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EVALUATION OF ELEMENTAL CONTENT IN AIRBORNE PARTICULATE MATTER IN LOW-LEVEL ATMOSPHERE OF BRATISLAVA

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Оценка элементного содержания аэрозольных частиц в низких слоях атмосферы Братиславы

В рамках проекта по изучению загрязнений воздуха в Братиславе в течение одного года проводился отбор аэрозолей и изучался их элементный состав. Для оценки уровней загрязнения и его потенциального вредного воздействия на окружающую среду в Братиславе в 2004 г. были отобраны 16 образцов воздушных фильтров. Методом инструментального нейтронного активационного анализа (ИНАА) были определены концентрации 30 химических элементов (Na , Al, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Ga, As, Se, Br, Rb, In, Sb, I, Cs, Ba, La, Sm, Dy, Tm, W, Au, Hg, Th, U). Дополнительно методом атомной абсорбционной спектрометрии (AAC) были определенены 6 элементов (Cr, Ni, Cu, Zn, Cd, Pb). Полученные результаты сравнивались с аналогичными данными для других европейских городов. С целью выявления возможных источников аэрозольных частиц рассчитывались факторы обогащения элементами земной коры. Для ряда элементов наблюдались повышенные концентрации в летние месяцы, в то время как другие концентрации оставались относительно стабильными в течение всего года.

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Evaluation of Elemental Content in Airborne Particulate Matter in Low-Level Atmosphere of Bratislava

A one-year study on total airborne particulate matter has been undertaken in the framework of air pollution project in Bratislava. Sixteen filter samples were collected in 2004 in order to evaluate the level of pollution and assess the potential environmental hazards in Bratislava. As a result of two irradiations with neutrons and four gamma-spectrometric measurements the concentrations of 30 chemical elements (Na , Al, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Ga, As, Se, Br, Rb, In, Sb, I, Cs, Ba, La, Sm, Dy, Tm, W, Au, Hg, Th, U) were determined using instrumental neutron activation analysis (INAA). Additionally, the concentrations of other 6 elements (Cr, Ni, Cu, Zn, Cd, Pb) were measured by atomic absorption spectrometry (AAS). The comparison with other European cities was carried out. Crustal enrichment factors were calculated in order to distinguish the possible sources of airborne particulate matter. For some elements elevated concentrations were observed for the summer months. The other concentrations were relatively stable over the year.

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR, and at Comenius University, Bratislava, Slovakia.

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INTRODUCTION

Elemental content of airborne particulate matter can provide important information on the degree of atmospheric pollution and further evaluation of the potential health risk to the population. Several epidemiological studies have shown positive correlation between different aerosol characteristics and increased human morbidity and mortality [5, 17]. Moreover, the airborne particles significantly influence several atmospheric processes, for example, creation of clouds, and changing the transfer of solar radiation [11, 14]. For these reasons it is necessary to know their chemical composition and physical characteristics in order to understand their behavior and impact.

Trace metals influence the toxicity of airborne particulate matter. Some heavy metals play an important role in the nutrition of plants, animals or humans (Mn, V, Cr, Ni, Cu, Zn), but if they occur in excess, they may produce certain toxic effects. The others (Cd, Hg, Pb) are toxic even in very low contents. Metals are redox active and can, therefore, induce or catalyze chemical change leading to production of free radicals, which have a known ability to cause tissue inflammation. Toxicological studies showed that ultrafine particles of less than 100 nm appear to have considerably enhanced toxicity per unit mass and that their toxicity increases as particle size decreases. The large surface area provided by ultrafine particles in contact with the lung, provides the opportunity for surface chemistry of the particles to have a profound effect [6]. Within the last few decades, experimental data have shown that high-dose as well as low-level exposure to certain metals induces subtle changes, including altered immunological competence [27].

Heavy metals are released to the environment from a great number of sources like different industrial activities or combustion of fossil fuels. These elements are components of traffic emissions and are emitted into the atmosphere in the form of fine dust and aerosols. Dry deposition velocity of heavy metals attached to airborne particles is usually less than $0.5\ \mathrm{cm \cdot s^{-1}}$ and medium residence time is supposed to be about 5 d [2]. Resuspended soil particles, volcanic aerosols and forest fires contribute to natural emissions of trace elements such as Cr, Mn, V, Cu, Mo, Ni and Zn [15]. On the other hand, the combustion of fossil fuels and traffic emissions are principal anthropogenic sources of Cu, Ni and Zn [16]. Pb, Sb, Br, Cr and V are elements associated with automotive exhaust products and domestic heating. The current anthropogenic metal emissions are up to several orders of magnitude higher than natural contents [2]. In order to undertake a strategy to improve the air quality, it is necessary to know the particular pollution sources. The fact that, a balance between sources, chemical transformations in the atmosphere, long-range transport effects and removal processes influences the composition of atmospheric particles and has to be taken into consideration.

INAA and AAS are widely used to determine the contents of trace elements in airborne particulate matter. As well known, INAA is the most suitable and appropriate analytical technique for multielemental analyses of aerosol samples [4, 9]. The main advantages of this method are high precision, high selectivity and sensitivity, small sample quantity needed, direct non-destructive method, etc.

This work has been focused on the determination of several elements in the atmospheric aerosol particles sampled in Bratislava within different seasons of the year 2004. The overall objective was to obtain the ranges of the concentrations in the total airborne particulate matter and to observe the seasonal variations within the year 2004.

MATERIALS AND METHODS

Collection of Samples. Sampling of aerosol particles has been continuously performed on a location situated at the urban site, at the meteorological station near the Faculty of Mathematics, Physics and Informatics, Comenius University, Bratislava ($48^\circ\ 09'02''$N, $17^\circ\ 04'14''$E). Bratislava is a city with a population of approximately 500,000. The chemical industry, technical glasswork, building industry, incineration plant, and car industry are located in the larger city area. Neither a ferrous or non-ferrous smelter, nor a power plant is in the vicinity.

Nitro-cellulose membrane filters (PRAGOPOR, pore size 0.85 μm) with collection efficiency approximately 100 % were used. A sampling device with an air-flow rate 30 $\mathrm{m}^3 \cdot \mathrm{h}^{-1}$ was installed at height 2.85 m above the ground. Filters were changed every week and about 3000 m^3 of air was pumped through each sample. Over the year 2004 there were 37 filter samples collected and 16 of them were analyzed for elemental content determination. Further details can be found in [24].

Sample Preparation, Activation and Measurement for INAA. After sampling the filters were divided into two parts for short- and long-term irradiations. One part was pressed into pellets using simple press-forms and heat-sealed in polyethylene foil bags ($\sim$ 0.4 g) for short-term irradiation. The special effort has to be taken to avoid contamination of the samples. Since the highest interest is in the determination of heavy metals, also the device for pressing is made from plastic.

INAA at the FLNP, JINR, Dubna, was applied for qualitative and quantitative determination of elements. Air filter samples were irradiated in the irradiation channels of the reactor IBR-2 by means of the REGATA experimental set-up. The irradiation facility is described elsewhere in [8].

Table 1. Characteristics of irradiation channels Ch1 and Ch2 of the reactor IBR [8]

Irradiation site	Neutron flux density $[(\mathrm{cm}^{-2} \cdot \mathrm{s}^{-1}) \times 10^{12}]$			T [°C] Inside Ch	Ch diameter [mm]	Ch length [mm]
	Thermal	Epithermal	Fast			
Ch1	Cd-coated	3.31	4.32	70	28	260
Ch2	1.23	2.96	4.10	60	28	260

To determine the short-lived isotopes the conventional NAA was used. Samples were irradiated for 10 min in the channel Ch2 (Table 1) and immediately measured for 5 min. It was recounted for the second time for 20 min. The time of irradiation depends on the type of sample and the element composition expected. It can vary from 3 to 20 min for the short-lived isotopes determination.

After the determination of short-lived isotopes, the samples are repacked into aluminum containers and re-irradiated in an epithermal cadmium-screened channel Ch1 for determination of long-lived isotopes. Usually samples are irradiated for 3 d. Unfortunately, the nitrocellulose material is not appropriate for long-term irradiation, and therefore, the nitrocellulose air filters were irradiated only for one hour and measured twice, after 4–5 d and 20 d of decay.

Gamma spectra were measured with high-purity germanium detectors with a resolution of 2.5–3 keV for the 1332.5 keV peak of ^{60}Co. The data processing was carried out using software developed in FLNP JINR.

Element contents were determined on the basis of certified reference materials and flux comparators [8]. For short-term irradiations in Ch2, a comparator of Au (10 μg) was employed.

For quality control, to provide content of elements yielding, short- and long-lived isotopes were determined using certified reference materials: Lichen IAEA-336, Cabbage IAEA-359 (International Atomic Energy Agency, Austria); standard reference materials: SRM-1573a (tomato leaves), and SRM-1633b (coal ash) from the NIST (National Institute of Standards and Technology, USA).

For long irradiation the reference materials were packed together with samples in each transport container. The reference material showing least deviation between measured and certified values of elemental content was chosen. Table 2 shows selected activated radionuclides, their half-lives, gamma peaks, the type of measurement used for element determination and accuracy of determination.

Table 2. Overview on elements determined in air filter samples by INAA

Element determined	Radionuclide	Half-life	Energy [keV]	Measurement*	Accuracy [%]
Na	^{24}Na	14.91 h	1368.5	B	10
Al	^{28}Al	2.241 m	1778.9	A	6
Cl	^{38}Cl	37.24 m	2167.7	B	29
K	^{42}K	12.36 h	1524.7	B	19
Ca	^{49}Ca	8.718 m	3084.0	A	14
Sc	^{46}Sc	83.83 d	889.0	D	32
Ti	^{51}Ti	5.76 m	320.0	A	34
V	^{52}V	3.743 m	1434.4	A	18
Cr	^{51}Cr	27.7 d	320.0	D	36
Mn	^{56}Mn	2.578 h	1810.7	B	5
Fe	^{59}Fe	44.5 d	1099.1	D	23
Ga	^{72}Ga	14.1 h	629.9	C	14
As	^{76}As	1.097 d	559.1	C	5
Se	^{75}Se	119.8 d	264.1	D	58
Br	^{80}Br	17.7 m	617.0	B	13
Rb	^{86}Rb	18.66 d	1076.6	D	59
In	^{116m}In	54.1 m	1294.0	B	25
Sb	^{122}Sb	2.7 d	564.2	C	51
I	^{128}I	24.99 m	442.7	A	7
Cs	^{134}Cs	2.062 y	795.8	D	65
Ba	^{139}Ba	83.1 m	165.8	B	45
La	^{140}La	1.678 d	1596.2	C	52
Sm	^{153}Sm	46.7 h	103.2	C	40
Dy	^{165}Dy	2.334 h	94.7	B	40
Tm	^{170}Tm	128.6 d	84.3	D	79
W	^{187}W	23.9 h	685.7	C	60
Au	^{198}Au	2.694 d	411.8	C	49
Hg	^{203}Hg	46.6 d	279.0	D	64
Th	^{233}Pa	27 d	312.0	D	40
U	^{239}U	23.5 m	74.7	B	27

* A: conventional NAA, short irradiation, first measurement
B: conventional NAA, short irradiation, second measurement
C: epithermal NAA, long irradiation, first measurement
D: epithermal NAA, long irradiation, second measurement

Sample Preparation and Measurement for AAS. The content of some environmentally meaningful elements such as Ni, Cu, Zn, Cd, and Pb, not detectable by INAA, was determined using atomic absorption spectrometry at the Institute of Geology, Faculty of Natural Sciences, Comenius University [12]. Cr was determined by AAS as well.

The aerosol filter samples were placed into PTFE vessels and 10 ml of concentrated HNO_3 (p.a., Merck, Germany) was added. After one week of digestion at laboratory temperature the solution was transferred into 25 ml volumetric flask and made up to the volume by redistilled water. A Perkin–Elmer Model 1100 (USA) atomic absorption spectrometer, equipped with a deuterium arc background corrector was used for determination of Cr, Ni, Cu, Zn, Cd and Pb concentrations in filter sample solutions. The concentrations of Hg were measured by Trace Mercury Analyzer TMA-254 (Czech Republic).

The quality assurance and relative determination of elemental contents were provided by the certified reference materials used NCSDC 73349 (branches and leaves, NCS China) and NCSDC 73350 (poplar leaves, NCS China). The reference materials were diluted and analyzed simultaneously with the filter samples. The volume concentrations were determined with accuracies 7% (Cr), 18% (Ni), 5% (Cu), 5% (Zn), 25% (Cd), and 5% (Pb).

RESULTS AND DISCUSSION

Elemental Content in Atmospheric Aerosol. A total of 30 elements was determined by INAA at IBR-2 reactor in Dubna in total airborne particulate

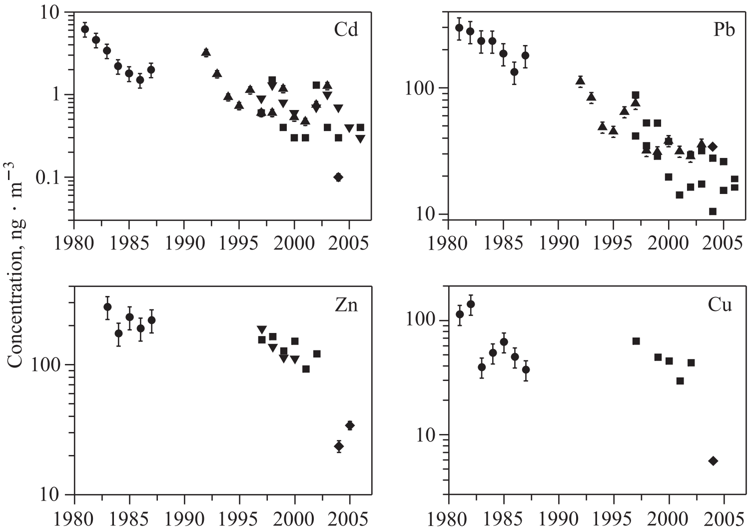

Fig. 1. Temporal variation of atmospheric concentrations of Cd, Pb, Zn, and Cu in Bratislava, circle — [1], triangle — (SHMU), diamond — our data. For comparison elemental concentrations in Prague (square) and Brno (reverse triangle) (CHMU) are also shown

matter. Another six elements were determined using AAS (Cr was determined by both techniques). These data can be considered representative of the elemental concentrations in the atmospheric aerosol of Bratislava and are unique in the Slovak Republic. For the first time such an extensive range of elements was determined at a long-term time scale.

Figure 1 shows a decreasing trend of air pollution by heavy metals in Bratislava since the year 1981. The emissions of Pb have decreased, reflecting the shift from leaded to unleaded gasoline. Since January 1, 2001 it is mandatory to use the unleaded gasoline with Pb concentration lower than $0.005\ \mathrm{g}\cdot\mathrm{l}^{-1}$ [20]. The next reason of this decreasing trend is the decline of the industry production in the Slovak Republic after the year 1989, since the fuel burning processes in thermal power plants and industry plants are a major source of atmospheric pollution with heavy metals. The emissions of pollutants were reduced also via application of more strict requirements in the environmental legislation.

Comparison to Other Locations. The results of the present study are compared to six other European cities in Table 3. The concentrations of almost all elements are lower in Bratislava compared to other localities, or comparable to Italian sites of Ponzone and Ispra, which are typical low pollution areas. Ponzone is a small town where the major part of wool industries is settled and Ispra is a residential settlement in north Italy. The city of Milan is the industrial centre of northern Italy, thus concentrations of almost all elements are obviously the highest in relevant aerosols. Only concentrations of K, Ca, Ti are higher in Colchester; Na, Al, Cl in Budapest and Ga in Krakow. Colchester is a relatively small town and with the exception of motor vehicle traffic, it is not expected to contribute high aerosol emissions to the surrounding area. Other aerosol inputs to this area may result from the London Metropolitan area and the North Sea. The main goal of the Krakow research study was to determine the contribution of traffic to the particulate air pollution, and to characterize transport of aerosols in urban area that are close to the vicinity (5 m) of a main road. The location of Szena Square in Budapest, has a more closed downtown character, and is affected by heavy traffic.

The low-level atmospheric pollution in Bratislava may be caused by the small number of pollution sources, and the high number of windy days per year is in particular typical of this location. This statement is supported by the negative correlation found between the wind velocity and the elemental concentrations in our samples [13]. The small contents of V, Ni, and As may be attributed to weaker frequency of oil and coal combustion in Bratislava, compared to other locations. Oil combustion is practically the only source of V, while Ni can also be emitted from high quality steel production plants and Ni smelters [25].

Crustal Enrichment Factors. The concept of enrichment factors (EF) was introduced by Rahn [18] to detect contributions of non-crustal sources to observed concentrations of elements. EF compares the ratio of the concentration of element

Table 3. Contents of stable elements $[\mathrm{ng} \cdot \mathrm{m}^{-3}]$ in atmosphere for eight European locations

Location	Bratislava	Colchester	Krakow	Budapest	Ponzone	Ispra	Milan
	(Slovakia)	(UK)	(Poland)	(Hungary)	(Italy)	(Italy)	(Italy)
	INAA,	[7]	[26]	[23]	[21]	[22]	[10]
Method	AAS	PIXE	PIXE	INAA, PIXE		INAA, ET-AAS	
Na	104		172	349			
Al	189		369	857			
Cl	49		179	249			
K	195	1590	247	353	493	413	1217
Ca	179	4332	1181	2002			
Sc	0.032				0.25	1.1	0.4
Ti	7.8	322	45.3	100	41	19	147
V	0.83	4	2.19	4.2	12.1	3.5	13.9
Cr	1.1	8		3.4	7	6.5	38.6
Mn	4.9	44	17.0	27	23	14	98
Fe	252	2653	788	1969	807	511	5800
Ni	0.45			2.9	20.5	12.2	25.1
Cu	8.0	13	18.7	32.7	21.4	10.5	185
Zn	28		60.5	147	98	119	392
Ga	0.02		0.87	0.13			
As	0.30			1.62	0.77	0.99	2.3
Se	0.42		0.77	0.48	0.6	0.037	1.8
Br	3.5			8.4	4.9	11	250
Rb	0.0001				1	0.76	5.2
Cd	0.11				0.75	0.51	3.42
In	0.0010						
Sb	1.0				4.9	4.5	68.3
I	0.66						
Cs	0.045				0.05	0.24	0.22
Ba	2.1		11.9	33.4	8	19.9	103
La	0.085				1.05	0.31	1.41
Sm	0.012						
Dy	0.010						
Tm	0.10						
W	0.22				0.07	0.2	2.3
Au	0.0002						
Hg	0.064						
Pb	22		45.9	29	36	98	475
Th	0.042				0.22	0.05	0.5
U	0.012						

$c(x)$ in question to that of a selected reference element $c(\mathrm{Al})$ in a sample, and the corresponding ratio in the average composition of the Earth's crust

$$\mathrm{EF} = \frac{(c(x)/c(\mathrm{Al}))_{\mathrm{sample}}}{(c(x)/c(\mathrm{Al}))_{\mathrm{crust}}}.$$

Aluminum (Al) was used as a soils (crust) reference element. The average elemental concentrations in humus horizon of soil for Slovakia are used in the EF calculations [3].

Enrichment indicates natural votilization, marine sources and anthropogenic activities. Rahn [19] suggested classification criteria of EF: if EF < 7, then air particulate is of crustal origin, and if EF > 7, then it is of the anthropogenic one. Enrichment factors were calculated for 24 elements. The results are presented in Fig. 2. The values of EF close to one indicate the soil origin of Ba, La, Th and U. Following Rahn's criteria, one can conclude that Cu, Zn, As, Se, Cd, Sb, Hg, and Pb are of the anthropogenic origin.

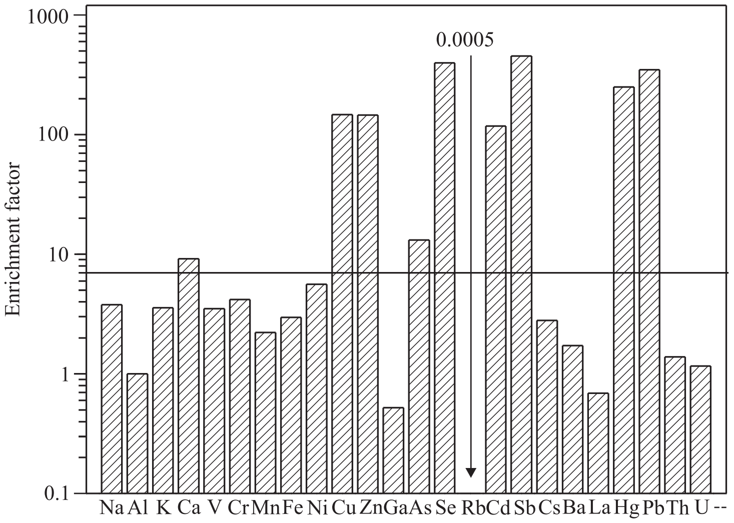

Fig. 2. Enrichment factors for investigated elements

Non-ferrous smelters are the sources of Cu, Zn, Cd and Pb. The largest source of airborne Cd in the environment, is the burning of fossil fuels such as coal or oil, and incineration of municipal waste materials. Cd may also be emitted into the air from zinc, lead, or copper smelters. Se enters atmosphere together with S due to fossil fuel combustion. Se can be also accumulated in aerosols of marine origin [3].

Seasonal Variations. Atmospheric concentrations of some elements indicate seasonal variations over the year with elevated values in summer as shown in Fig. 3. Most of them are crustal components (Ca, Ti, Mn, Ba, U), although they may also be emitted as fly-ash from the combustion of coal, and as dust from other mineral-related activities. Only Cu, according to the EF, was classified as anthropogenic. From this we can conclude that the seasonal variations of elements are not significantly influenced by human activities. The variations may be governed by atmospheric transport processes and circulation. Important role play the variations of emission rates of pollution sources within the year.

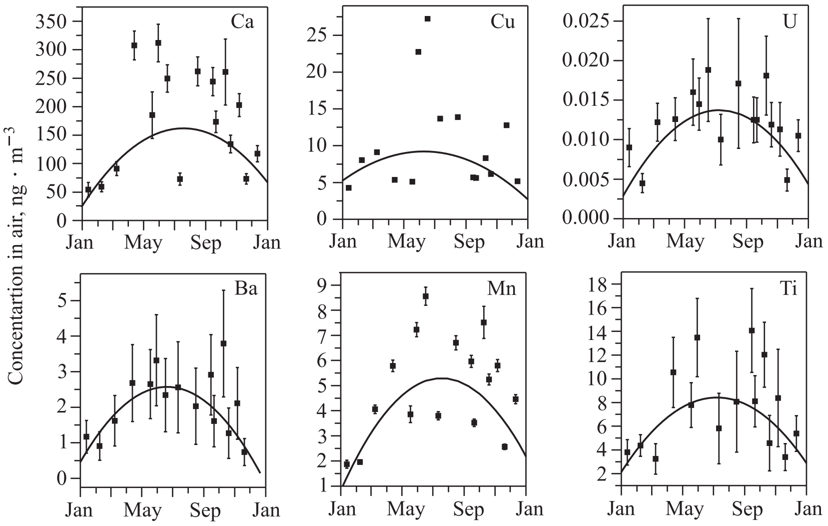

Fig. 3. Temporal variations of concentrations of Ca, Cu, U, Ba, Mn, and Ti [$\mathrm{ng} \cdot \mathrm{m}^{-3}$] in atmospheric aerosol in Bratislava during the year of 2004

The concentrations of these elements are correlated with the air temperature (the meteorological data are taken from the meteorological station at the Department of Astronomy, Physics of the Earth and Meteorology at our faculty) what indicates the relation to atmospheric circulation phenomenon [13]. The rise of concentrations might be caused by intensive vertical mixing within the troposphere typical of hot season, and hence stronger resuspension from the Earth's surface. Generally it can be concluded that the air temperature is the main meteorological factor influencing temporal variations of elemental content in low-level atmosphere. For the other elements the concentrations are relatively stable over the year. Measurements over more than one year would be needed to really prove the trend of seasonal variations.

CONCLUSIONS

Data on atmospheric concentrations of 34 elements including heavy metals, halogens and rare earths, uranium and thorium in particulate matter were obtained by INAA method. Decreasing trend of air pollution by heavy metals since the year 1981 was observed. It results mainly from the decline of the industry production in the Slovak Republic after the year 1989 and via application of more strict requirements in the environmental legislative. In comparison to seven European locations (Colchester, Krakow, Budapest, Ponzone, Ispra and Milan), Bratislava is the one with the lowest concentrations of most of the elements in atmospheric aerosol. The crustal enrichment factor analysis identified Cu, Zn, As, Se, Cd, Sb, Hg and Pb as anthropogenic pollution elements. For some elements mostly of soil origin like Al, Ca, Ti, Mn, Ba, U and for some originating from the sea salt spray like Na and Cl seasonal variations were observed. The increased concentrations in summer might be caused by intensive vertical mixing within the troposphere typical of hot season, and hence stronger resuspension from the Earth's surface. To sufficiently prove the trend of seasonal variations the measurements longer than one year would be necessary.

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