



WATER QUALITY SURVEY OF STREAMS FROM RETEZAT MOUNTAINS (ROMANIA)

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Research article, received 3 July 2016, accepted 14 November 2016

Abstract

The Retezat Mountains, located in the Southern Carpathians, are one of the highest massifs in Romania and home of the Retezat National Park, which possesses an important biological value. This study aimed at the investigation of water quality in creeks of the Southern Retezat (Piule-Iorgovanul Mountains) in order to provide information on pollutants of both natural and anthropogenic origin, which could pose a threat for the human health. Heavy metal and other inorganic ion contents of samples were analyzed with on-site and laboratory measurements to estimate water quality. The samples were investigated using microwave plasma - atomic emission spectrometry to quantify specific elements, namely aluminium, cadmium, cobalt, chromium, copper, iron, magnesium, manganese, molybdenum, nickel, lead and zinc. The results were compared with the European Union and Romanian standards regarding drinking water and surface water quality. The studied heavy metals have been found to be in very low concentrations or under the method's detection limit. Thus, in the microbasin corresponding to the sampling points, there seems to be no heavy metal pollution and, from this point of view, the samples comply as drinking water according to the European Union and Romanian recommendations. Our findings confirm that the Retezat Mountains are still among the least contaminated regions in Europe and that the ecosystem and the human health is not negatively influenced by water quality problems.

Keywords: water chemical analysis, surface water quality, heavy metals, MP-AES, Retezat National Park

INTRODUCTION

Heavy metals are considered common pollutants of the environment, having both natural and anthropogenic origins (Tchounwou et al., 2012; Bradl et al., 2005). The rapid development of modern world has accelerated their release into the biosphere (Mosa et al., 2016; Panagos et al., 2013). Some of the chemical species that contain heavy metals can be highly toxic when inhaled or ingested. They can have an impact on almost every organ and system in a living organism, posing a serious threat to the stability of the ecosystems and a danger for the human health (Jaishankar et al., 2014; Bradl et al., 2005). They constitute the main contaminant category that affects Europe, contributing to around 30-35% in soil and groundwater contamination (Panagos et al., 2013). Besides air pollution, heavy metals have been regarded, in the last decades, the greatest immediate health threat in Central and Eastern Europe (Fitzgerald et al., 1998). The influence of metal pollution on the river ecological status in Europe is evaluated according to the Water Framework Directive (Roig et al., 2016).

The Retezat National Park is located in the western part of the Southern Carpathian Mountain Range (Romania, Hunedoara County). It possesses a high biological value and has thus been added to the UNESCO's Man and the Biosphere reserves network. The park includes 19 peaks above 2000 m elevation and it has been proposed as a model for the conservation efforts in Romania and other countries (Bytnerowicz et al., 2005). While several studies from the scientific mainstream literature have dealt with the geology and hydrogeology of the area (Povară and Ponta, 2010) or the composition of mountain lake sediments (Catalan, 2015; Camarero et al., 2009; Rose et al., 2009), studies regarding the composition of surface creeks seem to be scarce or non-existent.

In this work, several springs and streams from the Southern Retezat were analyzed for the presence of heavy metals and other ions (cations and anions). The tested cations include ammonium (NH_4^+), arsenic (As^{3+}), calcium (Ca^{2+}), iron ($\text{Fe}^{2+}/\text{Fe}^{3+}$) and other heavy metals (e.g., lead Pb^{2+}), while the anions that were searched for

include halides (chloride Cl^- , bromide Br^- , iodide I^-), nitrite (NO_2^-) and sulfate (SO_4^{2-}), all of them being determined according to the chemical methods stipulated in the Romanian Pharmacopoeia (1993) or by using test strips. The results were supplemented by microwave plasma - atomic emission spectrometry (MP-AES) determinations for some metals, i.e. aluminium, cadmium, cobalt, chromium, copper, iron, magnesium, manganese, molybdenum, nickel, lead and zinc. Most of these species are included in the category of heavy metals (Duffus, 2002).

STUDY AREA

The studied area (Fig. 1) belongs to the southern part of the Retezat Mountains (Bytnerowicz et al., 2005), also known as Southern Retezat ("Retezatul Sudic" in Romanian), and is located on the southern slope of the Piule-Iorgovanu Mountains (Ardelean, 2010; Povară and Ponta, 2010), between 1529 and 1871 m of altitude. The stream water samples (Fig. 1) were collected at six sampling points at Scorota cu Apă and Scorota Seacă (the headwaters of Scorota River) in the upper part of the Jiul de Vest River basin (Iordache et al., 2015;

Ujvári, 1972). The microbasins corresponding to the sampling points contain active streams with lengths which vary between 100 and 500 m, and with a discharge usually between 2 and 5 L/s. The sampling points were allocated by considering these short lengths and their relative position to the Scorota sheepfold: near the sheepfold, which is also the lowest part of the grazing area, and in the higher limit of this area, with approximately 100 m of altitude between the different sample points.

From geological point of view, mostly sedimentary rocks cover the surface, like quartz sandstones, marl and marl-limestones, with patches of schists and limestones. The most important soil types are humus-iron-illuvial podzols, humus-silicate soils, brown podzols and brown acidic soils. These mountains are characterized by a rich flora and fauna. Subalpine meadows (grassland) predominate, being in contact with the upper limit of the coniferous domain, composed mainly of Norway spruce (*Picea abies*). *Carex* and *Festuca* meadows alternate with mountain pine, juniper shrubs and dwarf shrubs composed of *Vaccinium vitis-idaea* and *Vaccinium myrtillus* (Bytnerowicz et al., 2005; Kern and Popa, 2009; Măciu

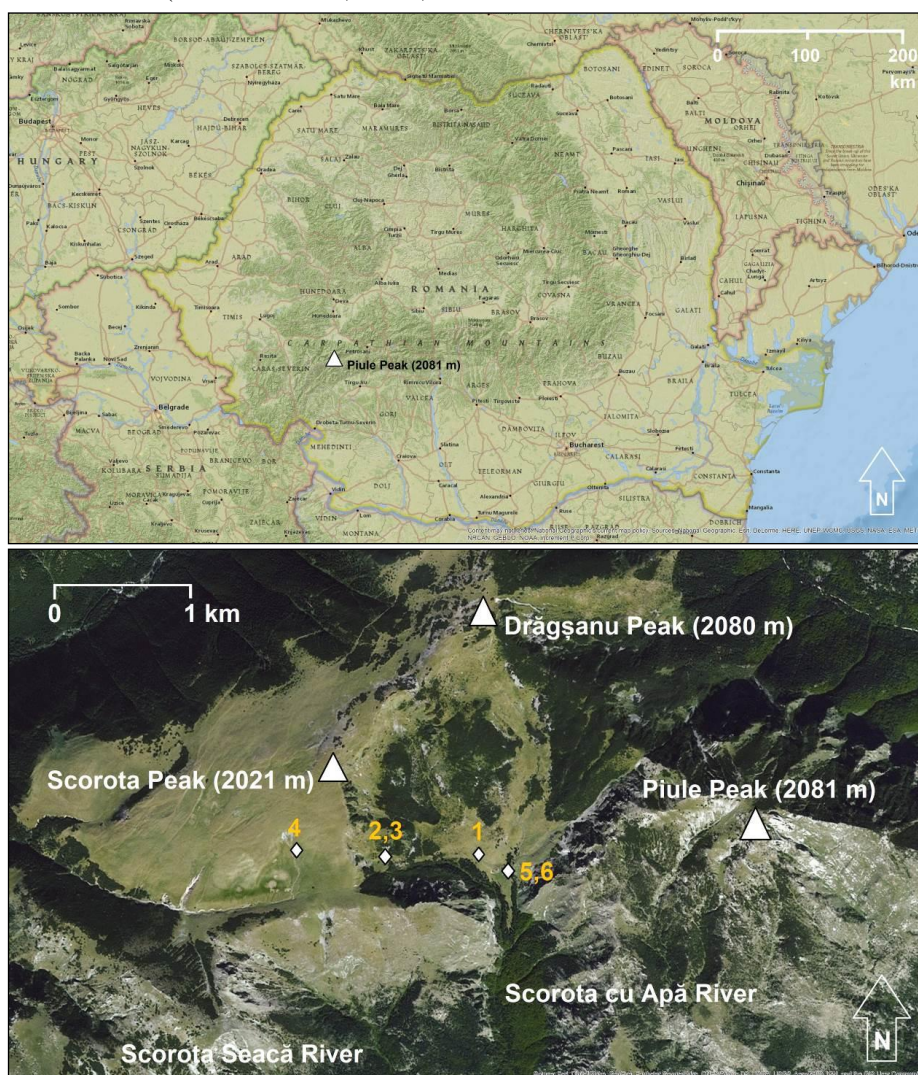


Fig. 1 Location of the sampling points



Fig. 2 Environment of the sampling sites (left side: juniper forest and *Festuca* meadows, characteristic for samples 1, 2, 3, 5 and 6; right side: eroded mountain slopes in the case of sample 4)

et al., 1982; Tulucan et al., 1999), as can be seen in Fig. 2 (left). The climate is continental and typical for high mountain areas (Povară and Ponta, 2010). Prior to sampling, weather was stable with constant atmospheric pressure. There was no record of rainfall or snowfall that could have produced signs of indirect contamination (e.g., acid rain or residual grazing waste).

The list of analyzed samples, together with time, location and on-site measured parameters, are given in Table 1. Samples 1-3 come from springs which originate in fluvial deposits from non-karst rocks (sandstones). For samples 2 and 3, the measurements were taken near a confluence of two very short creeks, which combine to form a right tributary of Scorota cu Apă stream. Sample 4, corresponding to the highest measured point, comes from a spring localized in non-calcareous detrital rocks; the sampling was done in a ravine with erosional slopes (Fig. 2, right). Samples 5 and 6 were collected near Scorota sheepfold from two creeks that flow over Holocene detrital deposits. The catchment is located in the *Festuca* meadows perimeter; sampling sites corresponding to samples 1-3, 5 and 6 are located in the juniper floor, where juniper clusters alternate with *Festuca* meadows.

Due to the fact that the sampling was performed at the beginning of November, any organic pollution that might have appeared because of grazing should have been washed by precipitations in the two months that have passed since the ending of the grazing period.

METHODS

Sampling and in situ measurements

Geographic coordinates and altitudes were established using a Magellan Meridian Platinum Mapping GPS receiver, while air temperature and pressure were recorded with a portable Auriol weather station. Sample temperature, pH and electrical conductivity (EC) were registered with a portable Hanna HI 98130 Combo pH&EC measuring device. Nitrites and sulfates were measured *in situ* using Merck test strips (Merckoquant® Nitrit-Test and Merckoquant® Sulfat-Test) (Table 1).

We have generally followed ISO 5667-3 guidelines for sampling. To prevent contaminations, thoroughly cleaned plastic recipients were used (Bradl et al., 2005; Ogoyi et al., 2011), prepared in the laboratory by protracted soaking with 2 M nitric acid followed by rinsing with double distilled water. They were also conditioned *in situ* with several aliquots of the water to be

Table 1 Sampling parameters

Parameter / Sample	1	2	3	4	5 ^a	6 ^a
Date (day/month/year)	01/11/2014	01/11/2014	01/11/2014	01/11/2014	02/11/2014	02/11/2014
Time (UTC+2 hours)	12:00	13:00	13:10	16:00	09:00	09:10
Geographic coordinates	45°17'57''N 22°53'23''E	45°17'55''N 22°52'57''E	45°17'55''N 22°52'57''E	45°17'57''N 22°52'30''E	45°17'53''N 22°53'33''E	45°17'53''N 22°53'35''E
Altitude [m]	1620	1759	1759	1871	1533	1529
Air pressure [mbar]	842.8	828.3	828.3	817.6	^b	^b
Air temperature [°C]	25	22	22	15	^b	^b
Sample temperature [°C]	4.8	5.4	5.9	4.9	5.0	4.0
pH [pH units]	8.40	7.92	8.02	8.00	8.13	8.18
EC [μS/cm]	~ 10	< 10	< 10	< 10	< 10	< 10

^a geographic coordinates and altitude were established using Google Earth (2015) software;

^b not measured.

sampled. After completing this protocol a volume of 350 mL of water was collected. To avoid the loss of elements by adsorption on the walls of the storage recipients, the samples were stabilized by acidification to pH ~ 1 by adding 20 mL of 10% nitric acid (Pascariu et al., 2013).

Laboratory analysis

All glassware needed for analysis was washed with 2 M nitric acid and thoroughly rinsed with double distilled water just prior of being used. Preliminary analyses were performed on filtrated samples the day after they were collected according to the general procedures stated by the Romanian Pharmacopoeia (1993). A blank solution (350 mL double distilled water with 20 mL 10% nitric acid) was also prepared in an identical plastic container and tested for comparison. The following aqueous reagents were used: Nessler's reagent (potassium tetraiodomercurate (II), K_2HgI_4 , and potassium hydroxide, KOH) for ammonium, sodium hypophosphite (NaH_2PO_2) in hydrochloric acid (HCl) for arsenic, ammonium oxalate ($(NH_4)_2C_2O_4$) for calcium, silver(I) nitrate ($AgNO_3$) for halides, potassium hexacyanoferrate(II) ($K_4[Fe(CN)_6]$) for iron, sodium sulfide (Na_2S) for heavy metals (e.g., lead) and barium chloride ($BaCl_2$) for sulfates. The chemical reactions that use these reagents are stated to have the following detection limits: 0.3 ppm for ammonium, 1 ppm for arsenic, 3.5 ppm for calcium, 0.5 ppm for chlorides, 0.5 ppm for iron, 0.5 ppm for lead and 3 ppm for sulfates (Romanian Pharmacopoeia 1993).

For MP-AES, an Agilent 4100 with web-integrated Agilent MP Expert software was used. The instrument was adjusted using as calibration standard the provided Wavelength Calibration Concentrate for ICP-OES & MP-AES (Al, As, Ba, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sr, Zn 50 mg/L, K 500 mg/L) and also an AAS standard solution for Ca, Fe and Mg. The following wavelengths (in nm) were measured: Al 396.152, Cd 228.802, Co 340.512, Cr 425.433, Cu 324.754, Fe 259.940, Mn 403.076, Mg 285.213, Mo 379.825, Ni 352.454, Pb 405.781, and Zn 213.857. In contrast to atomic absorption spectrometry (AAS), which is based upon the absorption of a characteristic radiation, atomic emission spectrometry (AES) uses the emission of a characteristic wavelength for the

determination of the analyte element. Plasma emission spectrometry utilizes a plasma as the excitation source for atomic emissions, which, in MP-AES, is formed via the use of a microwave field source. AES belongs to the most useful and commonly used techniques for the analysis of heavy metals, providing rapid and sensitive results in a variety of sample matrices, although the detection limits are higher than those of AAS (Bradl et al., 2005; Higson, 2006).

RESULTS AND DISCUSSION

The average water temperature was 5.0 °C, the mean pH value was 8.11, while the measured EC value was around or below 10 $\mu S/cm$ for all samples (Table 1). The in situ tests using test strips did not indicate the presence of nitrites or sulfates (nitrite ion concentration less than 1 mg/L, sulfate ion concentration less than 200 mg/L, according to test strips instructions). Also, the very low measured EC indicates that the total dissolved solids (TDS) must be under 10 ppm (Lenntech, 2016).

The samples were tested for the presence of ammonium, arsenic, calcium, halides (chloride, bromide, and iodide), iron, heavy metals (e.g., lead) and sulfates. Except for a very faint opalescence obtained when sample 1 was tested for calcium, all these tests were negative, an observation that supports the very low measured EC for all samples (Table 1).

MP-AES results are summarized in Table 2, while the drinking water standards from EU (1998) and Romanian "Law no. 311 from June 28, 2004" (Romanian Government 2004) are given in Table 3 for comparison with the analyzed samples. As can be seen, except for a somewhat increased iron content in sample 1 (probably due to the humus-iron-illuvial podzols, which are present in the area), the studied streams are within the limits specified for drinking water (Brad et al., 2015) by all specified standards. The metal contents are generally low and there is no observable trend downstream on any of the elements.

The Romanian environmental legislation regarding surface water quality, stipulated in "Order no. 161 from February 16, 2006" (Romanian Government 2006), is summarized for the considered ions in Table 4. This surface water quality classification is useful in order to establish the ecological status of all marine and continental

Table 2 MP-AES cation content results, in mg/L

Sample	Concentration											
	Al	Cd	Co	Cr	Cu	Fe	Mg	Mn	Mo	Ni	Pb	Zn
1	*	*	0.03	*	*	1.61	0.53	*	0.03	*	0.01	*
2	*	*	0.03	*	*	0.05	1.10	*	0.02	*	0.01	*
3	*	*	0.02	*	*	0.13	1.84	*	0.02	*	*	*
4	0.01	*	0.02	*	*	0.08	0.18	*	0.02	*	*	*
5	0.01	*	0.02	*	*	0.14	0.88	*	0.02	*	*	*
6	*	*	0.02	*	*	0.05	0.54	*	0.02	*	*	*

* under detection limit (< 0.005 mg/L).

Table 3 Drinking water standards comparative table; all values are in units of mg/L unless stated otherwise (European Union, 1998; Romanian Government, 2004)

Parameter	EU standards 1998	Romanian law no. 311/2004
pH [pH units]	6.5-9.5	6.5-9.5
EC [mS/cm]	2.500	2.500
TDS	#	#
Temperature [°C]	#	#
Aluminium (Al ³⁺)	0.200	0.200
Ammonia/ammonium (NH ₃ +NH ₄ ⁺)	0.50	0.50
Cadmium (Cd ²⁺)	0.0050	0.0050
Calcium (Ca ²⁺)	#	#
Chromium (Cr ³⁺ +Cr ⁶⁺)	0.050	0.050
Cobalt (Co ²⁺ +Co ³⁺)	#	#
Copper (Cu ²⁺)	2.0	0.1*
Iron (Fe ²⁺ +Fe ³⁺)	0.200	0.200
Lead (Pb ²⁺)	0.010	0.010
Manganese (Mn ^{x+})	0.050	0.050
Magnesium (Mg ²⁺)	#	#
Molybdenum (Mo ^{x+})	#	#
Nickel (Ni ²⁺)	0.020	0.020
Zinc (Zn ²⁺)	#	5.000
Chloride (Cl ⁻)	250	250
Nitrite (NO ₂ ⁻)	0.50	0.50
Sulfate (SO ₄ ²⁻)	250	250

not mentioned;

* is allowed as 2.0 mg/L if the distribution piping material contains copper.

Table 4 Surface water quality classes depending on cation content, as stated in Romanian "Order no. 161 from February 16, 2006"; units are in mg/L, unless stated otherwise

Parameter	Order no. 161 (2006)				
	I	II	III	IV	V
pH [pH units]	6.5 – 8.5				
EC [mS/cm]	No guideline				
TDS	Not mentioned				
Temperature [°C]	No guideline				
Aluminium (Al ³⁺)	Not mentioned				
Cadmium (Cd ^{x+})	0.0005	0.001	0.002	0.005	>0.005
Chromium, total (Cr ³⁺ +Cr ⁶⁺)	0.025	0.050	0.100	0.250	>0.250
Calcium (Ca ²⁺)	50	100	200	300	>300
Cobalt (Co ³⁺)	0.010	0.020	0.050	0.100	>0.100
Copper (Cu ²⁺)	0.020	0.030	0.050	0.100	>0.100
Iron, total (Fe ²⁺ +Fe ³⁺)	0.3	0.5	1.0	2	>2
Lead (Pb ^{x+})	0.005	0.010	0.025	0.050	>0.050
Magnesium (Mg ²⁺)	12	50	100	200	>200
Manganese, total (Mn ²⁺ +Mn ⁷⁺)	0.05	0.1	0.3	1	>1
Nickel (Ni ^{x+})	0.010	0.025	0.050	0.100	>0.100
Zinc (Zn ²⁺)	0.100	0.200	0.500	1.000	>1.000
Chloride (Cl ⁻)	25	50	250	300	>300
Sulfate (SO ₄ ²⁻)	60	120	250	300	>300

aquatic ecosystems, including rivers and lakes, both natural and artificial. The evaluation of the considered quality elements, like chemical and physical-chemical parameters, can indicate the presence of certain natural environments, minor alterations of these or the degree of anthropic impact, and, respectively, the status of water bodies quality in a certain amount of time. There are five ecological states being defined for natural rivers and lakes: very good (I), good (II), moderate (III), poor (IV) and bad (V). According to Table 4, the streams corresponding to samples 3-6 belong to class I. An exception could be the stream that provided sample 1, which belongs to class IV according to the iron content. Also, according to the lead content, the streams that provided samples 1 and 2 could belong to class II or III, but these low measured lead levels may more realistically be accounted for by the MP-AES precision limit.

Our findings support the previous studies which state that the Retezat Mountains are among the least contaminated regions in Europe (Catalan, 2015; Catalan et al., 2009). Regarding the studied parameters and considering the low levels of dissolved ions, water quality was found to be good or very good. Thus, heavy metals do not pose any ecological or human risk in the studied area.

CONCLUSIONS

MP-AES, alongside some classical analytical procedures, were used to analyze the water quality of springs and creeks from the Retezat National Park. For all tested samples, heavy metals were at very low levels or under the detection limit for the chemical reactions employed and the MP-AES method applied. From this point of view, the samples comply as drinking water according to the EU and Romanian recommendations. The average water temperature was 5.0 °C, the mean pH value was 8.11, while the measured EC value was around or below 10 µS/cm for all samples, the later also confirming the very small ion content present in the analyzed mountain streams. In the microbasin corresponding to the sampling points, there seems to be no heavy metal pollution. Also, no other potential sources of chemical pollution was recorded in the studied perimeter during our survey.

Acknowledgements

Part of this paper was presented at The 17th DKMT Euroregional Conference on Environment and Health, June 5-6, 2015, Szeged, Hungary. Some of the research was done at the Center of Genomic Medicine of the "Victor Babeş" University of Medicine and Pharmacy of Timișoara, POSCCE 185/48749, contract 677/09.04.2015.

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