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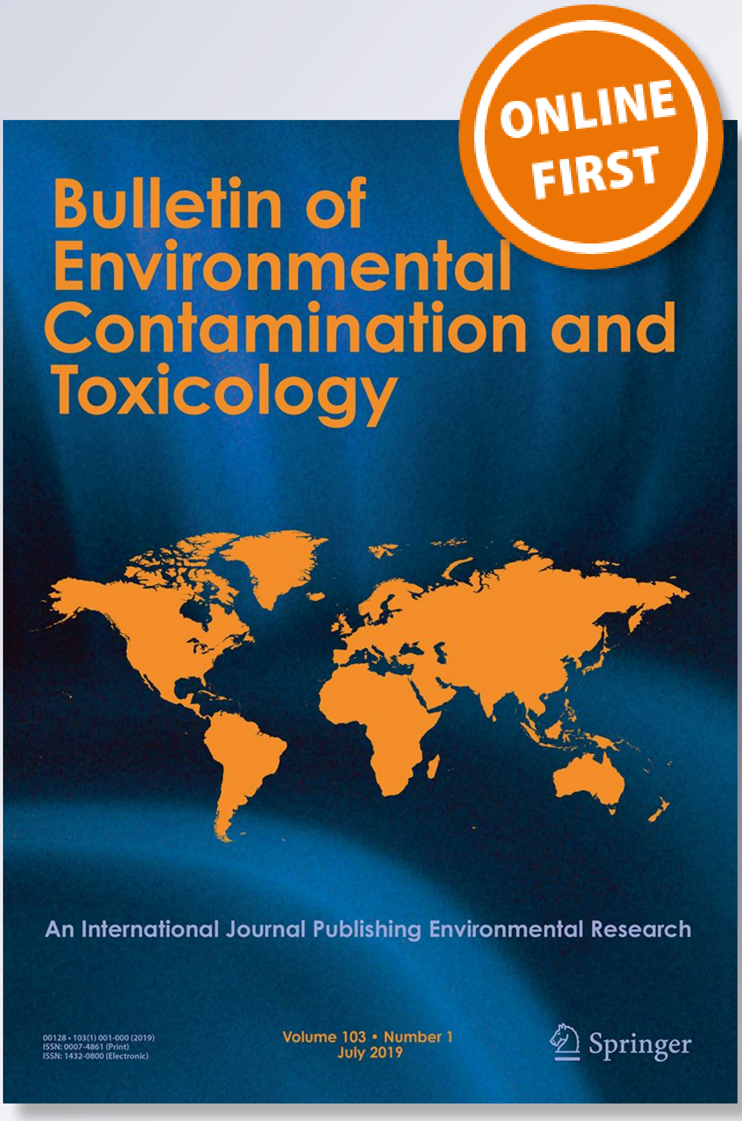

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Heavy Metal Atmospheric Deposition Study in Moscow Region, Russia

Konstantin Vergel[1] · Inga Zinicovscaia[1,2,3] · Nikita Yushin[1] · Marina V. Frontasyeva[1]

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Abstract
The air quality in north-eastern part of Moscow region was evaluated by trace metals atmospheric deposition using moss *Pleurozium schreberi* as bioindicator. Thirty six elements were determined in analyzed samples by Neutron activation analysis and Atomic absorption spectrometry. Principal component analysis was used to identify and characterize different pollution sources. Maps showing the geographical distribution of the factor scores were built using ArcGis software. Median values of the elements studied were compared with data obtained for other regions in Russia. The present survey showed that industrial activity, thermal power plants and transport still have the largest anthropogenic impact on air pollution in studied region.

Keywords Air pollution · Trace metals · Contamination factor · Biomonitoring · Atomic absorption spectrometry · Neutron activation analysis · Source markers · Factor analysis

The use of mosses as biomonitors was introduced in Scandinavian countries more than five decades ago, and it is currently widely accepted as a method to assess the atmospheric deposition of metals (Rühling and Tyler 1968). The most important property of mosses, that make them suitable measure heavy metal pollution on a large time scale, is related to the fact that they take up nutrients directly from wet and dry deposition, and the mineral adsorption occurs over their entire surface (Rühling et al. 1987, 1998).

Today, the use of native terrestrial mosses as biomonitors is a well-recognized technique in studies of atmospheric contamination (Harmens et al. 2010, 2015) and is applied as a practical mode in establishing and characterizing deposition sources (Stafilov et al. 2018; Maxhuni et al. 2016; Qarri et al. 2014).

In Russia, moss biomonitoring was applied in north-western regions: Leningrad Region, Kola Peninsula (Reimann et al. 1999), Karelia (Kharin et al. 2001). Later it was expanded to a number of central regions (Tula, Tver, Moscow, etc.), South Ural, as well as in Kaliningrad (Ermakova et al. 2004a, b; Gorelova et al. 2016; Vergel et al. 2009; Koroleva 2010).

The first attempt to apply moss biomonitoring technique in the north-eastern part of the Moscow region was made in 2004. According to data presented in Vergel et al. (2009) the main source of As, Th and U are considered heat power plants, while main source of Ag, Hg, Ni, Sb, W, are Zn were industrial activity and transport. The aim of the present study was: (i) to present results from the 2015/2016 moss survey in this region based on analysis of terrestrial moss samples by Neutron activation analysis and Atomic absorption spectrometry; (ii) to compare the obtained results with data obtained in 2004; and (iii) to identify the possible sources of heavy metals in atmospheric deposition.

Materials and Methods

Moscow region is a federal subject of the Russian Federation. With a population of 7,095,120 living on a territory of 44,300 square kilometers it is one of the most densely populated zones in the country. Moscow region borders Tver region in the northwest, Yaroslavl region in the north, Vladimir region in the northeast and east, Ryazan region in the southeast, Tula region in the south, Kaluga region in the southwest, and Smolensk region in the west. In the center the federal city of Moscow is located. Moscow region is highly

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industrialized, the main industrial branches being metallurgy, oil refining, mechanical engineering, food, energy, and chemical industries. Moscow region is characterized by the dense transport network, including roads, railways and waterways along the largest rivers (Volga, Moscow-river and Oka), lakes and reservoirs (Anonymous 2015).

Sampling was performed in June 2014 at a total of 39 sampling sites in the north-eastern part of the Moscow region (Fig. 1a) in accordance with the CLRTAP (2015) manual for moss sampling. Collected samples were cleaned of foreign materials adhered to the surface of the samples such as tree bark, lichens, soil dust, and dead materials and dried at 40°C to constant weight.

Sample preparation for neutron activation analysis and atomic adsorption analysis was done in accordance with procedure described in Zinicovscaia et al. (2017) study.

The elemental composition of moss samples was determined by Neutron activation analysis (NAA). NAA is a high sensitive bulk analysis method with multi-element capability (Pomme et al. 1997). NAA was carried out at the pulsed fast reactor IBR-2 of the Frank Laboratory of Neutron Physics, JINR, Dubna, Russia (Frontasyeva 2011; Pavlov et al. 2016). A total of 33 elements (Na, Mg, Al Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Zn, As, I, Se, Mo, Br, Rb, Sr, Sb, Cs, Ba, La, Ce, Sm, Hf, Tb, W, Th, and U) were determined using both short and long time activation. More details concerning irradiation time and gamma spectra processing can be found in Zinicovscaia et al. (2018).

Due to low sensitivity of NAA for Cd and Pb and low concentration of Cu in analyzed samples, their content was determined using a Thermo Scientific™ iCE™ 3000 Series Atomic absorption spectrometer with electrothermal (graphite furnace) atomization. The calibration solutions were prepared from a 1 g/L stock solution (AAS standard solution; Merck, Germany).

The quality control of NAA results was assured by simultaneous analysis of samples and standard reference materials: NIST 1549 (Apple leaves), NIST 1547 (peach leaves), NIST 1572 (Citrus leaves), NIST 1632c (Trace elements in coal), NIST 2710 (Montana Soil) and NIST 2711 (Montana Soil). The obtained values for concentrations of standard reference materials differed from the certified values in the range (2%–10%).

In the case of AAS, quality control was performed by using the standard reference materials NIST 1570a (Spinach Leaves) and NIST 1575a (Pine Needles). The method of standard additions was applied, and it was found that the recovery of the investigated elements ranged between 96.5% and 103.9%.

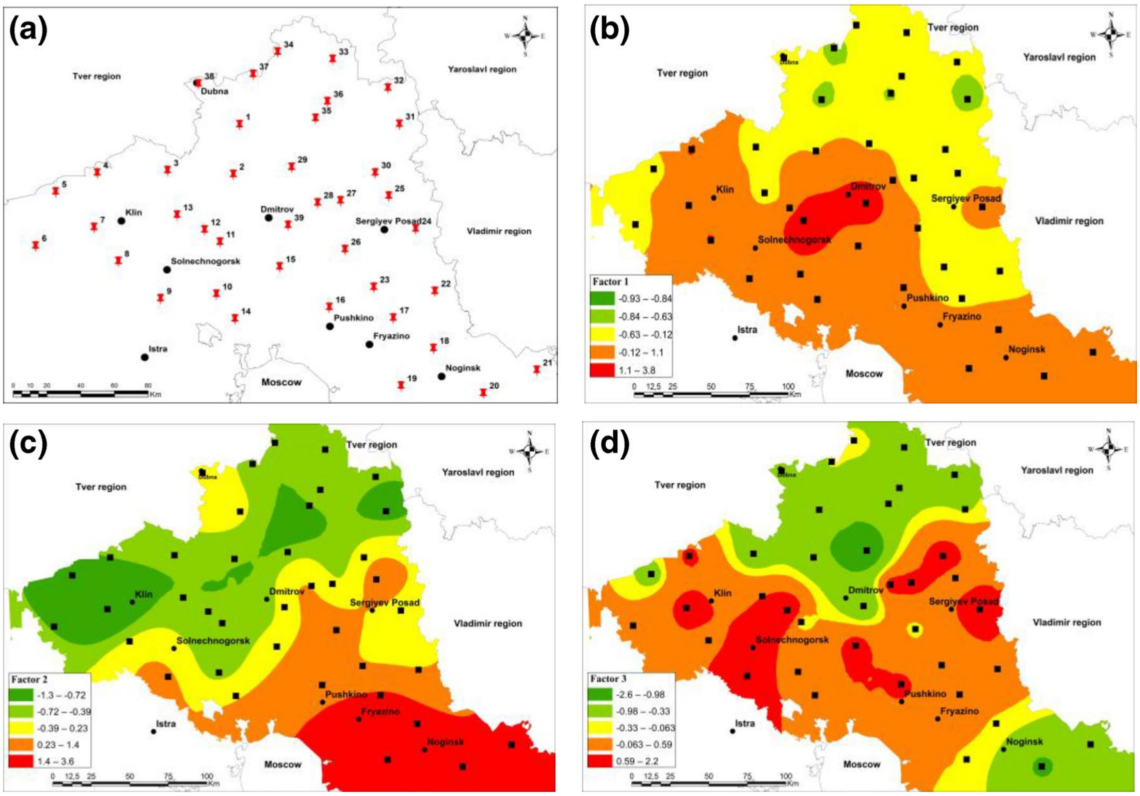

Fig. 1 Maps showing the sampling points (**a**) as well as the distribution of Factors 1 (**b**), 2 (**c**) and 3 (**d**) respectively

Descriptive statistics and multivariate statistics were done by means of Stat-Soft Statistica 7. Factor Analysis (FA) was used to investigate association of chemical elements and perform variable reduction. Prior to FA, data deviating from normal distribution were transformed using natural logarithms.

Maps showing the geographical distribution of the factor scores were built using ArcGis software.

Results and Discussion

The results of descriptive statistics analysis of content of heavy metals in the moss samples along with the corresponding data from similar studies carried out in Moscow region in 2004 (Vergel et al. 2009) as well as Tula, Ivanovo and Tikhvin regions are shown in Table 1.

The most important parameters, such as mean, median, minimum, maximum, coefficient of variation (CV%), the first and third quartiles were calculated. According to data presented in Table 1, Al, Cl, Sc, Ti, Sb, La, Ce, Sm, Tb, Hf and Th show high values of variability (CV% > 75%). The main source of these elements can be considered soil. For other determined elements CV value was less than 75%, indicating low spatial variability and thus likely dominant influence of regional source types. The Mann–Whitney test was applied to reveal differences between data obtained in present study and in Vergel et al. (2009) work. Statistically significant differences ($p < 0.05$) between 2015 and 2004 data were obtained for Cl, Ca, K, Co, As, Mo, and Hf, for other elements there were no significant differences ($p > 0.05$).

In the studied region content of potentially toxic elements (Ti, V, Fe, Co, Ni, Cu, Zn, As, Sb and U) were higher in comparison with Tikhvin region, while Cr was significantly lower. In Tikhvin region the main source of Cr, Fe, Ni and V is ferroalloy plant located in Tikhvin (Vergel et al. 2014). In comparison with Tula region the obtained mean values for all elements, except for Mn and Sb, were lower. In Tula region are functioning around 470 enterprises of chemical, metallurgy, defense, machine-building industry, fuel and energy and mining complex, which are the main sources of air pollution in the region. The main source of V, Zn and Mn can be considered metallurgical enterprise "Vanadium-Tula". Ferrous metallurgy, metal processing and defense industries, as well as ore processing are the main source of Fe, Cr, Co, Cu, Ni, As, Cd, Mo, Sr, Ba, Sb, W and Sm (Gorelova et al. 2016; Ermakova et al. 2004b). High content of As, U, Th is associated with coal mining, refining and coal combustion industries, while of Cl, Br and I with production of chlorine and organic compounds (Ermakova et al. 2004b). The release of V, Ni and lanthanoid elements reflect the impact of refinery industry and heavy oil combustion (Moreno et al. 2010). In Ivanovo region, content of Mn, Co and Ni was higher than in Moscow region, while content of Al, Cr, Fe, Zn and Sb was lower. The main source of air pollution in Ivanovo region is engineering plant located in Rodniki town (Dunaev et al. 2018).

Factor analysis with Varimax raw rotation was applied to identify possible sources of the elements determined in the mosses. The matrix of the dominant rotated factors is given in Table 2. Three factors with the variability of the established elements of 70% have been identified: Factor 1 which includes the elements Na, Mg, Al, Cl, Ca, Sc, Ti, V, Cr, Fe, Ni, Co, As, Sr, Cd, La, Ce, Sm, Tb, Hf, Th and U; Factor 2 with the elements Ni, Cu, Mo, Sb, Pb and W and; Factor 3 with elements Mn, Zn, Br, Rb, I, Ba, and Cs.

Factor 1 (Fig. 1b) covers 44% of the total variability and can be defined as lithogenic, geogenic and anthropogenic association. Presence of Al and insoluble trace elements such as Sc, REE, Hf, and Th in this group is associated with the influence of mineral particles released to the atmosphere by wind, and their spatial distribution mainly depends on urban activities not related to industrial activities, for example transportation (Stafilov et al. 2018; Barandovski et al. 2015). High contents of elements of this association have been found in samples taken from the vicinity of Klin, Dmitrov, Solnechnogorsk and satellite towns (Pushkino, Fryazino, Noginsk).

Vanadium (V), Ni, La and Ce are associated with heavy fuel oil combustion, such as in oil refineries, institutional heating, power generation using fuel oil, or oil refineries (Moreno et al. 2010). Arsenic, U, Cd, and Cr are considered as indicators of emission from fossil fuel combustion processes (Zinicovscai et al. 2018; Korzekwa et al. 2007). Main sources of Fe, Co, Cd, As, Ni, and U can be considered metallurgy industry and mechanical plants. Calcium release in the atmosphere is associated limestone mining located in Dmitrov and Noginsk. High content of the elements of the first factor near Klin may be associated with cement production company and location of the polygon for holding municipal solid waste "Aleksinskii mine", while near Dmitrov and Solnechnogorsk by functioning of mechanical plants and location of oil stores. High content of toxic elements in the eastern part of Moscow can be explained by well-developed industrial activity, thermal power plants and developed network of transportation routes. In this area are located metallurgical and mechanical plant, engineering plants (Mytischi, Balashikha, Zheleznodorozhniy, Electrostal), cement production (Podolsk, Pushino), ect.

Factor 2 (Ni, Cu, Mo, Sb, Pb and W), with 14% of the total variance, is an anthropogenic factor and is associated with urban and industrial activities. From Fig. 1c it appears that the highest contents of these elements are found in samples collected in the eastern part of Moscow where the main part of industrial enterprises are located.

Table 1 Descriptive statistic of measurements for moss samples as well as comparison of the obtained values with literature data the present study and literature data.

Element	Range	Md	Mean ± st. dev	Q_1	Q_3	CV	Moscow region (Vergel et al. 2009)	Tikhvin region (Vergel et al. 2014)	Tula region (Gorelova et al. 2016)	Ivanovo region (Dunaev et al. 2018)
Na	71–726	230	255 ± 140	143	343	57	240	166	436	190
Mg	1010–4970	1860	2095 ± 800	1530	2530	39	1963	395	3496	2530
Al	0.045–0.69	0.12	0.01 ± 0.12	0.07	0.2	82	0.08	0.06	0.46	0.08
Cl	47–1040	108	220 ± 250	82	271	115	182	102	499	161
K	0.23–1.7	0.85	0.9 ± 0.35	0.7	1.1	40	1.08	0.7	1.3	0.98
Ca	0.24–0.9	0.47	0.5 ± 0.17	0.3	0.6	33	0.35	0.2	0.75	0.36
Sc	0.08–1.3	0.26	0.2 ± 0.25	0.14	0.35	81	0.16	0.10	0.9	0.29
Ti	35–1050	146	210 ± 200	97	234	96	–	69	363	
V	0.94–11	2.5	2.9 ± 1.80	1.73	3.43	63	2.3	1.3	8.8	2.4
Cr	0.72–9.5	3.2	3.7 ± 1.9	2.49	4.72	52	3.1	17	8.9	2.3
Mn	76– 48	347	390 ± 200	260	494	52	405	422	210	706
Fe	0.03–0.34	0.1	0.1 ± 0.07	623	1320	60	0.08	0.04	0.32	0.06
Co	0.14–2.1	0.56	0.6 ± 0.36	0.35	0.78	58	0.34	0.25	1.1	0.82
Ni	0.66–8.4	3.2	3.7 ± 2	2.12	5.54	56	2.4	2.5	3.9	5.2
Cu	2.9–21	7.1	7.3 ± 3	5.2	8.1	44	–	4.3	–	–
Zn	21–159	50	56 ± 25	40	64	45	51	34	52	44
As	0.12–1.1	0.32	0.3 ± 0.17	0.22	0.4	52	0.19	0.12	0.8	0.4
I	0.36–2.4	1.5	1.4 ± 0.5	0.99	1.82	37	–	0.6	1.4	–
Mo	0.06–1.9	0.18	0.2 ± 0.4	0.13	0.23	59	0.37	–	0.24	0.11
Se	0.09–0.4	0.16	0.16 ± 0.06	0.12	0.2	36	0.18	0.09	0.18	0.13
Br	0.7–5.1	1.9	1.9 ± 0.55	1.5	2.3	29	1.7	1.7	3.8	7.4
Rb	7.4–65	19	20 ± 8	14	25	40	17	26	12	15
Sr	7.7–50	17	17 ± 7	12	24	40	17	10	33	24
Cd	0.1–0.7	0.3	0.3 ± 0.13	0.22	0.39	43	–	–	0.5	0.43
Sb	0.04–1.5	0.3	0.4 ± 0.3	0.19	0.50	80	0.22	0.09	0.17	0.19
Cs	0.1–0.7	0.18	0.2 ± 0.11	0.14	0.22	53	0.16	0.3	0.34	0.15
Ba	7.5–188	48	53 ± 38	18	72	73	48	21	60	87
La	0.3–4.2	0.84	1.1 ± 0.89	0.42	1.23	85	0.67	0.38	3.1	0.61
Ce	0.6–7.5	1.6	2.1 ± 1.6	1.06	2.45	75	2.1	0.71	6.4	1.1
Sm	0.04–0.7	0.13	0.17 ± 0.14	0.072	0.23	83	0.12	0.05	0.5	0.15
Tb	0.005–0.1	0.02	0.02 ± 0.02	0.007	0.025	94	0.013	0.008	0.08	0.02
Hf	0.07–2.4	0.29	0.4 ± 0.51	0.17	0.56	112	0.15	0.05	0.9	0.12
W	0.1 – 200	0.47	0.4 ± 36	0.2	0.55	69	0.35	0.16	0.26	0.2
Pb	0.12–2.2	0.67	0.7 ± 0.43	0.4	0.96	58	–	–	–	–
Th	0.07–1.5	0.23	0.3 ± 0.27	0.11	0.34	93	0.19	0.09	0.95	0.16
U	0.01–0.19	0.08	0.08 ± 0.04	0.04	0.11	55	0.08	0.03	0.24	0.07

in mg/kg, except for Al, K, Ca and Fe where contents are expressed in % dw

Md median value, *SD* standard deviation, *CV* coefficient of variation, Q_1 25th percentile, Q_3 25th percentile

Tungsten (W) is a heavy metal that is widely used in industry, such as steel, aerospace, and electronics industry (Plattes et al. 2007). Molybdenum (Mo) is mainly used in metallurgical applications and in the production of flame retardants, pigments and catalysts for high temperature chemical processes (Korzekwa et al. 2007; Verbinnen et al. 2012). Thus, Ni, Cu, Mo and W can be emitted by metallurgical plants, machinery companies, plants for the nuclear fuel production located in Mytischi, Balashikha, Zheleznodorozhniy, Electrostal, etc. The enormous traffic density in this zone also contributes to the increased

Table 2 Matrix of the first three factors loading

Element	F1	F2	F3	Element	F1	F2	F3	Element	F1	F2	F3
Na	**0.94**	0.27	0.04	Ni	**0.58**	**0.50**	0.19	I	0.20	0.32	**0.51**
Mg	**0.82**	−0.06	0.46	Co	**0.86**	0.32	0.15	Ba	0.51	0.27	**0.54**
Al	**0.92**	0.01	0.18	Zn	0.30	0.47	**0.50**	Cs	0.30	−0.10	**−0.69**
Cl	**0.52**	−0.22	0.38	Cu	0.24	**0.80**	0.05	La	**0.97**	0.06	0.11
K	0.25	0.11	0.17	As	**0.77**	0.23	0.19	Ce	**0.95**	−0.06	0.11
Ca	**0.56**	0.29	0.50	Se	0.34	0.29	0.14	Sm	**0.93**	0.11	0.08
Sc	**0.97**	0.16	0.06	Br	0.42	−0.08	**0.63**	Tb	**0.98**	0.04	0.10
Ti	**0.66**	−0.03	0.34	Rb	−0.16	−0.05	**− 0.51**	Hf	**0.88**	−0.08	0.09
V	**0.89**	0.22	0.17	Sr	**0.72**	0.29	0.47	W	0.24	**0.89**	0.00
Cr	**0.83**	0.37	−0.05	Mo	0.04	**0.95**	0.01	Th	**0.96**	0.07	0.11
Mn	0.30	−0.01	**0.68**	Cd	**0.52**	0.06	0.37	U	**0.59**	0.35	0.33
Fe	**0.91**	0.35	0.04	Sb	0.12	**0.90**	0.10	Pb	−0.5	**0.53**	0.19

Statistically significant values are given in bold (r2 > 0.5, $p < 0.01$)

pollution of the region. The elements such as Pb, Mo, and Sb are characteristic for road traffic (Aničić et al. 2007).

Factor 3 (Fig. 1d) represents 11% of the total variance. This factor is loaded by Mn, Zn, Br, I, Ba and negatively loaded by Cs and Rb. Manganese does not occur as the free metal and is found in more than 100 minerals including various sulfides, oxides, carbonates, silicates, phosphates (Qarri et al. 2014). Moscow region is rich in carbonate and phosphate rocks, consequently Mn may originate from these rocks. Barium can be emitted in atmosphere by petroleum industry, steel industry, production of semiconductors (Kravchenko et al. 2014). Bromine and iodine emission can be associated with chemicals, pharmaceutical, fertilizers and pesticides production. Negative load for Cs and Rb indicates on participation of these elements in ion-exchange processes.

For a better interpretation of the results, various descriptors such as the contamination factor C_F, Geo-accumulation Index I_{geo} and pollution load index (*PLI*) were calculated (Zinicovscaia et al. 2016). As reference material for calculation moss collected in the Central Forest Nature Reserve was used.

All tree indices were calculated for the 13 elements and are presented in Table 3. According to the scales proposed by Fernández and Carballeira (2001) there is no contamination with As, Mn, Ni, and Cd since C_F less than 2 can easily be obtained from natural variation (Qarri et al. 2015). Chromium, V, Zn, Cu and Fe are associated with the third scale (slightly polluted areas), while Sb and Co are associated with the moderate pollution. C_F value obtained for Pb indicates on moderate pollution, while value obtained for W point at severe pollution. I_{geo} data are in good correlation with C_F values and point out an unpolluted to severe industrial pollution localized around main urban and industrial centres. The distribution map of PLI index values (Fig. 2) shows that main air pollution sources are located in immediate proximity to Moscow. The values of PLI confirm generally moderate pollution in the studied region.

Table 3 Mean values of the contamination factor C_F and Geo-accumulation Index I_{geo} for studied region

Element	C_F	I_{geo}	Element	C_F	I_{geo}
Mn	0.46	−1.69	Co	3.56	1.25
As	0.39	−1.93	W	13	3.12
Fe	2.52	0.75	Cu	2.44	0.7
Sb	3.81	1.35	Ni	0.91	−0.71
V	1.62	0.11	Cr	2.36	0.65
Cd	0.86	−0.8	Pb	5.5	1.9
Zn	1.22	−.29			

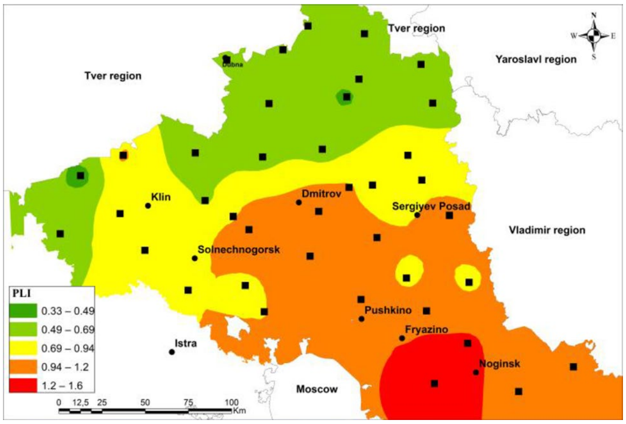

Fig. 2 The map of the most polluted sites based on PLI ranges of 13 elements

Using neutron activation analysis in combination with Atomic absorption spectrometry it was possible to determine 36 major and trace elements in the studied moss samples. According to contamination factor and geo-accumulation index values Cr, Fe, Co, and W cause moderate or severe environment pollution. The main sources of air pollution in Moscow region can be considered well developed industrial activity, thermal power plants, and transport.

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