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The distribution of platinum in the environment in large cities: a model study from Brno, Czech Republic

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Abstract

Due to increasing car traffic, concentrations of platinum (Pt) in all environmental compartments have significantly increased in the last two decades. Primarily, contamination of larger cities and specific environmental compartments has mostly been examined. Only limited data on Pt pollution of middle-sized cities are available. In this article, we study Pt concentrations in tunnel dust, soil and plant material collected in the middle-sized city of Brno, Czech Republic, in 2015–2016. Prior to analyses of samples, we optimized and validated preconcentration as a step preceding to AAS analysis in order to improve the LOQ and LOD values. Platinum concentration in tunnel dust ranged from 66.3 to 192 ng g^{-1} before cleaning the tunnels and from 29.2 to 38.2 ng g^{-1} after it. As expected, the Pt contents in soil and plant material were two to three orders lower. The concentrations in soil fluctuated from 10.5 to 15.7 ng g^{-1} Pt; they were comparable to Pt levels in grass that oscillated from 10.0 to 11.6 ng g^{-1} Pt. This implies relatively easy dissolution of Pt and its transport from soil to grass, which is in contrast to previous assumption that Pt is solubilized very slowly in environmental compartments. The Pt contents in all samples collected outside the traffic routes did not exceed the limit of quantification. Therefore, concentrations of Pt in the parts of the city exposed to high car traffic are several orders higher than those found in non-exposed sites; levels of Pt are increasing fast, which should initiate more comprehensive and intense research on the subject.

Keywords Atomic absorption spectrometry · Plant material · Platinum · Soil · Solid-phase extraction · Tunnel dust

Introduction

In the last two decades, levels of platinum (Pt) and its compounds in all environmental compartments have significantly increased. The main reason is rising car traffic, which is considered as one of the major sources of pollution (Wang and Li 2012). The types of environmental pollutants are progressively changing. A significant variation was induced by the ban of leaded petrol, effective in the Czech Republic since January 1, 2001, which was accompanied by the use of catalytic converters of exhaust gases. The above change triggered emissions of new pollutants such as platinum metals and their compounds. Requirements for quantitative and

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R. Komendova komendova@fch.vutbr.cz qualitative composition of exhaust emissions are influenced mainly by approved regulations on EURO emission limits, which restrict the concentrations of carbon monoxide, hydrocarbons, nitrogen oxides and particulate matter. These standards, however, do not apply to Pt metals emissions.

The catalytic converters are devices made for catalytic oxidation and reduction in hot toxic exhaust fumes to non-toxic or less toxic products. As a rule, platinum or alloys of palladium and rhodium are used as catalysts; the above leads to gradual abrasion of their active surface and to release of small particles of Pt metals and their compounds into the environment (Rosner et al. 1991; Spaziani et al. 2008; Dubiella-Jackowska et al. 2009; Reith et al. 2014).

In nature, emitted Pt and its compounds can virtually contaminate all environmental compartments. Their mobility and bioavailability are enhanced by their solubilization. Platinum present in road dust occurs in various forms such as oxides, chlorides or metallic Pt; their water solubility depends on pH, redox potential and presence of common metal ions and complexing agents in water. Solubility of Pt in rain water in the pH range 5–8 fluctuates between



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0.010 and 0.025%, and at pH 1, it reaches the value of about 0.5%. It was shown that presence of sulfur in soil or water positively influences solubility of Pt; there are studies which report solubility of up to 10% of Pt contained in fine dust particles (Zereini and Alt 2000; Ravindra et al. 2004). In general, solubility of particles is given by their size and chemical nature; only around 10% of the released Pt particles are rather quickly soluble in water. The rest, however, is not inert either and can be subjected to a number of slower reactions, which results again in the presence of soluble forms increasing thus mobility and bioavailability of Pt particles (Rosner et al. 1991; Bencs et al. 2011).

Even at low concentrations, solubilized Pt metals can enter the food web (Pawlak et al. 2014) and, consecutively, cause serious health problems to animals and people (Wiseman and Zereini 2009). It was shown that Pt(IV) ion with high redox potential can oxidize sulfur in side chains of proteins and denature them, thereby causing variety of problems such as reduction in integrity of cell membranes, inhibition of cell functions or changes in synthesis of other proteins.

Furthermore, many Pt complexes exhibit bactericidal effect caused by inhibition of nucleic acid synthesis and cell division (Rosner et al. 1991). Problems associated with damage of kidney glomeruli (Rosner et al. 1991) were also observed in higher organisms exposed to various doses of Pt salts. Platinum complexes containing halogen ligands belong to the most important allergens, and their aggressiveness increases with higher number of halogen atoms in the molecule. Exposure to the above complexes induces health problems such as autoimmune disorders, asthma or dermatitis (Moldovan et al. 2002; Ek et al. 2004).

As aforementioned, concentration of Pt metals in environmental compartments is progressively increasing due to high traffic. After emission of exhaust fumes, Pt comes into a direct contact with road surface, vegetation (Komendova 2018), buildings or soil that are located in immediate vicinity of roads. Concentration of Pt in road dust may reach values of about 100 ng g^{-1} ; its concentration decreases with increasing distance from roads and with rising depth of the soil layer (Zereini and Alt 2000; Hooda et al. 2007; Mihaljevič et al. 2013). In general, the amount of Pt released from automotive catalysts ranges in orders of ng km⁻¹ according to distance travelled; levels of $pg m^{-3}$ were found in air close to busy roads, and Pt amounts oscillating within ng g⁻¹ range were determined in road dust, soil, vegetation and bodies of animals (Zimmermann et al. 2003). Platinum is also accumulating in marine sediments. The research conducted in the 1980s reported Pt metals levels of 150 pg L^{-1} and, after two decades, their 50 times increase (Zereini and Alt 2000; Abdou et al. 2016). That means that concentration of Pt metals in environment did not exceed critical level. As a result, Pt occurring in environmental compartments has not been regarded as a substantial problem; there is only scarce information on its distribution available.

Analyses of Pt metals in environmental matrices require an approach sensitive enough to determine their trace concentrations. Therefore, either the use of a sensitive, usually costly analytical technique is required, or the samples are preconcentrated, and a less sensitive device can be used.

An ICP-MS device, whose detection limits usually range within units of ng L^{-1} , is regarded as a sufficiently sensitive instrument for Pt metals analyses. The preconcentration step is typically followed by AAS analysis, with detection limits in μ g L^{-1} . Nevertheless, the preconcentration step can improve sensitivity of the above method by several orders enhancing it thus to performance shown by more sensitive analytical techniques. In addition, preconcentration process removes accompanying compounds such as other ions or natural colloids.

Direct analyses of trace amounts of Pt in biological materials (Zimmermann et al. 2003) and soils or road dust (Spaziani et al. 2008) were conducted as reported. The techniques used included ET-AAS atomic spectrometry (Leopold et al. 2008), ICP-OES, ICP-MS (Bencs et al. 2003; Niemelä et al. 2004; Goncalves et al. 2008), DRC-ICP-MS (Kan and Tanner 2004), ICP-MS/MS (Machado et al. 2017) or voltammetry (Orecchio and Amorello 2011). The advantages of separation and preconcentration techniques using solid sorbents such as solid-phase extraction (SPE) were accounted and proved in many studies (Vlašánková et al. 1999; Vlašánková and Sommer 1999; Komendová-Vlašánková and Sommer 2002; Chwastowska et al. 2004; Castillo et al. 2012; Kononova et al. 2012).

In particular, researchers (Komendová-Vlašánková and Sommer 2002) developed a procedure for separation and preconcentration of trace amounts of Pt on C18 silica gel. This method had been tested only with spiked materials. As a novelty, it was applied for determination of Pt in real soil and grass samples within this study.

As aforementioned, the steadily increasing intensity of traffic, especially in industrially developed countries, leads to a tremendous increase in concentration of Pt metals in environmental matrices. However, up to now, only little attention has been paid to the above rise. Contamination of larger cities and specific environmental compartments has mostly been examined. Only limited data on pollution of middle-sized cities are available. Therefore, the aim of this work was to (1) optimize and validate the separation and preconcentration method elaborated by Komendová-Vlašánková and Sommer for ICP-MS in 2002 to be applicable for AAS analyses using a certified tunnel dust standard and (2) use this method for determination of Pt in soil and grass samples monitoring thus volume of automobile transport in Brno, Czech Republic. Brno serves as a model of a Central European city with population of around 400 thousands inhabitants; the city has experienced a substantial increase in car traffic, industrial production and transport tunnels building in the last 20 years.



Materials and methods

Soil, tunnel dust and plant samples

Three types of samples exposed to car traffic were analyzed: soils, plant tissues and tunnel dust. Dust samples were obtained mainly from walls of tunnels and underpasses located within the city of Brno. The samples were collected in the Husovice, Pisárky and Dobrovského tunnels, each having one upward and downward tube. The tunnels are cleaned twice a year, in spring and autumn (see Table 1). They are linked to each other by connecting roads; the tunnels show traffic volume of about 40 thousand cars per 24 h. The dust settled in tunnels was collected in two steps. First sampling was carried out shortly before cleaning the tunnels; then, the second one was done a month after the cleaning. To ensure consistency of dust particles, dust in the form of powder was scraped from the walls using a broom at a height of about 1 m above the road.

Collection of soil and grass samples was carried out at several sites located in Brno, namely around the high traffic (average traffic over 40,000 cars per 24 h), medium traffic (average traffic 20,000 cars per 24 h) and low traffic roads (1000 cars per 24 h in average) lined with grass strips. The samples were collected around the following streets categorized into three above classes: high traffic streets (Svatoplukova, Zvonařka and Úvoz); medium traffic streets (Lipová, Černovická and Jedovnická); and low traffic streets (Podbělová, Barvičova and Střední). Soil, as the underneath of grass, was always sampled together with it at one sampling site situated close to the roadway. The grass was then separated from the soil, and the samples were subsequently dried at room temperature to constant weight. This process was followed by homogenization, quartering and decomposition procedures. Analyzed samples collected in the areas near the traffic-affected roads from the above three categories were compared with the samples from traffic-free locations, which were supposed to show significantly lower levels of contamination. These no traffic samples were obtained in Lužánky Park, Líšeň Calcite Quarry and Mariánské Valley (see Table 1).

Decomposition of road dust samples was carried out in a microwave extractor using *aqua regia* (HCl/HNO₃ 3:1). Samples (0.25 g) were quantitatively transferred into six TeflonTM patrons and mixed with 10 mL of *aqua regia*. The cartridges were then placed in the microwave extractor and decomposed subsequently according to the program described in Table 2. After decomposition, the content of the patrons was quantitatively filtered through a glass filter. Consequently, the filtrate was evaporated in Teflon[®] dishes on a hot plate. Quantitative conversion of the residue to a 25-mL volumetric flask was done via addition of 0.1 mol L⁻¹ HCl to avoid sorption of Pt on the vessel walls.

Decomposition of soil and grass samples was carried out using the classical wet way in a 250-mL boiling reflux flask with *aqua regia*. The sample (10 g of soil, 5 g of grass) was quantitatively transferred to a round-bottom flask, wetted with HNO₃ (15 mL) and left in it until the next day, when HCl (45 mL) was added. The processed sample solution was brought to boiling point via a heating mantle. After 2 h at reflux, water (25 mL) was added and the solution of the specimen was boiled for additional 15 min and filtered then. Subsequently, the solvent was evaporated using a hot plate and quantitative conversion of decomposed sample into a 50-mL volumetric flask filled up with 0.1 mol L⁻¹ HCl was done.

Instrumentation

An atomic absorption spectrometer ZEEnit 60 (Analytik Jena, Germany) with transversely heated graphite furnace, a Zeeman background corrector, a platinum hollow cathode lamp by Photron Australia and an autosampler were used for determination of Pt. Optimal measurement parameters of temperature program are given in Table 3. The method for determination of Pt by the ET-AAS ZEEnit 60 device has extensively been studied and optimized. Used wavelength, temperature program, slit width and the effect of acidity on the signal were optimized. The effect of the Septonex[®]surfactant and influence of other accompanying elements contained in real samples after acid digestion have also been studied (Komendová-Vlašánková and Sommer 2002; Kosárová et al. 2015).

The values of instrumental limits obtained from calibration function measured using ET-AAS were determined as follows: Limit of detection (LOD) was calculated at 1.14 µg L⁻¹ and limit of quantification (LOQ) was gained at 3.79 µg L⁻¹. All results obtained within this study are calculated as the average results of three separate independent determinations; each of them was done five times using an atomic absorption spectrometer. The resulting values of Pt content found in environmental samples are given as mass concentration (after conversion of the weight of analyzed material) in ng g⁻¹, which corresponds to volume concentration in µg L⁻¹.

Procedure

A Milestone MLS-1200 Microwave Laboratory System was used for decomposition of tunnel dust samples. Its carousel was equipped with six extraction thimbles made from TeflonTM. The optimal program for decomposition of samples included five heating operation steps: 2 min at 200 W; 2 min at 400 W; 5 min at 0 W; 10 min at 600 W; and final cooling of patrons for 20 min.

The solid-phase extraction sorption system for separation and preconcentration of Pt from decomposed samples consisted of a peristaltic PCD 82.4 K pump with four ISMATEC ISO649 positions (Czech Republic), silicon tubes, SPE Bond Elut-C18 columns and a BAKER J. T. spe-12G vacuum suction device with 12 slots for location of the SPE columns.



Table 1Characterization ofsample collection sites

Sample num- ber	Sample type	Site of sample collection	GPS
Tunnels			
1	t	Husovice tunnel, upward	49°12′59.746″N, 16°37′52.961″E
2	t	Husovice tunnel, downward	49°12′59.746″N, 16°37′52.961″E
3	t	Pisárky tunnel, upward	49°11′9.584″N, 16°33′58.241″E
4	t	Pisárky tunnel, downward	49°11′9.584″N, 16°33′58.241″E
5	t	Dobrovského tunnel, upward	49°13′7.414″N, 16°35′24.872″E
6	t	Dobrovského tunnel, downward	49°13′7.414″N, 16°35′24.872″E
7	t	Pisárky underpass	49°11′38.509″N, 16°34′9.729″E
8	t	Husovice tunnel, upward	49°12′59.746″N, 16°37′52.961″E
9	t	Pisárky tunnel, upward	49°11′9.584″N, 16°33′58.241″E
High traffic			
10	S	Svatoplukova street ^{ht}	49°12′34.941″N, 16°38′32.812″E
11	g		
12	s	Úvoz street ^{ht}	49°12′2.857″N, 16°35′34.972″E
13	g		
14	S	Zvonařka street ^{ht}	49°11'13.378"N, 16°37'6.311"E
15	g		
Medium traffic	2		
16	8	Černovická street ^{mt}	49°10'42.132"N, 16°38'45.299"E
17	g		
18	s	Jedovnická street ^{mt}	49°12'40.931"N, 16°40'24.794"E
19	g		
20	s	Lipová street ^{mt}	49°11'37.349"N, 16°34'36.931"E
21	g		
Low traffic			
22	S	Podbělová street ^{lt}	49°12′50.626″N, 16°41′10.465″E
23	g		
24	8	Barvičova street ^{lt}	49°11′58.021″N, 16°34′42.110″E
25	g		
26	S	Střední street ^{lt}	49°12'38.491"N, 16°36'22.711"E
27	g		
No traffic			
28	S	Lužánky park ^{nt}	49°12′24.419″N, 16°36′31.801″E
29	g		
30	8	Kalcit quarry ^{nt}	49°13′22.881″N, 16°41′28.775″E
31	g		
32	S	Mariánské valley ^{nt}	49°12′59.445″N, 16°43′9.494″E
33	g		

Each sample was labeled with a number. The samples were classified into the following categories: t tunnel dust, tb tunnel dust before cleaning, ta tunnel dust after cleaning, s soil, g grass. Four categories of sampling sites were distinguished: ht high traffic, mt medium traffic, lt low traffic and nt no traffic places

The sorption procedure involved four consecutive steps: (1) conditioning the sorbent; (2) sorption of the sample; (3) washing the sorbent; and (4) elution of the analyte. The octadecyl silica gel was used as a sorbent for SPE. The conditioning solution consisted of 10 mL 0.005 mol L⁻¹ Septonex[®] in 0.1 mol L⁻¹ HCl; the washing was performed with distilled water and 10 mL of acetonitrile was used as an eluent. The flow rate was set at 1 mL min⁻¹. The 95% sorption efficiency for Pt was confirmed through SPE optimization process in model solutions. Interferences of elements were not observed, because they were eliminated during the separation procedure.

Reagents

Platinum certified reference material ASTASOL[®] containing 1.000 ± 0.002 g L⁻¹ Pt in the form of chloro complex

 Table 2
 Program decomposition for tunnel dust samples and cleaning patron extractor

Decomposition power	Time (min)	Cleaning power	Time (min)
200 W	2	200 W	2
400 W	2	400 W	2
0 W	5	0 W	2
600 W	10	600 W	5
Cooling	20	Cooling	15

Table 3 The temperature program and measurement conditions applied for ZEEnit 60, Jena

Process	Tempera- ture (°C)	Ramp (°C s ⁻¹)	Time of retention (s)	Time (s)
Drying	90	5	20	34
Drying	105	3	20	25
Drying	110	2	10	12.5
Pyrolysis	1600	250	10	16
Atomization	2300	1400	8	8.5
Cleaning	2400	500	4	4.2

Other conditions: graphite cuvette within platform, slot width 0.2 nm, used wavelength 265.9 nm, current 8 mA, injection volume $20 \,\mu L$

in 5% HCl (Analytika, Prague, Czech Republic) was used for measurement of the calibration curve and verification of the method effectiveness. N-(α -Carbethoxypentadecyl)trimethylammonium bromide (Septonex[®]) was used as a cationic surfactant for SPE treatment of decomposed samples (GNB Inc., Czech Republic). All other used chemicals and reagents were of analytical grade purity.

The hydrophobic Bond Elut C18 sorbent in original columns (500 mg in 3-mL cartridges), particle size 40 μ m (Agilent Technologies, HPST s.r.o., Prague, Czech Republic), was used as a stationary phase for preconcentration.

Standard Reference Material of tunnel dust BCR-723 with declared content of Pt metals (Zischka et al. 2002; Sutherland 2007) was analyzed to verify the methods for determination of Pt in environmental samples. This standard was prepared in the same way like the other samples (see the above description). Certified concentration of Pt in the standard preparation was declared in the range of 81.3 ± 2.5 ng g⁻¹.

Results and discussion

Optimization and validation of preconcentration procedure

At first, we focused on optimization and validation of preconcentration procedure, which leads to an increase in concentration of Pt in a sample and simultaneously to removal of ions and compounds potentially masking determination of the analyte. In principle, the Bond Elut C18 is the most hydrophobic bonded silica sorbent for retaining nonpolar compounds in existence. Platinum, in the form of a chloro complex, creates ion associates with a cationic surfactant and this associate exerts high affinity to the octadecyl-modified silica gel sorbent. The sorption procedure described in (Komendová-Vlašánková and Sommer 2002) was optimized with model samples to reach maximal sorption recovery of Pt. We tested a wide range of the following conditions: sorption rate; the type and concentration of association agents; type, pH and volume of eluent mixtures; acidity of sorbed and measured solutions; and various interferences originating from presence of interfering ions, which occurred in digested real samples of soil, plant material and tunnel dust. After ten repeated measurements under the optimal conditions, the recovery rate ranging between $95.2 \pm 1.5\%$ was achieved. It was also found that this sorption process is reproducible and robust, which means that it does not depend on type of matrix and Pt concentration. In other words, large excesses (thousand times in comparison with Pt concentration) of either accompanying anions (chlorides, nitrates, nitrites, sulfates) or cations (sodium and potassium ions, calcium and magnesium ions, iron ions, aluminum ions and other heavy metal ions) did not interfere with the determined analyte. Therefore, the preconcentration method was proved to be suitable for determination of Pt in sampled soils, dust and plants in the range of several concentration orders. This method was also validated using the BCR certified reference material; $83.0 \pm 3.6 \text{ ng g}^{-1}$ Pt was found, which is in a good agreement with a certified reference value of 81.3 ± 2.5 ng g⁻¹.

Determination of Pt in tunnel dust, soils and plant material

Platinum was determined in nine samples of tunnel dust, 12 soil samples and 12 corresponding samples of grass collected at the same place (Table 1). All results are summarized in Fig. 1 which shows that Pt concentrations in tunnels reached their highest levels before cleaning when they ranged from 66.3 to 192 ng g^{-1} . Cleaning of tunnels, which is carried out twice a year, resulted in a significant decrease in Pt levels that oscillated between 29.2 and 38.2 ng g^{-1} . We hypothesize that the concentration of Pt measured after cleaning reflects capability of tunnel dust to accumulate it within a month, which represents sampling time after tunnel cleaning. The concentration found before cleaning reflects capacity of the tunnel to retain Pt particles within 6 months when the tunnels are regularly cleaned. Comparison of averaged values (Fig. 2) implies that accumulation rate decreases in time. We assume that this is caused by progressive





Fig. 1 Platinum concentration in ng g⁻¹ determined in tunnel dust, soil, and plant samples

saturation of tunnel dust absorption capacity and by outlet of the dust from the tunnel. This is partially reflected in elevated concentration of Pt in underpass (sample 7).

Platinum concentrations ranged from 10.5 to 15.7 ng g^{-1} in soils and oscillated from 10.0 to 11.6 ng g^{-1} in grass; the soil and grass samples were collected in the vicinity of roads with high car traffic. In the samples collected in the area with medium volume of automobile traffic, Pt contents ranged from 6.02 to 8.44 ng g^{-1} in soil and from 5.61 to 7.85 ng g^{-1} in grass. The area with low car traffic, located mainly in the outskirts of the city, was characterized by lower values ranging from 4.00 to 6.48 ng g^{-1} in soil and from 3.99 to 5.02 ng g^{-1} in grass. As expected, these values are one to two orders lower than those found in tunnel dust, which was caused by better diffusion of emission exhausts into free space and by removal of dust by rain precipitations. Surprisingly, concentration of Pt in soil and grass (dry mass) reached comparable levels, which implies relatively easy transport of Pt from soil to grass. As aforementioned, bioavailability of Pt is determined by its solubility (Pawlak et al. 2014). The finding on comparable concentrations in grass and soils is in contrast to the observation showing that only a low portion of Pt is rapidly (within a vegetation period) solubilized. Nevertheless, the influence of Pt dispersed in air plays also significant role.

Rest and recreational areas of Brno such as parks and valleys showed concentrations of Pt below the quantification limits of the suggested technique; this affirms the assumption that traffic represents the main source of higher concentration of Pt in environmental compartments.

Figure 3 shows average Pt content found in different samples with respect to the frequency of car traffic volume excluding the tunnels. Dependency of Pt concentration on traffic volume indicates that an increase in Pt concentration is not linearly proportional to rise in number of cars; the difference between categories of traffic volume 1 and 20 thousand cars per hour is really minor. We assume that this may be attributed to speed of cars and driving style, but this supposition goes beyond the scope of this paper.

The findings of the authors corroborate with results of other researches who reported data from other cities in Europe and also around the world (Bencs et al. 2011; Dubiella-Jackowska et al. 2009; Hooda et al. 2007; etc.). The obtained results show that the inhabitants of large cities are continuously exposed to emissions of Pt metals. Given the ever-increasing vehicular traffic (Matthey 2017) and irreplaceability of Pt metals in catalysts, the population will be exposed to rising concentrations of these substances. With regard to possible allergenic reactions to Pt compounds, the findings of this study are supposed to contribute to





introduction of complete monitoring and determination of Pt effects in such polluted environment.

Conclusion

The results of this study show that the environmental compartments of middle-sized cities exposed to car traffic are contaminated with platinum (Pt). It was demonstrated that the environmental Pt concentrations depend on intensity of automotive traffic. Tunnels represent specific urban sites, where dust containing Pt accumulates. Levels of Pt in tunnels are several times higher than those in soil or grass; after cleaning of tunnels, concentration of Pt is rapidly restored. Compared with Pt levels in tunnels, its lower concentrations in open places are given by conditions for dispersion of dust particles to the atmosphere and also by the effects of weather, especially of rainfall events.

■Pt in grass (ng/g)

☑ Pt in soil (ng/g)

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