Dynamics and assessment of some mineral elements level (Ca, Mg, K, Na) for the silver fir species in the Tarnita area, Romania

[1]Sidor C. Gheorghe, [1,2]Cuciurean C. Ilie, [1,3]Buculei Amelia

[1] "Marin Drăcea" National Research-Development Institute in Forestry, Station Câmpulung Moldovenesc, Romania; [2] "Ștefan cel Mare" University of Suceava, Faculty of Forestry, Suceava, Romania; [3] "Ștefan cel Mare" University of Suceava, Faculty of Food Engineering, Suceava, Romania. Corresponding author: A. Buculei, ameliab@fia.usv.ro

Abstract. Significant long-term monitoring requires extensive research into the basic characteristics of forest biodiversity (composition, structure and function). This topic has been addressed by many researchers, but no data have been obtained on the dynamics and evolution of mineral accumulation in annual silver fir growth rings in areas with high pollution levels compared to unpolluted areas. The goal of this research was to develop a database on the level of concentrations of the mineral elements of the tree in the Tarnita area, Romania, accumulated over 60 years, including the period when the mining activity was intense, respectively, the period when the activity was significantly reduced. The results showed that there is a major change in the concentrations of the mineral elements studied (Ca, Mg, K, Na) in the case of trees in the heavily polluted area compared to trees in the unpolluted area.
Key Words: monitoring, chemical elements, dendrochemistry, air pollution, silver fir.

Introduction. Long-term observations are essential for the surveillance of forest ecosystems, because such systems have high capacities to store atmospheric inputs and feedback loops may be slow. Predicted climate change and severe weather events will impact forest ecosystems. These impacts can include heat and water stress, increased vulnerability to pests, or a change in species composition. Soil and water concentrations are characteristics of an uncontrolled forest ecosystem. The primary focus of research on minerals and heavy metals in forest ecosystems has been on potassium, magnesium, calcium, lead, cadmium, arsenic, copper and other heavy metals (Michopoulos et al 2022). Remote forests have low concentrations of these minerals in water, soil and plant tissue and therefore serve as a comparison to previously polluted agricultural and forestry ecosystems (Michopoulos et al 2022). Monitoring the health of forest ecosystems subject to atmospheric pollution is one of the main tasks of the International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests (ICP Forests). For example, in Estonia, oil-shale burning power plants are the main sources of heavy metals which may affect forest ecosystems (Bussoti et al 2017). The heavy metal content of needles, bedding, fine roots and soil organic horizons in six conifer stands of the ICP and ICP Integrated Forest Monitoring Network was analyzed. The highest concentrations of lead, cadmium, chromium and nickel were found in soil organic horizons, whereas biogenic zinc and copper showed the highest concentrations in the fine roots indicating the active uptake of these micro-elements in the soil organic layers (Bussoti et al 2017). Along with nitrogen and phosphorus, calcium is the fourth most abundant element in mineral nutrition and plays important physiological and structural roles in vegetable metabolism.

The minerals rich in calcium and the low cationic exchange capacity in addition to the activity of aluminum reduces the quantity of calcium available for forest plants (Kudrevatykh et al 2021). Focusing mainly on forests dominated by pine, spruce and birch, some researchers studied the concentration of calcium in the foliage relative to the properties of the climate and soil (Malinova et al 2017). Minerals are very important for developing all woody plants. The mineral content depends on both the specific composition of the planting and the climatic and soil factors of certain habitats. The aim of another research was to compare the chemical content of Scots pine, Norwegian spruce and silver fir in myrtles forests. In this way, the content of minerals and heavy metals from wood cores from the Pressler driller in the central and peripheral areas was determined, by the use of X-ray fluorescence analysis. Pine and spruce wood contain above all calcium and potassium, and later smaller magnesium, iron, sulphur, copper, phosphorus. Copper and lead were twice as important in the middle part of the stem, calcium and potassium in the peripheral part (Kilyusheva et al 2017).

Studies of the relationships between the micro-element composition of plants in the surface layer and soil are also of great interest for research. A research study established how the lithological substrate or parent material in the Medvednica Natural Park region affects calcium, magnesium, potassium and sodium content in the soil (Bakšić et al 2015). Other researchers compared the structure of the field layer and chemical composition of the soil in dark conifer forests of North-Eastern Europe (Kudrevatykh et al 2021). In this way, nutrient dynamics along a rotation of the Douglas fir have been studied by chemical characterization of the solutions that are transferred into the ecosystem (Kudrevatykh et al 2021).

While differences in the composition of the soil solution were more related to soil characteristics than to population age, a significant difference in the quality of throughfall, stemflow and forest floor solutions were directly associated with tree growth and stand characteristics (Augustynczik et al 2019).

Although some studies that were related have been carried out on the dynamics and evolution of mineral accumulation in annual silver fir growth rings in areas with high pollution levels compared to unpolluted areas, no data has been obtained on the dynamics and the evolution of the accumulation of mineral elements in the fir growing rings when the pollution level was identified in Tarnita area. The purpose of this study was to create a database for the evolution of mineral elements accumulated in the over years old 60growth rings.

Material and Method

Sample collection. The research was carried out in the Tarnita area, Romania, an area affected by the extraction and processing of non-ferrous metals, this area being the subject of several studies over time (Popa & Barbu 2001; Flocea 2013). In order to determine the dynamics and evolution of the accumulation of mineral elements in the fir growth ring, growth samples were taken from trees in the heavily polluted area (located in the immediate vicinity of the pollution source) and from the unpolluted area (located at a fairly long distance where local pollution due to mining operations did not have an effect). The characteristics of experimental surfaces are related in Table 1.

Table 1

Characteristics of experimental surfaces

Series code	*Species*	*No of samples*	*Period length*	*Location*	*Forest district*	*Production units*	*U.A.*	*Distance from pollution source (km)*	*Pollution degree*
T.Br. P.	Silver fir	1	1961-2020	Tarnita	O.S. Stulpicani	V Tarnița	111E	1.2	Intensive pollution
T.Br. N.	Silver fir	1	1961-2020	Tarnita	O.S. Stulpicani	V Slătioara	4B	12.0	Unpolluted

The areas selected for harvesting growth samples are part of the Forest District Stulpicani in the Tarnita area, from two different production units (U.P.V Tarnita and U.P. VIII Slatioara, Figure 1).

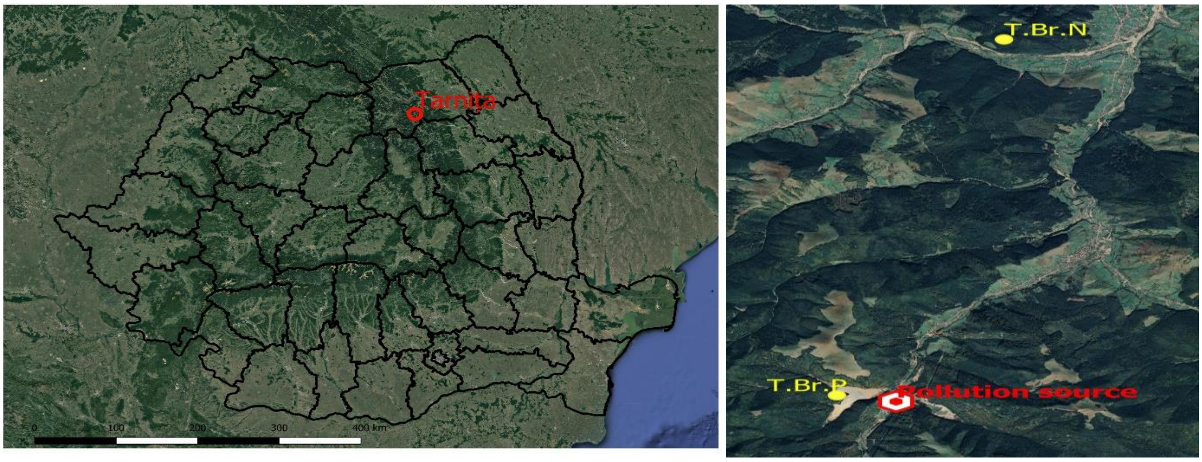

Figure 1. Localization of research.

The growth samples were selected to include trees over 60 years old to show the evolution of mineral elements accumulated in the growth rings, both during the period when the mining activity in Tarnita was carried out at a very high level, and in the period after, when the activity was significantly reduced.

A radial growth sample for the fir species was taken from each area. The collection was carried out with the Pressler drill with an internal diameter of 12 mm, the samples being placed in special cardboard tubes until the time of their processing. The samples were subsequently cut at a periodicity of 5 years, from 1961 to 2020 (there is a collection of samples starting from 1961), resulting in a length of the analyzed series of 60 years. After cutting the growth cup into sequences of 5 annual rings, a total of 12 samples were produced, each of which was placed in plastic containers, sealed and labeled according to the surface, species, pollution intensity and the years specific to each sample.

Mineral elements determination. The methods used to determine mineral elements are standardized (Table 3). For analytical purposes, between 0.5 and 1 g of each sample was completely dissolved in a 5/1 ratio of nitric acid to water. The resulting solution was analyzed by the ICP MS method, with a Perkin Elmer ICP MS Elan DRC II equipment. Among the general analytical techniques used by other researchers to determine inorganic metal impurities such as titration, ion chromatography, capillary electrophoresis, inductively coupled plasma (ICP MS Elan DRC II) it is a method that improves performance and has a high performance sample insertion system with the ability to run any reaction gas. The intensity of the energy emitted at a specific wavelength of a particular element is proportional to the amount of that element in the material analyzed (Vallapragada et al 2011).

Table 2

Parameters of the ICP-MS (Perkin Elmer Elan DRC-e)

Characteristics	
Sample units	µg L^{-1}
RF Power	1000 W
Sampler Cone	Nickel 1.1mm
Sample gas flow rate	1.002 L/m
Nebulizer gas flow	0.81
Scanning system	Peak hopping
Reaction gas	CH_4(1ml/min) Cell gas A 0.2; RPq 0.8 BEC (ppb) 0.1463 (As 74.9216)
Blank vacuum	After internal standard
Number of readings/ replicates	20/ replicate 3
Curve type	Simple linear

Characteristics	
Take up time	110 s
Washing time	48 (+/- rpm)
Read time leg, speed	20 (+/- rpm)
Resolution	LR-300, MR-4000, HR -10000
Sample per peak	50(LR) 20(MR, HR)

Table 3

Analytical method

Element analyzed	*Analytical method/ standard*
Potassium (K)	EPA Method 0258.1 Potassium, SR EN ISO 17294-1:2007/ISO 11466:1995
Calcium (Ca)	EPA Method 0215.1, 0215.2 Calcium, SR EN ISO 17294-1:2007/ ISO 19730:2008
Magnesium (Mg)	EPA Methods 0242.1, 0242.2 Magnesium, SR EN ISO 17294-1:2007/ SR ISO 14869:2012
Sodium (Na)	EPA Methods 0242.1, 0242.2 Magnesium, SR EN ISO 17294-1:2007

Chemical and standard solutions. All required stock solutions were prepared on the day of experience. Detection limit is the minimal concentration of the analyte to be detected, but not limited, and the limit of quantification is the lowest concentration of the analyte that can be quantified under defined experimental conditions (AFNOR 2009; Legouta et al 2020). A standard solution of known concentration, stable over time, was used to trace the linear calibration curve. The standard deviation of the signal and the Fischer function that was compared to the PG factor (ratio of standard deviation squares) were calculated for each element. Factor F<PG confirms that the deviation between the dispersions is not significant and that the calibration curve has been drawn correctly. The verification of the accuracy of the method was carried out by the analysis with a standard calibration solutions were prepared by successively diluting a high purity multielement standard (29-element ICP-MS 10 mg L^{-1} standard, 5%HNO_3 matrix, Perkin Elmer life and analytical Sciences), which is a certified reference material (ISO 17034). The correlation coefficient for calcium, magnesium and potassium turned out to be 0.9995, indicating good linearity.

Statistical analysis. Statistical analysis and processing of data of elemental concentrations contained in the tree rings was carried out using the RStudio program (R Core team 2019).

Results and Discussion

The analysis of the dynamics of mineral elements (Ca, Mg, Na and K) identified in the growth rings of fir trees in the Tarnita area over the 60 years showed different variations and concentrations depending on the area analyzed. The results are comparable to other specialty studies (Iacoban et al 2019). In the case of Ca concentrations there is an intensive increase at the heavily polluted area compared to the unpolluted area, between 1965 and 1970. Since 1975, there has been a decline in the concentration of this element, but nonetheless a slight increase in values can be observed between 1995 and 2005 (Figure 2).

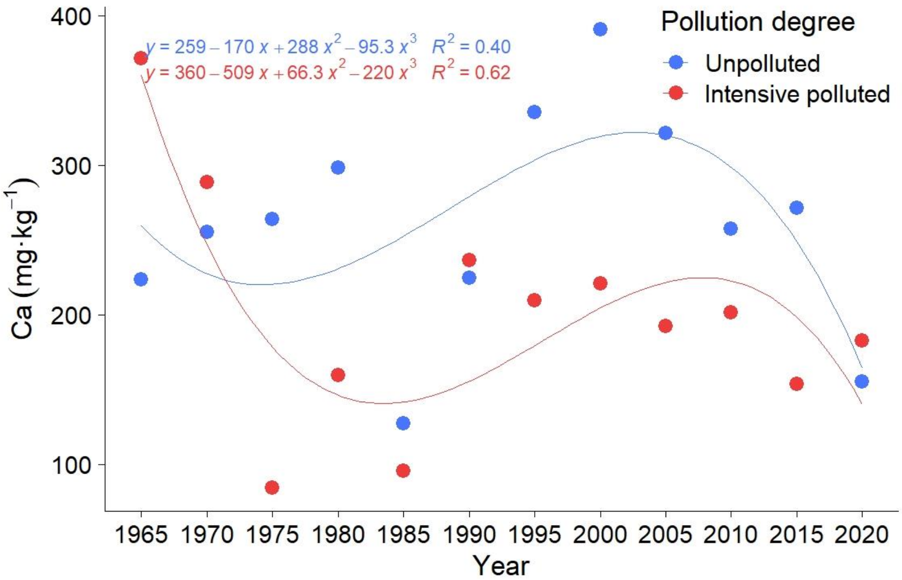

Figure 2. Dynamics of concentration for Ca in the period 1965-2020.

The Mg content was higher during the period 1965-1985 and over the past decade, the recorded values for this element were very low, both in the heavily polluted area and in the non-polluted area (Figure 3).

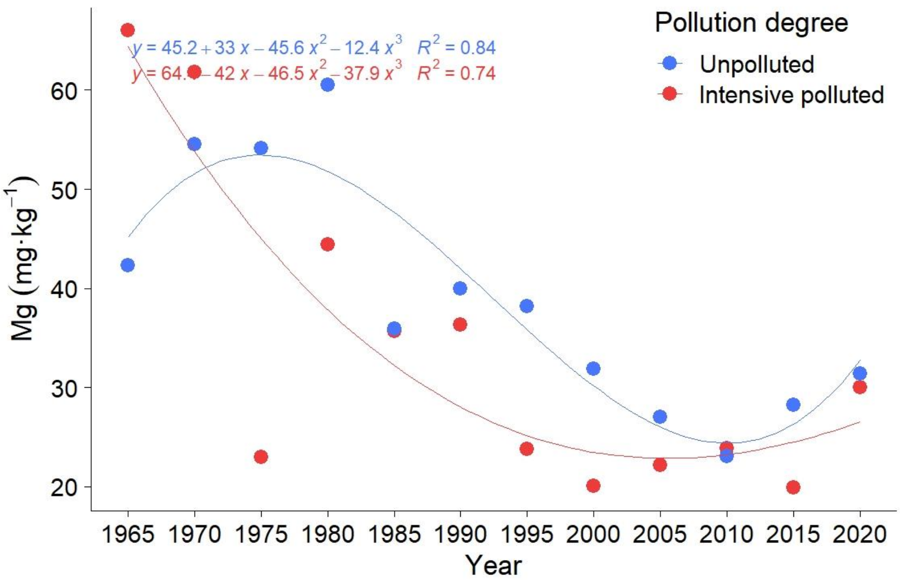

Figure 3. Dynamics of concentration for Mg in the period 1965-2020.

Iacoban et al (2019) found high variation in heavy metal concentrations between trees, in the same place. While the Mg content was higher near the landfill for silver fir (*Abies alba* Mill.) and European beech (*Fagus sylvatica* L.) the difference was only statistically significant for beech. A specific relationship was found between the level of contamination with heavy metals in the Tarnita region and the accumulation of certain heavy metals in forest trees. Even though the trees studied here were affected by heavy metal pollution, they also reacted against it by limiting the absorption of metal ions. In addition, because of their long lifespan, even trees located 6 km away from the deposit of contaminated residues could have been contaminated. These toxic heavy metals also appear to affect nutrient metabolism in wood species (Iacoban et al 2019).

The evolution of the mineral elements content in the growth rings, during both periods showed a high variation in concentration for the Na for year 1965, 20 mg kg^{-1} respectively 5 mg kg^{-1} for the year 2020 (Figure 4).

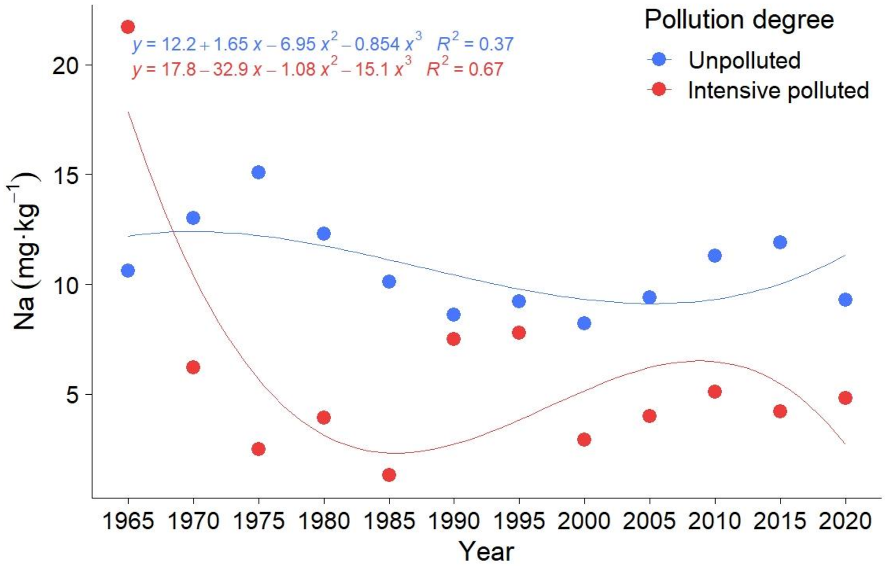

Figure 4. Dynamics of concentration for Na in the period 1965-2020.

The K concentration showed a very high-level trend during the period when the mining activity in Tarnita was carried out, and a significantly decreasing level in the period after, when the activity was significantly reduced (Figure 5).

Some authors reported a high variation for concentrations of macronutrients, in sapwood and heartwood of *Fagus sylvatica*. For potassium, a variation found between 950 and 1,355 mg kg^{-1}, 700-1,110 mg kg^{-1} for calcium and 180-310 mg kg^{-1} for magnesium (Kavvadias et al 2001; Malinova et al 2019).

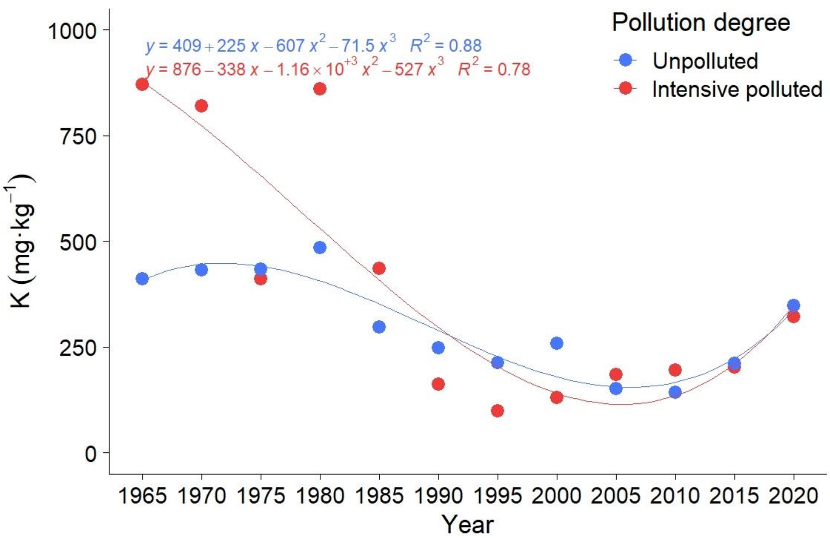

Figure 5. Dynamics of concentration for K in the period 1965-2020.

The result obtained in this study are comparable with the result from another study carried out by Kavvadias et al (2001). It has been showed that the concentrations of macronutrients in four forest ecosystems in northern Greece were substantially greater at fir than black pine. The tree's nutrient accretion varied between 3.2-12.3 kg ha^{-1} year^{-1} for K, 3.7-47.9 kg ha^{-1} year^{-1} for Ca, 1.8-8.6 kg ha^{-1} year^{-1} for Mg and 0.25-0.80 kg ha^{-1} year^{-1} for Na. Average element quantities (kg ha^{-1} year^{-1}) of the forest black pine between, 40.4 and 113.1 for K, 99.3 and 506.9 for Ca, 39.9 and 285.5 for Mg, and between 3.1 and 18.2 for Na. Over the past decade, there has been a positive relationship between mineral concentration levels and nutrient accumulation (Kavvadias et al 2001). The data for all the obtained elements concord with the results obtained from a study of the condition of forest soils in Europe (Ashwood et al 2019).

In the period 1965-1985, the content of the studied elements is very high. Among the mineral nutrients, Mg, K, Na and Ca showed a decreasing trend between 1995-2005. There was found a specific relationship between the contamination level with heavy metals of Tarnita area and the accumulation of some of them in the forest trees (Iacoban et al 2019). The levels of individual minerals and Ca, Mg, K and Na are approximately the same, whereas the correlation between horizons is statistically significant ($p<0.01$). The researchers proved in 2019 that the content of Ca and Mg was higher for the less polluted site, for silver fir (*A. alba*) and European beech (*F. sylvatica*), suggesting better nutritional status for these trees compared to those near the dump. In the case of silver fir, the calcium and magnesium content were negatively correlated with the diameter of the trees harvested from both sites.

Iacoban et al (2019) showed that the content of Ca and Mg in the entire core in silver fir and European beech wood collected from two differently polluted sites on the Tarnita mining area, it was negatively and statistically significantly correlated with the diameter of the tree for the *A. alba* trees sampled from both places. Among the mineral nutrients, Mg and Ca showed a decreasing trend, while Na and K concentrations tended to increase as the tree diameter increased. In the case of the first two elements, the intensity of the relationship was average and statistically significant. The authors of these studies (Kilyusheva et al 2017; Iacoban et al 2019) showed that the woody species studied seem to react against heavy metal pollution as a function of various levels of metal ions. The levels of Ca and Mg decreased, suggesting that increased the concentration of heavy metals Pb, Cd, Cu, Zn, As) influenced by the change in pH and the presence of certain anions (Kilyusheva et al 2017; Iacoban et al 2019).

Calcium is associated with traffic sources and concentrations are expected to decline sharply in the background as a function of distance from the roadway (Kord et al 2010; Tomasevic et al 2011). Earlier bioindicator studies have shown high levels of calcium in pine needles from road salts used for de-icing, sodium chloride and calcium chloride mixed (Ukonmaanaho et al 2017; Schmitt et al 2017). For trees, this tendency was particularly pronounced for K and Ca, as the flow concentrations of the stems differed considerably between the three size classes. In the case of palm trees, K concentrations have risen significantly, from smaller to larger (Puletti et al 2019). For pine and spruce, a sequence of mineral accumulation was Ca > K > Mg > Fe > S > P. As the tree weakened, the calcium content was reduced and the heavy metal content increased (Kilyusheva et al 2017). During their search, other scientists found a statistically significant difference in Ca and Na concentrations at two sites, in the topsoil of Medvednica Nature Park and Central Croatia (Bakšić et al 2009; Tomasevic et al 2011; Schmitt et al 2017).

Ferrara et al (2017) showed that Beech-fir stands developed on the soils above basic igneous rocks are characterized by a higher Ca, Mg, K and Na content, and consequently higher pH values. The authors remarked a significant Pearson correlation coefficient between exchangeable/available and total 0–70 cm soil pools for Ca, Mg, K and P, respectively 0.50, 0.82, 0.83 and 0.53, as shown in other forest soils (Ferrara et al 2017). Furthermore, no relationship was observed between the elements available in the soil and the contributions of atmospheric deposition. It is a negative or non-significant positive correlation of Pearson, with no data presented, at two sites studied (Legouta et al 2020). Other studies showed that during storms there are significant changes in the mineral elements in the composition of silver trees (Bakšić et al 2015).

Some studies also showed that the accumulation of elements by plants depends on the properties of the rocks that comprise the soil. It was found that trees in high spruce and fir forests have higher Ca content in their shoots and higher K, Mg, P, Zn, Mn and Al content in the roots. Bilberry-feathermoss fir-spruce forest plants have higher concentrations of K, S, and P in the shoots, and their roots contain more Ca, Mg, Zn, Fe, Mn, and Al (Lehndorff et al 2006; Watmough et al 2014; Michopoulos et al 2022).

It is important to note that land cover biodiversity affects the functioning of ecosystems through two different mechanisms (Prescher et al 2018). The impact of vegetation cover on the chemical composition of rainfall depended on the region of the country and the predominant species in a particular tree plantation. Pine and oak stands enriched precipitation with components that leached out of the canopy (K, Mn, Mg) to a greater degree than spruce and beech stands (Kowalska et al 2016).

Conclusions. Based on the obtained results it can be concluded that there is a decrease in the concentrations of the studied elements (Ca, Mg, K, Na) in the Tarnita area, between 1980, when the mining activity was reduced and 1990, respectively, when the exploitation plant was closed. The results obtained showed the impact of pollution on the forest ecosystem between 1965 and 2020. The aspects and results reported in this study can be developed a reference database for future research.

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Conflict of interest. The authors declare no conflict of interest.

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Authors:
Sidor Cristian Gheorghe, "Marin Drăcea" National Research-Development Institute in Forestry, 73 bis, Calea Bucovinei, 725100 Campulung Moldovenesc, Suceava, Romania, e-mail: cristi.sidor@yahoo.com
Cuciurean Cosmin Ilie, "Marin Drăcea" National Research-Development Institute in Forestry, 73 bis, Calea Bucovinei, 725100 Campulung Moldovenesc, Suceava, Romania, e-mail: cosmin.cuciurean@icas.ro
Buculei Amelia, "Marin Drăcea" National Research-Development Institute in Forestry, 73 bis, Calea Bucovinei, 725100 Campulung Moldovenesc, Suceava, Romania, e-mail: ameliab@fia.usv.ro

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