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## ***Trans*- and *cis*-resveratrol concentration in wines produced in Serbia**

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**Abstract:** Resveratrol, which occurs in two isomeric forms, *trans* and *cis*, is a phytoalexin with numerous pharmacological activities, such as anti-cancer, antiviral, neuroprotective and anti-aging. Red wine is the main source of the compound and an easy way of including resveratrol in the human diet. In this study, the most popular commercial Serbian wines (red, white and rosé-type) were analyzed for their content of *trans*- and *cis*-resveratrol. The analysis was performed by HPLC with a UV detector. Prior to the injection, phenolic compounds were extracted onto a LiChrolut RP18 bonded silica cartridge. The concentration of *trans*-resveratrol ranged from 0.11 to 1.69 mg L<sup>-1</sup> and *cis*-resveratrol from 0.12 to 1.49 mg L<sup>-1</sup>.

**Keywords:** *trans/cis*-resveratrol; wine; HPLC.

### INTRODUCTION

Wine is defined as the fermented juice of *Vitis vinifera* grapes used as a beverage, while viticulture is the cultivation of grapes especially for wine making.<sup>1</sup> Wine has been produced and enjoyed for thousands of years. References to wine can be seen in writings dating back to the days of Hammurabi and to the Bible.

Wines are known to contain many biologically active compounds. The amounts and compositions of these compounds depend on the type of grapes and their degree of ripeness, climate and soil of the viticultural area, as well as vinification techniques.

There are many different types of wines that can be produced by a variety of different methods. From red to rosé to white and from fortified to sparkling,

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wines can be produced with a variety of different flavors, aromas, in addition to alcohol contents.

Phenolic compounds are an important group of substances in wine that contribute to several sensory characteristics, such as color, flavor and astringency. Furthermore, it has been reported that phenols have multiple biological effects, such as antioxidant activity, anti-inflammatory action, inhibition of platelet aggregation and antimicrobial activity.<sup>2</sup>

Several beneficial physiological properties have been attributed to wine. It has been shown to have anticancer (chemo-preventive), anti-inflammatory, anti-fungal, and anti-microbial properties. There is also considerable evidence that a correlation exists between red wine consumption and the prevention of coronary heart disease, due to its beneficial anti-oxidant effect on low-density lipoprotein (LDL), which causes arteriosclerosis.<sup>3</sup> The cancer chemopreventive properties of resveratrol were first appreciated when Jang *et al.* demonstrated that resveratrol possesses cancer chemopreventive activity against all the three major stages of carcinogenesis *i.e.*, initiation, promotion and progression.<sup>4</sup> Some findings suggest that resveratrol may also be anticarcinogenic and a potent chemopreventive agent for breast cancer.<sup>5</sup> Traditional Japanese and Chinese folk medicine use root extract of the weed *Polygonum cuspidatum*, which contains resveratrol, to fight liver, skin and circulatory-diseases.<sup>6,7</sup>

One of the important phenolic bioactive constituents in wine is resveratrol (3,4',5-trihydroxystilbene), a naturally occurring phytoalexin produced by some spermatophytes, such as grapevines, in response to injury. In grape species, resveratrol reaches concentrations of 50–400 µg/g fresh weight in the leaves. Resveratrol is also synthesized in the berries and in lignified plant tissues. Concentrations in the skin (pericarp) of the berries are high compared with those in the flesh. During mashing, a part of the resveratrol from the skins is dissolved in the must. However, only low levels occur in white wine ( $\leq 1 \text{ mg L}^{-1}$ ), whereas red wines contain on average  $2 \text{ mg L}^{-1}$ .<sup>8–10</sup>

Resveratrol exists in *cis*- and *trans*- isomeric forms (Fig. 1), but the *cis*-isomer is present only in small amounts.<sup>11–13</sup> However, *cis*-resveratrol and its glucoside have been detected in almost all wines analyzed to date, regardless of the origin and the technology applied. Both isomeric forms were detected in white, rosé, and red wine. The *cis*-isomer is probably transformed during the vinification process but usually does not reach the concentration of the *trans*-isomer in the wine.<sup>14</sup>

Resveratrol is the parent molecule of a family of polymers named viniferins. Plants also synthesize glucosides (piceid = resveratrol 3-*O*- $\beta$ -glucoside).

The concentrations in the form of *trans*- and *cis*-isomers of aglycone and glucosides are subject to numerous variables. In red wine, the concentrations of the *trans*-isomer, which is the major form, generally ranges between 0.10 and  $15 \text{ mg L}^{-1}$ .<sup>2</sup>

Wines of the rosé type exhibit intermediate values between red and white wines. This is presumably due to the longer extraction time during contact between grape skin and juice in the production of red wine.<sup>15,16</sup>

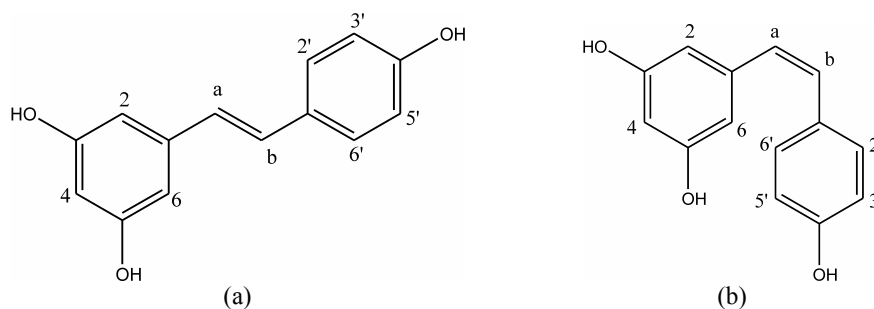


Fig. 1. a) *trans*-resveratrol, b) *cis*-resveratrol.

Resveratrol concentrations increase during fermentation of the skins but the amount extracted is dependent on the variety and enological conditions.<sup>17–19</sup> The extraction of resveratrol from the skin may be facilitated by the production of ethanol during the fermentation process.

Resveratrol is synthesized in response to microbial infection or stress.<sup>18</sup> However, it is also produced after chemical treatment, such as herbicide or fungicide application, and by exposure to UV light.<sup>20,21</sup>

The complexity of the wine matrix makes analysis by a single technique difficult. Usually, purification prior to the analysis is crucial. Thus, solid phase microextraction, solvent extraction, and elution through preconcentration columns are performed. High performance liquid chromatography (HPLC) and gas chromatography (GC) are the two major techniques used. Furthermore, these instruments can be coupled to various types of detectors, including UV, photodiode array (DAD),<sup>22</sup> chemiluminescent detection (CL),<sup>23</sup> fluorescence detection (FD),<sup>24</sup> and mass spectrometry (MS).<sup>13,25</sup>

The aim of the present study was to determine the *trans*- and *cis*-resveratrol content in 18 commercial samples of wines produced in Serbia. At the present time, there are no reports in the literature about the resveratrol content in wines from Serbia.

## EXPERIMENTAL

### *Wine samples and standards*

The analyses were performed on eighteen commercial Serbian wine samples (10 red, 7 white and 1 rosé), from six different producers. All wines were stored in the dark at 4.0 °C and analyzed immediately after bottle opening. *trans*-Resveratrol was purchased from Sigma Chemical Co. (St. Louis, MO). *cis*-Resveratrol is not commercially available and was therefore obtained through UV-photoisomerization (280–310 nm, for 10 h) of a standard solution (100 mg L<sup>-1</sup>) of *trans*-resveratrol. Under these conditions, 80 % of the *trans*-resveratrol

was converted into the *cis*-isomer (examined by HPLC and  $^1\text{H-NMR}$  spectroscopy). The  $^1\text{H-NMR}$  spectra and HPLC chromatograms of *trans*-resveratrol prior and after UV treatment are shown in Figs. 2–4.

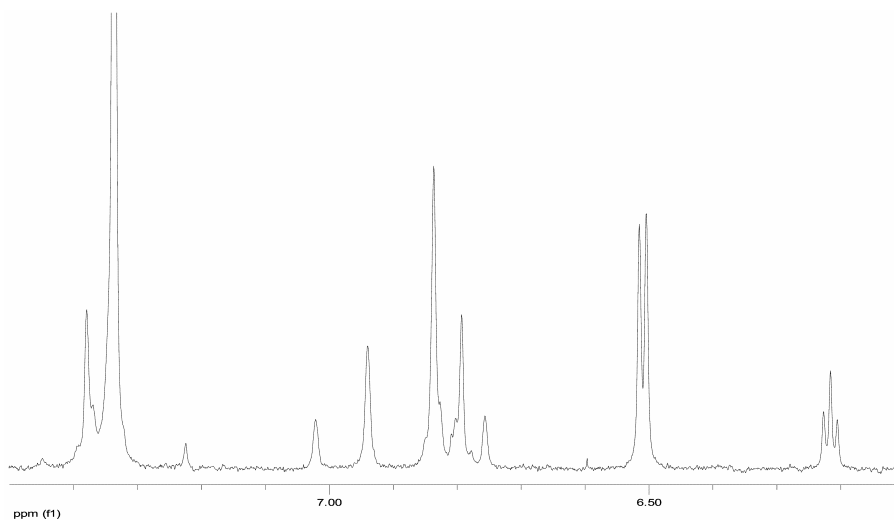


Fig. 2.  $^1\text{H-NMR}$  (200 MHz) spectra of *trans*-resveratrol in  $\text{CDCl}_3$  before UV-photoisomerization ( $\delta/\text{ppm}$ : 6.51 (2H, *d*, H-2/6,  $J = 2.0$  Hz), 6.21 (1H, *t*, H-4,  $J = 2.0$  Hz), 7.35 (2H, *d*, H2'/6',  $J = 8.5$  Hz), 6.81 (2H, *d*, H3'/5',  $J = 8.5$  Hz), 6.80 (1H, *d*, Ha,  $J = 16.5$  Hz), 6.97 (1H, *d*, Hb,  $J = 16.5\text{Hz}$ )).

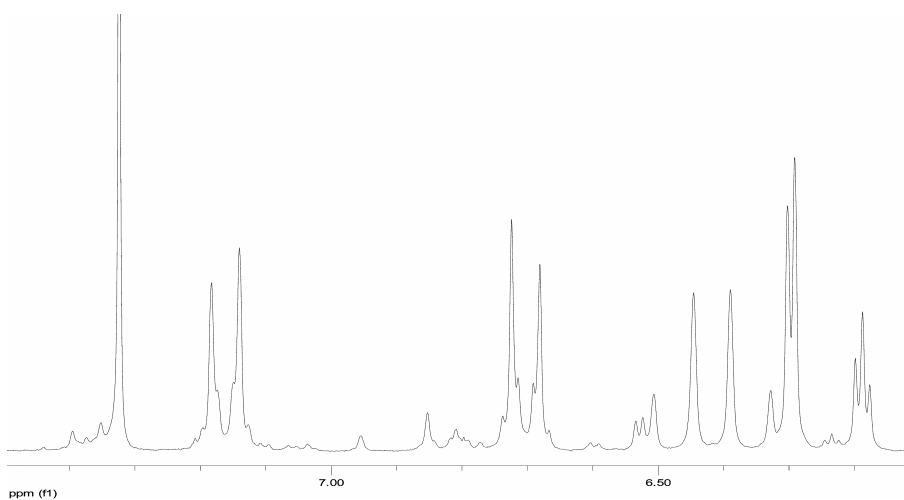


Fig. 3.  $^1\text{H-NMR}$  (200 MHz) spectra in  $\text{CDCl}_3$  of *cis*-resveratrol obtained after UV-photoisomerization of *trans*-resveratrol ( $\delta/\text{ppm}$ : 6.30 (2H, *d*, H-2/6,  $J = 2.0$  Hz), 6.19 (1H, *t*, H-4,  $J = 2.0$  Hz), 7.16 (2H, *d*, H2'/6',  $J = 8.5$  Hz), 6.70 (2H, *d*, H3'/5',  $J = 8.5$  Hz), 6.35 (1H, *d*, Ha,  $J = 12.0$ , Hz), 6.47 (1H, *d*, Hb,  $J = 12.0$  Hz)).

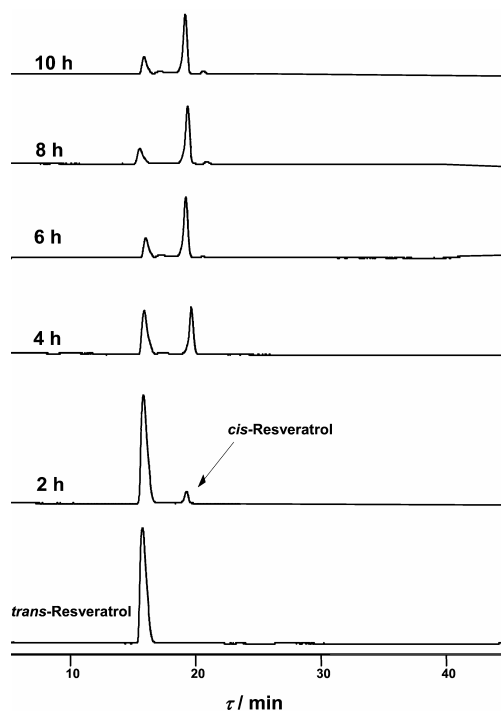


Fig. 4. LC chromatograms of the *cis*-resveratrol obtained during irradiation of *trans*-resveratrol.

#### Analytical HPLC procedure

A Hitachi HPLC system consisting of two pumps (Model 655A-11), a Hitachi automated gradient controller (Model L-5000) and autosampler Hitachi (Model 655A-40) was used. The chromatograms were recorded using a Gilson 117 UV dual wavelength detector. A Baseline Workstation CSW32 and a personal computer were employed for data storage and evaluation. The analytical column was a Bishoff Hyperchrome, ODS Hypersil (25 cm×4.6 mm ID, 5 μm particle diameter).

The HPLC column was initially equilibrated with acetonitrile–acetic acid–water (20:2:78, v/v) as solvent A for 10 min. The chromatographic separation was performed using a six stage linear gradient: from 100 to 90 % of A in 8 min, from 90 to 85 % of A in 12 min, from 85 to 70 % of A in 15 min, from 70 to 50 % of A in 5 min and from 50 to 0 % of A in 5 min, with a total flow rate of 1.0 ml min<sup>-1</sup>. The total gradient time was 42 min. A mixture of acetonitrile–acetic acid–water (90:2:8, v/v) was used as solvent B. The eluent was monitored at 306 and 286 nm, the optimum UV absorbances of *trans*- and *cis*-resveratrol, respectively.

#### Calibration and recovery

Calibration graphs were obtained by plotting the peak area against the concentration. Six standards of *trans*- and *cis*-resveratrol covering the range 0.10–15 and 0.25–10 mg L<sup>-1</sup> were made up in methanol and analyzed in duplicate. The constructed calibration curves showed excellent linearity (Table I).

The precision of the method was confirmed by repetitive analyses, calculating the average relative standard deviation (*RSD*) for 6 replicate determinations. The limit of detection (*LOD*, *S/N* = 3) of the individual compounds was calculated at their absorbance maxima. The

recoveries for *trans*- and *cis*-resveratrol were determined by adding known amounts of resveratrol to the wine sample and by performing assays before and after addition. The recovery was 94.56 and 95.61 % for *trans*- and *cis*-resveratrol, respectively (Table I).

TABLE I. Analytical characteristics for the determination of resveratrol in wine samples by HPLC–UV ( $t_R$  – retention time;  $\lambda$  – wavelength of absorbance maxima;  $R^2$  – correlation coefficient;  $RSD$  – repeatability)

Compound	$t_R$ / min	$\lambda$ / nm	Linearity interval mg L <sup>-1</sup>	$LOD$ / mg L <sup>-1</sup>	$R^2$	$RSD$ %	Recovery %
<i>trans</i> -Resveratrol	16.02	306	0.10–15	0.02	0.9998	3.3	94.56
<i>cis</i> -Resveratrol	18.53	286	0.25–10	0.05	0.9994	5.8	95.61

#### *Solid-phase extraction (SPE) of wine samples*

The determination of polyphenolic compounds in wine samples usually requires extraction and preconcentration procedures prior to the HPLC analysis. This is because wine has very complex composition and many phenolic compounds are present at very low concentrations.

A sample of wine was first filtered through a 0.45  $\mu$ m membrane filter. The solid-phase extraction was performed using a LiChrolut RP18 bonded porous silica cartridge (3 ml, 300 mg), obtained from Merck. The cartridges were previously conditioned with 10 ml of methanol, followed by 5 ml of water. Then, 5 ml of wine were introduced. Then the cartridge was dried under vacuum and, finally, the compounds were eluted with 1 ml of methanol. All analyses were performed in triplicate and the data are presented as mean  $\pm$  error (95 % confidence level,  $F = 4$ ,  $n = 5$ ).

#### *NMR analysis*

The <sup>1</sup>H-NMR (200 MHz) spectra were recorded on a Varian Gemini 2000 spectrometer in CDCl<sub>3</sub>.

## RESULTS AND DISCUSSION

The results of the chromatographic analyses of the 18 different Serbian wines are summarized in Table II. Typical chromatograms of red and white wine are shown in Figs. 5 and 6, respectively.

TABLE II. Concentrations of resveratrol in Serbian wines (mg L<sup>-1</sup>)

Wine and vintage	Wine producer	Alcohol content vol. %	<i>trans</i> -Resveratrol	<i>cis</i> -Resveratrol
Cabernet Sauvignon <sup>a</sup> 2000	Navip, Zemun	12.0	0.61 $\pm$ 0.04	0.27 $\pm$ 0.04
Cabernet Sauvignon <sup>a</sup> 2002	Podrum Radovanović, Krnjevo	12.5	1.69 $\pm$ 0.04	0.81 $\pm$ 0.04
Cabernet Sauvignon <sup>a</sup> 2004	Faculty of Agriculture, Uni- versity of Bel- grade, Zemun	13.0	0.18 $\pm$ 0.04	0.19 $\pm$ 0.04
Cabernet Sauvignon <sup>a</sup> 2002	Rubin, Kruševac	12.0	0.36 $\pm$ 0.04	0.42 $\pm$ 0.04

TABLE II. Continued

Wine and vintage	Wine producer	Alcohol content vol. %	<i>trans</i> -Resveratrol	<i>cis</i> -Resveratrol
Cabernet Sauvignon <sup>a</sup> 2004	VinoVita, Trstenik	12.5	1.00±0.04	1.49±0.04
Merlot "Dionis" <sup>a</sup> , 2000	Navip, Zemun	12.0	1.00±0.04	0.53±0.04
Merlot <sup>a</sup> , 2003	Faculty of Agriculture, Uni- versity of Bel- grade, Zemun	13.5	0.11±0.04	0.12±0.04
Royal <sup>a</sup> , 2000	Navip, Zemun	11.5	0.72±0.04	0.66±0.04
Pinot Noir <sup>a</sup> , 2001	Rubin, Kruševac	11.5	1.31±0.04	0.82±0.04
Vranac <sup>a</sup> , 2000	Rubin, Kruševac	11.5	0.84±0.04	0.20±0.04
Rosé <sup>b</sup> , 2004	Faculty of Agriculture, Uni- versity of Bel- grade, Zemun	12.0	0.29 ± 0.04	< 0.05 ± 0.04
Chardonnay <sup>c</sup> , 2002	Podrum Radovanović, Krnjevo	12.5	0.29±0.04	< 0.05±0.04
Chardonnay <sup>c</sup> , 2004	Faculty of Agriculture, Uni- versity of Bel- grade, Zemun	12.0	< 0.02 ± 0.04	< 0.05±0.04
Chardonnay blanc <sup>c</sup> 2002	Navip, Zemun	12.4	0.19±0.04	< 0.05±0.04
Chardonnay <sup>c</sup> , 2003	Rubin, Kruševac	12.0	0.15±0.04	< 0.05±0.04
Chardonnay <sup>c</sup> , 2002	Erdevik, Erdevik	12.5	0.34±0.04	0.40±0.04
Sauvignon <sup>c</sup> , 2003	Rubin, Kruševac	12.1	0.11±0.04	< 0.05±0.04
Graševina <sup>c</sup> , 2000	Erdevik, Erdevik	11.5	0.33±0.04	0.58±0.04

<sup>a</sup>Red wine; <sup>b</sup>rosé wine; <sup>c</sup>white wine

Only free forms of resveratrol were analyzed. The contents of resveratrol in red wines showed a relatively high variability. The concentration of *trans*-resveratrol ranged from 0.11 to 1.69 mg L<sup>-1</sup> and that of *cis*-resveratrol from 0.12 to 1.49 mg L<sup>-1</sup>.

The obtained resveratrol concentrations were compared with those reported for foreign wines. The concentrations of *trans*-resveratrol in the analyzed wines from Serbia were significantly lower than in wines produced in other countries. The average levels of *trans*-resveratrol in red and white wines vary greatly from one region to another. Although significant differences were found between regions, no specific region was significantly different from the others. A comparison of the content from different regions is given in Table III.

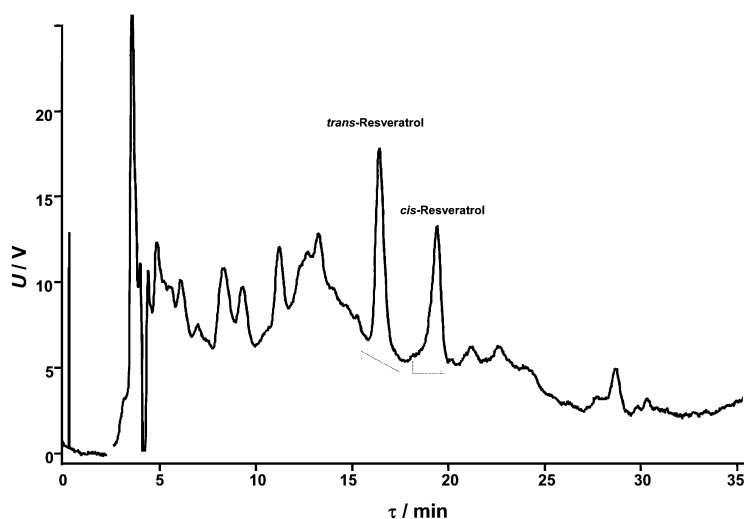


Fig. 5. Chromatogram of the red wine Cabernet Sauvignon (Vino Vita, Trstenik).

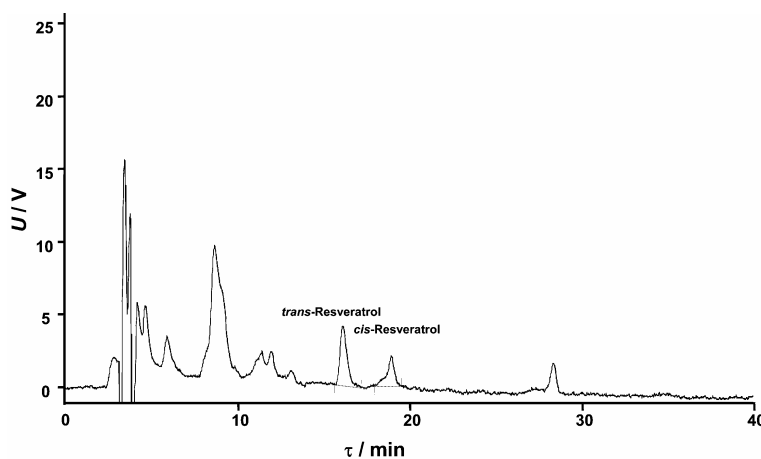


Fig. 6. Chromatogram of the white wine Chardonnay (Erdevik, Erdevik).

These differences could be attributed to environmental conditions, such as humidity and fungal disease, which are factors influencing the production of *trans*-resveratrol by grapevines.<sup>26</sup> However, Soleas *et al.*<sup>26</sup> stated that the difference between the statistical parameters, sample pretreatment (direct injection or not) and the chromatographic method (GC or HPLC) might cause different result. For example, wines from Italy presented mean values of resveratrol varying from 0.03 to 8.87 mg L<sup>-1</sup>.

Seven white wines were analyzed. As it can be seen (Table II), the highest amount of *trans*-resveratrol was found in Chardonnay 2002 (0.34 mg L<sup>-1</sup>). The highest value for *cis*-resveratrol in white wines was 0.58 mg L<sup>-1</sup> (Table II).



TABLE III. Previous reports of *trans* and *cis*-resveratrol concentrations (mg l<sup>-1</sup>) in red and white wines

Country	<i>trans</i> -Resveratrol		<i>cis</i> -Resveratrol		Reference
	Lowest	Highest	Lowest	Highest	
Red wines					
Brazil	0.82	5.75	1.70	22.9	27, 28
Italy	0.03	8.87	0.13	2.55	13, 24, 29, 30
France	0.30	7.62	0.30	5.30	31–36
Spain	0.18	8.00	0.02	2.48	33, 37–40
Canada	0.15	5.79	1.48	6.52	27, 41
China	0.07	3.20	–	–	23
Portugal	0.20	5.70	0.03	9.50	32, 36, 41, 42
Greece	0.37	2.53	–	–	34, 43, 44
Chile	0.80	1.57	0.14	1.23	33, 36
Czech Republic	0.92	6.25	0.68	2.80	45
Slovenia	0.90	8.70	–	–	39
USA	0.23	5.81	0.07	2.96	33, 36
Korea	0.19	3.30	–	–	46
Japan	0.001	2.30	–	–	14, 18
Hungary	0.10	14.3	–	–	47
Australia	0.20	10.6	–	–	36, 38
White wines					
Italy	0.02	0.55	–	–	24, 29, 30
France	0.30	4.00	0.10	0.10	35, 49
Spain	0.07	2.50	n.d.	0.18	37, 38, 50, 51
Portugal	0.03	2.60	0.90	1.70	32, 41
Greece	0.03	0.14	–	–	43
Slovenia	n.d.	0.60	–	–	39

### CONCLUSIONS

In this study, for the first time, focus was directed on the determination of *trans*-resveratrol and *cis*-resveratrol levels in red wines produced in Serbia.

The resveratrol content of a wine is related to the length of time the grape skins are present during the fermentation process. Thus, the concentration is significantly higher in red wines than in white wines, because the skins are removed earlier during white wine production, reducing the amount extracted.

*cis*-Resveratrol has been detected in almost all hitherto analyzed wines, regardless of the origin and the technology applied. Generally, the high levels of *cis*-resveratrol in Serbian wines support the assumption that resveratrol is initially produced as the *trans*-isomer and the *cis*-isomer is derived by subsequent isomerization of the *trans*-isomer, mainly during fermentation.

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## ИЗВОД

КОНЦЕНТРАЦИЈА *trans*- И *cis*-РЕЗВЕРАТРОЛА У ВИНИМА ПРОИЗВЕДЕНИМ У СРБИЈИ

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Методом течне хроматографије (HPLC) испитан је садржај слободног облика *trans*- и *cis*-резвератрола у осамнаест комерцијалних узорка (10 црвених, 7 белих и 1 розе) српских вина. Сви узорци су пре хроматографије екстраховани SPE техником на LiChrolut RP18 колони. *trans*-Резвератрол је детектован у 17 од 18 анализираних узорка вина са просечном концентрацијом од 0,78 mg l<sup>-1</sup> за црвена вина и 0,23 mg l<sup>-1</sup> за бела вина. Највиша концентрација *trans*-резвератрола је нађена у узорку црвеног вина Cabernet Sauvignon бербе 2002 године. *cis*-Резвератрол је детектован у 12 од 18 анализираних узорка вина са просечном концентрацијом 0,55 mg l<sup>-1</sup> за црвена вина, док је у белим винима од анализираних 7 узорка детектован само у 2 узорка са концентрацијом 0,12 и 0,49 mg l<sup>-1</sup>. Висок садржај *cis*-резвератрола у неким узорцима је вероватно последица изомеризације *trans*- у *cis*-резвератрол током процеса производње вина.

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