

## Analysis of micro and macroelements in must and wine of three *Vitis vinifera* L. varieties, using FAAS-technique

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**Abstract.** There are some factors which influence the must and wine composition: grape varieties, soil type, the climate, type of culture, agrochemical products (pesticides and fertilizers) and the practices used for wine making. These factors are directly involved in wine quality and in wine characterization. The aim of this paper is to determine the micro- and macroelements of must and wine on three cultivars Fetească albă, Fetească regală and Italian riesling, located in Turulung Vii vineyard, NW Romania. The quantitative analysis of five elements: Na, Ca, Mg, Fe and Co were detected using flame atomic absorption spectrometry (FAAS). With exception of Fe in wine (2.61 mg/L – Italian riesling, significant lower than 3.66 mg/L - Fetească regală), Italian riesling recorded highest values of mineral concentrations of both must and wine samples. The concentration of Co was below the detection limit of the device for all three varieties. The concentration of micro-, macroelements from wine are directly influenced by the concentration of micro-, macroelements from must. Wine is a complex matrix, which in additions to its basic components: water, alcohol and sugars contains high varieties of inorganic and organic compounds. It should be noted that certain inorganic elements in must and wine as Na, Ca, Mg, Fe and Co at different stages of wine are of great interest because of legal requirements. Determination of minerals in wine is important not only for the wine industry, in terms of problems of quality wine, but this can be viewed from the perspective of health of consumers of wine. The main propose of this research is to evaluate the minerals content (Na, Ca, Mg, Fe and Co) of must and wine samples and their transfer from must into wine. The biological material used was composed by the varieties: Fetească albă, Fetească regală and Italian riesling grafted on rootstock Kobber 5BB, grown in Turulung Vineyards area. Metals concentration in must samples for the five elements is in normal range, the highest concentrations were recorded by Italian riesling variety ( $33.72 \pm 2.22$  mg/L-Na;  $216.27 \pm 5.06$ mg/L-Ca;  $218.29 \pm 1.74$  mg/L-Mg;  $22.53 \pm 2.03$  mg/L-Fe). The highest concentrations in samples of wine were recorded at the same variety Italian riesling ( $24.42 \pm 3.84$  mg/L-Na;  $85.60 \pm 2.15$  mg/L-Ca;  $102.67 \pm 4.63$  mg/L-Mg), and the highest concentration of Fe are registered at the Fetească regală ( $3.66 \pm 0.11$  mg/L). Regarding the translocation factors between wine and must, the highest values are recorded for macroelements (Na, Mg, Ca) followed by microelements (Fe and Co). Based on this research we can say that the highest concentrations of metals analyzed were identified in samples of must comparative with wine sample. We also can observe a significant decrease in the concentration of metals in wine, this reduction is attributable to the fermentation process of must. The concentration of micro- and macroelements from wine are directly influenced by the concentration of micro- and macroelements from must.

**Key Words:** Minerals, grapevine products, FAAS method, NW Romania.

**Rezumat.** Vinul este o matrice complexă, care pe lângă componentele de bază: apă, alcool și zaharuri conține și o mare varietate de compozi anorganici dar și organici. Trebuie remarcat faptul că anumite elemente anorganice din must dar și din vin cum sunt Na, Ca, Mg, Fe și Co la diferite etape ale procesului de vinificație sunt de mare interes, din cauza cerințelor legale. Determinarea conținutului de minerale din vin este important nu numai pentru industria vinului, în ceea ce privesc problemele de calitate a vinului, dar acest fapt poate fi privit și din perspectiva sănătății consumatorilor de vin. Scopul principal al acestei cercetări este acela de a evalua conținutul în minerale (Na, Ca, Mg, Fe și Co) din probele de must și vin, precum și transferul lor în vin. Materialul biologic utilizat a fost format din soiurile: Fetească albă, Fetească regală și Riesling italian altoite pe portaltolul Kobber 5BB cultivate în arealul Turulung Vii. Concentrația metalelor analizate în probele de must pentru cele 5 elemente este în limite normale, cele

mai mari concentrații au fost înregistrate de către soiul Riesling italian ( $33.72 \pm 2.22$  mg/L-Na;  $216.27 \pm 5.06$  mg/L-Ca;  $218.29 \pm 1.74$  mg/L-Mg;  $22.53 \pm 2.03$  mg/L-Fe). Cele mai mari concentrații în probele de vin s-au înregistrat la același soi, Riesling Italian ( $24.42 \pm 3.84$  mg/L-Na;  $85.60 \pm 2.15$  mg/L-Ca;  $102.67 \pm 4.63$  mg/L-Mg), iar în cazul Fe cele mai mari concentrații s-au înregistrat la soiul Fetească regală ( $3.66 \pm 0.11$  mg/L). Privitor la factorul de translocare dintre vin și must, cele mai mari valori au fost înregistrate în cazul macroelementelor (Na, Mg, Ca) urmate de microelemente (Fe și Co). Pe baza prezentei cercetări putem afirma că cele mai mari concentrații ale metalelor analizate s-au identificat în probele de must, comparativ cu probele de vin. De asemenea se observă o scădere semnificativă a concentrației de metale din vin, această reducere este pusă pe seama procesului de fermentare la care este supus mustul. Concentrația macro-, microelementelor din vin este direct influențată de concentrația macro-, microelementelor din must.

**Cuvinte cheie:** Minerale, produse viticole, metoda FAAS, NV României.

**Introduction.** It's well-known that the wine, as a product obtained from the fermentation of grape must, is one of the most highly consumed beverages all of the world. The assessment of its quality is important for consumers and for manufacturers also. Wine contains a large number of inorganic ions (such as Na, Ca and Mg) and organic compounds (organic acids, flavanols, flavonols, aminoacids, anthocyanins and polysaccharides) (Pyrzynska 2007; Jäntschi et al 2013).

There are some sources contributing to the presence of inorganic elements in must and wine. For example the mineral content of the soil and the plant's capacity to uptake and accumulate these elements in the grape berry, after that its passes into must and finally in the wine (Laurie et al 2010; Pyrzynska 2007). Other sources derived from the human activity are pollution of the surrounding industries like metallurgy, fuel and energy production, mining and smelting of metalliferous (Bora et al 2015a,b; Damian et al 2008; Bora et al 2013), fertilizers and pesticides, air pollution, irrigation water, and the contamination during the winemaking process (Pessanha et al 2010; Vystavna et al 2014; Angelova et al 1999).

Minerals are of particular importance in oenology. Some of these minerals are required for fermentation processes, entering into the composition of enzymes, other mineral substances influence the redox process of the wine and some are essential for the metabolism of wine yeast and bacteria (Pereira 1988; Aceto et al 2002). The metals present in small amounts provide a better clarity of the wine, but also preserving flavors, while contributing to raising the food value of wine (Garoglio 1973, cited by Cotea 2010).

The most widely used techniques for the determination of major and trace elements in wine are AAS and inductively coupled plasma mass-spectrometry (ICP-MS) respectively (Ough & Amerine 1988; Aceto et al 2002; Laurie et al 2010; Banović et al 2009). The same techniques were applied for analysing the trace elements in grape juice from Brasil (Bragança et al 2012) and Romania (Dehelean et al 2012). Analysis of the must and wine in Douro region, Portugal, was performed by Total Reflection X-ray Fluorescence (TXRF) (Pessanha et al 2010).

Determination of the composition of wines is important issue but is difficult. There is a multiplicity of factors can influence the composition of the wine. They are linked to environment surroundings, including soil, climate and some processes such as wine fermentation process (Rovio et al 2011).

The aim of this investigation is to determine the amounts of Na, Ca, Mg, Fe and Co and the mode of their uptake by the grapes (must), as well as their transfer into the wine.

**Material and Method.** Three vine varieties for high quality white wines were used in the research: Fetească albă, Fetească regală and Italian riesling, grafted on the rootstock Berlandieri x Riparia Kobber 5 BB and cultivated in the area of Turulung Vii ( $47^{\circ}56'$  North,  $23^{\circ}5'$  East), Satu Mare county, NW Romania.

The grape samples were collected in 2013 at full maturity, and 10 kg of grapes/variety were collected from 10 vines/repetition. Tree repetitions/variety were used, placed in randomized blocks. The grapes were harvested from the lower, middle and top of each vine, grapes exposed to the sun, but also from shaded, thus obtaining a homogeneous sample (Bora et al 2014). For grape sampling disposable rubber gloves were used. After sampling, the samples were placed in sealable plastic bags, they were

numbered and shipped as soon as possible to the laboratory. The grape samples were pressed with the laboratory press (manually) and the must was obtained, followed by the process of microvinification which resulted in the samples of wine.

The wines produced in the laboratory (microvinification) had only sulphitation treatments (SO<sub>2</sub>), without interfering with other stabilization techniques that alter the content of macro- and microelements.

Determination of Na, Ca, Mg, Fe and Co was performed using Perkin Elmer AAnalyst 800, (Shelton, USA) and the method of analysis used is flame atomic absorption spectrometry (FAAS). Must and wine samples were diluted with ultrapure water so that the metallic ions concentration analyzed being in the detection limit of the apparatus.

Atomic absorption spectroscopy (AAS) is the most commonly used technique for metal determination in must and wines. Several methods are proposed in literature, this methods use ICP-AES (Eschner et al 1989; Thiel & Danzer 1997; Fournier et al 1998), flame – AAS (Caputi & Ueda 1967; Gonzales et al 1988), or GF-AAS (Soares et al 1995; Bruhn et al 1996; Chmilenko & Baklanova 1997). Wine from an analytical point of view is a fairly complex matrix owing to the content of its organic compounds. Above all, ethanol influences transport properties of the sample toward atomization devices due to changes in density and surface tension, with respect to aqueous standard solutions. In metal determination various types of interferences are to be expected but the knowledge of the concentration of few major components allows the adoption of simple quantitation procedures. In particularly important and easy to apply is the matrix matching method, which offers the possibility for obtaining a simple external calibration by preparing standard solutions as similar as possible to the samples. In fact atomic absorption spectrometry (AAS) techniques are generally little prone to interference caused by organic compounds due to high temperatures involved in atomization steps (Aceto et al 2002).

The standard solution developed for the calibration curves of each of the ions analyzed were prepared daily by dilutions of 1000 mg/L certified chemicals standard solutions (Merck, Darmstadt, Germany). The intermediate solutions were stored in polyethylene bottles and glassware was cleaned by soaking in 10 % v/v H<sup>+</sup>NO<sub>3</sub><sup>-</sup> for 24 hours and rinsing at least three times with ultrapure water. Solutions were prepared using ultrapure water obtained from a system Barnstead Easypure RoDi model D13321 (England), and suprapure quality chemicals (Merck, Darmstadt, Germany).

For each analyzed element was prepared in the same conditions a blank sample (control) to check the purity of the reagents used for the determinations. The variation coefficients were under 10% and detection limits (mg/L) were determined by the calibration curve method. Limit of detection (LOD), limit of quantification (LOQ) and other operation conditions are shown in Table 1.

Table 1

Instrumental conditions for determination of each mineral by FAAS

| <i>Element</i> | <i>Wavelength (nm)</i> | <i>Slit (nm)</i> | <i>Correlation coefficient</i> | <i>Flame (≈ 2300° C)</i> | <i>Background correction</i> | <i>LOD* (mg/L)</i> | <i>LOQ** (mg/L)</i> |
|----------------|------------------------|------------------|--------------------------------|--------------------------|------------------------------|--------------------|---------------------|
| Na             | 589.0                  | 0.2              | 1.000000                       | air-acetylene            | -                            | 0.012              | 0.039               |
| Ca             | 422.7                  | 0.7              | 1.000000                       | air-acetylene            | -                            | 0.092              | 0.306               |
| Mg             | 202.6                  | 0.7              | 1.000000                       | air-acetylene            | Deuterium                    | 0.190              | 0.632               |
| Fe             | 248.3                  | 0.2              | 0.999972                       | air-acetylene            | Deuterium                    | 0.109              | 0.366               |
| Co             | 240.7                  | 0.2              | 0.999900                       | air-acetylene            | Halogen                      | 0.119              | 0.399               |

\* Detection Limit

\*\*Quantification limit

Statistical analyses were performed using the statistical software package SPSS (version 20.0; SPSS Inc., Chicago, IL, USA). The data were expressed as mean ± standard deviation (SD) of three replications for each sample analyzed. In order to determine the significance differences among values, analysis of variance (ANOVA) and Duncan multiple

range test (MRT) were performed. Person's correlation was done using version 22 of SPSS (SPSS Inc. Chicago, IL, USA).

## Results and Discussion

**Macro- and microelements from must.** The concentration of macro-, microelements from must, largely reflect the mineral concentration from ground support growth of the vine (Bertoldi et al 2011; Chopin et al 2008; Marengo & Aceto 2008; Pessanha et al 2010), in other words, growth and fructification vine is directly influenced by the local environment where it was planted (Rogiers et al 2006; Pohl 2007; Dalipi et al 2015).

Mineral substances increase the food value of grapes, must and the wine, their role is very important in human body (K, Mg, and Ca). Exceptions are minerals contamination, especially Pb, this heavy metal is very toxic for human and animal organism. These minerals are necessary for the redox processes from must and wine, and in small quantities of this mineral are involve in physico-chemical stabilization processes (Țârdea 2007).

Regarding the Na concentration in the must (Table 2), the highest concentration was recorded in the Italian riesling variety ( $33.72 \pm 2.22$  mg/L), followed by Fetească albă ( $28.25 \pm 0.93$  mg/L) and Fetească regală ( $27.01 \pm 1.71$  mg/L). The differences between variants were statistically assured ( $F = 13.044$ ,  $p = 0.007$ ) (Table 2). The obtained results for the Na concentration are comparable to those obtained by Magdaș et al (2012) (24.49 mg/L).

The presence of Ca in must is normal and derives mostly from grapes, where it accumulates until ripening (Țârdea 2007; Cotea 2010). Țârdea 2007 claims the concentration of Ca in must may be comprised between 50-200 mg/L. The highest concentration of Ca was recorded in the must of Italian riesling grape variety ( $216.27 \pm 5.06$  mg/L), followed by Fetească albă ( $209.34 \pm 4.39$  mg/L) and Fetească regală which has the lowest concentration of Ca ( $187.77 \pm 5.80$  mg/L) compared to the other two varieties examined. The differences between variants were statistically assured ( $F = 25.276$ ,  $p = 0.001$ ) (Table 2). Results are significantly lower than those obtained by Sani (2012) (817 ppm).

Table 2  
The content of macro-, microelements in must (mg/L) from Turulung Vii area, year 2013

| Variety           | Na                 | Ca                  | Mg                  | Fe                 | Co   |
|-------------------|--------------------|---------------------|---------------------|--------------------|------|
| Fetească albă     | $28.25 \pm 0.93^b$ | $209.34 \pm 4.39^a$ | $196.06 \pm 7.77^b$ | $15.34 \pm 0.72^c$ | BLD* |
| Fetească regală   | $27.01 \pm 1.71^b$ | $187.77 \pm 5.80^b$ | $178.24 \pm 3.00^c$ | $19.08 \pm 1.25^b$ | BLD  |
| Italian riesling  | $33.72 \pm 2.22^a$ | $216.27 \pm 5.06^a$ | $218.29 \pm 1.74^a$ | $22.53 \pm 2.03^a$ | BLD  |
| Average           | $3.21 \pm 0.05$    | $204.46 \pm 5.08$   | $197.53 \pm 4.17$   | $18.98 \pm 1.33$   | -    |
| Minimum values    | $27.01 \pm 1.71$   | $187.77 \pm 5.80$   | $178.24 \pm 3.00$   | $15.34 \pm 0.72$   | -    |
| Maximum values    | $33.72 \pm 2.22$   | $216.27 \pm 5.06$   | $218.29 \pm 1.74$   | $22.53 \pm 2.03$   | -    |
| F (Fisher Factor) | 13.044             | 25.276              | 49.935              | 18.723             | -    |
| Sig.              | $p = 0.007$        | $p = 0.001$         | $p \leq 0.000$      | $p = 0.003$        | -    |

Average value  $\pm$  standard deviation (n=3). Different letters are significantly different for  $P \leq 0.05$  between varieties. Within a column, the difference between any two values, followed by at least one common letter, is insignificant.

\*BLD= below limit of detection

Mg is a macroelement involved in grapevine nutrition and directly involved in the formation of chlorophyll. It accumulates in low concentrations in grapes. During the alcoholic fermentation, it enables phosphorylation of sugars. The values obtained for Mg concentration were within the maximum value of  $218.29 \pm 1.74$  mg/L recorded in Italian riesling variety and the minimum of  $178.24 \pm 3.00$  mg/L recorded in Fetească albă. The differences between variants were statistically assured ( $F = 49.935$ ,  $p \leq 0.000$ ) (Table 2). Results are significantly lower than those obtained by Magdaș et al (2012) (219.39 mg/L).

The concentration of Fe in must ranged from the maximum value of  $22.53 \pm 2.03$  mg/L recorded by Italian riesling, followed by Fetească regală ( $19.08 \pm 1.25$  mg/L), while the lowest concentration of Fe in must was recorded in Fetească albă ( $15.34 \pm 0.72$  mg/L). The differences between variants were statistically assured ( $F = 18.723$ ,  $p = 0.003$ ) (Table 2). Cotea (2010) stated that the small amounts of Fe come from grapes as a result of the nutrition process of the vine (2-5 mg/L) and is called physiological iron.

Regarding the concentration of Co, the metal is below the limit of detection in must.

Țârdea (2007) say, that the vine takes minerals in the soil solution in the form of cations and anions. The predominant minerals are phosphorus, copper, zinc, lead, aluminum. The mineral substances quantity of grapes is highly variable from 1-3% depending from variety of vine, type of soil in the vineyard and the annual rainfall.

**Macro- and microelements from wine.** The content of wine in minerals, varies depending on the grape variety from which it originates, the area of production, the raw material processing technology, and applied treatments on must and wine. Red wines, but also flavored ones, contain more minerals than white wines. During the fermentation of must, but also during the storage of wine, the mineral content decreases due to assimilation by yeasts, but also due to precipitation and their deposit (Cotea 2010).

It should be noted that certain inorganic elements in wine such as Cu, Zn, As, Cd, Pb, Co and Fe at different stages of wine are of great interest because of legal requirements (Voica et al 2009). Determination of mineral wine is extremely important not only for the wine industry, as concern wine quality problems, but this can be viewed from the perspective of health wine consumers (Rovio et al 2011).

Table 3  
The content of macro-, microelements in wine (mg/L) from Turulung Vii area, year 2013

| Variety           | Na<br>60 mg/L**    | Ca                 | Mg                  | Fe                | Co   |
|-------------------|--------------------|--------------------|---------------------|-------------------|------|
| Fetească albă     | $23.16 \pm 1.87^a$ | $60.04 \pm 1.95^b$ | $60.41 \pm 3.17^b$  | $3.35 \pm 0.02^b$ | BLD* |
| Fetească regală   | $23.69 \pm 3.33^a$ | $53.52 \pm 1.81^c$ | $51.31 \pm 2.35^c$  | $3.66 \pm 0.11^a$ | BLD  |
| Italian riesling  | $24.42 \pm 3.84^a$ | $85.60 \pm 2.15^a$ | $102.67 \pm 4.63^a$ | $2.61 \pm 0.02^c$ | BLD  |
| Average           | $23.76 \pm 3.01$   | $66.39 \pm 1.97$   | $71.46 \pm 3.38$    | $3.21 \pm 0.05$   | -    |
| Minimum values    | $23.16 \pm 1.87$   | $53.52 \pm 1.81$   | $51.31 \pm 2.35$    | $2.61 \pm 0.02$   | -    |
| Maximum values    | $24.42 \pm 3.84$   | $85.60 \pm 2.15$   | $102.67 \pm 4.63$   | $3.66 \pm 0.11$   | -    |
| F (Fisher Factor) | 0.123              | 220.429            | 181.977             | 147.672           | -    |
| Sig.              | $p = 0.886$        | $p \leq 0.000$     | $p \leq 0.000$      | $p \leq 0.000$    | -    |

Average value  $\pm$  standard deviation (n=3). Different letters are significantly different for  $P \leq 0.05$  between varieties. Within a column, the difference between any two values, followed by at least one common letter, is insignificant.

\*BLD = below limit of detection

\*\*MLA = maximum limit allowed (codex OIV)

Sodium, as macroelement, is widespread in nature, but in wine it can be found in very low concentrations (10-40 mg/L). In the case of wines produced from grapes that were harvested from the sea areas or planted vines on halomorphic soils and indigenous vine plantations, sodium concentration may increase. The OIV codex allow a maximum sodium content of 60 mg/L (2.6 mEq/L). Regarding the Na concentration in wine (Table 3), it can be seen that, in statistical terms, the three studied species are equal ( $23.16 \pm 1.87$  mg/L Fetească albă,  $23.69 \pm 3.33$  mg/L Fetească regală, respectively  $24.42 \pm 3.84$  mg/L Italian riesling). The differences between variants were statistically assured ( $F = 0.123$ ,  $p = 0.886$ ) (Table 3).

The results are comparable to those obtained by Galgano et al (2008) ( $28.47 \pm 14.87$  ppm), Avram et al (2014) (33.10 mg/L), and higher than those presented by Kment et al (2005) ( $14.7 \pm 12.7$  mg/L). The values of Pérez et al (2011) are significantly higher than those presented by the present study ( $57.7 \pm 30.5$  mg/L); and as those

presented by Paneque et al (2010) ( $42.4 \pm 9.8$  mg/L); Laurie et al (2010) ( $30.3 \pm 13.3$  mg/L).

Comparing the results for sodium with the maximum set by the OIV codex (60 mg/L), it can be noted that all tested cultivars registered Na concentrations well below this limit. In the case of Murfatlar vineyard, Romania, the Sauvignon wines had a sodium content of 48-58 mg/L, Chardonnay wines of 60-70 mg/L (Marin et al 1996, cited by Țârdea 2007).

Wine always contains less calcium than the must of origin, responsible for the decrease in the calcium concentration is the alcohol which helps the insolubilization of calcium tartrate. White wines have a much higher concentration of calcium than red wines. Normally the concentration of Ca in must is between 80-150 mg/L (Cotea 2010).

The values of Ca concentration in the wine samples from Turulung Vii ranged from  $85.60 \pm 2.15$  mg/L in Italian riesling, followed by Fetească albă ( $60.04 \pm 1.95$  mg/L). The opposite was found in Fetească regală which has the lowest concentration ( $53.52 \pm 1.81$  mg/L). The differences between variants were statistically assured ( $F = 220.429$ ,  $p \leq 0.000$ ) (Table 3).

The results are similar to those obtained by Odăgeriu (2006), who analyzed white wines from the Bucium-Iași area and reported a Ca content between 78-98 mg/L, comparable to those obtained by Galgano et al (2008) ( $83.17 \pm 13.91$  ppm); Pérez et al (2011) ( $80.5 \pm 15.7$  mg/L); Paneque et al (2010) ( $68.3 \pm 16.2$  mg/L); Laurie et al (2010) ( $75.2 \pm 12.2$  mg/L), and much lower than those presented by Kment et al (2005) ( $108 \pm 45$  mg/L); Avram et al (2014) (40.78 mg/L). In the results presented by Iochims dos Santos et al (2010), the values are much lower than the ones shown by us ( $42 \pm 12$  mg/L), but the research has been conducted on a red variety - Cabernet sauvignon.

Magnesium is better represented in the wine than calcium. Its salts are stable, and the concentration does not decrease after the end of the fermentation by precipitation, as is the case of the calcium concentration.

Regarding the concentration of Mg in wine, it can be noticed that the Italian riesling variety has the highest values ( $102.67 \pm 4.63$  mg/L), followed by Fetească albă ( $60.41 \pm 2.35$  mg/L) and Fetească regală ( $51.31 \pm 2.35$  mg/L). Magnesium is present in wine in higher quantity than calcium, concentrations are relatively constant in both red and white wines (Țârdea 2007). The differences between variants were statistically assured ( $F = 181.977$ ,  $p \leq 0.000$ ) (Table 3).

The results presented by Iochims dos Santos et al (2010) show values lower than those shown in our study ( $58 \pm 15$  mg/L) and comparable to those obtained by Galgano et al (2008) ( $116 \pm 16.51$  ppm), Pérez et al (2011) ( $112.5 \pm 41.8$  mg/L), Kment et al (2005) ( $75.4 \pm 16.8$  mg/L), Paneque et al (2010) ( $65.9 \pm 9.4$  mg/L), Laurie et al (2010) ( $96.4 \pm 17.4$  mg/L), Avram et al (2014) (72.56 mg/L).

The range for the concentration of Fe in wine was comprised between the maximum value obtained in Fetească regală ( $3.66 \pm 0.11$  mg/L) and the minimum, recorded in Italian riesling ( $2.61 \pm 0.02$  mg/L). The presence of iron in large quantities in wine is not desired, since it is the main cause favoring flaws in wine (white phosphato-ferric scrapping and black tanato-ferric scrapping, which degrades the color of wine). Normally, through the nutrition of vine, wine contains small amounts of iron 2-3 mg/L, but it can further be enriched with iron of an exogenous origin, which is due to utensils and equipment used in viticulture and winemaking. In the current study, the iron concentration obtained is normal. The differences between variants were statistically assured ( $F = 147.672$ ,  $p \leq 0.000$ ) (Table 3).

Comparing our results to the international literature, it can be concluded that the results presented by Iochims dos Santos et al (2010) ( $2.97 \pm 1.2$  mg/L) are comparable to ours. Similar results were obtained by Galgano et al (2008) ( $3.92 \pm 1.16$  ppm), Kment et al (2005) ( $2.64 \pm 1.06$  mg/L), Paneque et al (2010) ( $3.51 \pm 2.69$  mg/L), but the results presented by Pérez et al (2011) ( $1.55 \pm 0.96$  mg/L) and Laurie et al (2010) ( $1.64 \pm 1.36$  mg/L), are much lower than those obtained at Turulung Vii.

The concentration of Co in the wine samples is below the detection limit of the device.

**Pearson correlation coefficients of micro-, macroelements in must and wine.** To reveal if the concentration of micro- and macroelements in must influence the concentration of the micro- and macroelements from wine, in this sense, we have been performed Pearson correlations, between average values of metals from must and wine.

Based on Pearson correlation coefficients presented in Table 4, in this case were obtained many strong relationships between micro- and macroelements from must and wine Ca&Na (0.757\*\*); Mg&Na (0.778\*\*); Fe&Na (0.775\*\*); Mg&Ca (0.990\*\*); Fe&Ca (0.945\*\*); Fe&Mg (0.930\*\*).

Table 4  
Pearson correlation coefficients of micro-, and macroelements in must and wine\*

|    | <i>Na</i>        | <i>Ca</i>        | <i>Mg</i>        | <i>Fe</i>        | <i>Co</i>      |
|----|------------------|------------------|------------------|------------------|----------------|
| Na | 1.000            | 0.757**          | 0.778**          | 0.775**          |                |
|    | <i>p</i> = ...   | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> = ... |
| Ca | 0.757**          | 1.000            | 0.990**          | 0.945**          |                |
|    | <i>p</i> ≤ 0.000 | <i>p</i> = ...   | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> = ... |
| Mg | 0.778**          | 0.990**          | 1.000            | 0.930**          |                |
|    | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> = ...   | <i>p</i> ≤ 0.000 | <i>p</i> = ... |
| Fe | 0.775**          | 0.945**          | 0.930**          | 1.000            |                |
|    | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> ≤ 0.000 | <i>p</i> = ...   | <i>p</i> = ... |
| Co |                  |                  |                  |                  |                |
|    | <i>p</i> = ...   | <i>p</i> = ...   | <i>p</i> = ...   | <i>p</i> = ...   | <i>p</i> = ... |

\*significant correlation for *p* < 0.05, at the 95% confidence level; N = 18

In other words, the concentration of micro- and macroelements from wine is directly influenced by the concentration of micro- and macroelements from must. The most important aspect is the concentration of these metals, or the quantity in which are found in wine.

**Translocation factor of metals in system must-wine.** In order to reveal traceability of analyzed metal ions (Na, Ca, Mg, Fe, Co), on the must-wine chain, the transfer factor was calculated (Table 5). The translocation (transfer) factor (TF) was calculate as the ratio between metal concentration of wine and must  $TF_{wm} = C_{wine}/C_{must}$  (Li et al 2010; Peter et al 2011).

Table 5  
Translocation factors in system wine-must

| <i>Variety</i>   | <i>Na</i> | <i>Ca</i> | <i>Mg</i> | <i>Fe</i> | <i>Co</i> |                |
|------------------|-----------|-----------|-----------|-----------|-----------|----------------|
| Fetească albă    | 0.820     | 0.287     | 0.308     | 0.218     | 0.000     |                |
| Fetească regală  | 0.877     | 0.285     | 0.288     | 0.192     | 0.000     |                |
| Italian riesling | 0.724     | 0.396     | 0.470     | 0.116     | 0.000     |                |
| Average          | 0.807     | 0.323     | 0.356     | 0.175     | 0.000     | Na>Mg>Ca>Fe>Co |
| STDEV            | 0.077     | 0.063     | 0.100     | 0.053     | 0.000     |                |
| RSD %            | 9.571     | 19.671    | 28.103    | 30.349    | -         | Fe>Mg>Ca>Na>Co |

SDTV= standard deviation

RSD%= relative standard deviation

In the case of the transfer factor between wine and must, the highest values were obtained in the analyzed macroelements (Na>Mg>Ca) followed by microelements (Fe>Co).

On the other hand, it was shown that the values of the transfer coefficients in wine and must represent subunitary values, fact explained by the physico-chemical and biological transformations occurring when must turns into wine. These transformations reduce the concentration of metals, fact demonstrated both by the lower values of metals analyzed from wine compared to must, or by the subunitary values of transfer factors.

**Conclusions.** Regarding the cultivars taken for testing, based on analyzes, it can be concluded that the highest values of three mineral concentrations (Mg, Ca, Na) of both must and wine samples have been registered in the Italian riesling cultivar. On the other hand, Fetească regală has the highest concentration of Fe in wine samples, followed by Fetească albă cultivar. The concentration of Co in must and wine samples was below the detection limit of the device for all three varieties. Comparing the results for Na with the maximum set by the OIV codex (60 mg/L), it can be noted that all tested cultivars registered Na concentrations well below this limit. It can be noted that the metal ions analyzed have a higher concentration in must compared to those from wine. This reduction in concentration is mainly due to the alcoholic fermentation, where most of these metals pass under insoluble compounds form and are found in the yeast. Person's correlation analysis revealed a number of strong inter-elemental relationships. The concentration of micro-, and macroelements from wine is directly influenced by the concentration of micro-, and macroelements from must. The most important aspect is the concentration of these metals or the quantity in which are found in wine.

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