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SOME METALS AND POLYCYCLIC AROMATIC HYDROCARBONS IN FUGITIVE PM10 EMISSIONS FROM THE COKING PROCESS

Coke oven batteries significantly contribute to fugitive emission of dust (particulate matter, PM) from the coking process. The dust contains harmful substances many of which such as heavy metals and polycyclic aromatic hydrocarbons (PAH), although mentioned in the Pollution Release and Transfer Registers (PRTR), so far have not been included into balances of emissions from hard coal coking. The goal of the presented work was to determine chemical composition of fugitive PM sampled on the battery roof. Three batteries in various technical conditions were examined for the PM emission. The results may provide information to help assessment of fugitive emissions from coking and for filling gaps in PRTR.

1. INTRODUCTION

The European Pollution Release and Transfer Registers (E-PRTR) are established by the UN-ECE Protocol on Pollutant Release and Transfer Registers to the Aarhus Conventionwhich was adopted by the European Community and 23 member countries in Kiev in May 2003. The basis for its establishing is the European PRTR Regulation 166/2006/EC (E-PRTR Regulation), in force since February 2006. E-PRTR replace the European Pollutant Emission Registers (EPER) – the basis for reporting on emissions in 2001 and 2004.

The E-PRTR Regulation is to facilitate and enhance public access to environmental information and decision-making by establishing a register (E-PRTR) of essential data on emissions from European industrial facilities. The register enables social control over environmental pollution – prevention and/or reduction of emissions – by making the environmental data available to general public.

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Yearly E-PRTR report on the sites where the threshold values of concentrations established for selected substances are exceeded. So far, the Polish practice (reports of the Central Statistic Office, fines for emission violation) has consisted in reporting on substances listed in law acts concerning payments for use of the natural environment and on substances whose permissible emission were determined by integrated permits [1].

Guidance Document for the Implementation of the European PRTR [2] comprises lists of characteristic substances emitted from production processes of key industrial branches and defines thresholds of their ambient concentrations exceeding of which is to be reported by the installation operator in the yearly report.

As far as coking is concerned, not only PRTR should report on the mass of emitted PM10 but also on masses of some substances comprised in PM10 such as polycyclic aromatic hydrocarbons (PAH)

- benzo(a)pyrene,
- benzo(b)fluoranthene,
- benzo(k)fluoranthene,
- indeno(1,2,3-cd)pyrene,
- naphthalene,

and metals:

- arsenic and its compounds (as As),
- cadmium and its compounds (as Cd),
- chromium and its compounds (as Cr),
- cuprum and its compounds (as Cu),
- mercury and its compounds (as Hg),
- nickel and its compounds (as Ni),
- lead and its compounds (as Pb),
- zinc and its compounds (as Zn).

PAH and metals in the dust emitted from the coking process come from the battery charge – a mixture of various sorts of coal. Emission rates of these substances, apart from the technical condition of a battery itself, depend on the chemical composition of the coal.

In recent years, because of intensive use of coal in energy production and industry, researchers focused on inorganic trace elements [3–5] and organic PAH [6–9] in coals and fly ashes. In general, the term "trace element" relates to a chemical element present in natural material at mass concentrations lower than 0.1% [10].

Geochemical investigations of coal and other fossil fuels for toxic trace elements have been intensified in recent years due to growing awareness of the adverse environmental effects of these elements and advancements in analytical methods [11]. Many of these elements may be found on various lists of potentially toxic substances. In the American Clean Air Act Amendment (1990), 189 potentially hazardous air pollutants (HAP) are listed out of which 15 are trace elements that probably originate

from the use of coal [15]: antimony (Sb), arsenic (As), beryllium (Be), cadmium (Cd), chlorine (Cl), chromium (Cr), cobalt (Co), fluoric (F), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), selenium (Se), thallium (Th) and uranium (U) [12, 13].

It is not known much about emission of metals from the coking process in Poland. This shortage of knowledge may equally well be attributed to both the deficiencies of the past legislation and lack of standardized methods for measurement of fugitive emission. At present, the only coking installations whose environmental documentation limits emission of some metals are the installations working within integrated coking plant-steelworks systems.

There are no unified standards for PAH either. Some operators of installations use the notion of emission of tar substances (determined gravimetrically), some use PAH meaning sixteen EPA compounds or nine PAHs from regulations concerning exposure to hazardous substances at the workplace. Benzo(a)pyrene and naphthalene are balanced for each Polish coking installation separately.

The contribution of the substances listed in PRTR to emission from point sources of a coking installation may be estimated from the results of routine (periodic) monitoring. The routine monitoring of fugitive emission from the coking process is not mandatory; it is not forced by integrated permits. Some methods exist for direct measuring of such emissions, e.g. the methods of virtual duct or casings. In practice, application of the method of casings to measurement of fugitive emission of some metals or PAH from coking, although possible, is time and money consuming.

A reasonable way for small installation to cope with the problem of PRTR and detailed emission balances is invention and application of simple methods for assessing emission rates.

The goal of the work was to collect the PRTR required data on the composition and chemical profile of fugitive PM10 emitted from the coking process.

PM10 has been selected for analysing due to two reasons. The former was the necessity of getting ride of coarser particles. The particles coarser than PM10 (breeze coke, mineral particles) in samples collected on the battery roof might be secondary emission from the ground or battery roof. The emission of particles and condensing vapours that escape through leaks of coking (heating) equipment was decided to be the concern of the work. The other reason was the interest in widening of the chemical profile of PM10. The data concerning dust would be scientifically appreciable if, as data on PM10, they were applicable in other investigations, e.g. modelling of PM10 immission with the use of receptor methods.

The work included collection of diurnal samples of PM10 on roofs of three coke oven batteries, the batteries differing in technical states, and determination of the chemical composition of sampled dust (PAH and metals) with the use of liquid chromatography and X-ray fluorescence.

Because coal and coke dusts from coal and coke handling within a cooking plant are also present in fugitive emissions, chemical analyses of coal mixtures charged into

ovens as well as produced coke and tar were analysed with the use of the methods mentioned above and applied in the PM analyses.

2. EXPERIMENTAL

Coking plants. The experiment was conducted in three coking plants, which belonged to the company Kombinat Koksochemiczny "Zabrze" S.A.:

- coking plant "Jadwiga" in Zabrze,
- coking plant "Dębieńsko" in Czerwionka-Leszczyny,
- coking plant "Radlin" in Radlin.

The coking plant "Jadwiga" in Zabrze is a single-battery coking plant, having a coke sorting plant and a wet quench tower with water curtain and cellular packing for cleaning and separation of vapours. The battery is a 54 oven Ja-64 battery, commissioned in 1969, reconstructed under hot conditions (successive cold reconstructions of particular chambers) in 2004/2005. The battery works in stamp charging technology, has two gas main collectors connected by the common suction main with a pressure regulator. The coke oven gas is treated conventionally in the by-product plant where volatile products of coking (tar, benzol) are recovered and the gas is cleaned.

The dimensions ot the oven chamber are: length – 14 070 mm, effective length – 13 210 mm, height – 3300 mm. The volume of charge is 19.20 m^3, and average coking time – 20 h 35 min. The coking plant produces domestic and blast-furnace coke.

The coking plant "Dębieńsko" in Czerwionka-Dębieńsko is also a single-battery coking plant. It has a D-82 top charged battery, re-started in January 1985 after a capital repair. The battery consists of two blocks of 28 twin flue coke ovens, underfired with rich coke oven gas, with individual cross-regenerators and two-side waste gas driving off system. The coking plant has a wet quenching tower and a coke sorting plant. Coke oven gas is cleaned in a conventional installation to recover volatile products of coal coking before using for the battery firing.

In the coking plant "Radlin" the sampler was located on the battery No. 1-bis. It is a new PWR battery (underfired with rich coke oven gas, with two-chamber cross-regenerators and twin heating flues) designed by GIPROKOKS. It consists of 86 ovens in two 43 oven blocks and is the only in Poland wide-chamber battery with stamp charging system – at present at the commissioning stage.

- The hot oven chamber dimensions are: length– 16 120 mm, height – 5070 mm, average width – 488 mm, volume – 35.8 m^3, volume of charge – 32.02 m^3, nominal coking time – 20 h 30 min.

The cokery's by-product plant is equipped with a catalytic ammonia cracking installation and Claus reactors for desulphurisation of coke oven gas.

Sampling and gravimetric analyses. Samples were taken with the use of an Atmoservice PNS sampler which, being capable of automatic filter changing, provides up to 15 days of maintenance-free sampling. The sampler is compliant with the European regulations concerning PM10 sampling[*]. PM10 weights were determined gravimetrically in accordance with the PN-EN 12341 norm. The samples for examinations for PAH were collected on glass filters, samples to be examined for elemental composition – on PTFE filters, both 47 mm Whatman's. The filters were conditioned in the weighing room at 20±1 °C of the relative humidity of 50±5% for 48 h before and after sampling (the weighing room conditions were monitored and recorded). They were weighed on a Mettler Toledo microscales (resolution 2 µm) and an electric charge neutralizer was used.

Five fourteen day measuring campaigns were performed – two in each of "Jadwiga" and "Radlin", and one in "Dębieńsko". PTFE and glass filters in the sampler magazines were arranged alternately. Every other filter was the PTFE one, later analyzed for metals, the rest were the glass ones to analyse for PAH.

Each day of each measuring campaign, the samples of coal mixture, coke, and tar, which later underwent the same analyses as PM10, were taken. They were averaged according to the KK Zabrze S.A. quality control procedures.

Method for PAH determination. The method for determination of PAH in dust involved extraction of the compounds from dust collected on a filter in cyclohexanone ($CH_2(CH_2)_4CO$), evaporation of the solvent, dissolving of the dry residue, and determination of PAH (anthracene, chrysene, benzo(a)anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenzo(a,h)anthracene, indeno(1,2,3-cd)pyrene, benzo(g,h,i)perylene) by the high performance liquid chromatography method.

A high performance liquid chromatograph from BISCHOFF Chromatography was used. The apparatus had an auto-sampler (automatically taking and injecting samples), two pumps, central processor, gradient mixer, fluorescence detector, degasser, and C-18 packed column (placed in a column thermostat) enabling separation of particular PAH from their mixture.

The investigations were done in the KK "Zabrze" S.A. laboratory, accordingly to the procedure accredited by PCA (Polskie Centrum Akredytacji, Polish Centre for Accreditation). In accordance with the procedure, to assure proper quality, injections of reference material at the concentrations of 0.02–0.04 µg/cm³ or 0.20–0.40 µg/cm³ were done for each series of the analysed samples.

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[*]EN 12341 norm: *Air quality. Determination of the PM 10 fraction of suspended particulate matter. Reference method and field test procedure to demonstrate reference equivalence of measurement methods.* Polish version: *PN-EN: Jakość powietrza. Oznaczanie frakcji PM_{10} pyłu zawieszonego. Metoda odniesienia i procedura badania terenowego do wykazania równoważności stosowanej metody pomiarowej z metodą odniesienia.*

Method for determination of selected metals. Dust was examined for 42 elements (Na-Pb, except Zr, Nb, Tc, Ru, In, Hf, Ta, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, Re, Os, Ir, Pt, Hg) with the use of energy dispersive X-ray fluorescence (EDXRF). A PANalytical Epsilon 5 spectrometer was used. The Epsilon 5 had a water cooled X-ray tube working in the range of 25–100 kV, gadolinium anode, beryllium 150 µm thick side window, a system of nine secondary targets (Al, Ti, Fe, Ge, Zr, Mo, Ag, Ce_2O_3, Al_2O_3). It had a Ge(Li) detector with the resolution of 140 eV, 30 mm^2 active area, 8 µm thick beryllium window, and worked in the energy range 0.7–100 keV.

Determinations were done in vacuum, the analysis lasted for 4800 s (whole runtime of the analytical program with secondary targets: Al, Ti, Fe, Ge, Zr and Al_2O_3). Concentrations of particular elements were computed with the use of the Epsilon 5 software. They were determined by comparing the ovtained results with calibration curves. The curves were determined by analyzing the Micromatter Inc. thin layer standards. To maintain the quality of determinations, daily calibration of energy (automatic, performed by the apparatus), weekly measurements of the NIST 2873 standard (recovery within 80–120% of the certified value), monthly monitor measurements (observation of X-ray tube efficiency and sensitivity of detector), and drift correction were performed. The detection limits of the procedure were determined by measuring two blinds (PTFE filters) 30 times (each) in the same as for the samples conditions. The detection limits were computed as the standard deviations (σ) of the series.

3. RESULTS AND DISCUSSION

Ranges of concentrations or percentages of the investigated substances in dust emitted from the coke oven batteries are presented in the paper. In Tables 1–4, percentages of tar substances and PAH in dust, tar, coal and coke sampled at the three coke oven batteries are presented.

Coal mixtures contained 1.59 wt. % of tar substances on average – similarly for all cokeries. Percentage of total PAH in the mixtures was from 0.02 to 0.03%. Among all examined PAH, anthracene concentrations in samples of the coal mixtures were the most scattered – 0.13 to 1.23%, depending on a coking plant (Table 2).

Average mass contributions of tar substances and PAH to coke were also similar in all coking plants. Percentage of naphtalene in total PAH varied considerably between 2.62 and 13.84 (Table 3).

No significant differences were visible in contents of total tar in PAHs from the coking plants (Table 4). The highest average contribution of total PAH to tar was found in 'Jadwiga.' The differences in the chemical composition of coke and tar be-

tween cokeries might be due to differences in technical condition of the installations, and consequently, to the level of degasification of coal in the coking process.

Average percentage of tar substances and PAH in sampled PM10

Species	"Jadwiga"	"Radlin"	"Dębieńsko"	Average
Tar substances in a sample	18.24	12.24	3.99	11.49
PAH in a sample	2.98	2.77	0.64	2.13
PAH in tar substances.	16.34	22.61	15.93	18.29
Naphthalene in PAH*	0.85	3.11	0.51	1.49
Anthracene in PAH*	0.53	0.06	0.95	0.51
Benzo(b)fluoranthene in PAH	9.35	6.63	3.73	6.57
Benzo(k)fluoranthene in PAH	4.47	3.18	2.34	3.33
Benzo(a)pyrene in PAH	8.45	5.49	3.89	5.94
Indeno(123cd)pyrene in PAH	5.78	2.96	3.03	3.92
Benzo(b)fluoranthene in a sample	0.28	0.18	0.02	0.16
Benzo(k)fluoranthene in a sample	0.13	0.09	0.01	0.08
Benzo(a)pyrene in a sample	0.25	0.15	0.02	0.14
Indeno(123cd)pyrene in a sample	0.17	0.08	0.02	0.09

*Result burdened with a considerable error due to the method of sampling.

Average percentage of tar substances and PAH in sampled coal mixtures

Species	"Jadwiga"	"Radlin"	"Dębieńsko"	Average
Tar substances in a sample	1.65	1.57	1.56	1.59
PAH in a sample	0.03	0.02	0.03	0.03
PAH in tar substances	2.08	1.23	1.84	1.72
Naphthalene in PAH*	4.05	10.04	5.19	6.43
Anthracene in PAH*	1.29	0.13	0.80	0.74
Benzo(b)fluoranthene in PAH	3.85	6.75	2.98	4.53
Benzo(k)fluoranthene in PAH	2.10	3.73	1.70	2.51
Benzo(a)pyrene in PAH	3.85	2.27	3.54	3.22
Indeno(123cd)pyrene in PAH	1.58	3.02	2.60	2.40

*Result burdened with a considerable error due to the method of sampling.

The highest content of tar substances in dust was found on the battery roof in "Jadwiga", the lowest – in "Dębieńsko". In "Dębieńsko" it was lower than in other cokeries. Average weight contribution of PAH to tar substances in PM10 was similar for all cokeries and equal to 18.29% onin average (Table 1).

Table 3

Average percentage of tar substances and PAH in sampled coke

Species	"Jadwiga"	"Radlin"	"Dębieńsko"	Average
Tar substances in a sample	1.18	0.81	1.18	1.05
PAH in a sample7	0.01	0.01	0.01	0.01
PAH in tar substances.	0.16	1.38	0.75	0.76
Naphthalene in PAH*	13.84	2.62	3.14	6.53
Anthracene in PAH*	2.17	1.07	0.62	1.29
Benzo(b)fluoranthene in PAH	2.93	7.81	3.10	4.61
Benzo(k)fluoranthene in PAH	1.42	4.02	2.02	2.49
Benzo(a)pyrene in PAH	0.85	5.16	1.42	2.48
Indeno(123cd)pyrene in PAH	2.31	4.10	1.17	2.53

*Result burdened with a considerable error due to the method of sampling.

Table 4

Average percentage of tar substances and PAH in sampled tar

Species	"Jadwiga"	"Radlin"	"Dębieńsko"	Average
Tar substances in a sample	39.45	37.20	41.51	39.39
PAH in a sample	17.42	14.12	14.41	15.31
PAH in tar substances.	44.15	37.95	34.72	38.94
Naphthalene in PAH*	3.15	4.91	3.36	3.81
Anthracene in PAH*	5.00	5.16	5.13	5.10
Benzo(b)fluoranthene in PAH	2.63	2.88	2.99	2.83
Benzo(k)fluoranthene in PAH	1.53	1.63	1.59	1.58
Benzo(a)pyrene in PAH	3.13	3.36	3.24	3.24
Indeno(123cd)pyrene in PAH	1.50	1.27	1.71	1.49

*Result burdened with a considerable error due to the method of sampling.

The convergence of the PAH profiles in "Radlin" and "Jadwiga" in suspended dust may be attributed to the technology – both cokeries work in a stamped charging system. Lower content of tar substance in dust in "Dębieńsko" may be due to the coking plant top type charging system and only one gas receiver, which limit potential points of leakages. Moreover, the emission from this kind of charging is negligible while fugitive emission from the stamped charging, where the coal cake is introduced into an open chamber, is considerable despite hydroinjection applied. In total PM10 related PAH, in all cokeries, benzo(b)fluoranthene (6.57% on average) and benzo(a)pyrene (5.94% on average) prevailed. The results concerning naphthalene and anthracene are burdened with a considerable error due to their physicochemical prop-

erties (new method for their sampling should be developed). Results of examinations for heavy metal content of coal, coke, tar and dust are presented in Figs. 1–4.
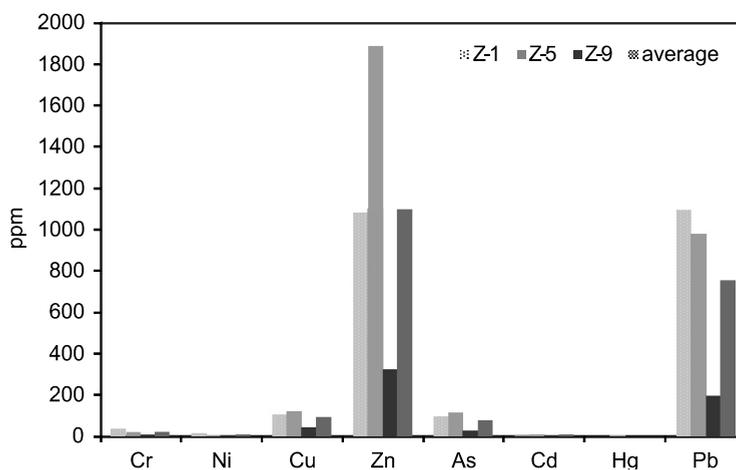

Fig. 1. Average metal content in samples of dust from
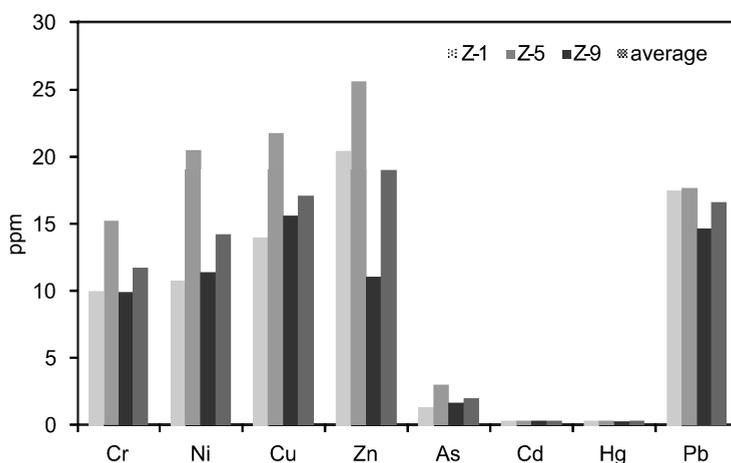"Jadwiga" (Z-1), "Radlin" (Z-5), and "Dębieńsko" (Z-9)

Fig. 2. Average metal content in samples of coal from
"Jadwiga" (Z-1), "Radlin" (Z-5), and "Dębieńsko" (Z-9)

The ranges of mass concentration (contribution to mass) of 42 elements in PM10 are presented in Table 5. Contents of selected metals in coal were comparable for all three installations although in "Radlin" Cr, Ni, Cu, As and Zn contents were higher (Fig. 2). The metal contents in coke were similar as in the coals under investigation (Fig. 3). Compared to other metals, the percentages of Zn and Pb in tar were elevated. Percentages of Cr, Ni, Cu, As, Cd, and Hg in tar were very low (Fig. 4). The elemental

profile of dust was similar to that of tar except for Zn and Pb whose percentages in dust were much higher (Fig. 1).

Results of the elemental analyses of dust from the battery roofs in the three cokeries and the data presented in Table 1, which confirm high contribution of tar substances to PM10, proved that PM10 is very rich in the elements that easily migrate to tar (in a gas collecting main, during condensation in normal conditions).
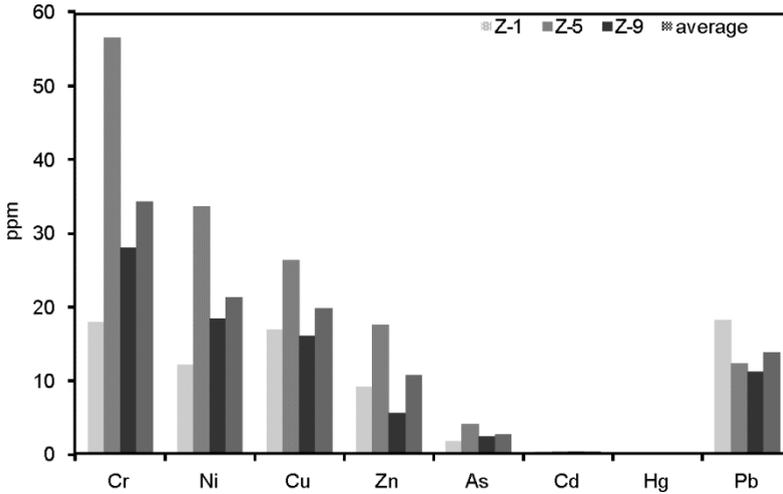

Fig. 3. Average metal content in samples of coke from
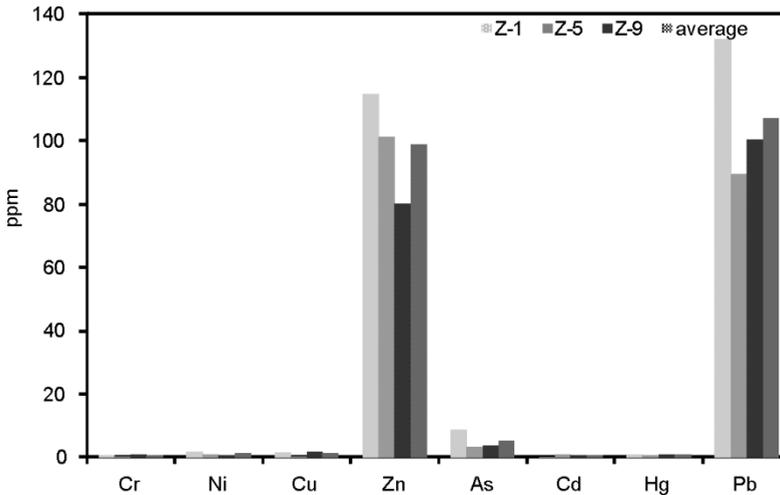"Jadwiga" (Z-1), "Radlin" (Z-5), and "Dębieńsko" (Z-9)

Fig. 4. Average metal content of samples of tar
from "Jadwiga" (Z-1), "Radlin"(Z-5), and "Dębieńsko" (Z-9)

T a b l e 5

Ranges of concentrations [ppm] of particular elements in dust emitted from coking

Element	"Jadwiga"		"Dębieńsko"		"Radlin"	
	Maximum	Minimum	Maximum	Minimum	Maximum	Minimum
Na	12546.13	2150.14	3365.00	801.00	15128.75	47.07
Mg	6250.13	1496.92	2872.00	1898.00	6266.07	216.82
Al	22919.21	3180.24	17466.00	1538.00	28747.15	1934.80
Si	34968.08	3675.11	24182.00	2668.00	31903.26	1905.44
P	1671.37	393.74	900.00	529.00	5053.62	398.29
S	26771.45	16305.26	12966.00	7375.00	32034.89	2200.45
Cl	58942.48	4531.84	31849.00	1382.00	76145.40	713.52
K	5829.42	1943.04	2775.00	1077.00	9780.05	494.38
Ca	10638.42	2482.20	4472.00	774.00	9515.16	500.19
Sc	0.00	0.00	12.00	12.00	0.00	0.00
Ti	855.97	158.88	472.00	64.00	835.74	39.53
V	21.08	6.67	3.00	3.00	0.00	0.00
Cr	170.34	5.11	15.00	2.00	60.32	3.49
Mn	949.97	150.55	267.00	89.00	1055.56	36.04
Fe	18192.42	4537.24	7592.00	2013.00	16662.84	932.96
Co	10.42	10.42	0.00	0.00	0.00	0.00
Ni	23.16	7.81	4.00	4.00	0.29	0.29
Cu	196.55	49.81	94.00	22.24	346.83	15.99
Zn	1555.98	375.51	562.00	202.00	3483.36	126.72
Ga	0.00	0.00	0.00	0.00	0.00	0.00
Ge	11.67	11.67	9.20	9.00	0.00	0.00
As	289.51	7.66	94.00	10.00	294.48	1.74
Se	26.05	2.11	11.00	11.00	0.00	0.00
Br	507.90	80.47	269.00	23.01	512.54	10.19
Rb	26.21	5.11	13.00	3.00	30.16	0.00
Sr	108.10	41.67	42.00	7.00	95.81	0.00
Y	34.74	34.74	0.00	0.00	0.00	0.00
Mo	81.06	8.71	13.00	13.00	120.64	1.16
Rh	104.22	3.83	8.00	3.00	150.79	14.33
Pd	11.58	11.58	2.00	2.00	135.72	0.87
Ag	57.90	1.28	6.13	4.00	150.79	3.31
Cd	62.24	27.86	12.00	1.00	316.67	0.29
Sn	29.48	2.55	20.00	12.00	256.35	5.23
Sb	32.76	1.67	41.00	7.00	153.50	5.47
Te	0.00	0.00	7.67	6.00	49.12	2.74
I	216.18	22.63	40.00	8.00	96.20	1.49
Cs	0.00	0.00	6.00	6.00	105.56	0.00
Ba	215.05	11.58	109.00	6.00	364.06	22.09
La	0.00	0.00	0.00	0.00	0.00	0.00
W	0.00	0.00	0.00	0.00	0.00	0.00
Au	1.28	1.28	0.00	0.00	0.00	0.00
Pb	1922.35	123.89	578.00	59.82	2431.50	40.40

Like for naphthalene and anthracene, the Hg content in dust is underestimated due to the lack of capacity to assess the Hg contents in gas phase. Due to high temperature of the coke oven battery, substances such as naphthalene, anthracene and mercury escape as gases in fugitive emission. Besides the metals obligatorily reported in PRTR (Fig. 1), also the contents of Na, Mg, Al, Si, Cl, Fe, and S h in PM10 from the battery roofs were high (Table 5). The elemental profile of PM10 was similar in all three cokeries.

4. CONCLUSIONS

• Seventy three 24 h samples of dust were taken on roofs of three coke oven batteries and analysed for chemical composition (elemental composition and selected PAH) and contents of tar substances. Samples of coal and products of coking were taken simultaneously.

• For each installation, average percentages of particular substances in dust, coal, coke, and tar were determined. The standard health and safety at work (HSW) procedures for dust examinations used in industry and some routine measurements were applied.

• Necessity of PRTR for fugitive emissions of the substances under examination may be verified by using the usual suspended dust emission factors and the average percentages of these substances in dust. The averaged results from the three coking plants may be used as the average values for the substances. It is justified by the fact of application of the factor methods to assess emission rates of metals and PAH, where the factors are unified for each of industry branches, in the European countries and the USA.

• Measurements of dust concentrations on a battery roof and balancing of elements in input coal and products together with utilisation of data on mass contributions of selected substances to dust yield results more adequate than balancing of raw material and products because of relatively simple methods of metering and small number of analytical stages.

• Results concerning naphthalene and mercury were highly uncertain.

To receive more credible results for naphthalene, anthracene, and mercury, further investigations are needed. It is necessary to develop a method for sampling that would enable determinations of as well gas as solid phase of these substances. It would require development of a sampling head capable of simultaneous sampling of dust with these substances and their gaseous forms.

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