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Multivariate analysis of the contents of metals in urban snow near traffic lanes in Novi Sad, Serbia

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Abstract: During December 2009, snow was collected at twenty two locations across the urban area of Novi Sad, directly from roads and from traffic islands near crossroads. The total metal concentration was determined for each of ten metals (Al, Ca, Cu, Fe, K, Mn, Na, Ni, Pb and Zn) using the ICP-OES analytical technique. Ni was found to have the lowest concentration (0.0265 mg dm^{-3}). Na was the metal with the highest concentration (10786 mg dm^{-3}), which was the consequence of sodium chloride being used as a de-icing salt on the roads. The metal with the second highest concentration at all locations was Ca; this was most likely the result of soil dust. The Spearman rank correlation coefficients between analyzed metals were calculated to determine how the concentrations of the metals were related. Cluster analysis was performed on the obtained data sets, using both the hierarchical and partitioning methods in order to identify associations among metals and/or locations. It was shown that traffic density was not the most important factor that caused the differences between the concentrations of the metals in the samples.

Keywords: metal concentration; snow; traffic; correlation; cluster analysis.

INTRODUCTION

There has been an interest in investigating the chemistry of urban snow in recent years.[1–4] Road runoff after the melting of snow presents a significant source of pollution in the environment. Snow melting usually exerts an acute impact on waters receiving road runoff. Snow on the road contains high concentrations of pollutants from both natural and anthropogenic sources including

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heavy metals.[1,5,6] A part of this snow is ploughed to the roadside and therefore is a contributing factor in the pollution of urban land in the vicinity of the roads, especially traffic islands.

The natural sources of road snow pollutants are materials from surrounding soils (windblown soil dust), particles from atmospheric deposition and biological materials from vegetation.[7,8] Road-deposited particulate matter can be attributed to anthropogenic sources, such as emissions from industrial plant processes, vehicle exhaust emissions and wear of automobile parts.[9–11] Traffic intensity is one of the most important factors influencing pollution in road runoff.[12,13]

Significant differences in the levels of pollutants in urban snow have been found in studies performed in different regions, thus indicating the need for local data. This is caused by differences in climate, surrounding land-type and land-use, vehicular traffic density, neighbouring industrial plants, *etc*.[14] Pollutant load is very high during snow melting in regions where snow cover lasts several months.[6] In northern countries, various efforts have been made to address this problem, but little is known about the pollution of urban snow in the Balkan region. In regions where the snow cover does not remain for longer periods, the environmental impact of snowmelt is not as acute but it should be investigated. The present case study was performed in the city of Novi Sad (Serbia). In regions such as Serbia, snow has a short lifespan and usually does not remain for longer than ten days. The intention of the study was to determine whether there was any significant pollution in urban road/roadside snow.

The melt water with all its pollutants is often directly discharged without treatment into the receiving water. In all urban areas in the city of Novi Sad, water that enters the sewage system discharges into the Danube River without any chemical or mechanical treatment. During snowmelt, pollutants are usually rapidly released from snow piles[6] and the pollutant load can rise suddenly thus having a significant impact on the receiving watercourses.[3] The toxicity of metal pollutants in receiving waters is mostly connected to the dissolved fraction, but knowledge about total metal concentration is also very important.[15–17]

In this study, two statistical techniques: descriptive statistics and multivariate analysis of experimental data were employed. Correlation analysis is often performed to estimate the extent of a relationship between any pair of variables, *e.g.*, in a group of selected metals.[18,19] Cluster analysis, a multivariate statistical technique, has been widely used to interpret complex data and to identify sources of pollution.[20,21]

The objectives of the work presented in this paper were: to analyze the metal concentration in snow collected: *i*) directly from the road near crossroads and *ii*) from traffic islands in the urban area of Novi Sad and to identify pollution sources using statistical analysis of the data.

EXPERIMENTAL

Novi Sad is the second largest city in Serbia, located on the Danube River in the southern part of The Pannonian Plain. The urban area of the city is 129.7 km². The population was estimated to be 370,000 at the end of 2009. Novi Sad has a moderate continental climate, with an average of 22 days of complete sub-zero temperatures. January is the coldest month, with an average temperature of −1.9 °C.

From December 20 until December 22, 2009, snow was collected at twenty-two locations across Novi Sad. The snowfall started on December 15 and continued intermittently until December 23. The snow cover persisted for ten days due to cold weather, with temperatures constantly below 0 °C. The snow depth was in the range of 18–25 cm. Sampling locations are shown on the map of Novi Sad presented in Fig. 1.

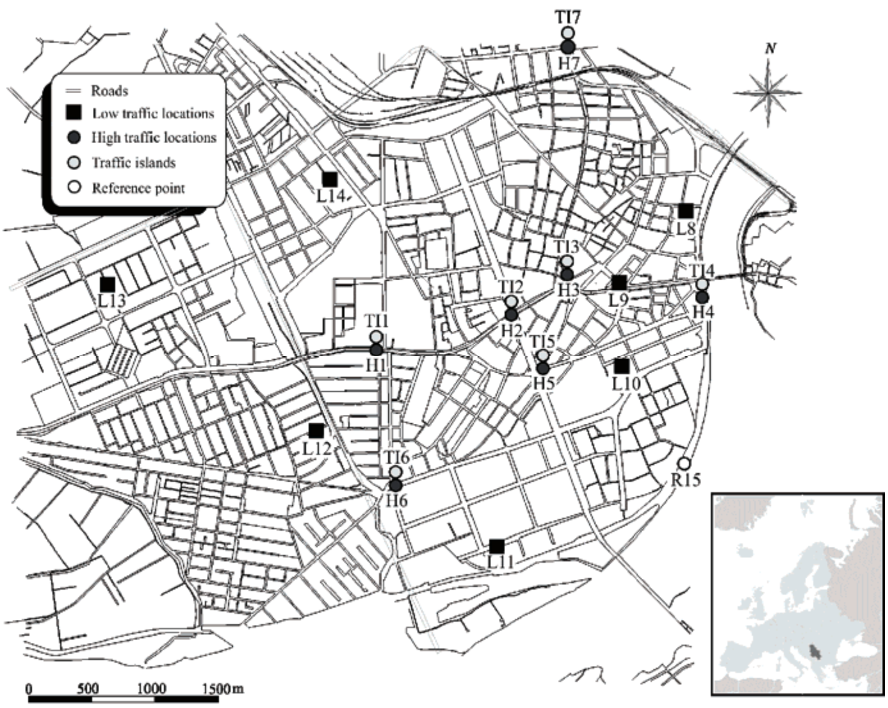

Fig. 1. Sampling locations on the map of the city of Novi Sad.

Fourteen of the sampling points were situated directly on roads near crossroads and were divided into two subgroups. For subgroup one, "high traffic", the average traffic volume at the crossroads was in the range of 400–800 vehicles per hour, while for the second, "low traffic", it was 30–150 vehicles per hour. Seven sampling points were located on traffic islands separating lanes of opposing traffic. Sampling locations in the high traffic subgroup, low traffic subgroup and traffic island subgroup are denoted by H1–H7, L8–L14 and TI1–TI7, respectively. The reference sampling point is R15.

Samples were collected using polyethylene (PE) bags, PE trowels and PE gloves. Prior to sampling, the trowels were cleaned with nitric acid and rinsed with distilled water. One–two litres of snow were collected at each sampling site, except at the reference site where fifteen litres of snow was collected.

Before analysis, samples were allowed to melt slowly. To avoid potential problems with detection limits, only the reference sample was 50-fold pre-concentrated. Pre-concentration was realized by non-boiling evaporation. The melted samples were acidified with nitric acid to a pH of less than 2. Sub-samples of 100 mL, taken from homogenized acidified samples, were acid digested with nitric acid. Prior to analysis, the sub-samples were filtrated through a blue ribbon filter (Whatman) with an average retention 2–4 µm. The filtrates were used for measurements. The total concentration of metals was measured using ICP-OES (Vista-Pro, Axial; Varian) in accordance with the US EPA method 200.7:2001. Quality assurance and quality control (QA/QC) were conducted by performing laboratory blanks and NIST standard reference material 1643e (trace elements in water). Deviations from the obtained results were within ±10 % of the certified value. The limits of detection for the examined metals were: 15 (Al), 1 (Ca), 6 (Cu), 4 (Fe), 5 (K), 0.5 (Mn), 1.5 (Na), 3 (Ni), 15 (Pb) and 3 µg dm^{-3} (Zn).

All statistical analyses were performed using the software package Statistica 10.[22] Non-parametric correlation analysis was performed due to the non-normal distribution of some parameters and because non-parametric correlation analysis is less sensitive to outliers. The Spearman rank correlation coefficients between the analyzed variables were calculated. Only correlation coefficients significant at the 0.05 level are discussed within this paper. A cluster analysis was also performed on the obtained data sets, using both hierarchical and partitioning methods by minimizing the squared Euclidean distances.

RESULTS AND DISCUSSION

Basic statistical parameters of the data are given in Table I. Concentrations of metals at reference site were: 2.6 (Al), 545 (Ca), 1.5 (Cu), 11 (Fe), 129 (K), 1.6 (Mn), 603 (Na), 0.7 (Ni), 1.3 (Pb) and 14 µg dm$^{–3}$ (Zn). Concentrations of all metals investigated from locations H1–H7, L8–L14 and TI1–TI7 were several

TABLE I. Results of multi-element analysis

Parameter	Element									
	Al	Ca	Cu	Fe	K	Mn	Na	Ni	Pb	Zn
					Low traffic					
c_{min} / mg dm^{-3}	0.61	102.70	0.10	4.68	3.23	0.27	233.80	0.030	0.06	0.34
c_{max} / mg dm^{-3}	2.96	586.50	0.26	29.39	19.24	2.01	5104.50	0.058	0.49	1.39
c_{mean} / mg dm^{-3}	1.86	347.43	0.18	17.09	10.66	1.10	1924.30	0.042	0.27	0.92
SD / mg dm^{-3}	0.94	164.48	0.06	8.57	5.49	0.52	1676.18	0.011	0.13	0.32
					High traffic					
c_{min} / mg dm^{-3}	1.27	192.90	0.14	9.81	5.67	0.52	1149.80	0.027	0.20	0.45
c_{max} / mg dm^{-3}	6.14	849.40	0.32	30.82	34.25	1.99	10786.00	0.086	1.37	2.37
c_{mean} / mg dm^{-3}	2.38	453.34	0.20	15.97	11.96	0.95	3494.91	0.045	0.46	0.94
SD / mg dm^{-3}	1.70	273.62	0.06	7.01	9.96	0.48	3393.07	0.024	0.41	0.64
					Traffic islands					
c_{min} / mg dm^{-3}	0.22	34.85	0.04	1.78	0.88	0.10	235.30	–	0.05	0.12
c_{max} / mg dm^{-3}	0.74	116.30	0.09	5.72	3.20	0.30	2174.50	–	0.23	0.43
c_{mean} / mg dm^{-3}	0.35	59.34	0.05	3.01	1.92	0.15	983.31	–	0.10	0.19
SD / mg dm^{-3}	0.18	26.65	0.02	1.35	0.73	0.07	624.20	–	0.06	0.11

orders of magnitude higher than ambient background levels measured at the reference site. Reference sampling point (R15) was located in a residential area in Novi Sad near the Danube River away from roads and industrial plants. Therefore, the reference sample was believed not to be directly polluted by traffic or human activities, but affected only by atmospheric pollutants. The lowest metal concentration was found for Ni (0.0265 mg dm^{-3}) while, as expected, the highest was for Na (10786 mg dm^{-3}). The high concentration of Na, which exceeds the other concentrations by orders of magnitude, is a consequence of sodium chloride being used as a de-icing agent on roads during winter. Sodium chloride has only been considered a pollutant since 2001.[23] The metals, in descending order of mean concentration were Na > Ca > Fe > K > Al > Mn > Zn > Pb > Cu > Ni. This order was the same for both high traffic and low traffic locations. The order was the same for traffic islands, but the Ni concentration was below the limit of detection. This indicates only small variations in the composition of the snow at the selected locations in urban areas of Novi Sad. It also indicates that the samples were in general impacted by the same source(s). It is considered that the main pollution sources in the area studied in this work may be traffic, oil refining and combusting for home heating in some parts of the city. The only large facility is the oil refinery located in the northeast part of the city. The second highest concentration of Ca at all locations was, most probably, due to contribution from soil particles containing minerals. Soil dust is the most likely source of this high level of Ca as well as of K. Another source of Ca and K was the de-icing salt. Namely, according to its Quality and Safety Certificate, the de-icing salt used by the Public Utilities Company of the city of Novi Sad contained 0.281 Ca, 0.040 Mg and 0.012 % K. The salt was imported from Ukraine (produced in salt mines in Soledar).

The calculated ratio between mean values of metal concentration at high and low traffic locations, shown in Fig. 2a, indicates the impact of traffic volume. The most significant differences in concentrations of elements that mainly originate from anthropogenic sources at locations near the high and the low traffic crossroads were for Na (1.73) and Pb (1.58). For a number of years, only unleaded petrol has been in use in many countries, but in Serbia, leaded petrol was still in use at the time of sample taking. Therefore, it was assumed that the lead content was mostly a result of traffic pollution. Considering de-icing salt as the main source of Na, it is evident that more salt was applied at high traffic locations.

The main source of metals at traffic island locations was snow ploughed from the roads. As expected, the mean concentration of metals at traffic islands was considerably lower compared with those at high traffic density locations. The ratio between mean values of metal concentration at traffic islands and high traffic locations was not significantly different, as can be seen in Fig. 2b.

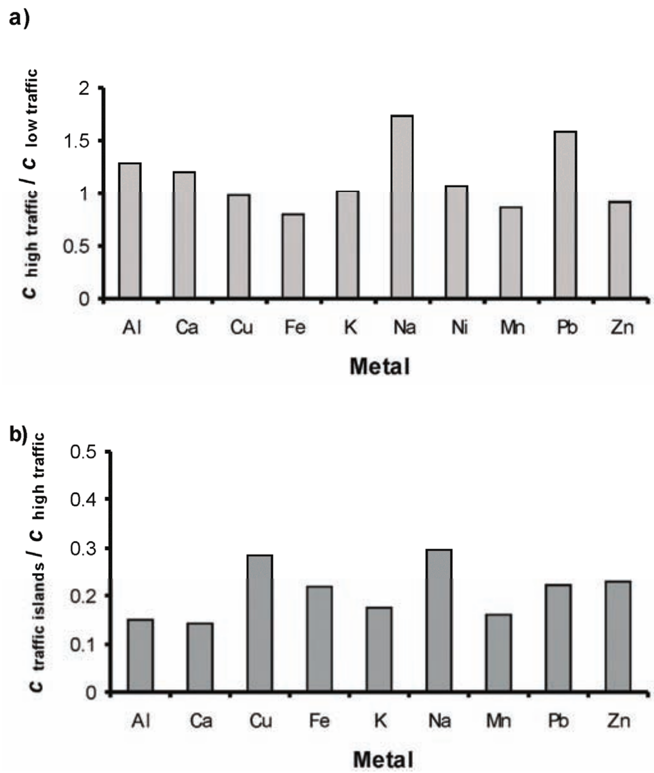

Fig. 2. Ratio between the mean values of the metal concentrations at: a) high and low traffic locations and b) traffic islands and high traffic locations.

The results of this study were compared with the results of the total metal concentrations in urban snow reported for Cu, Zn and Pb,[3] as well as for Al, Ca, Cu, Fe, Na, Pb and Zn.[1] Concerning Cu, Zn and Pb, the results of this study were in the same range as those reported in the literature.[3] Mean values of Cu and Zn were of the same order of magnitude but lower than the values measured in Innsbruck[3] for both high and low traffic locations. The mean Pb concentration at low traffic locations was approximately the same, while at high traffic locations the result of this study was higher than that reported by Engelhard *et al.*[3] The results of this study were compared with the values reported by Glen and Sansalone[1] for snow collected on highways in Cincinnati, OH (USA). The maximum concentrations for Cu, Al, Fe and Zn were one order of magnitude lower that the corresponding concentrations reported in the literature.[1] The smallest difference between the results reported here and in the literature[1] concerns Ca and Pb. The highest concentration level obtained for Na was one order of magnitude greater than that reported by Glen and Sansalone.[1]

Correlation analysis was performed to estimate the extent of the relationship between any two variables in the group of investigated metals. The Spearman rank correlation coefficient values were calculated for high and low traffic locations data and the results are given in Table II. Interestingly, all correlation coefficients significant at the 0.05 level were positive. The correlation analysis results showed the absence of significant correlations between the concentration of Na and any other metal considered in the study. This result is consistent with the fact that the total metal concentration was measured. An observation on polluted snow from other studies[23,24] was that increased values of dissolved metal concentrations are correlated with greater levels of de-icing salt concentrations. The chloride ion is highly mobile and causes the washout of pollutants, such as heavy metals, from deposited sediments.[25–27]

TABLE II. Spearman rank correlation coefficient values between the measured parameters for combined data: high + low traffic locations; * – values significant at the 0.05 level

	Ca	Cu	Fe	K	Mn	Na	Ni	Pb	Zn
Al	0.45	0.81*	0.86*	0.30	0.52	–0.03	0.87*	0.67*	0.45
Ca		0.39	0.32	0.68*	0.68	0.52	0.57*	0.85*	0.60*
Cu			0.67*	0.23	0.28	–0.28	0.73*	0.56*	0.25
Fe				0.28	0.70	–0.05	0.83*	0.51	0.64*
K					0.56	0.46	0.36	0.43	0.56*
Mn						0.28	0.64*	0.64*	0.88*
Na							0.09	0.30	0.36
Ni								0.66*	0.67*
Pb									0.51

In agreement with expectations, metals originating predominantly from natural sources were strongly correlated (at the 0.05 significance level), *e.g.*, strong positive correlations were found between Ca and K. Furthermore, the results indicated a strong positive correlation between the Fe and Ni concentrations, which could be a result of attrition of steel parts. The strong positive correlation between Cu and Ni could be a consequence of the emission of these two metals from brake-lining wear, and metal plating parts.[16,28] This was also the case for Ni and Zn, for which a strong statistically significant positive correlation was found. Potential sources of Zn are tire wear and motor oil.[10] The statistically significant positive correlation between Al–Ni, Al–Fe, Al–Cu, Fe–Cu, Fe–Zn is consistent with the fact that the traffic related emission sources contribute to the levels of these elements in the environment.[9,25]

Interestingly, a very strong, statistically significant, positive correlation was found between Ca and Pb, which means that there could be some kind of relationship between them. This result is not consistent with the mentioned assumption that the main source of lead was vehicle exhausts. As already stated, the main source of Ca at all locations was probably minerals from soil dust particles.

Cluster analysis is a very efficient tool for the identification of metal sources.[29] The main purpose of cluster analysis is to split a number of variables into groups that have similar characteristics or behaviour.[30,31] Cluster analysis can be performed either by clustering variables or samples. In multi-element analysis, obtained concentration levels are usually very different. Therefore, the data must be "prepared" for cluster analysis by using appropriate standardization techniques. The Z-scale standardization was used in this analysis. Although some cluster analysis procedures do not require the data to be normally distributed, the Box–Cox transformation[32] was performed as well. In this study, the most common approach, hierarchical clustering, was used to cluster variables. Hierarchical clustering was first performed on the data from the high and low traffic locations using the Ward method[33] with squared Euclidean distances as a measure of similarity. This approach resulted in grouping the metals into two clusters with significant differences between them. The dendrogram of the hierarchical cluster analysis of total metal concentrations at the high and low traffic locations is shown in Fig. 3. The first cluster consists of Al, Cu, Fe and Ni, divided into two sub-clusters: Al–Cu and Fe–Ni according to the degree of association between the metals. As mentioned in the subsection concerning correlation analysis, very strong statistically significant correlation coefficient values were found for pairs of metals within this group. These metals are typical road run-off pollutants and many sources for all of them are traffic related: moving engine parts and brake-lining attrition, auto body rust and wire corrosion.[25,34] The second cluster contained K, Ca, Pb, Mn, Zn and Na. Such a groupation in the second cluster was unexpected because of the close linkage between Ca and Pb, and between Mn and Zn. It is assumed that Ca and Mn mainly originated from natural geochemical sources (soil erosion products, windblown soil dust) while Pb and Zn may have derived mostly from traffic pollution (leaded gasoline, tire wear, motor oil). While interpreting the results, it was born in mind that sampling locations with high (low) anthropogenic input could additionally be influenced by high (low) natural input. In other words, high anthropogenic input does not exclude high natural input.

When hierarchical clustering was performed for all data, including those from traffic island locations, three distinct clusters were obtained. Fig. 4 displays the hierarchical dendrogram for high traffic + low traffic + traffic island data. Ni was not detected on traffic island locations and therefore was not included in the analysis. As could be seen in Fig. 4, cluster 1 contained Al, Cu and Fe; cluster 2 K, Ca, Pb, Mn, and Zn, while cluster 3 contained only Na. As already mentioned, the main source of Na is de-icing salt and the results of the multivariate statistical analysis are consistent with this fact.

The partitioning method was used to cluster sampling locations. In contrast to the hierarchical method, this method requires the number of clusters to be predetermined. Considering the vehicular traffic density as one of the primary variables that influences metal concentration, a hypothesis that all samples could be divided into two groups was proposed. The idea was to check how the samples would cluster if there was the possibility to separate them into two groups. The *K*-means algorithm with a predetermined number of clusters was used. The partitioning method was initially performed on data for both high and low traffic metal concentration values, then traffic density data (an additional variable) was added. The results were the same. One of the clusters included locations L2, L4, H5 and H7. The other included L1, L3, L5, L6, L7, H1–H4, and H6. Each cluster comprised sampling locations from both high and low traffic locations. Thus, the results showed that traffic density was neither the only nor the most important factor that divided the samples into two groups.

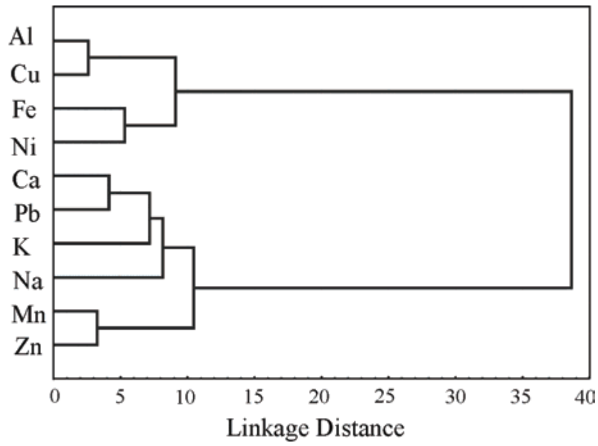

Fig. 3. The dendrogram of the hierarchical cluster analysis of the total metal concentrations for combined data: high traffic locations + low traffic locations.

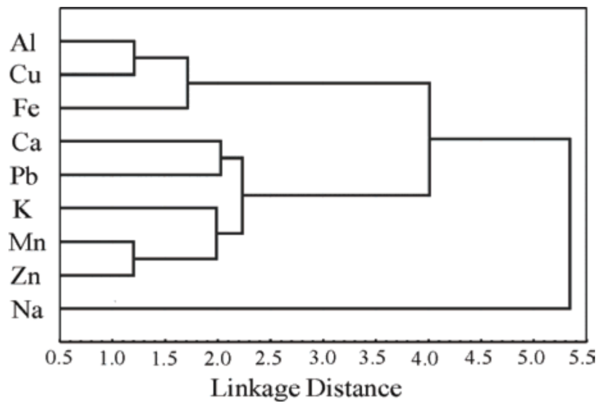

Fig. 4. The dendrogram of the hierarchical cluster analysis of total metal concentrations for combined data: high traffic locations + low traffic locations + traffic islands.

CONCLUSIONS

The metal contents of twenty-two snow samples taken directly from the roads and traffic islands in the urban area of Novi Sad were determined using the ICP-OES analytical technique. The small variation in the composition of snow at the selected locations indicates that the samples were, in general, impacted by the same source(s).

The hierarchical clustering applied to the variables (metals) for samples taken directly from the roads comprised two distinct groups of metals according to the degree of their association. The results of metal clustering were consistent with the fact that very strong statistically significant correlation coefficient values were obtained between pairs of metals within both groups. The partitioning method of cluster analysis was also performed on sampling locations. The results showed that the traffic density was neither the only nor the most important factor that divided the samples into two groups.

The results of non-parametric correlation analysis and multivariate statistical analysis revealed that common sources (most likely traffic related) mainly contribute to the contents of Al, Cu, Fe and Ni. Associations between some investigated elements (Ca, K, Mn, Pb and Zn) may suggest inputs from both anthropogenic activities and natural geochemical sources. Considering all of the analyses conducted in this study, no categorical conclusion concerning the pollution sources could be made.

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И З В О Д

МУЛТИВАРИЈАЦИОНА АНАЛИЗА САДРЖАЈА МЕТАЛА У СНЕГУ СА УРБАНОГ ПОДРУЧЈА ГРАДА НОВОГ САДА

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Током децембра 2009. године сакупљен је снег на двадесет и две локације на подручју града Новог Сада и то директно са асфалтних путева и са пешачких острва. Методом ICP-OES спектроскопије одређена је укупна концентрација следећих метала: Al, Ca, Cu, Fe, K, Mn, Na, Ni, Pb и Zn. Најмања и највећа концентрација метала у испитиваним узорцима утврђене су за никл и натријум, редом. Други по опадајућем редоследу концентрација био је калцијум и то на свим локацијама. У циљу одређивања повезаности метала израчунате су вредности Спирмановог (Spearman) корелационог коефицијента за парове метала. Такође, да би се утврдила повезаност између самих метала као и између метала и локација извршена је и кластер анализа добијених података и то користећи хијерархијски и партициони метод. На основу извршених анализа утврђено је да

густина саобраћаја није најбитнији фактор који је узроковао разлике у концентрацијама метала у испитиваним узорцима.

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