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MERCURY AND METHYLMERCURY IN TERRESTRIAL SOILS IN DIFFERENTLY CONTAMINATED SITES IN SLOVAKIA

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Abstract

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Mercury (Hg) and methylmercury (MeHg) are global pollutants that might affect the function of terrestrial ecosystems. We were investigating the concentrations of MeHg and Hg_{total} in the soil profiles under forest and meadow at 9 sites in Slovakia and analyzing the relation between MeHg-to-Hg_{total} and Hg pollution. The Hg_{total} and MeHg concentrations in Rudňany soils (up to 104.000 and 50 $ng.Hg.g^{-1}$, respectively) were much higher than those in the other soils (up to 1000 and 4 $ng.Hg.g^{-1}$, respectively). The soils from the polluted site (Rudňany) had very low MeHg-to-Hg_{total} ratios (median: 0.037%) comparing to the ratios in soils from less contaminated sites (median: 0.48%). In Žiar basin, the MeHg-to-Hg_{total} ratios in forest floors were the lowest at the closest sites to the actual and past pollution sources. The high MeHg concentrations in Rudňany soils suggest the enhanced importance of Hg methylation in Hg contaminated terrestrial soils. For the first time, the MeHg-to-Hg_{total} ratios were demonstrated to be negatively related to the Hg pollution in terrestrial soils. The ratios in forest floors reflected the Hg pollution better than those from meadow and mineral soils.

Key words: methylmercury, total mercury, soil, Slovakia

Introduction

Mercury (Hg) is released into the environment by both natural sources and human activities and today it is an ubiquitous pollutant. Human activities like combustion of fossil fuels, artisanal gold mining (Nriagu, Wong, 1998) and several industrial, chemical and pharmaceutical application of Hg have significantly increased the Hg release into the environments (Allan, 1999).

Organomercury compounds have usually higher toxic effect than their corresponding inorganic forms. The methylmercury (MeHg) in the environment is generally thought to

originate from natural process instead of anthropogenic emission, e.g. *in situ* Hg methylation in wetland soils and aquatic ecosystem (St. Louis et al., 1996) and *in vivo* methylation by organisms (Jereb et al., 2003). Methylmercury in terrestrial soils may dominantly originate from the atmospheric deposition. Methylation of Hg seems less important in terrestrial soils than wetland soils due to the higher demethylation rate in aerobic environments compared to anaerobic environments (Ullrich et al., 2001). Therefore, the MeHg concentrations in terrestrial soils were usually much lower comparing to those in wetland soils. Little information about the MeHg in Hg contaminated upland soils. Thus, to estimate the relevance of methylation in strong contaminated upland soils is still impossible.

The ratio of MeHg-to-Hg_{total} in precipitation may be useful as the indicator of polluted areas with lower ratios (< 1.0%) indicating a more polluted site (Downs et al., 1998). In sediments, the MeHg-to-Hg_{total} ratios increased usually with increasing distance from the pollution source (Suchanek et al., 1998), because high Hg concentrations may depress Hg methylation or may favour demethylation (Ullrich et al., 2001). In the past, the relation of MeHg-to-Hg_{total} ratios to Hg contamination in terrestrial soils was rarely investigated.

The atmospheric Hg emission in Slovakia amounted to 12.479 $t.yr^{-1}$ in 1990 and 4.450 $t.yr^{-1}$ in 2000. The atmospheric Hg levels at more than 34% of the tested sites in the Slovakia exceeded 5 $ng.m^{-3}$ (Hladíková et al., 2001), the WHO recommends guideline value. In sense of Kabata-Pendias, Pendias (1992) values of total Hg content in soil are ranging from 50 $ng.Hg.g^{-1}$ to 300 $ng.Hg.g^{-1}$ in the world. According to Čurlík, Šefčík (1999) the average value in Slovakia is 80 $ng.Hg.g^{-1}$ in forest floor and 50 $ng.Hg.g^{-1}$ in substratum. Content ranges (20–98 000 or 446 000 $ng.Hg.g^{-1}$) are higher than ones in the world, where limit values of Hg in soils are 1000–2700 $ng.Hg.g^{-1}$. In Žiar basin, the Hg concentrations in soils could reach 2460 $ng.Hg.g^{-1}$ (Iwaco, 1994), which was over the Slovak soil guideline value B (2000 $ng.Hg.g^{-1}$) for noticeably Hg contaminated soils (in sense of Resolution of Ministry of agriculture of the Slovak republic No. 531/1994-540). The past studies indicated the potential problem of soil pollution caused by Hg in Slovakia, but little information was available about MeHg.

The objectives of this study were to determine the concentrations of MeHg and Hg_{total} in the soils in the middle and east Slovakia and to analyse the relation of MeHg-to-Hg_{total} ratios to soil Hg pollution. The results of this investigation may provide helpful information to access the environmental risk and human hazards in the middle and east Slovakia.

Materials and methods

Site description and soil sampling

Forest and meadow soils were taken at transect Žiar nad Hronom-Vtáčnik (abbreviated as transect Žiar), Banská Štiavnica, and Rudňany. Just at these sites are according to both the soil monitoring (Kobza et al., 2002) and geochemical atlas (Čurlík, Šefčík, 1999) the most common exceed Hg limits in soils in Slovakia. The most dangerous is place around Rudňany, strictly among towns Rožňava – Gelnica – Spišská Nová Ves.

The investigated transect Žiar is located in Žiar basin in the middle of Slovakia (Fig. 1) and includes 7 sites (Table 1). An aluminium smelter near Ladomerská Vieska has operated since 1953 and additionally gallium

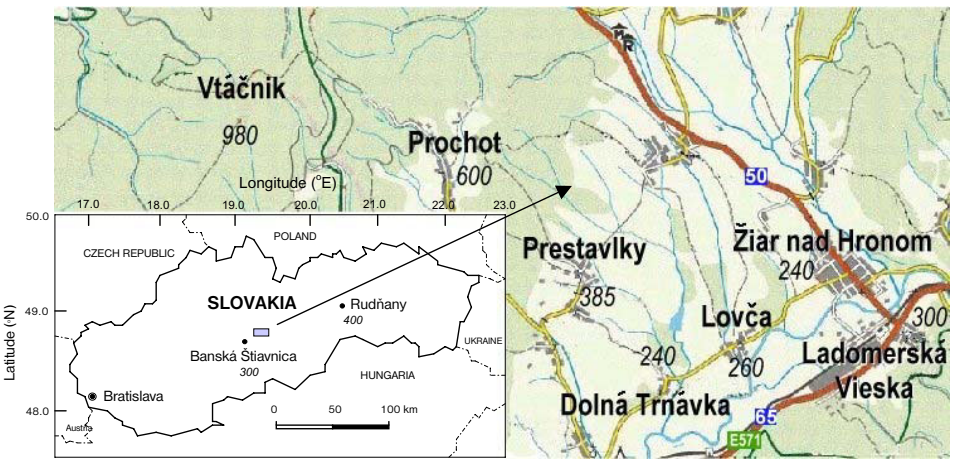

Fig. 1. Map of sampling locality in Slovakia. ▭: transect Žiar nad Hronom-Vtáčnik. The altitude of each locality is shown.

was produced per amalgamation with Hg until 1993. A brown-coal power plant Zemianske Kostoľany in Hornonitrianska basin is 26 km west from Žiar basin.

Banská Štiavnica is 21 km southwest from Žiar basin (Fig. 1) and located in the volcanic mountains Štiavnické vrchy. The pollution of heavy metals in this area is caused by mining in the past (10-19 century). The site Rudňany is located in east Slovakia (Fig. 1) and is strongly contaminated by Hg, that is not only geochemical anomaly but also has anthropogenic origin (Maňkovská, 1996). Mercury has been produced together with iron and copper at this site in the past (1332–1992).

Forest and meadow soils were taken in summer 2003. Soils were sampled from the depth to 30 cm at 10 cm interval. Fresh soils and forest floors were sieved to 2 mm and homogenized. After freeze-drying, soil materials were ground and stored at –20°C in the dark before analysis.

Reagents

Methylmercury (MeHg), 95%, as chloride was purchased from Alfa Aesar, Karlsruhe, Germany. Triethyltin chloride, 98%, was purchased from Merck, Darmstadt, Germany. The stock solution (10 μg.Hg.ml^{-1}) of MeHg was prepared in methanol and stored at -40°C in the dark. A working solution with a concentration of 0.1 μg.Hg.ml^{-1} was prepared before each use by dilution of the stock solutions with methanol (Merck, p.a. grade). Triethyltin used as internal standard was prepared in the same way.

De-ionized water was purified in a Milli-Q system (Milli-Q system (Millipore, Milford, MA, U.S.A.). Sodium tetra(*n*-propyl)borate ($NaBPr_4$), 98%, was synthesized by Merseburger Spezialchemikalien, Merseburg, Germany. The derivatization reagent, 2% $NaBPr_4$ solution, was prepared before each usage by dissolving reagent in Milli-Q water. The acetate buffer was prepared by dissolving 1 mole sodium acetate in 1 liter of Milli-Q water followed by adjusting pH to 4 with glacial acetic acid.

Analysis of methylmercury

For analysis of MeHg in soils, 0.3–1.0 g samples were extracted with 4 ml 1 M $CaCl_2$, 0.1% tropolone in glacial acetic acid together with 5 ng internal standard triethyltin. The aliquot merged with 80 ml Milli-Q water was

T a b l e 1. Carbon contents, pH, and type (FAO) of soils

	Vegetation, Soil type (FAO)		pH (H_2O) meadow	pH (H_2O) forest	C [%] meadow	C [%] forest
Transect Žiar	**Ladomerská Vieska**					
	Forest floor			7.19		22.5
	0–10 cm	oak forest,	6.61	6.79	1.91	4.92
	10–20 cm	cambisols (typical)	6.71	6.00	1.57	1.06
	20–30 cm		6.77	5.74	0.84	1.75
	Lovča					
	Forest floor			6.35		6.99
	0–10 cm	oak forest,	6.41	5.93	1.60	2.68
	10–20 cm	gleysol (typical)	6.42	5.66	1.43	2.74
	20–30 cm		6.64	5.73	1.30	1.51
	Žiar nad Hronom					
	Forest floor			7.20		13.6
	0–10 cm	oak forest,	7.52	7.22	4.08	2.99
	10–20 cm	cambisol (luvisol)	7.38	7.31	1.81	0.67
	20–30 cm		7.23	7.54	1.08	0.47
	Dolná Trnávka					
	Forest floor		6.41		14.8	
	0–10 cm	oak forest,	6.23	6.53	4.40	1.93
	10–20 cm	gleysol (typical)	6.14	5.88	4.18	1.00
	20–30 cm		5.98	5.65	3.97	0.73
	Prestavlky					
	Forest floor			6.20		15.1
	0–10 cm	oak and beech forest,	7.41	5.28	2.88	2.25
	10–20 cm	gleysol (typical)	7.35	5.63	1.08	1.61
	20–30 cm		7.35	5.85	3.59	1.51
	Prochot					
	Forest floor			6.09		16.3
	0–10 cm	oak and beech forest,	6.23	5.92	4.19	3.25
	10–20 cm	cambisol (typical)	5.88	6.22	2.27	1.65
	20–30 cm		5.78	6.44	1.72	1.21
	Vtáčnik					
	Forest floor			4.61		22.4
	0–10 cm	beech forest,	6.32	4.83	9.67	6.17
	10–20 cm	andosol (typical)	6.45	4.86	1.37	3.76
	20–30 cm		6.25	4.78	0.85	2.77
	Banská Štiavnica					
	Forest floor			5.16		7.65
	0–10 cm	oak forest,	5.77	4.67	5.42	3.53
	10–20 cm	cambisol (dystric)	5.65	4.50	3.97	1.90
	20–30 cm		5.35	4.48	1.56	2.43
	Rudňany					
	Forest floor			5.14		25.4
	0–10 cm	oak forest,	6.63	5.01	2.58	19.9
	10–20 cm	ranker	6.91	3.93	2.12	2.05
	20–30 cm		6.97	4.05	0.57	2.11

adjusted to pH 4 with acetate buffer and derivatizated by 10 mg $NaBPr_4$ in glass volumetric flask. The solution was then extracted with 1 ml cyclopentane by vigorous shaking for 10 minutes. The cyclopentane extract was centrifuged, cleaned-up with silica gel and analyzed with a coupling of a gas chromatograph (HP 6890) to an inductively coupled plasma mass spectrometer (ICP-MS ELAN 5000, Perkin-Elmer SCIEX) (Huang et al., 2003).

A certified sediment IAEA-356 was used for quality control. Additionally, 0.5-1 g soils were spiked with 5 ng MeHg as Hg in 10 ml glass centrifuge tubes for testing the recovery quality. The samples were then stored in the dark and at the room temperature overnight. Recoveries of spiked MeHg from mineral soils and forest floors were 88% and 95%, respectively.

Analysis of total mercury

Soils (0.5 g) were first digested with 3 ml HNO_3 (65%) + 0.5 ml HCl (30%) by High Pressure Accelerated Solvent (HPA-S, Anton Paar, Austria). In the 3-step program, a first heating to 80°C, was followed by heating to 170°C and finally to 270°C, lasting for 90 minutes. The supernatant was then filtered with membrane filter, diluted to 25 ml with Milli-Q water for further analysis with ICP-MS (Agilent 7500c, Japan). Quality control of Hg_{total} from soils was conducted with certified materials, 7002 light sandy soils, 7003 silty clay loam (Analytika Co, the Czech Republic) and SO-3 soil (Canda Centre for Mineral and Energy Technology) with recoveries from 93% to 119%.

For analysis of carbon contents, 0.5 g soil material was analyzed by CHN-O Rapid, Elementar, Germany. Measurements of soil pH were conducted using a pH electrode in a 1:2.5 soil/water ratio.

Results

The MeHg concentrations in soils from transect Žiar and Banská Štiavnica ranged from 0.04 to 4.25 $ng.Hg.g^{-1}$ (Fig. 2). In Rudňany the MeHg concentrations in soils were up to 50 $ng.Hg.g^{-1}$ and were apparently higher than those from transect Žiar and Banská Štiavnica. The MeHg concentrations in forest and meadow soils were usually the highest in either forest floors or upper mineral soils and decreased with depth. Generally, the MeHg concentrations in the upper horizons of forest soils were higher than of meadow soils with the exception of Prestavlky, where the MeHg concentrations in the meadow soils were much higher than those in the forest soils.

The Hg_{total} concentrations in soils from transect Žiar and Banská Štiavnica were between 26 and 916 $ng.Hg.g^{-1}$ (Fig. 3). In most cases, Hg_{total} concentrations were higher in forest soils than those in meadow soils, except Prestavlky. In forest soils, concentrations of Hg_{total} were highest in forest floors and decreased with depth. Similar pattern of Hg_{total} vertical distribution were observed in most meadow soils, but gradients were less steep than those in forest soils. In transect Žiar, Hg_{total} concentrations showed the steepest gradient in forest soils from Ladomerská Vieska and Vtáčnik. In meadow soils in Banská Štiavnica Hg_{total} concentrations increased with depth. All soils in Rudňany had much higher Hg_{total} concentrations than those in transect Žiar and Banská Štiavnica. The highest Hg_{total} concentration in this study was observed in the forest floor in Rudňany (104 000 $ng.Hg.g^{-1}$).

The MeHg and Hg_{total} concentrations in soils correlated rarely to soil parameters, such as carbon contents, pH and conductivity. However, the vertical distribution of MeHg and Hg_{total} in each soil profile depend on carbon contents in most cases (Table 1).

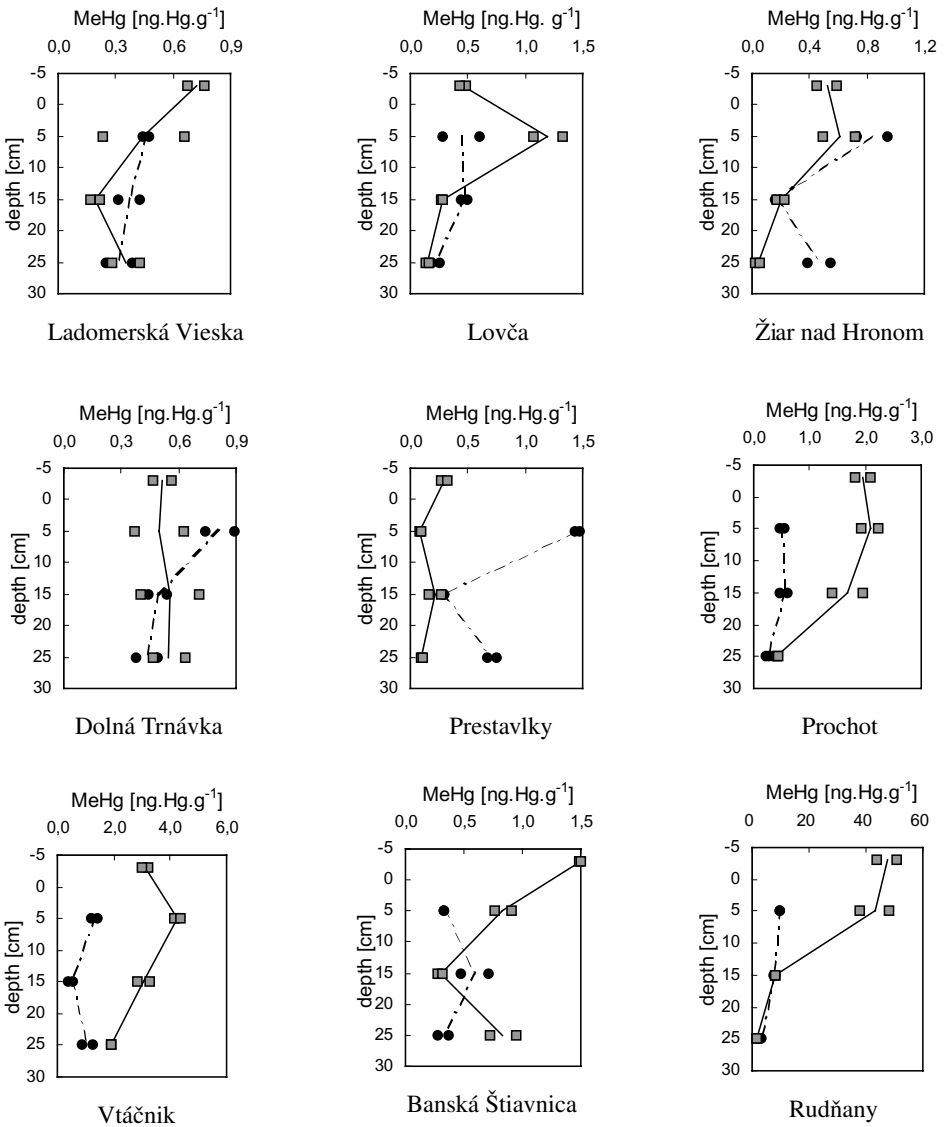

Fig. 2. Methylmercury (MeHg) concentrations in forest soils and meadow soils from transect Žiar nad Hronom–Vtáčnik, Banská Štiavnica and Rudňany. —□— forest soils, —◆— meadow soils.

The ratios of MeHg-to-Hg_{total} in soils from transect Žiar and Banská Štiavnica ranged from 0.039% to 2.94%, with 0.48% as median value (n = 96). The soils from Rudňany had much smaller MeHg-to-Hg_{total} ratios (0.017–0.050%, with 0.037% as median value, n = 14) than those from transect Žiar and Banská Štiavnica. The MeHg-to-Hg_{total} ratios in the forest

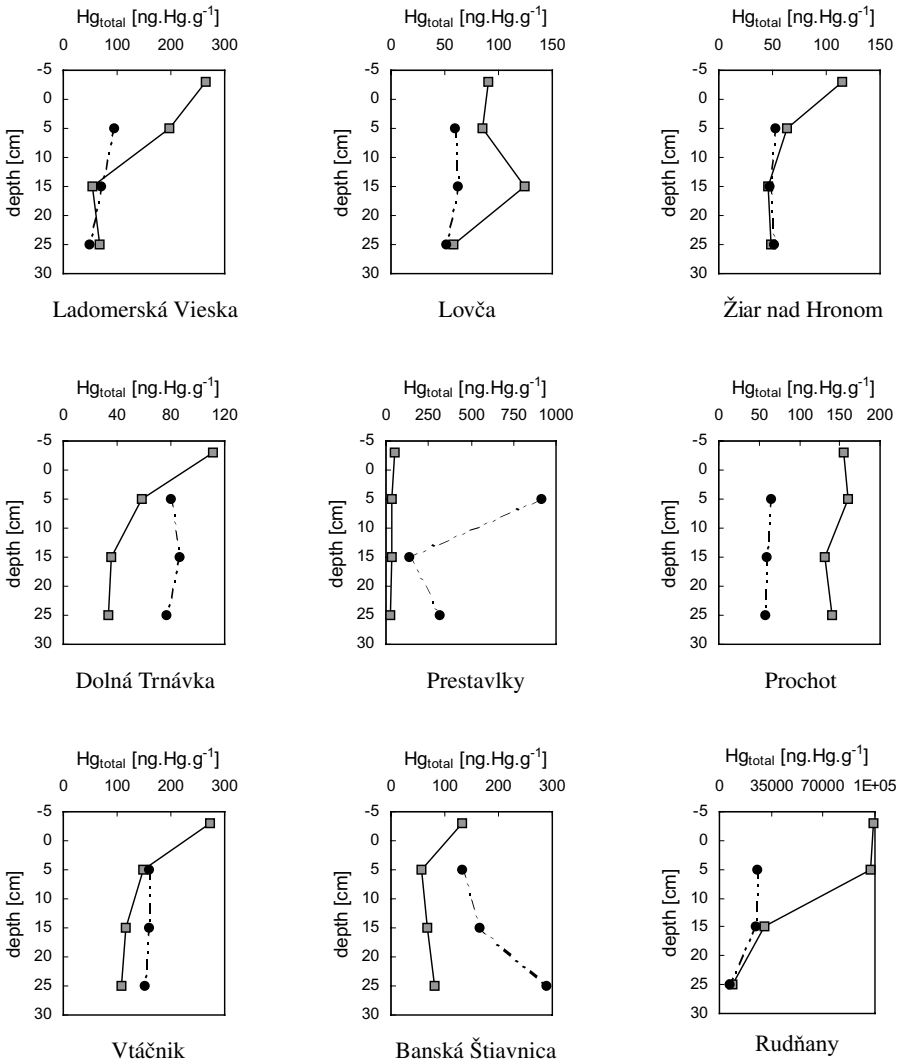

Fig. 3. Total mercury (Hg_{total}) concentrations in forest soils and meadow soils from transect Žiar nad Hronom–Vtáčnik, Banská Štiavnica and Rudňany. —□— forest soils, – - ◆ - - meadow soils.

floors from transect Žiar showed a very clear pattern: Ladomerská Vieska (0.27%) < Lovča (0.50%), Žiar nad Hronom (0.45%) and Dolná Trnávka (0.46%) < Prestavlky (0.55%) < Prochot (1.26%) > Vtáčnik (0.19%). However, such pattern was not so remarkable in meadow soils and mineral soils.

Discussion

In Rudňany the Hg_{total} concentrations in soils exceed the Slovak guideline value C 10 000 ng.Hg.g^{-1} (in sense of Resolution of Ministry of agriculture of the Slovak republic No. 531/1994-540), due to pollution from ore mining in the past (Maňkovská, 1996). Older data (Banásová, Pišút, Holub, 1993) are stated contents 200 000 ng.Hg.g^{-1} in soils near Rudňany. These results suggest that decontamination of Hg in soils is necessary at site Rudňany and more detailed investigation is required for the further remediation. In transect Žiar, the Hg_{total} concentrations in forest floors were the highest in Ladomerská Vieska and Vtáčnik, approaching the Slovak guideline value A (300 ng.Hg.g^{-1}) for Hg contaminated soils (in sense of Resolution of Ministry of agriculture of the Slovak republic No. 531/1994-540). The Hg_{total} concentrations in forest soils from the both sites declined from the forest floor to the mineral soil more steeply than those from the other forest sites, suggesting the influence of atmospheric pollution, since the both sites are near to the aluminium smelter and the brown-coal power plant, respectively. Anthropogenic Hg pollution affected the other soils in transect Žiar and Banská Štiavnica little, because the Hg_{total} concentrations were at a similar level to the soils regarded as less contaminated or remote (Liu et al., 1996; Schwesig et al., 1999).

At most sites investigated, MeHg and Hg_{total} had higher concentrations in forest soils than in meadow soils. The Hg deposition in forest areas was generally much higher than in the open area, because litterfall and Hg atmospheric deposition to the foliages except for the wet precipitation may result in extra Hg input to forest soils (St. Louis et al., 1996; Schwesig et al., 2000). The high Hg_{total} concentrations in the deep horizons of soils in Banská Štiavnica reflected geogenic Hg (Bedrna, 2002). Despite of geogenic Hg, the vertical distribution of MeHg and Hg_{total} in the upland soil seems mainly governed by soil organic matters. In most forest soils, both MeHg and Hg_{total} concentrations were highest in upper soils and decreased dramatically with increasing depth. The soil organic matters may complex Hg compounds and retain them strongly. Thus, only little amount of Hg compounds may leach into deeper horizons. The carbon contents in meadow soils were lower comparing to forest floors (Table 1) and the bioturbation may prompt transport of Hg compounds. Therefore, the decrease of MeHg and Hg_{total} concentrations with the depth in most meadow soils was mild in comparison with those in forest soils. The higher Hg_{total} concentrations in some meadow soils as compared to forest soils (e.g. in Prestavlky) may be explained by additional input through organic and inorganic fertilization. However, we are not aware of the past management of these soils and the related Hg input.

The origin of MeHg in the terrestrial soils is more likely to be the atmospheric deposition. In situ Hg methylation seems to be less important in the terrestrial soils than in the wetland soils due to the higher demethylation rate in aerobic compared to anaerobic environments (Ullrich et al., 2001). Nevertheless, the MeHg from atmospheric deposition alone cannot explain the high concentrations of MeHg in the Rudňany soils, which comparable to those in wetland soils (Schwesig et al., 1999), because the MeHg concentrations in precipitation vary much less spatially compared to Hg_{total} (Downs et al., 1998). The high MeHg

concentrations in Rudňany soils suggest Hg methylation as a source of MeHg in the highly contaminated upland soils. High Hg_{total} concentrations in contaminated soils may stimulate Hg-methylation, even though high Hg_{total} concentrations may inhibit the microbial methylation activity or enhance the demethylation rates in soils (Gilmour, Henry, 1991 and Oremland et al., 1995). In Prestavlky the much higher MeHg concentrations in the meadow soils than those in the forest soils may also be explained by Hg methylation induced by higher Hg_{total} concentrations in the meadow soils.

The MeHg-to-Hg_{total} ratios in the forest floors in transect Žiar increased from Ladomerská Vieska to Prochot and declined in Vtáčnik, corresponding well to the influence of the aluminium smelter in Ladomerská Vieska and the power plant near Vtáčnik as point sources of Hg. The MeHg-to-Hg_{total} ratios in the forest floors reflect Hg pollution better than those in meadow and mineral soils, probably because forest floor retain strongly deposited Hg and the bioturbation prompt Hg transport in meadow soils. The MeHg-to-Hg_{total} ratios in Rudňany soils were an order of magnitude lower than those from the other sites, reflecting the strong Hg contaminated in Rudňany soils.

Conclusion

The Rudňany soils are still strongly contaminated by Hg even after more than 10 years of the termination of mines. In Žiar basin, most soils are regarded as uncontaminated based on Slovak guideline value. The MeHg concentrations at strongly contaminated soils could be an order of magnitude higher than those in uncontaminated soils. The MeHg-to-Hg_{total} ratios in soils reflect the Hg pollution, especially those in forest floors.

Translated by the author

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Karpinska R.: **Ortuť a metylortuť v terestrických pôdach v rôzne kontaminovaných lokalitách Slovenska.**

Ortuť a metylortuť sú globálne znečisťujúce látky, ktoré môžu ovplyvniť funkciu terestrických ekosystémov. Skúmali sme koncentrácie MeHg a Hg_{total} v pôdnych profiloch lúk a lesov v 9 lokalitách Slovenska a analyzovali vzťah medzi pomerom MeHg/Hg_{total} a znečistením Hg. Koncentrácie Hg_{total} a MeHg v pôde z lokality Rudňany (do 104.000 a 50 ng.Hg.g^{-1}) boli v porovnaní s ostatnými pôdami omnoho vyššie (do 1000 a 4 ng.Hg.g^{-1}). Pomer MeHg/Hg_{total} v lokalite Rudňany (kontaminovaná oblasť) bol nízky (medián: 0.037 %) v porovnaní s pomerom MeHg/Hg_{total} v pôdach z menej kontaminovaných lokalít (medián: 0.48 %). V Žiarskej kotline bol pomer MeHg/Hg_{total} v humusovej vrstve najnižší na miestach blízko súčasných a minulých zdrojov znečistenia. Vysoké koncentrácie MeHg v pôde v Rudňanoch naznačujú zvýšený význam metylácie Hg v terestrických pôdach kontaminovaných Hg. Inverzný vzťah bol po prvýkrát pozorovaný v terestrických pôdach v pomere MeHg/Hg_{total} a Hg_{total}. Pomer MeHg k Hg_{total} v humusovej vrstve odzrkadľuje znečistenie Hg lepšie ako tento pomer v minerálnej a lúčnej pôde.