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The effects of the cherry variety on the chemical and sensorial characteristics of cherry brandy

NINOSLAV NIKIĆEVIĆ¹, MILOVAN VELIČKOVIĆ¹, MILKA JADRANIN², IVAN VUČKOVIĆ³, MIROSLAV NOVAKOVIĆ², LJUBODRAG VUJISIĆ³, MIROSLAVA STANKOVIĆ², IVAN UROŠEVIĆ¹ and VELE TEŠEVIĆ^{3*}

¹Faculty of Agriculture, University of Belgrade, Nemanjina 6, 11080 Zemun, ²Institute of Chemistry, Technology and Metallurgy, University of Belgrade, Njegoševa 12, 11000 Belgrade and ³Faculty of Chemistry, University of Belgrade, Studentski trg 16, 11000 Belgrade, Serbia

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Abstract: The chemical and sensorial characteristics of cherry brandy produced from five cherry varieties (Oblacinska, Celery's 16, Rexle, Heiman's Ruby and Heiman's Conserve) grown in Serbia were studied. Gas chromatography and gas chromatography–mass spectrometry analysis of these distillates led to the identification of 32 components, including 20 esters, benzaldehyde, 6 terpenes and 5 acids. The ethyl esters of C₈–C₁₈ acids were the most abundant in all samples. The benzaldehyde content was quantified by high performance liquid chromatography with UV detection. The average benzaldehyde concentration in the samples ranged between 2.1 and 24.1 mg L⁻¹. The total sensory scores of the cherry brandies ranged between 17.30 to 18.05, with the cherry brandy produced from the Celery's 16 variety receiving the highest score (18.05).

Keywords: aroma; benzaldehyde; cherry brandy; GC/MS; cherry varieties.

INTRODUCTION

Cherries are divided into sweet cherries (*Prunus avium*) and sour cherries (*P. cerasus*). There is archaeological evidence of sweet cherry about 5000 to 4000 BC in Switzerland, France, Italy, Hungary, Germany and England. The first description of sweet cherry was by Theophrastus (ca. 300 BC), who named it *kerasos*, after the town Kerasun in ancient Pontus on the Black Sea, but the town may have been named after the fruit. By Roman times, cherries were a common fruit and were described by Pliny and Virgil, but generally as wild trees.¹

Sour cherry fruits contain many volatile compounds and a number of these compounds, including benzaldehyde, linalool, hexanal, (2*E*)-hexenal, (2*E*), (6*Z*)-

* Corresponding author. E-mail: vtesevic@chem.bg.ac.rs
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-nonadienal, phenylacetaldehyde and eugenol, contribute to the fruit flavour and aroma.² The majority of the quantitative and qualitative changes in volatile production occur during fruit development and ripening.³

The typical flavour of sour cherries is produced during processing into wine, liquor, juice, jam or fruit sauce. Benzaldehyde was determined to be the most important aroma compound in sour cherries,⁴ but benzyl alcohol, eugenol and vanillin are also important.⁵

Distilled spirits are produced from stone fruits such as cherry (Kirschwasser, Cherry, Kirsch), plum (Zwetschgenwasser, Slivovitz), yellow plum, and apricots not only in many regions of Europe, but also in many other parts of the world. The flavour of stone fruit spirits is mostly affected by the aroma compound benzaldehyde, which originates from the enzymatic degradation of amygdalin in the stones of the fruits, passing into the mash during fermentation and later into the distillate at rather high levels.

Kirschwasser is mainly produced in southern Germany, France and Switzerland by crushing different kinds of sweet cherries, and leaving the mashed mass to ferment for several weeks. The fermented mash is then distilled in copper stills on open fire or vapour, whereby the first running and the tailings are removed. The resulting distillate has an alcohol content of approx. 60 vol. % and more and is marked as a clear, colourless fruit spirit with an alcohol content of 40–50 vol. %. Kirschwasser is also used as an additive for different liqueurs (*e.g.*, Curacao, Cherry Brandy, Maraschino *etc.*).

Aroma compounds, their levels, odour attributes and thresholds are most important for quality and authenticity of distilled spirits and liqueurs. The composition of the volatile aroma compounds in distilled spirits has been widely investigated using gas chromatography and mass spectrometry.⁶ By direct injection of an alcoholic distillate, it is possible to determine more than 50 components within levels between 0.1 and 1.0 mg L⁻¹; special methods of extraction can be used to increase this number up to more than 1000 volatile substances. However, sensory analysis is still indispensable to describe and evaluate spirit drinks.⁷

The production of unique fruit brandies, the prominent place belonging to a sour cherry brandy, has a long tradition in Serbia. Favourable microclimatic conditions and pedological properties of Serbian soil resulted in Serbia holding fourth place in Europe for the production of this fruit. Sour cherry accounts for 9 % of the total fruit production in Serbia. For its importance in Serbia, sour cherry ranks third, following plum and apple. The annual production is of 89.814 t or 11.25 kg per capita.

In addition to production factors (alcoholic fermentation, distillation, distillate aging), the choice of the appropriate cultivar is of critical importance for the end quality of sour cherry brandy.

The aim of this study was to compare the influence of the cherry variety (Oblačinska, Celery's 16, Rexle, Heiman's Ruby, and Heiman's Conserve) on the composition of the volatile compounds in alcoholic distillates and on the sensorial characteristics.

EXPERIMENTAL

Materials and methods

Chemicals and reagents. Ethanol, NaCl, anhydrous sodium sulphate and dichloromethane were purchased from Merck (Darmstadt, Germany).

Samples. In the present study, the fruits of five sour cherry cultivars that had been grown near Belgrade (the experimental orchard of Radmilovac, property of the Faculty of Agriculture, University of Belgrade) were used. Healthy and technically mature fruit of the following cherry cultivars was used in the experiments: Oblačinska, Celery's 16, Rexle, Heiman's Ruby and Heiman's Conserve.

Fermentation was performed with the autochthonous micro flora over a period of 10 to 15 days. The distillation was performed with a traditional copper alembics of 80 L, which is a simplified type of the Charentais-type distiller. The fermented raw material was transferred to the vessel up to 3/4 of its capacity. Before the beginning of heating, the alembic was hermetically closed with dough in order to prevent any vapour leakage. The first distillation of the fermented mash was performed without separation of a head. Redistillation was realised using the same distiller, but now with separation of 1 % of head, the heart (when average alcoholic strength of the heart was 60 % v/v) and a tail. The heart, containing 60 % v/v of ethanol was diluted with distilled water down to a strength of 45 % v/v. All samples were filled into glass bottles and stored in the dark at 4 °C until analysis. All the tested samples were distinguished by a characteristic aroma and flavour and were colourless.

Alcoholic strength

The ethanol content in the distillates was determined using a pycnometric method according to European Union regulations.⁸

GC and GC/MS analysis volatile compounds

For a typical experiment, a 100-mL aliquot of each beverage was mixed with 50 mL of dichloromethane and continuously extracted (2 h). Then the extract was dried (2 h) over anhydrous sodium sulphate, and concentrated to 1.0 mL under nitrogen.

Gas chromatographic analysis was performed using an HP 5890 gas chromatograph equipped with a flame ionization detector (FID) and a split/splitless injector. The separation was achieved using an HP-5 (5 % diphenyl and 95 % dimethylpolysiloxane) fused silica capillary column, 30 m×0.25 mm i.d., 0.25 µm film thickness. The temperature of the GC oven was programmed from 50 °C (6 min) to 285 °C at a rate of 4.3 °C min⁻¹. Hydrogen was used as the carrier gas; flow rate: 1.6 mL min⁻¹ at 45 °C. The injector and detector temperatures were 250 and 280 °C, respectively. Injection mode: splitless delay, 1 min. The injection volume of the beverage extract was 1.0 µL.

Gas chromatographic–mass spectrometric analysis (EI) was performed using an Agilent 5973 Network chromatograph coupled to an Agilent 5973 MSD spectrometer. The separation was achieved on an Agilent 19091S-433 HP-5MS fused silica capillary column, 30 m×0.25 mm i.d., 0.25 µm film thickness. The temperature of the GC oven was programmed from 60 °C to 285 °C at a rate of 4.3 °C min⁻¹. Helium of grade 5.0 was used as the carrier gas; the inlet pressure was 25 kPa; the column flow: 1 mL min⁻¹ at 210 °C. The injector temperature

was 250 °C. The splitless injection mode was employed with a delay of 1 min. MS conditions: source temperature, 200 °C; interface temperature, 250 °C; *E* energy, 70 eV; mass scan range, 40–350 amu; scan speed, 1.1 scan s⁻¹. Identification of the components was based on the retention indices and comparison with reference spectra (Wiley 07 & NIST 05). Percentage (relative) of the identified compounds was computed from the GC peak area. All analyses were performed in triplicate and the data are presented as mean±error (95 % confidence level, *F* = 4, *n* = 3).

HPLC analysis and benzaldehyde content

The samples were filtered through a 0.45 µm nylon membrane and 10 µL directly injected into the chromatographic system. Benzaldehyde identity was confirmed by retention time and by spiking the sample with the standard.

The separation was performed with an HPLC apparatus (1100 Series Agilent Technologies) comprising an on-line degasser, binary pump, auto injector, column oven and photodiode array (PDA) detector, equipped with a Zorbax Eclipse XDB-C8 column (Analytical, 150×4.6 mm², 5 µm ID) maintained at 25 °C. The mobile phase was a mixture of solvent A (water) and solvent B (methanol) according to a combination of gradient and isocratic modes: 95 % A, 0 min; 90 % A, 4 min; 85 % A, 8 min; 80 % A, 12 min; 60 % A, 16 min; 0 % A, 20–25 min and 0–95 % A, 25–26 min (26 min stop time and 5 min post time), at a flow-rate of 1.0 mL min⁻¹. Detection was accomplished using a diode array detection system, storing the signals over the spectral range 190–400 nm. To obtain quantitative data, the primary detection wavelength used was 254 nm. A personal computer system running Agilent ChemStation software was used for data acquisition and processing. Quantifications were realised by the external standard method and calibration curves were constructed through linear regression of the data obtained for the mean peak area after triplicate injection of solutions containing 1.5, 5, 10, 15, 20 and 30 µg mL⁻¹ benzaldehyde. The constructed calibration curve showed excellent linearity (correlation coefficient: 0.99954). All analyses were performed in triplicate and the data are presented as mean ± error (95 % confidence level, *F* = 4, *n* = 3).

Sensory analysis

Sensory assessment of cherry brandy samples was performed using a modified Buxbaum model of positive ranking. This model is based on five sensorial experiences rated by a maximum of 20 points. The samples of cherry brandies were subjected to sensory evaluation by a panel comprising five qualified testers, all of them highly experienced in sensory testing.

Statistical analysis

The statistical significance of difference between the analyzed samples was evaluated by analysis of variance (one-way ANOVA) followed by the Tukey test.

RESULTS AND DISCUSSION

The volatile compounds identified in the five cherry spirits are presented in Table I. In total, 32 aroma compounds were identified, including esters, acids, benzaldehyde and the monoterpene linalool.

Numerically, esters were the main group in all the distillates. Fatty acid ethyl esters were by far the most abundant. Esters are mostly formed from esterification of fatty acids with alcohols during the fermentation and ageing process. Ester formation can be influenced by many factors, such as fermentation tempe-

ature, oxygen availability and fermentation strains. Ethyl esters are present in other drinks, such as whiskey, cognac and rum, as the result of yeast metabolism during fermentation and are associated with pleasant fruity flavours.

TABLE I. Aroma composition of the studied cherry brandies (mean±standard error (*SE*), %)

Component	Cherry variety					<i>R</i> ^a
	Oblačinska	Rexle	Heiman's Ruby	Heiman's Conserve	Celery's 16	
Isoamyl acetate	2.56±0.01	9.30±0.02	7.50±0.1	9.02±0.02	9.52±0.02	876
Benzaldehyde	0.27±0.03	3.55±0.02	0.58±0.03	0.58±0.03	2.59±0.02	961
Ethyl hexanoate	2.96±0.02	2.11±0.02	1.92±0.03	2.06±0.03	2.02±0.02	996
Linalool	0.29±0.02	0.74±0.03	0.58±0.03	0.64±0.03	0.70±0.02	1098
Methyl octanoate	0.56±0.03	0.34±0.03	0.11±0.03	0.23±0.02	0.31±0.03	1125
Limonene	tr.	tr.	tr.	tr.	tr.	1031
<i>cis</i> -Linalool oxide	tr.	tr.	tr.	tr.	tr.	1074
<i>trans</i> -Linalool oxide	tr.	tr.	tr.	tr.	tr.	1088
Octanoic acid	1.21±0.01	2.00±0.02	1.80±0.02	1.64±0.03	1.11±0.02	1143
Ethyl octanoate	19.0±0.03	17.13±0.03	16.76±0.03	17.02±0.03	16.24±0.02	1195
2-Phenylethyl acetate	0.21±0.03	0.51±0.03	0.49±0.03	0.48±0.02	0.81±0.02	1312
Methyl decanoate	0.34±0.01	0.26±0.03	0.25±0.02	0.22±0.03	0.26±0.02	1326
Benzyl acetate	tr.	tr.	tr.	tr.	tr.	1163
Ethyl benzoate	tr.	tr.	tr.	tr.	tr.	1170
α -Terpineol	tr.	tr.	tr.	tr.	tr.	1189
Decanoic acid	4.64±0.02	6.30±0.02	5.26±0.02	6.08±0.02	6.75±0.03	1354
Ethyl 9-decenoate	2.15±0.02	1.62±0.02	1.13±0.02	1.11±0.02	1.32±0.03	1362
Ethyl decanoate	28.44±0.01	23.99±0.01	28.36±0.02	26.67±0.03	24.88±0.02	1394
Isoamyl octanoate	0.40±0.02	0.41±0.02	0.75±0.02	0.77±0.02	0.54±0.02	1446
Undecanoic acid	2.83±0.02	2.93±0.02	3.26±0.03	3.26±0.01	2.93±0.02	1467
Ethyl undecanoate	9.11±0.02	6.65±0.02	9.53±0.02	8.85±0.01	8.03±0.02	1496
3-Methylbutyl dodecanoate	0.34±0.02	0.31±0.02	0.60±0.02	0.62±0.02	0.46±0.02	1505
Nerolidol	tr.	tr.	tr.	tr.	tr.	1534
Dodecanoic acid	0.20±0.02	0.34±0.02	0.33±0.01	0.25±0.02	0.28±0.02	1561
Ethyl dodecanoate	1.00±0.02	0.74±0.02	1.18±0.02	1.10±0.02	0.79±0.02	1576
3-Methylphenyl butanoate	0.23±0.02	0.32±0.02	0.34±0.02	0.36±0.02	0.39±0.02	1591
Tetradecanoic acid	1.56±0.01	0.72±0.02	0.25±0.02	0.53±0.02	0.48±0.02	1663
Ethyl 9-hexadecanoate	1.25±0.02	0.61±0.02	0.15±0.02	0.69±0.02	0.46±0.02	1972
Ethyl hexadecanoate	6.78±0.02	5.35±0.02	5.36±0.02	5.08±0.02	4.62±0.01	1993
Ethyl linoleate	3.36±0.02	3.46±0.02	2.41±0.02	2.33±0.02	2.72±0.02	2177
Ethyl oleate	7.16±0.02	6.58±0.02	7.20±0.02	6.83±0.02	6.04±0.01	2180
Ethyl stearate	0.30±0.02	0.31±0.02	0.12±0.02	0.22±0.02	0.25±0.02	2194

^aRetention index on HP-5 and according to *n*-paraffins

The concentrations of long chain acid ethyl esters (C₈–C₁₈) in the investigated distillates were similar. Ethyl octanoate and ethyl decanoate, which are considered important contributors to the aroma of alcoholic distillates, were found in the highest concentrations. Distillates obtained from Oblačinska and Heiman's Ruby cherry varieties (cv) had the highest content of these compounds (≈28 %).

They were also reported as important aroma constituents in several fruit species. For example, ethyl hexanoate, ethyl octanoate and ethyl decanoate, were identified as odour active compounds in apple and apricot distillates.⁹

Salo *et al.*¹⁰ identified ethyl esters of fatty acids with even numbers of carbons, between 6 and 12, as the major contributors to whisky flavour. Jounela-Eriksson¹¹ reported that if ethyl esters were added or removed from the spirits, a negative effect on the overall odour intensity resulted. Postel and Adam¹² and Schreier *et al.*¹³ also showed that ethyl esters could be used to analytically differentiate between cognacs and other groups of grape brandies.

The most significant acetate esters present in cherry distillates are isoamyl acetate and 2-phenylethyl acetate. These esters are present in plum,¹⁴ apricot,⁹ apple⁹ and cornelian cherry¹⁴ brandy and are mainly responsible for the floral and fruity aroma of the distillates.¹⁵

The isoamyl acetate concentrations in the present samples ranged between 2.56 % (Oblačinska cv) and 9.52 % for distillate obtained from Celery's 16 cv. Highest content of 2-phenylethyl acetate (0.81 %) was found in the distillate obtained from Celery's 16 cv.

Free fatty acids are normal components of distilled alcoholic beverages and are mainly produced *via* yeast metabolism of carbohydrates. Long chain fatty acids, octanoic, nonanoic, dodecanoic, tetradecanoic and hexadecanoic acid, have a smaller effect on the flavour distillates.¹⁶ Five fatty carboxylic acids were identified in the analyzed samples.

Table I shows that decanoic acid has the highest mean value of all these acids, followed by undecanoic acid, octanoic acid, tetradecanoic acid and dodecanoic acid.

Terpenoids in distillates are formed in the fruits during the fermentation period, and pass into the distillate during distillation.¹⁷

In the present study, only six terpenes, *i.e.*, limonene, *cis/trans*-linalool oxide, linalool, α -terpineol and nerolidol, were detected. Despite the low concentration of these compounds, their presence is relevant because they harmonically synergize to produce the characteristic cherry aroma.

Linalool is a naturally-occurring terpene alcohol found in many flowers and spice plants with many commercial applications, the majority of which are based on its pleasant scent (floral, with a touch of spiciness). Linalool was previously reported as a constituent of the endogenous fruit aroma of sour cherry fruits. Li-

nalool has an aroma with a sweet, floral alcoholic note and its aroma threshold is in the range of 4–10 ppb.¹⁸

The linalool content of the analyzed samples was in the range of 0.29–0.74 %. The highest content was found in the Rexle cv distillate (0.74 %), and then in the Celery's 16 cv distillate (0.70 %). In contrast, the Obláčinska cv distillate, rated the lowest (17.30 %) of all the investigated distillates, had the lowest content of linalool.

Products of cyanogenic glycoside decomposition are important components of the aroma of some alcoholic beverages. These constituents occur mainly in fruit brandies produced from stone fruits, lending them the characteristic aroma of bitter almonds. The fruits of *Prunus* genus plants (plums, sour cherries, sweet cherries, apricots, peaches, etc.) contain amygdalin in the stones and prunasin mainly in the vegetative parts of the plants. Through enzymatic hydrolysis with the participation of β -glucanase and hydroxynitrilase or at elevated acidity, amygdalin is decomposed to form benzaldehyde, HCN and two glucose molecules.^{2,19,20}

The aroma of sour cherries was studied by Schmid and Grosch,² Poll and Lewis²¹ and, more recently, by Schwab and Schreier⁴ who studied the glycosidically bound aroma components. Benzaldehyde was found to be the most important aroma compound in sour cherries.²¹

Benzaldehyde content of the analyzed samples was in the range of 2.1–24.1 mg L⁻¹ (Table II). The highest benzaldehyde content was found in the Rexle cv distillate (24.1 mg L⁻¹), followed by the Celery's 16 cv distillate.

TABLE II. The content of alcohol and benzaldehyde in the investigated cherry brandies

Sample	Alcohol content, vol.%	Concentration of benzaldehyde, mg L ⁻¹
Obláčinska	41.5	2.1±0.5
Rexle	42.0	24.1±1.5
Heiman's Ruby	41.0	3.6±0.6
Heiman's Conserve	42.0	3.7±0.7
Celery's 16	42.5	14.6±1.1

Analysis of variance at the 0.05 level shows the population means were not significantly different (Table III).

TABLE III. Statistical significance of the difference between the data pairs evaluated by analysis of the variance (one-way ANOVA) followed by the Tukey test. Analysis of variance followed by the least significance at $p < 0.01$

Sample	Rexle	Heiman's Ruby	Heiman's Conserve	Celery's 16
Obláčinska	$p < 0.01$	$p < 0.01$	$p < 0.05$	$p < 0.01$
Rexle		$p < 0.01$	$p < 0.01$	$p < 0.01$
Heiman's Ruby			No statistical significance	$p < 0.01$
Heiman's Conserve				$p < 0.01$

Sensory evaluation

Total sensory quality of cherry brandies was between 17.30 and 18.05, which are very high scores (Table IV). According to the results of the performed sensory ranking, the best-rated brandy was the sample produced from Celery's 16, which was rated with a very high score by the five examiners (total sensory characteristics 18.05). It had the highest content of benzaldehyde and linalool as well as the most harmonious proportion of these two compounds to other aromatic components.

TABLE IV. Sensory analyses of the cherry brandies

Cherry brandy sample	Assessment characteristics					
	Colour (max 1 pt.)	Clearness (max 1 pt.)	Typicality (max 2 pts.)	Odour (max 6 pts.)	Taste (max. 10 pts.)	Total (max 20 pts.)
Oblačinska	1	1	2	5.10	8.20	17.30
Celery's 16	1	1	2	5.50	8.55	18.05
Rexle	1	1	2	5.40	8.50	17.90
Heiman's Ruby	1	1	2	5.30	8.20	17.50
Heiman's Conserve	1	1	2	5.20	8.20	17.40

CONCLUSIONS

All the investigated cultivars yielded brandies of very good to excellent quality. The dominant content of benzaldehyde, the significant amount of aromatic organic acids, as well as the harmonious proportion of the two characteristic and the most aromatic compounds, benzaldehyde and linalool, together with other compounds, esters and organic acids, are the main reasons why the sour cherry cultivars Celery's 16 and Rexle were rated best compared to the distillates from the other investigated cultivars.

The aromatic ester ethyl decanoate was present in the highest percent of all aromatic compounds found in the investigated distillates. Its content in some distillates (Oblačinska cv and Heiman's Ruby cv) accounted for nearly 1/3 of all the aromatic compounds. In addition, it is interesting to note that the *Oblačinska* cv distillate had the highest content of the aromatic esters ethyl octanoate and ethyl hexadecanoate. However, the presence of these esters is not followed by the presence of two key compounds benzaldehyde and linalool; hence, this might be the reason for the lower sensory rating of this distillate compared to others.

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ИЗВОД

УТИЦАЈ ВАРИЈЕТЕТА ВИШЊЕ НА ХЕМИЈСКЕ И СЕНЗОРНЕ КАРАКТЕРИСТИКЕ
РАКИЈЕ ВИШЊЕВАЧЕ

НИНОСЛАВ НИКИЋЕВИЋ¹, МИЛОВАН ВЕЛИЧКОВИЋ¹, МИЛКА ЈАДРАНИН², ИВАН ВУЧКОВИЋ³,
МИРОСЛАВ НОВАКОВИЋ², ЉУБОДРАГ ВУЈИСИЋ³, МИРОСЛАВА СТАНКОВИЋ²,
ИВАН УРОШЕВИЋ¹ и ВЕЛЕ ТЕШЕВИЋ³

¹Пољопривредни факултет, Универзитет у Београду, Немањина 6, 11080 Земун, ²Институт за хемију,
технолозију и металургију, Универзитет у Београду, Њежишева 12 11000 Београд и ³Хемијски факултет,
Универзитет у Београду, Студентски брз 16, 11000 Београд

Испитиване су хемијске и сензорске карактеристике ракије вишњеваче произведене из пет варијетета вишње (Облачинска, Celery's 16, Rexle, Heiman's Ruby и Heiman's Conserve) гајених у Србији. Методама гасне хроматографије и комбинацијом гасне хроматографије и масене спектрометрије у екстрактима идентификована су 32 једињења, 20 естара, бензалдехид, 6 терпена и 5 киселина. Етил-естри C₈-C₁₈ киселина су најобилнији у свим узорцима. Садржај бензалдехида је одређиван методом течне хроматографије уз UV детекцију. Просечна количина бензалдехида у испитиваним узорцима била је између 2,1 и 24,1 mg L⁻¹. Оцене сензорског испитивања ракија вишњевача су у распону од 17,30 до 18,05, док је најбоље оцењена (18,05) ракија произведена од варијетета Celery's 16.

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