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Migration of Cadmium and Antimony from Zinc Oxide Emitted from a Sintering Machine in Zn and Pb Pyrometallurgical Process into Environment

*Zdzislaw Adamczyk, Katarzyna Nowińska**
Silesian University of Technology, Gliwice, Poland
**corresponding author's e-mail: katarzyna.nowinska@polsl.pl*

1. Introduction

The Miasteczko Slaskie Zinc Smelting Plant is the only zinc and lead manufacturer in Europe that uses the ISP (Imperial Smelting Process) pyrometallurgical process. The basic production departments of the smelting plant include Sinter Unit and Shaft Furnace (Zhao 2013, Guo et al. 2002, Habashi 1986).

A number of wastes are generated in the manufacturing process, such as: dusts, sludges, dross and slag.

Due to the diversity of the feed materials, i.e. mixture of zinc and lead concentrates with materials recycled from the process, these wastes differ widely in chemical and mineral composition (Gregurek et al. 2015, Rao 2006).

It is estimated that approximately 48 Mg of dust are emitted annually from the process (Adamczyk et al. 2010). The dust contains many toxic elements that may have an adverse environmental impact (Kitamura and Kubota 2000).

The subject of the study were the dusts emitted from the Sinter Unit because of the accompanying elements, including antimony and cadmium, that are present in Zn and Pb ores. The dust separation efficiency of filters at the Sinter Unit is very high at 99.99%, which strongly reduces dust emissions into the air. This, however, is not enough to completely eliminate Sb and Cd emissions with these dusts. These emissions are as follows: Cd – 0.0045 Mg/year, Sb – 0.0039 Mg/year.

Studies of the possibility of migration of chemical elements contained in the dust emitted to the soil and water environment by the Miasteczko Slaskie Zinc Smelting Plant forms a basis for determining the mechanisms of pollutant transformations in that environment.

Based on the data on the mean chemical composition of the dust from the Sintering Machine and on the results of dust phase composition determination

(Adamczyk et al. 2010, Nowińska et al. 2015) the fraction of zinc oxide among all other phase components (zinc sulphide, lead sulphide, lead sulphate, iron oxide and lead oxide) is 4.25 wt. %.

Zinc oxide grains are not uniform in terms of phase composition - they contain inclusions of other phases (Ruetten 2009). The latter include PbO and FeO which, on the average, constitute 0.54 wt. % and 1.95 wt. %, respectively, of the zinc oxide (Adamczyk and Nowińska 2015) Zinc oxide grains contain admixtures of trace elements, such as: Si, Al, Mn, Mg, Ca, K, Ag, As, Cd, Cu, Se, Sb and Sn. These elements most probably constitute substituents of the main elements in grains composed of ZnO, PbO and FeO. The total content of these elements is 1.629 wt. %. on the average (Adamczyk et al. 2015). These elements may form admixtures both in zinc oxide, as well as in the phases that constitute inclusions therein. Therefore, when considering the possibility of migration of these elements into the environment after the decomposition of zinc oxide, the content of these elements in inclusions, i.e. in PbO and FeO, should be indicated (Borch et al. 2010, Barakat 2003, Cabała et al. 2013, Voliante 2010).

The aim of the study was to determine the possibility of migration of Cd and Sb released from zinc oxide emitted by the Sintering Machine to the environment.

Both cadmium and antimony are toxic. Toxicity of cadmium comes from its high affinity with many chemical groups which are biologically important. This element forms covalent and ionic bonds with oxide, hydrogen and sulphur atoms, which create a number of compounds in plant and animal cells (Kaczyńska et al. 2015).

Antimony has high biogeochemical affinity with arsenic, wherein the antimony compounds are less toxic. Toxicity of Sb increases with decreasing of its oxidation state, toxicity of Sb (III) is tenfold higher than of Sb (V).
However, it needs to be emphasised that toxicity of elements depends on many parameters such as the considered organism, the route of exposure, or the presents of other contaminants (Diatta et al. 2008, Filella et al. 2009, Nadgórska-Socha et al. 2013, Niedzielski et al. 2000).

2. Methods

The estimation of the load of Cd and Sb introduced into ground waters as a consequence of zinc oxide decomposition was made for the environmentally worst case scenario: all of the zinc oxide from dust fallout from the Sintering Machine into the soil and water environment is decomposed. Two averaged dust fallout from samples collected over six months were used as objects of the study.

For these estimates to be correct additional considerations must be taken into account. The annual dust emission from the smelting plant is 48 Mg, of that

3.36 Mg is emitted by the Sintering Machine. The average annual dust fallout calculated on the basis of 25 measurement points located around the Smelting Plant is 59 g/(m^2 · year), of that 4.13 g/(m^2 · year) is attributed to the Sintering Machine (Melaniuk-Wolny 2001).

Chemical composition of dusts was determined by means of roentgen microanalyser JCXA 733 by Jeol equipped with energy-dispersive spectrometer ISIS 300 by Oxford Instruments. The focal beam of diameter range equal to 1-2 μm and accelerating voltage equal to 20kV were applied during the analysis. Fine-phase samples were additionally analysed with the use of the focal beam of diameter equal to ca. 50 μm, corresponding to maximum scanned area of 50×50 μm.

The chemical composition of dust was estimated as an average of results obtained from ten measurements of 9 microzones laid out for every sample.

The mobility of Cd and Sb present in zinc oxide was determined using geochemical modelling based on Eh-pH diagrams using HSC Chemistry 9.6 software. These diagrams were plotted for conditions characteristic of the soil and water environment of the Smelting Plant area:

- average precipitation – 700 mm/year,
- average temperature in winter – -5°C,
- maximum temperature in summer – + 25°C.

Studies were performed within the water stability region, within Eh range of -0.8-+1.4V and pH range of 4-8 (pH of soils in areas adjacent to the Smelting Plant is within 4.5-7.5). The diagrams show the stability regions of the forms of occurrence of trace elements contained in zinc oxide at extreme seasonal temperatures, i.e. -5° and +25°C.

To plot the diagrams it was necessary to make assumptions on the concentration of the elements in question in the soil and water environment. These concentrations were assumed to be equal to permissible limits specified in the Ordinance of the Minister of Environment of 24 July 2006 on the conditions of discharging wastewater to waters and soil and on substances particularly harmful to aqueous environment (Dz.U. 2006, No. 137, item 984): 2 mg Zn/dm^3, 0.2 mg Cd/dm^3, 0.3 mg Sb/dm^3. As CO_2 occurs naturally in water, it was also taken into account when plotting the diagrams, and the concentration thereof (20 mg CO_2/dm^3) was also assumed to be as specified in the Ordinance mentioned above.

3. Results

In order to determine the migration of elements into the environment, only two of them were considered, namely Sb and Cd. Discussion of all potential migrating elements would require a much broader study. In addition, these two elements can pose a hazard to the environment due to their toxicity. The content of these elements in zinc oxide grains was 0.1302 wt. % Sb and 0.1460 wt. % Cd (Adamczyk and Nowińska 2015).

Taking the average content of PbO and FeO in zinc oxide grains, the weighted average for Sb and Cd content in the phases of the zinc oxide grains is as follows:

- Cd – 0.1459 wt. % w ZnO, 0.00005 wt. % in PbO and 0.00005 wt. % in FeO,
- Sb – 0.1300 wt. % in ZnO, 0.0001 wt. % in PbO and 0.0001 wt. % in FeO.

The amount of zinc oxide in the dust emitted from the Sintering Machine, under the assumptions made, is 0.1429 Mg, which corresponds to 0.1756 $g/(m^2 \cdot year)$ in the dust outfall (Table 1). Along with zinc oxide, trace elements are discharged into the environment, among them Cd and Sb, in amounts of 0.0049 Mg/year and 0.0044 Mg/year, respectively, and 0.0060 $g/(m^2 \cdot year)$ Cd and 0.0054 $g/(m^2 \cdot year)$ Sb in fallen dust.

The worst case scenario may be that when all of the zinc oxide in fallen dust (0.1756 $g/(m^2 \times year)$) undergoes decomposition in the soil and water environment. In such case all trace elements bound into that phase will migrate into that environment. Under such assumptions, the maximum concentration of zinc in ground water may reach 0.20 mg Zn/dm^3 (with annual precipitation equal to 700 mm). Under these conditions, the concentrations of cadmium and antimony in ground water will reach 0.009 mg Cd/dm^3 and 0.008 mg Sb/dm^3, respectively.

The plotted Eh and pH diagrams indicate that in the pH range of 11.6 to 8.0 ($\Delta pH = 3.6$) zinc oxide will decompose in the soil and water environment at -5°C into $Zn_5(OH)_6(CO_3)_2$, and at pH < 8.0 it will decompose into Zn^{2+} and HCO_3^- (Fig. 1). This decomposition will occur irrespective of the Eh value in the environment.

With increasing temperature the stability region of zinc oxide is reduced. At 25°C the pH range is 9.6-7.2 ($\Delta pH = 2.4$), thus Zn^{2+} and HCO_3^- are formed as a result of $Zn_5(OH)_6(CO_3)_2$ decomposition at pH < 7.2. Also in this case the decomposition occurs within the entire range of Eh water stability values. It is worth noting that $Zn_5(OH)_6(CO_3)_2$ is a carbonate mineral, called hydrozincite from zones of zinc and lead ores oxidation (Cabała 2009). Another constituent forming as a result of zinc oxide decomposition may be $ZnCO_3$ (smitsonite), which is one of the products of Zn ores weathering – smithsonite, like hydrozincite. Its presence, however, is in fact largely reduced, as conditions of its formation require specific temperatures (ca. 10°C) and relatively high pH values (11.0-11.5).

Table 1. Content of Zn, Cd and Sb in dust emissions and fallen dust from the stack of the Sintering Machine (E3) with zinc oxide amount taken into account

Emission (Mg/year)	Amount of ZnO in dust emissions from E3	0.1429
	Zn fraction in emissions from E3	0.1146
	Fraction of Cd contained in ZnO in emissions from E3	0.0049
	Fraction of Sb contained in ZnO in emissions from E3	0.0044
Dust fall ($g/(m^2 \cdot year)$)	Amount of ZnO in fallen dust from E3	0.1756
	Fraction of Zn in fallen dust from E3	0.1409
	Fraction of Cd contained in ZnO in fallen dust from E3	0.0060
	Fraction of Sb contained in ZnO in fallen dust from E3	0.0054

The Pourbaix stability diagrams suggest that the conditions prevailing in the soil and water environment of the smelting plant area will favour complete decomposition of ZnO to Zn^{2+} and HCO_3^- ions.

Under such Eh/pH conditions Cd and Sb will be released into the environment during zinc oxide decomposition. These elements will take on the following forms (Figs. 2 and 3):

- Cd – ionic: Cd^{2+}, solid: $CdCO_3$ – ottavite.
- Sb – hydrolytic: $Sb(OH)_6$, $Sb(OH)_3^0(aq)$, HSb_2S_4, solid: Sb_2S_3 – stibnite, Sb_2O_4.

irrespective of seasonal temperature changes (-5...+25°C).

Within the pH range characteristic of the soil and water environment in the area covered by the study (pH = 4.5-7.5), only the following ions will be present: Cd^{2+}, $Sb(OH)_6^-$ and $HSb_2S_4^-$ migrating along with ground waters.

Analysis of Eh-pH diagrams shows that Eh value has a major impact on occurrence forms of Sb in hypergenic environment in the area covered by the study. Forms of antimony in hypergenic environment are stable throughout the pH = 4.5-7.5 range.

Characteristic form of the presence of antimony under reducing conditions (with the limit values $Eh_L = -0.05V$ and $-0.02V$) is $HSb_2S_4^-$, where Sb is on +3 oxidation degree. Above the Eh limit value, for the corresponding pH values (Fig. 3), $HSb_2S_4^-$ is hydrolyzed according to reaction:

$$HSb^{(+3)}{}_2S_4^- + 3H_2O \rightarrow 2Sb^{(+3)}(OH)_3^0(aq) + 4S^{2-} + H^+ \qquad (1)$$

Under oxidizing conditions (Eh_L = 0,15V and 0.35V) (Fig. 3) at a concentration of antimony 10^{-10}mol/dm^3, Sb (III) is oxidized according to reaction:

$$Sb(+3)(OH)_3^0\ (aq) + 1/2O_2 + 2H_2O \rightarrow Sb^{(+5)}(OH)_6^- + H^+ \quad (2)$$

While at a concentration of antimony 10^{-7} mol/dm^3, with the increase of Eh value, the following reaction will take place:

$$HSb^{(3+)}{}_2S_4^- + H_2O \rightarrow Sb^{(3+)}{}_2S_3 + S^{2-} + H^+ + H_2O$$

$$(Eh_L = -0.015V \text{ and } 0.05V) \quad (3)$$

$$Sb^{(3+)}{}_2S_3 + 6H_2O \rightarrow 2Sb^{(3+)}(OH)_3^0\ (aq) + 3H_2S$$

$$(Eh_L = -0.014V \text{ and } 0.06V) \quad (4)$$

$$2Sb^{(+3)}(OH)_3^0\ (aq) + 1/2\ O_2 \rightarrow Sb^{(3+)}\,Sb^{(5+)}\,O_4 + 3H_2O$$

$$(Eh_L = -0.02V \text{ and } 0.035V) \quad (5)$$

$$Sb^{(3+)}\,Sb^{(5+)}\,O_4 + 1/2O_2 + 8H_2O \rightarrow 2Sb^{(+5)}(OH)_6^- + 2H^+ + H_2O$$

$$(Eh_L = -0.014 \text{ and } 0.06) \quad (6)$$

Among the solid forms in hypergenic environment of the research area Sb will be represented by stibnite and Sb_2O_4. However, the research results show (Krupka and Serne 2002) that stability of Sb ions, and mainly occurrence of stable forms of Sb, depend on concentration of this element in the ground water (Fig. 3). At low concentration of Sb (< 10^{-10} molSb/dm^3) throughout the Eh and pH range antimony occurs only in hydrolytic forms, and its solid forms are not observed.

With increasing concentration of Sb (10^{-9} molSb/dm^3) next to hydrolytic forms, the solid forms of Sb occur, and further increase in the concentration of antimony (10^{-7} molSb/dm^3) causes the increase of the area of its stability fields (Fig. 3).

The attention should be paid to the fact, that under the environmental conditions the maximum concentration of Sb specified for research area (0.0054 g/(m^2 · year) correspond to the value of $6.30 \cdot 10^{-8}$ molSb/dm^3, while according to the Ordinance (Ordinance of the Minister of Environment of 24 July 2006 on the conditions of discharging wastewater to waters and soil and on substances particularly harmful to aqueous environment (Dz.U. 2006, No. 137, item 984)) the maximum concentration of Sb corresponds to the value of $2.47 \cdot 10^{-6}$ mol Sb/dm^3.

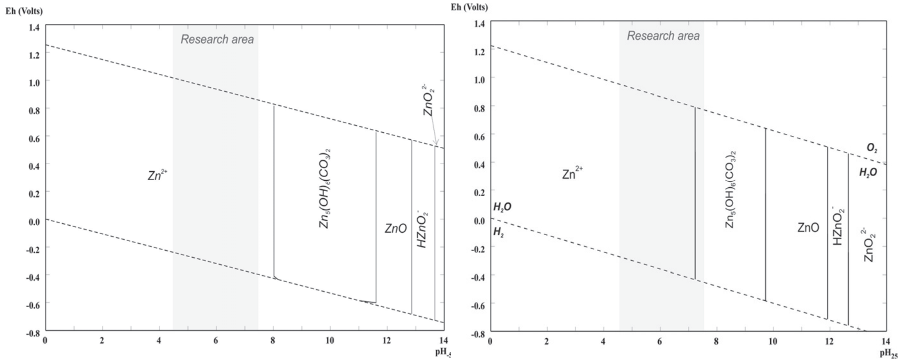

Fig. 1. Mobility of Zn from decomposition of zinc oxide emitted by the Sintering Machine (emission point E3) to the soil and water environment at -5 and 25°C

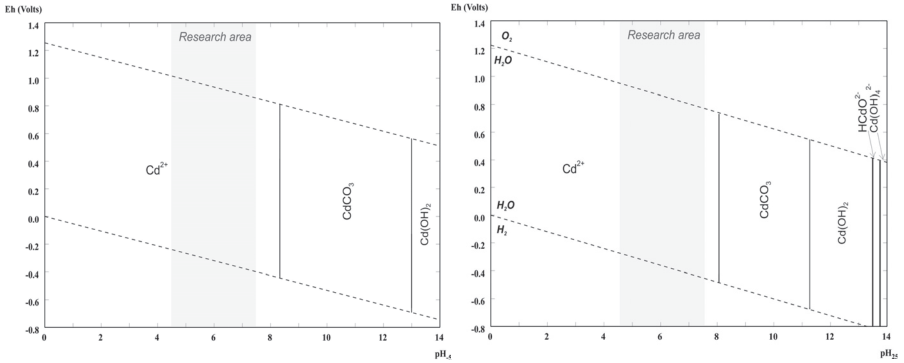

Fig. 2. Mobility of Cd from decomposition of zinc oxide emitted by the Sintering Machine (emission point E3) to the soil and water environment at -5 and 25°C

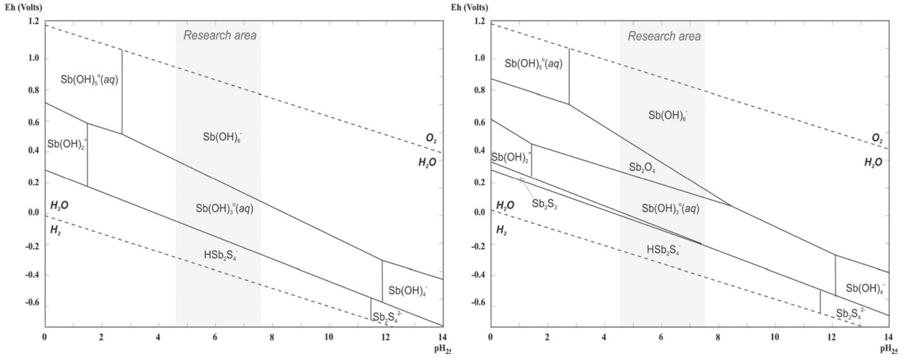

Fig. 3. Mobility of Sb from decomposition of zinc oxide emitted by the Sintering Machine (emission point E3) to the soil and water environment at 25°C

4. Conclusions

Zinc oxide is one of the main phases present in the dust discharged from the stack of the Sintering Machine at the Miasteczko Slaskie Zinc Smelting Plant. Along with zinc oxide, trace elements are discharged into the environment, among them Sb and Cd, in amounts of 0.0148 Mg/year and 0.0049 Mg/year, respectively, and 0.0182 g/($m^2 \cdot$ year) Sb and 0.0076 g/($m^2 \cdot$ year) Cd in fallen dust.

In a hypergenic environment zinc oxide is unstable and will decompose into ionic form Zn^{2+} over the whole pH range, and thereby trace elements Sb and Cd contained in zinc oxide will be released to the environment as a potential pollutant. However, under specific conditions, Zn may form hydrozincite and smithsonite. Hydrozincite is the more environmentally stable form, as its stability region has a wider range of pH and Eh than that of the less stable smithsonite. Smithsonite forms only at temperatures close to 10°C and within a narrow range of relatively high pH values of 11.0 to 11.5.

The plotted Eh and pH diagrams indicate that in the pH range of 4.5 to 7.5, and at temperatures of -5 to +25°C, which is characteristic of the soil and water environment of the area under study, cadmium is only in toxic ion form Cd^{2+} both under reducing and oxidizing conditions. Under reducing conditions antimony is on the third oxidation degree, in the mobile hydrolytic forms $Sb(OH)_6^-$, $Sb(OH)_3^0(aq)$, which are characterized by high toxicity. Under strongly oxidizing conditions (pH > 7,5 and Eh > 0.3 V) antimony is on 5 oxidation degree, in mobile form $Sb(OH)_6^-$ characterized by the lower toxicity.

With the increasing concentration of Sb (from 10^{-10} mol/dm^3 to 10^{-7} mol/dm^3, under oxidizing conditions) Sb_2S_3 and Sb_2O_4 are the products of hydrolysis and oxidation reactions, next to the dominant hydrolytic forms of Sb. The area of Sb_2S_3 and Sb_2O_4 stability fields increase with the increase of the Sb concentration.

In the initial period, antimony emitted with zinc oxide from sintering machine is characterized by low concentrations in ground-water environment. It fosters migration of Sb into this environment.

With increasing concentration of antimony concentration, the stable forms of Sb will occur. An important factor which has an impact on the Sb concentration in the ground-water environment, is the rate of decomposition of zinc oxide, which at this stage of research is difficult to determine.

Analysis of the results leads to the conclusion that both cadmium and antimony contained in the zinc oxide from the sintering machine used in the ISP process, due to the predominance of mobile, toxic forms of these elements in the soil and water environment of the smelting plant, pose a potential threat to that environment.

Single-stage extraction tests (Melaniuk-Wolny et al. 2014) also indicate that cadmium emitted with dusts during the oxidizing roasting at the "Miasteczko Śląskie" plant occurs in an exchangeable form, with more than 85% of the metal present in acetate-exchangeable form. The soils around the "Miasteczko Śląskie" plant exhibit poor buffering properties (Kicińska 2011), and hence the consequence of Cd presence in an exchangeable (mobile) form may be the migration of the metal into deeper soil strata and groundwater and the pollution thereof.

The presented results of the studies clearly indicate that, in order to eliminate toxic forms of cadmium and antimony (mainly Sb (III)) the Eh and pH of the soil in the research area should be raised by an appropriate culture of farming and fertilization.

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Abstract

The pyrometallurgical process of production of zinc and lead realized in The Zinc Smelting Plant "Miasteczko Śląskie" S.A. poses a potential threat to the natural environment. Technologies applied in the process produce toxic pollutants, among which one of the most important is dust which contains Pb, Zn, Cd, As, Sb, Tl, etc .

The detailed determination of chemical and mineral compositions of the dust allows to understand its behaviour in the environment and observe migration pathways. The paper presents results of investigations of the migration possibility to the soil and water environment of trace elements cadmium and antimony present in one of the main phases, zinc oxide, emitted with dusts from various operations of pyrometallurgical extraction of Zn and Pb at the Miasteczko Slaskie Zinc Smelting Plant, Poland.

The quantity of elements was estimated on the basis of: (i) dust fall, (ii) zinc oxide content in dust, (iii) element content in zinc oxide, and (iv) mobility of zinc oxide under the hypergenic conditions of the soil and water environment of the Smelting Plant area.

Among the elements considered, cadmium and antimony emitted with zinc oxide contained in dusts from the Sintering Machine will pose a potential hazard for the soil and water environment of the Miasteczko Slaskie Zinc Smelting Plant area.

Keywords:
pyrometallurgy, zinc, lead, zinc oxide, cadmium, antimony, Eh-pH diagrams, ground-water environment

Możliwość migracji kadmu i antymonu z tlenku cynku pochodzącego z maszyny spiekalniczej pyrometalurgicznego otrzymywania Zn i Pb do środowiska

Streszczenie

Proces pirometalurgicznego otrzymywania cynku i ołowiu metodą Imperial Smelting Process (ISP) stwarza potencjalne zagrożenie dla środowiska naturalnego. Proces technologiczny ISP jest źródłem toksycznych zanieczyszczeń, spośród których jednymi z najważniejszych są pyły zawierające w swym składzie między innymi takie pierwiastki jak Pb, Zn, Cd, As, Sb, Tl.

Dokładne poznanie składu chemicznego i mineralnego zanieczyszczeń pyłowych pozwala na określenie ich zachowania się w środowisku, prześledzenie drogi migracji. W pracy przedstawiono wyniki badań możliwości migracji do środowiska gruntowo – wodnego pierwiastków śladowych występujących w jednej z głównych faz – siarczku cynku – emitowanej z pyłami pochodzącymi z różnych odcinków technologicznych pyrometalurgicznego otrzymywania Zn i Pb w Hucie Cynku Miasteczko Śląskie S.A., Polska.

Ilość pierwiastków została oszacowana na podstawie: (i) opadu pyłu, (ii) udziału siarczku cynku w pyle, (iii) zawartości pierwiastka w siarczku cynku oraz (iii) mobilności siarczku cynku w warunkach hipergenicznych środowiska gruntowo-wodnego rejonu huty.

Potencjalne zagrożenie dla środowiska gruntowo-wodnego rejonu Huty Cynku „Miasteczko Śląskie" S.A., spośród rozpatrywanych pierwiastków, będą stanowić kadm oraz antymon emitowane wraz z siarczkiem cynku zawartym w pyłach Maszyny Spiekalniczej

Słowa kluczowe:
pirometalurgia, cynk, ołów, tlenek cynku, kadm, antymon, diagramy Eh-pH, środowisko gruntowo-wodne