

Integrated Approach to Determine Background Concentrations of Chemical Elements in Soils

G. O. Tepanosyan*, O. A. Belyaeva, L. V. Saakyan, and A. K. Sagatelyan

Center for Ecological–Noospheric Studies, National Academy of Science of the Republic Armenia, Yerevan, 0025 Armenia

*e-mail: gevorg.tepanosyan@cens.am, ecocentr@sci.am

Received March 14, 2016; in final form, September 19, 2016

Abstract—Analysis and calculation techniques of geochemical background concentrations of chemical elements in various systems is of paramount importance for applied geochemistry. Herein we assume the geochemical background as the average of natural variations in the concentrations of chemical elements determined at a territory that highly probable does not host any natural and/or anthropogenic sources of contaminating chemical elements. In the context of determining geochemical background, our research was focused on determining the concentrations of heavy metals in soils in the city of Yerevan with the application of an integrated approach. Comparison of the obtained background values with the mean concentrations of elements in the upper continental crust yields representative data, and the application of various statistical tests ($\pm 3\sigma$, $\pm 2\sigma$, and boxplots) is proved to equally efficient.

Keywords: geochemical background, heavy metals, urban soils

DOI: 10.1134/S0016702917060106

THEORETICAL ANALYSIS

Determining background concentrations of chemical elements in various biotic and abiotic systems is one of the pivoting problems of modern environmental science (Gałuszka, 2007). Usage of background concentrations of chemical elements in ecologic–geochemical studies makes it possible to distinguish between elements of natural and anthropogenic provenance, elucidate the spatial distribution of the concentrations of the elements, outline anomalies, identify the sources of the pollution, and estimate its intensity.

The concept of *geochemical background* was introduced by exploration geochemists to differentiate between positive and negative anomalies of chemical elements in a rock matrix (Matschullat et al., 2000). Progress in science resulted in the emergence of its branches that utilize geochemical techniques, but no consensus has been reached so far on the definition of the term of geochemical background. The concept of geochemical background is actively disputed in the literature and attracts much attention (Saet et al., 1990; Baize and Sterckeman, 2001; Bates and Jackson, 1984; Matschullat et al., 2000; Perez-Sirvent et al., 2009; Zhao et al., 2007).

Exploration geochemistry currently assumes background concentrations to be ranges of the concentrations of chemical elements, but not their anomalous concentrations, in certain naturally occurring materials, such as rocks, soils, plants, and water (Perez-Sirvent et al., 2009). A similar definition of geochemical back-

ground is also presented in the geological glossary (Bates and Jackson, 1984). From the standpoint of ecological geochemistry, it is important that stress is placed on the natural origin of the variations in the concentrations of chemical elements, and hence, background is defined in ecologic–geochemical studies as the mean values of the natural variations in the concentrations of chemical elements within a certain area that is homogeneous in geological and/or landscape–geochemical sense, with these variations produced without anthropogenic addition of chemical elements or any other external disturbances (Saet et al., 1990).

An interesting methodological approach to this problem was suggested in (Matschullat et al., 2000), in which the absence of any conventionally recognized definition of geochemical background was stressed and was suggested to regard it as a relative measure of various natural and anthropogenically-introduced concentrations of chemical elements and their compounds in a set of samples. In contrast to the foregoing definitions, this one stresses the applied aspect of the concept of geochemical background.

Depending on the landscape–geochemical specifics of territories to be studied, local and regional geochemical background are commonly distinguished, but again, no consensus is reached so far as to how to determine these values (Baize and Sterckeman, 2001; Matschullat et al., 2000; Portier, 2001). The reasons for this are the spatiotemporal variations in the concentrations of chemical elements, different geochemi-

cal characteristics of their chemical species, and the facts that these values can be determined by various techniques and with the application of various approaches (Kabata-Pendias and Pendias, 2001), and moreover, different techniques can be used to determine geochemical “normality” and “anomaly”.

Currently two major approaches are applied to determine background concentrations of elements: a direct one (geochemical or empirical) and indirect (statistical) (Gałuszka, 2007; Matschullat et al., 2000). When the direct (geochemical or empirical) techniques are used, the analyzed samples are collected at a certain “background territory”, which is believed not to be anthropogenically impacted (Baize and Sterckeman, 2001), and the background is thereby assumed as a constant value (arithmetic mean or median). Consequently, the determined values suggest a hypothetical basic criterion regardless of the natural variations in the concentrations of chemical elements in the examined environmental component. The distribution function is also thereby ignored. Direct techniques used to quantify a geochemical background involves certain drawbacks and is highly dependent on the expertise of the researcher, and hence, the conclusions can be biased (Matschullat et al., 2000).

When the indirect techniques are applied, the database is filtered to search for and then reject extremal values using various statistical tests. It is thereby assumed that the values caused by anthropogenic impact are “smoothed off” by filtering, so that the final data set reflects the natural distributions. Although the statistical techniques are advantageous in certain respects [a wide circle of tests, graphical representations, availability of several computer programs for data processing (Gałuszka, 2007)], these methods are criticized as ignoring the spatiotemporal variability of the geochemical data and the uncertainty of the preparatory processing and analysis of the samples, permissible errors (Rencz et al., 2006).

To eliminate certain drawbacks of the methods, an integral approach is applied, which involves a combination of direct and indirect techniques: analyses of the samples collected from an assumed background area are statistically processed (Gałuszka, 2007; Perez-Sirvent et al., 2009; Zgłobicki et al., 2011).

Herein the *geochemical background* is assumed to be the mean value of the natural variations of concentrations of chemical elements found in a territory that is thought to highly probably not host sources of anthropogenic or natural contamination with the elements.

The aim of this research was to determine the background concentration of heavy metals in soils of the city of Yerevan with the application of an integrated approach in the context of the problem of geochemical background.

The third regular 1 : 25000 soil geochemical survey was carried out in Yerevan in 2012 and required determining the background concentrations of heavy metals.

The city of Yerevan (40°10'40" N; 44°30'45" E) was selected for this study because its territory provokes much interest of geochemists. The geochemistry of this area has been studied for more than three decades, and heavy metals were always recognized as persistent local contaminants throughout this whole time span (Saakyan, 2008; Sagatelyan, 2004). Moreover, according to the geochemical classification of population centers (Perel'man and Kasimov, 1999) on the basis of concentrations of toxic chemical elements and their compounds in the soil-forming rocks, the territory of the city is classed with background areas (Kasimov et al., 2004) because its concentrations of toxic elements are closely similar to the mean concentrations of these elements in the Earth's crust (Sagatelyan, 2004). Anthropogenic activities at the territory gave rise to an anthropogenic biogeochemical province, which is characterized (in contrast to natural provinces) by high concentrations of chemical elements foreign to this landscape, with the distribution dynamics of these elements controlled solely by anthropogenic geochemical fluxes (Sagatelyan, 2004).

METHODS

Sampling Techniques, Preparatory Processing of the Samples, and Analysis

To determine the geochemical background of heavy metals in soils at the territory of Yerevan, soils were sampled at a background plot northeast of the city, outside its administrative boundary. The geology and landscape geochemistry of this plot are similar to those at most of the territory of the city (Fig. 1).

Soils were sampled and the quality of the fieldwork was controlled in compliance with standardized operation procedures (SOP) worked out at the Center for Ecological–Noospheric Studies, National Academy of Science of the Republic Armenia, on the basis of (*Methodical...*, 1982; Saet et al., 1990) and conventionally applied internationally standardized techniques ISO (Fomin and Fomin, 2001; ISO 10381-5:2005) and US EPA (US EPA Field sampling guidance document #1205). The survey of the background plot was conducted at a scale of 1 : 50000 (four soil samples per 1 km²). Soil samples were taken from horizon A₀ (depths of 0–5 cm). Each sample was produced by mixing no less than three and no more than five individual point samples collected within a single quadrangle of the sampling grid. We have collected 51 soil samples (Fig. 1).

In the laboratory, the samples were air dried, crushed, screened through sieves with 1 mm mesh, pulverized in a ball mill according to internationally adopted technique ISO-11464 (Fomin and Fomin, 2001), and then placed in hermitically sealed to analyze later. Bulk concentrations of Ti, Fe, Ba, Mn, Co, V, Pb, Zn, Cu, Ni, Cr, Mo, Hg, As, Ag, and Cd in the

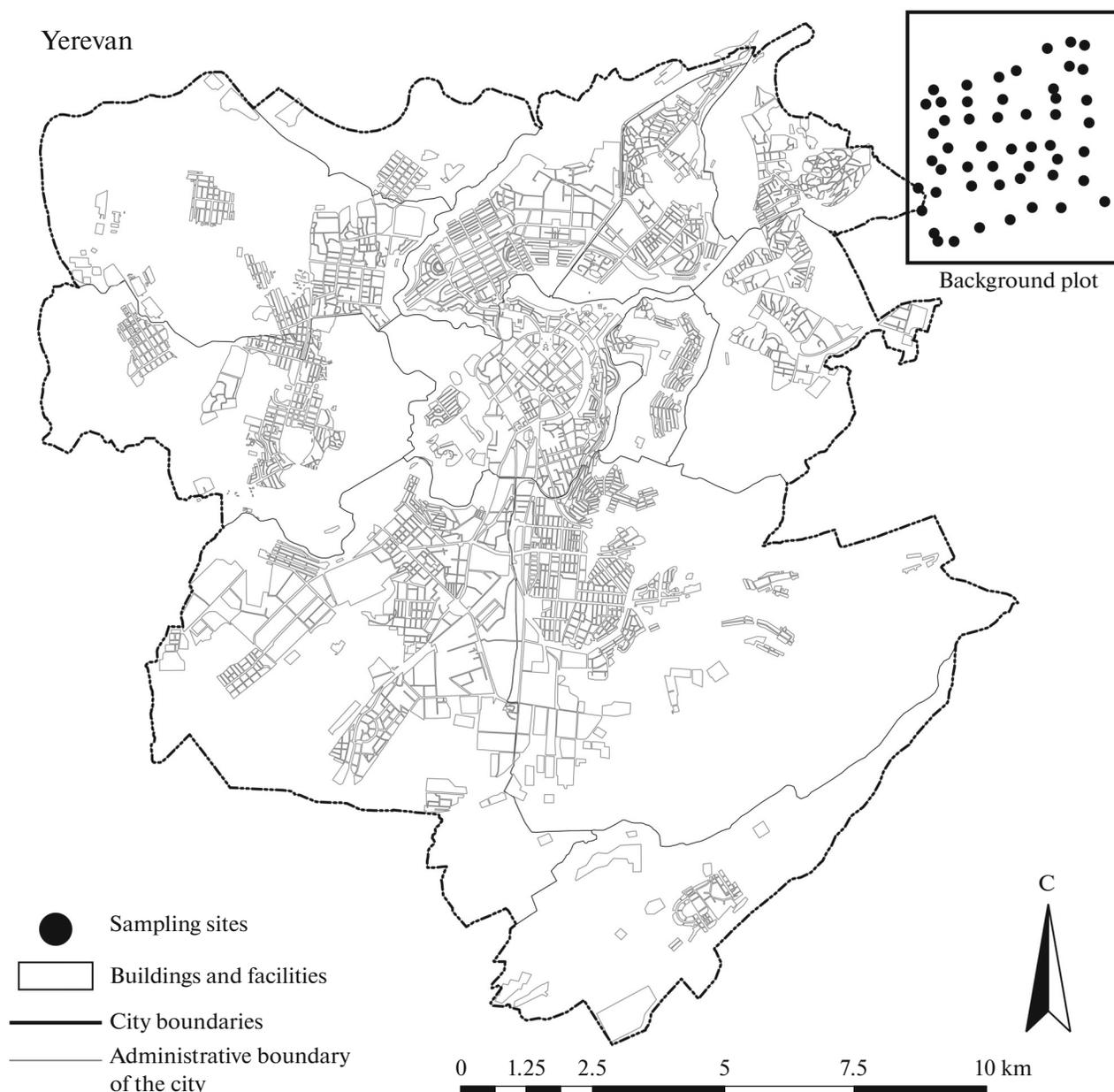


Fig. 1. Location map of the city of Yerevan and background plot.

samples were analyzed by XRF on an Olympus Innov X-5000 (USA) according to the standard technique EPA 6200. Each sample was analyzed three times, with the standard deviations of all elements not exceeding $\pm 5\%$. The quality was controlled (protocols QA/QC) using standard reference samples: soil standards NIST 2710a and NIST 2711a and a blank sample (SiO_2), which were certified at the National Institute of Standards and Technology (NIST) in the United States. The limits of detection (LOD), accuracy, and reproducibility of the analyses are presented in Table 1. The accuracy of analysis is defined as the percent dif-

ference (PD) between an analysis of a standard reference sample and its certified concentration (US EPA Method 6200):

$$PD = ((MV - CV)/CV) \times 100,$$

where MV and CV are the measured and certified values, respectively, and the permissible difference between the measured concentration of any element and its certified value, which should be no greater than 20%. The reproducibility of the analyses was determined using the relative percent difference (RPD)

Table 1. Limits of detection, accuracy, and reproducibility of X-ray fluorescence analysis (XRF)

Element	Limit of detection, ppm	Accuracy*, %	Reproducibility, %
Ag	0.02	13.0	10
As	0.1	9.2	6.4
Ba	20	4.8	2.4
Cd	0.03	5.7	10.0
Co	5	14.3	5.0
Cr	1.4	3.3	10.0
Cu	10	7.2	4.8
Fe	40	13.3	2.2
Hg	0.01	15.6	15.8
Mn	2	8.2	2.0
Mo	0.2	—	11.8
Ni	7.3	3.1	20.0
Pb	5	2.7	8.1
Ti	2	8.8	1.7
V	1	17.5	2.8
Zn	5	7.5	14.0

* No more than $\pm 20\%$ permissible (US EPA Method 6200).

between the results of replicate pairs of analyses of a single sample (Cicchella et al., 2005):

$$RPD = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100,$$

where X_1 and X_2 are the results of the pair of analyses; the permissible difference should be no greater than 20%.

Within the framework of the program of maintaining and controlling the quality of analytical operations according to methods EPA 6200 (US EPA Method 6200) and ISO 5725-1 (Fomin and Fomin, 2001), external quality control was conducted: 10% of the soil samples were analyzed by atomic absorption spectroscopy (AAS) on a PerkinElmer Analyst 800 for eight metals: As, Hg, Cd, Pb, Cr, Ni, Ag, and Mo. The correlation coefficients between the concentrations determined by XRF and AAS was 0.88–0.97, which verifies the reasonably good quality of the geochemical screening studies.

Statistical Analysis and Techniques Applied to Determine Background Concentrations

The data were statistically processed with the SPSS 20 program package to evaluate the absolute errors of the statistical distribution parameters of the concentrations of chemical elements in soils at the background plot (Beus et al., 1976). The distribution law of the concentrations of the elements was tested by

the $\pm 3\sigma$ method (Alekseenko et al., 2002; Beus et al., 1965, 1976). The geochemical background was estimated by the $\pm 3\sigma$, $\pm 2\sigma$, and boxplot techniques (Beus et al., 1976; Matschullat et al., 2000; Reimann et al., 2005; Reimann et al., 2008).

In statistically processing the data, values smaller than the limits of detection (<LOD) were substituted by $\frac{1}{2}$ LOD (Table 1) if no less than 15% such values occurred in the sample (Reimann et al., 2008).

The calculated background concentrations were then compared with the mean concentrations of chemical elements in the upper continental crust (Grigor'ev, 2002) and the mean concentrations in basalt (Meuser, 2010).

RESULTS AND DISCUSSION

Table 2 reports statistical distribution parameters of the concentrations of chemical elements in samples from the background plot. The percentage errors of the distribution parameters of heavy metals is reasonable low, with relatively high values found only for Mo, Pb, Ag, and Cd due to the appreciable variations in the concentrations of these elements. The variation coefficients are no greater than one, which justifies the selection of the assumed background plot (Johnson et al., 2011).

The small differences between the median and mean values (Table 2) suggests a certain symmetry, but the results of the $\pm 3\sigma$ method indicate that this sym-

Table 2. Statistical parameters of concentrations (ppm) of chemical elements in samples from the background plot ($n = 51$)

Element	Mean	Median	Standard deviation	Minimum	Maximum	Variation coefficient	Relative error, λ % ($p < 0.05$)
Ti	4667.73	4779.00	478.78	3602.00	5632.00	0.10	3.00
V	45.94	46.69	4.32	36.43	52.34	0.09	3.00
Cr	72.47	73.67	14.73	42.91	100.39	0.20	6.00
Mn	790.83	784.00	91.52	587.00	951.00	0.12	4.00
Fe	38450.46	38759.67	3669.16	29288.00	45928.33	0.10	3.00
Co	15.72	15.90	1.67	11.90	19.03	0.11	3.00
Ni	32.29	31.00	10.68	17.00	83.33	0.33	10.00
Cu	40.49	39.00	5.97	31.00	58.00	0.15	5.00
Zn	84.09	74.40	30.65	53.10	218.90	0.36	10.00
As	0.70	0.70	0.05	0.58	0.81	0.07	2.00
Mo	2.30	2.70	1.31	0.50	4.20	0.57	16.00
Ba	406.89	406.00	26.18	320.00	464.67	0.06	2.00
Hg	0.02	0.02	0.01	0.01	0.03	0.30	9.00
Pb	5.28	4.44	2.35	2.75	12.91	0.44	13.00
Ag	0.33	0.38	0.20	0.00	0.64	0.61	17.00
Cd	0.44	0.31	0.35	0.05	1.00	0.80	22.00

metry is within permissible ranges for Ti, V, Cr, Mn, Fe, Co, Cu, As, Mo, Ba, Hg, Ag, and Cd, i.e., the distribution of these elements complies with the normal law. Further statistical processing of the data has revealed that the Ni and Zn distributions are significantly affected by extremal values (Fig. 2), which led us to logarithmically transform the original data. As a result, we have found a lognormal distribution of these chemical elements.

Table 3 reports the geochemical background concentrations of the elements calculated by coupled application of the $\pm 3\sigma$, $\pm 2\sigma$, and boxplot methods. Data of different methods on As and Hg in Table 2 show practically identical results: 0.69 and 0.017 ppm, respectively. Similar values for Ti, V, Cr, Fe, Co, Ni, Cu, Zn, Mo, Ba, Ag, and Cd were obtained by the $\pm 3\sigma$ and boxplot methods (Table 3). The geochemical background values calculated with the $\pm 3\sigma$ and boxplot methods differ insignificantly. The geochemical background values obtained by the $\pm 3\sigma$, $\pm 2\sigma$, and boxplot methods are 785.47, 788.86, and 790.06 for Mn and 4.90, 4.80, and 4.70 for Pb, respectively.

However, the maximum differences between the geochemical background values obtained by the methods for Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, and Ba are within 0.15–1.2%, and the analogous differences for Mo, Pb, Cd, and Ag are higher: 1.7, 4.2, 4.0, and 3.2%, respectively.

This led us to conclude that the geochemical background concentrations obtained by the three methods for the heavy metals in question in soils at the background plot are similar. In view of this, we gave no preference to any of the techniques and assumed the

Table 3. Geochemical background calculated for soils at the background plot by the $\pm 3\sigma$, $\pm 2\sigma$, and boxplot methods

Element	Background, ppm		
	Calculation method		
	$\pm 3\sigma$	$\pm 2\sigma$	boxplots
Ti	4643.01	4653.72	4643.01
V	45.74	45.92	45.74
Cr	70.89	71.50	70.89
Mn	785.47	788.86	790.06
Fe	38274.91	38361.89	38274.91
Co	15.63	15.68	15.63
Ni	30.28	30.65	30.28
Cu	40.09	39.91	40.09
Zn	78.42	78.29	78.42
As	0.69	0.69	0.69
Mo	1.74	1.77	1.74
Ba	407.97	407.47	407.97
Hg	0.017	0.017	0.017
Pb	4.90	4.80	4.70
Ag	0.25	0.26	0.25
Cd	0.31	0.32	0.31

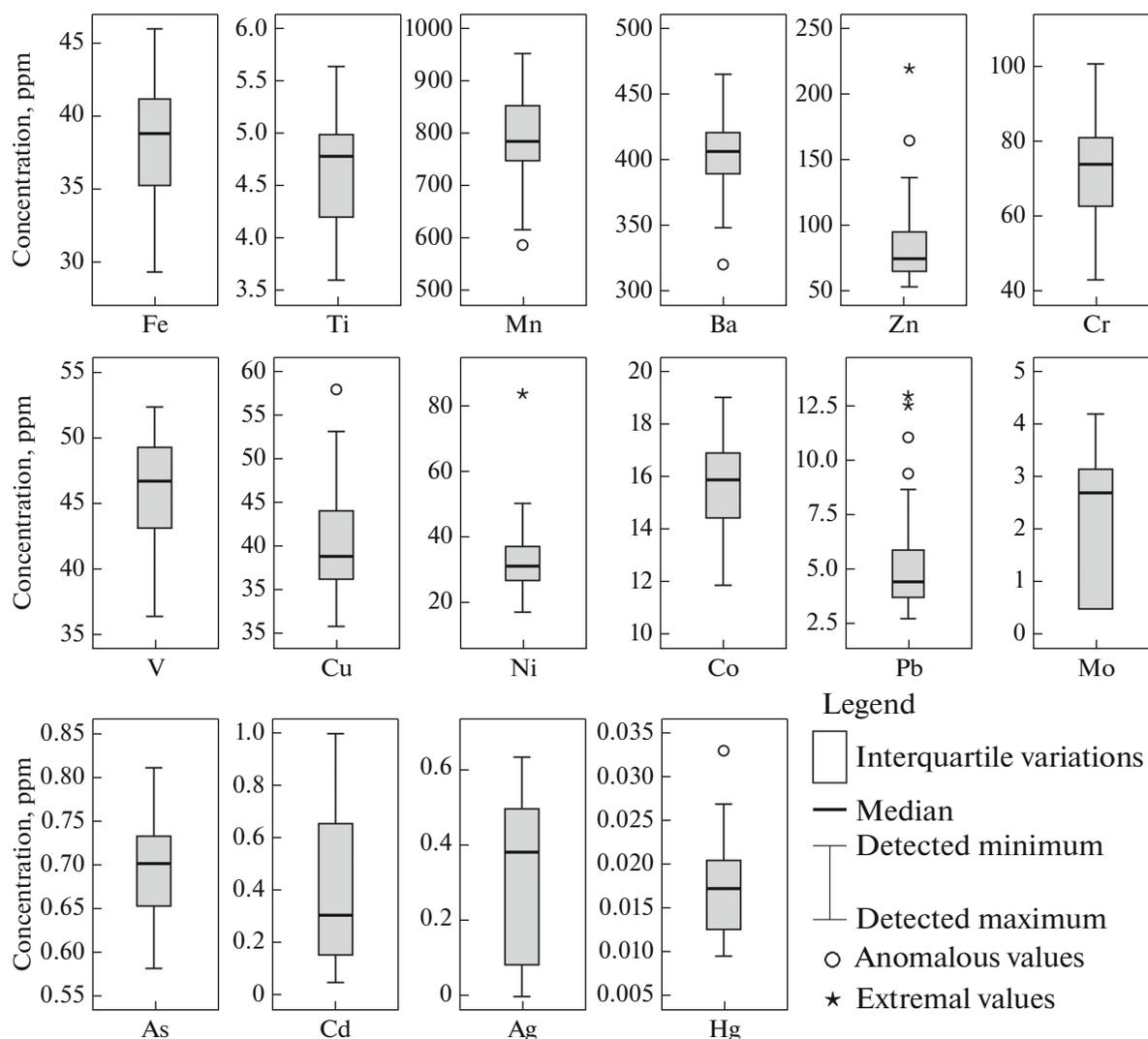


Fig. 2. Boxplots of the concentrations of heavy metals in soils at the background plot.

geochemical backgrounds to be the mean values of the three values for each element (Table 4). Note that the methods of $\pm 3\sigma$ (Beus et al., 1965, 1975), $\pm 2\sigma$ (Gałuszka, 2007; Matschullat et al., 2000; Reimann et al., 2005; Tume et al., 2014; Zgłobicki et al., 2011), and boxplots (Reimann et al., 2005; Reimann et al., 2008) for calculation geochemical background were employed by numerous researchers, but never all these techniques were applied simultaneously within the framework of a single project.

The geochemical succession of the ratios of the background concentrations of chemical elements to their concentrations in the upper continental crust (Grigor'ev, 2002) is as follows: $Ag_{(1.9)}-Ti_{(1.3)}-Sr, Mn, Mo_{(1.2)}-Cd, Cu, Zn_{(1.0)} > Zr, Fe, Co_{(0.9)}-Ba_{(0.8)}-Ni, Cr_{(0.5)}-Rb, V_{(0.4)}-Pb_{(0.3)}-Hg_{(0.2)}-As_{(0.1)}$.

Considering that lavas in the background plot are mostly of basaltic composition, concentrations of elements in the soils were also compared with the mean concentrations in the soil-forming basalt (Meuser, 2010). The background Cd, Hg, and Pb concentrations in the soils are, respectively, 3.1, 1.7, and 1.6 times higher than in the basalt. The background concentrations of other elements are much lower than their mean concentrations in the basalt. The geochemical succession of the ratios of the background concentrations of chemical elements in the territory of Yerevan to their mean concentrations in the soil-forming basalt is as follows: $Cd_{(3.1)}-Hg_{(1.7)}-Pb_{(1.6)} > Zn_{(0.78)}-As_{(0.46)}-Co_{(0.45)}-Cu_{(0.44)}-Cr_{(0.35)}-Ni_{(0.20)}-V_{(0.18)}$.

In conclusion, it should be mentioned that the geochemical background of the heavy metals discussed above can be used as reference material for soils in the territory of Yerevan.

Table 4. Geochemical background concentrations of heavy metals in soils at the territory of Yerevan, mean concentrations of the elements in the upper continental crust (Grigor’ev, 2002), and mean concentrations of the elements in the soil-forming basalt (Meuser, 2010)

Element	Concentration, ppm		
	background	mean concentration in the upper continental crust	mean concentration in the soil-forming basalt
Ti	4646.6	3400	—*
V	45.8	120	250
Cr	71.1	150	200
Mn	788	670	—
Fe	38304	40600	—
Co	15.7	17	35
Ni	30.4	62	150
Cu	40	39	90
Zn	78.4	78	100
As	0.69	6.5	1.5
Mo	1.75	1.5	—
Ba	407.8	510	—
Hg	0.017	0.072	0.01
Pb	4.8	17	3
Ag	0.25	0.13	—
Cd	0.31	0.3	0.1

* Dashes mean no data.

CONCLUSIONS

The geochemical background of the soils of Yerevan, a city with multiple randomly scattered sources of anthropogenic contamination, was determined using an integral approach. Comparing the calculated background values with the mean concentrations of the elements in the upper continental crust and the soil-forming basalt, we proved that a combination of the methods provides statistically representative results and that the $\pm 3\sigma$, $\pm 2\sigma$, boxplot statistical tests are equally efficient in solving such problems.

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Translated by E. Kurdyukov