants 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; PCBs; PCDDs; PCDFs; POPs

Introduction

Until 2003 the emission inventory data for the metallurgical sector in Poland was estimated on the basis of default emission factors taken mostly from the UNEP toolkit (Quaß et al. 2000). Because the formation of dioxins, furans, PCB and HCB is process specific, in particular for thermal processes in metal industry (Buekens et al. 2001, Luthardt et al. 2001), 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 selected metallurgical installations in Poland in order to update the national inventory of dioxin emission from the metallurgical industry.

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

Regarding the selection of plants and installations an effort was made to include into investigations each of the different production processes concerned and to ensure that the emission standard of the tested installation was typical for the given industrial process. At the time of measurement the technical process had to be run under typical, nominal load and according to 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 coordinated by COWI A/S, Denmark. Measurements were made since May until September 2004.

1 Plant Selection and the Description of Plant Profile

The measurements were conducted in 20 metal industry installations in Poland as they are presented in **Table 1**, which

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[1] Total TEQ is calculated from equation 3 as a sum of I-TEQ (dioxins) and WHO-PCB-TEQ

Table 1: List of selected and examined installations

Installation examined and its allocated identity number	
01 Foundry of spheroidical and cast iron	11 Aluminium scrap melting-natural gas furnace
02 Iron ore sintering plant	12 Aluminium scrap and cans melting-electric furnace
03 Roasting ? of copper ore	13 Zinc ore roasting (sphalerite)-H_2SO_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 [b]
06 Copper smelting (production of sulphuric acid)	16 Production of primary iron in a blast furnace
07 Foundry of cast iron [a]	17 Steel production in a basic oxygen furnace
08 Foundry of cast steel – electric arc furnace	18 Grey and spheroidical 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 [b]

[a] Measurements were performed on raw gas. The sampling of cleaned gas was not possible for this plant due to technical reasons.

[b] 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. Due to technical reasons, the measurement of primary off-gas for these plants was not possible. Sampling ports were installed on the top of the stack of 6 m diameter and 30 m height.

were selected and accepted for this work by the Polish Ministry of Environment. (for confidential reasons, only sector and profile of the plant is given).

1.1 Steel and iron metallurgy

There are four different processes representing this sector:

Sintering of iron ore. This process is commonly known as generating high concentrations of dioxins and PCB. As can be seen in the literature 1–50 g of the I-TEQ (only regarded to dioxins) are released per plant annually into the air by the European countries. However, recently obtained data for Polish plants indicate, that the overall range of dioxin emission is between 2 and 8 g I-TEQ per plant annually.

Steel production: Blast furnace and converter process. In both processes, a relatively low dioxin concentration is measured previously, which may result from the presence of a high SO_2 concentration causing blockage of the chlorination of aromatic compounds. This phenomenon (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/m^3 in the stack gases from these processes.

Melting of iron and steel scraps. Preliminary heating up the iron and steel scrap takes place in the presence of plastics, coatings, paintings, and sealing, as well as nonferrous materials. This leads to a strong emission of pollutants, including dioxins and PCBs.

1.2 Copper metallurgy

Two basic processes are operated in the Polish copper metallurgy, as described below. In both cases the emission concentration values for dioxins, PCBs and HCB from these processes are unknown.

Roasting of copper ore leads to a high concentration of SO_2, used directly for sulphuric acid production. Because of extremely high SO_2 concentrations, reaching up to 250 g/m^3, the dioxins formation in this process is negligible. Moreover, the process of H_2SO_4 production requires multi-step scrubbing of process gases leading to a very effective removal of dioxins and PCBs traces. The investigations performed confirm this theory.

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

Reactions of dioxins formation have never been studied under such circumstances and only measurements may confirm the actual emission concentration values.

1.3 Aluminium metallurgy

In all cases (four installations) the thermal process was using different technologies for melting aluminium scrap. The latter is smelted using an electric furnace; a gas chamber furnace, a gas and oil fired rotary furnace and an electric furnace with preheating to remove plastics and decoration coatings (aluminium beverage tins).

1.4 Zinc metallurgy

Two processes from zinc metallurgy were subject of this examination:

1) Roasting zinc ore releases a high SO_2 concentration. Similarly to copper ore the process gases are also used for production of sulphuric acid. This sulphuric acid production reduces dioxins from process gases to trace values, below 0.1 ng I-TEQ/m^3, in addition achieved by multistage scrubbing operations.
2) Remelting zinc waste obtained from the production of zinc electrodes used in electrolytic processes. In this process, an additive of ammonium chloride (NH_4Cl) is applied, another potential source of dioxin formation due to its chlorine content.

2 Sampling and Analysis Methodology

Although there is an established EU standard for the determination of PCDD and PCDF concentration in stack gas from waste incineration (European Standard EN-1948, 1996), this standard is not actually valid for the determination of these compounds considered here in flue gases from metal industry. Moreover, the EN-1948 standard is not validated yet for the determination of PCB and HCB. Therefore, the 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 the filter/condenser method as described in European Standard EN-1948:1996 part 1. In this work large volumes of flue gas were collected in average volumes of 10 Nm^3 [2] samples because it was assumed that concentrations of PCDFs and PCDDs would be at a ng/m^3 or even pg/m^3 range. Samples of stack gas were taken using the EMIOTEST 9265 sampling apparatus (WrocBaw, Poland). The apparatus is represented schematically in **Fig. 1**. This instrument was specially designed for isokinetic sampling of industrial gas samples for the determination of trace organic compounds and for the determination of PCDD, PCDF, PCB and HCB. Sampling nozzles (1) and probe (2) were made of titanium (1). Fly ash (if present) was collected using glass fibre filters of 0.2 µm pore size placed in a heated compartment (3). For stack gas or off-gas with high humidity an aqueous condensate containing dissolved compounds is collected. Vapour phase PCDD, PCDF, PCB and HCB are adsorbed on a PU-foam plug placed in a cartridge (4). In this work the product No: 20038 obtained from 'Supelco', Bellefonte, USA was used.

Gas drying was performed with silica (5) and gas volume, humidity, temperature and pressure are recorded in a central control unit (6). A pulse damper (7) is used for stabilizing the operation of the sampling gas pump (8).

All compartments were spiked before sampling with appropriate ^{13}C sampling standards for sampling recovery con-

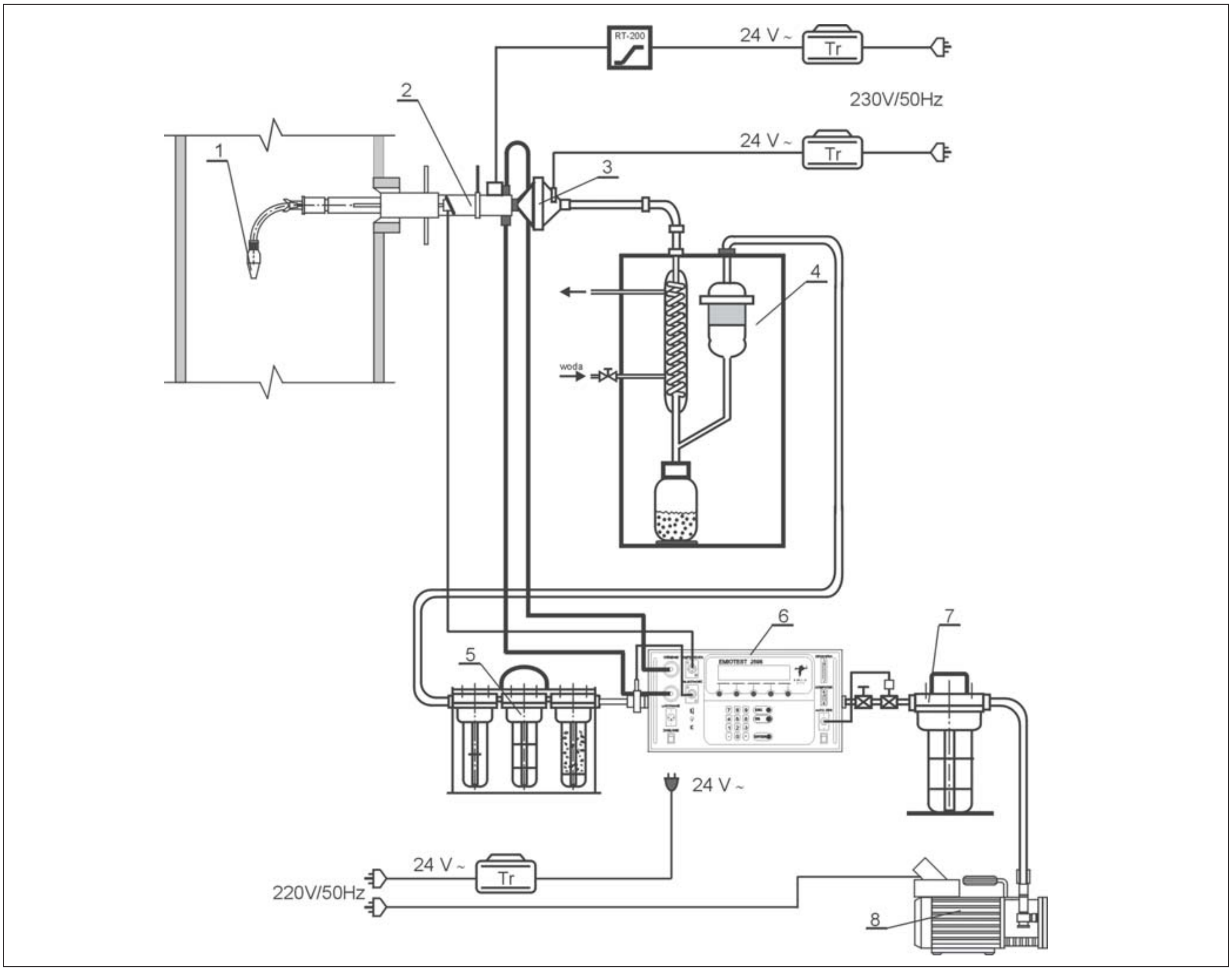

Fig. 1: Sampling apparatus used in the determination of PCDD, PCDF, PCB and HCB in stack and off-gases in metal industry in Poland in 2004

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[2] Nm^3 is one cubic meter of stack or off-gas in standard conditions (0°C, 0..1013 MPa, so called: 'Normal cubic meter')

trol, according to EN-1948 part 1. No ^{13}C-PCB or ^{13}C-HCB were used.

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

This procedure guarantees 90–95% collection efficiency of PCDDs, PCDFs, PCBs and HCB from flue gas. The limit of Detection (LD) for PCDFs/PCDDs, calculated for stack gas samples was 0.002–0.005 ng I-TEQ/Nm^3. The LD for HCB was in the range of 0.05–0.1 ng/Nm^3

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

2.2 Sample extraction, clean-up and analysis

Sample 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, regarding 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 (60 m, 0.25 mm) as routinely used, low polarity capillary column and DB-17 (30 m, 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, WHO-PCB congeners are presented that are used in the determining 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-TE 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 I-TEFi values (as recommended with EN–1948 EU standard)

PCDD i-congener	Value of the TEFi	PCDF i-congener	Value of the TEFi
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 TEFj values [1]

WHO-PCB j-congener	Value of the TEFj	WHO-PCB j-congener	Value of the TEFj
PCB77	0.0001	PCB126	0.1
PCB81	0.0001	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

[1] WHO-PCB TEFj were applicable mostly in food analysis so far

Calculation of the I-TEQ mass value was performed in accordance with the **Eq. 1:**

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

where:

m_i = 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 **Eq. 2**:

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

where:

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

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

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

4 Results and Discussion

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

Measurements were carried-out during typical operating conditions and at the nominal capacity of the plant, to obtain the most representative data for the process considered.

Table 5: PCDD/Fs, PCBs and HCB 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 O_2 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] [a]	Total PCB [ng/Nm^3] [b]	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

[a] T-TEQ values are calculated from the formula (3) as a sum of respective data from column II and III

[b] Total PCB were calculated as a sum of the masses of 7 I-PCB congeners as it is given in Table 2

Table 6: Emission factors estimated during the measurement program

Type of activity	Emission factor(s) µg I-TEQ/Mg of product This work	Emission factor(s) µg I-TEQ/Mg of product Previous data [a]
Iron and steel metallurgy plants		
Iron ore sintering plant	1.47; 1.10	5
Primary Iron production-Blast Furnace (BF)	0.01	0.03–0.13
Primary and secondary steel production-Basic Oxygen Furnace (BOF)	0.02	0.5–10
Iron casting-hot air cupola (good APCs)	0.06; 4.11	0.03–10
Iron casting-gas rotary kiln	0.02	4.3
Secondary steel production; steel scrap melting-electric arc furnaces	0.62; 0.02 [b]	5
Steel casting-electric arc furnace	0.03	1.0
Non-ferrous metallurgy plants		
Primary copper production from concentrate with H_2SO_4 production; copper slag recycling	0.005; 0.004; 0.002	0.01
Copper scrap melting	0.007	50
Secondary aluminium production; aluminium scrap and cans melting	8.65; 3.05; 1.69; 0.34	150
Primary zinc production	0.012	0.15–2.4
Zinc casting	0.02	No data available

[a] Inventory of Dioxin and Furan Releases (2002)
[b] Only secondary off gas was measured in the plants

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

In plant 7 the measurement was performed on the raw gas upstream of the cleaning system. Then the gas is released directly to the atmosphere, without any further installation (stack, duct), to possibly fix a sampling port. So in this case the real emission can only be estimated from the efficiency of the cleaning device.

Also for plants 15 and 20 the secondary off-gases were taken. Measurements on the primary off-gas were not possible mostly due to technical limitations and personal safety problems.

The highest dioxin content was recorded for iron ore sintering plants (01, 19) as well as for aluminium scrap melting (09–12). The average annually dioxin emission for iron ores sintering is in the range of 1.5–4 g I-TEQ (only for dioxins) per plant what is less, than obtained for the same plants in 2002 (Report 2005). For casting of iron and steel (melting of iron and steel scraps) relatively a high concentration of dioxins was obtained in some cases (07, 08) as well. The main reason is 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 aluminum cans preheating and melting is realized.

In most of the measured plants neither thermal nor catalytic/sorption processes are used for dioxin abatement.

Most installations are equipped with electro filters or fabric filter bag houses. Only for copper and zinc industry, where sulphuric acid is produced, scrubbing towers are used for SO_2 removal and H_2SO_4 production. Simultaneously, dioxins concentration is decreased because of the very low volatility at low temperature and an the very effective dust removal in the scrubbing process (UNEP 2005).

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

The highest emission factor value is calculated for plants: 7, 9 and 12 (foundries of cast iron, secondary aluminium production – aluminium scrap melting – Electric Furnace and secondary aluminium production – aluminium scrap and cans melting). Emission factors obtained for iron ore sintering indicate that the previous data (5 µg I-TEQ/t) has been overestimated. It also has been shown for primary iron (Blast Furnace), as well as for primary and secondary steel production.

That 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, hence this process is the major source to release dioxins, PCBs and HCB pollution to the environment.

The level of Dioxin emission from secondary aluminium production depends on the raw material composition and the applied APCS pls explain). The default emission factor proposed to apply previously was 150 µg I-TEQ/t of aluminium with an uncertainty range of 50–450 µg I-TEQ/t.

The Standardized Toolkit (2003) does not provide any default emission factors for aluminium casting. As pure ingots are used for the production, the emission factors are estimated to be considerably lower than the EF for secondary aluminium production. The toolkit applies an EF of 1 µg I-TEQ/t for zinc and zinc casting in furnaces without APCS and 0.3 for casting of zinc alloys. The data obtained from the European Dioxin Inventory (Quass 1997) applies emission factors for secondary zinc in the range of 0.15–2.4 µg I-TEQ/t.

As it can be seen for copper scrap melting and secondary aluminium production previously proposed data have been significantly overestimated.

5 Conclusion

The emission factors obtained in this work will be used for national emission inventory purposes instead of the factors proposed by Toolkit or taken from previous measurements. However, consideration should be given to the fact that the measurements in most cases are related to single installations. Therefore, the need for further development of national factors for the metallurgical industry in Poland is desired.

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Short CV:
Professorship from Krakow University of Technology – Chemistry Department in 2004

Scientific Specialisation:
Analytical chemistry, chromatographic analysis, organic trace analysis, GC-MS/MS analysis, PCBs, PCDDs/PCDFs, PBDE, waste disposal and thermal treatment methods, incineration and microwave waste material decomposition processes, PCDDs and PCDFs formation in combustion gases. Hospital and hazardous waste incineration processes.
Editor and Organiser of the annual national Polish conferences 'Dioxins in Industry and the Environment' (Dioksyny w przemysle i srodowisku) 1994–2005
Awarded from Allianz in Science in Poland with special Nike prize in 2000 – scientific work for Krakow
Awarded with Prof. Waksmundzki medal from Committee of Analytical Chemistry of Polish Academy of Sciences in 2001

Hobby: Entomology and macrophotography

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