

## Differences in concentration of principal volatile constituents in traditional Greek distillates

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### Abstract

Tsipouro is a strong distilled spirit produced from the wine-press residue. Some by-products of the alcoholic fermentation, such as acetaldehyde, ethyl acetate and amyl alcohols are mainly responsible for the flavour of alcoholic beverages and their amounts specify the quality of the distillate. In this study ten of the major volatile compounds were determined in commercially produced (bottled) and homemade tsipouro samples by GC analysis, and compared to other alcoholic distillates. Homemade tsipouro was found to have slightly higher concentrations for most of the studied compounds. It should be also pointed out that the toxic compound methanol was detected far below the acceptable legal limits.

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### 1. Introduction

The alcoholic beverage “tsipouro” is produced in Greece by distillation of the residual from wine-making, mostly the marc. Similar spirits have equivalent appellations, for example as Italian “grappa”, French “eau-de-vie de marc”, Spanish “aguardente”, Portuguese “bagaceira”, Cypriote “zivania”, Slavic countries “rakija” and the Georgian product “tshi-atshia”.

The EU regulation 1576/89 established the general production procedures for all these distillates and fixed common analytical composition limits. National regulations can specify certain production procedures and in particular cases, more restrictive analytical parameters for particular geographical denominations (EEC, 1989; Versini, 1993).

During grape marc distillate production, the slow distillation rate permits the separation of the distillate into three fractions: the “head”, the “heart” and the “tail” (Da Porto, 1998; Silva & Malcata, 1999). Methanol distills off throughout the entire distillation process

and shows a slight increase in the “tail” fraction (Da Porto, 1998). The definition of the times when the distillation cuts are made is still done in an empirical mode based on the particular skill of the distillery (Da Porto & Longo, 1997; Silva & Malcata, 1999).

In Greece before 1988, production licenses for tsipouro were granted for certain days of the year only to farmers, who were usually grape growers in poor areas. Today, the situation has changed as tsipouro has been acknowledged and appreciated by consumers and is appreciated by wine drinkers worldwide. Homemade tsipouro, which can be commercially available, is mostly consumed undiluted and its ethanol content is usually higher than 50% v/v. Commercially produced tsipouro is bottled after dilution with water to obtain a product of alcohol content between 37.5% and 50% vol. (Da Porto, 1998; Soufleros, 1987).

In the region of Epirus (in north-western Greece) tsipouro is produced by distillation of marc without adding any aromatic plants or herbs; therefore its aroma is attributed to the grape cultivars used, volatile alcoholic fermentation by-products and/or to products of chemical reactions between the above mentioned compounds. Traditional stills are normally made of copper. It is the preferred material, for its ease of working, good heat conduction, and wear resistance (Da Porto, 1998). These are used for homemade

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distillate production (Soufleros, 1987). Commercial tsipouro is the only one actually bottled and available for foreign sales while homemade is only for regional sale by the individual producing it within the territory where it is produced.

Homemade distillates attracted the researchers' interest because of the way they are produced and because of the alcoholic fermentation by-products, such as methanol, mainly produced in wines that remain in tanks for long periods with the pomace. This implies certain difficulties such as marc contamination with molds and spoilage bacteria producing off-flavours (Da Porto, 1998).

In the present study a gas chromatographic method was used for the quantification of the principal volatile substances (acetaldehyde, ethyl acetate, methanol, 1-propanol, 2-methyl-1-propanol, amyl alcohols, ethyl lactate, 1-hexanol, furfural and 2-phenyl ethanol) in homemade and commercially bottled tsipouro samples produced in the region of Epirus–Greece. The identification of the above compounds was performed by GC/MS analysis. Comparison of homemade and commercially produced (bottled) tsipouro is necessary to assess the best distillation procedures to be followed by individual distillers. Analysis was also conducted for the three distillation fractions (head, heart and tail) taken from one of the homemade tsipouro as well as for the samples taken from different production years for one of the bottled distillates.

## 2. Materials and methods

### 2.1. Samples

Five groups of different brands (A–E) of bottled tsipouro were analyzed. Each group consisted of five samples of the same commercial distillate. The same procedure was followed for the collection of five groups of homemade tsipouro samples (F–J). All the above samples (bottled and homemade) were produced in the region of Epirus, Greece (vintages 1998–1999). The raw material for the investigated distillates came from the following grape cultivars: *Vitis vinifera* var. Debina for samples A and H; a mixture of equal quantities of var. Xinomavro, Roditis and Red Cabernet for samples B and C; a mixture of equal quantities of var. Debina and Roditis for samples D and E; var. Isabella for sample F; a mixture of equal quantities of var. Kerino, Sultana and Red Cabernet for sample G; var. Aigiorgitiko for samples I and J. All samples were analyzed immediately after distillation.

During the production of sample H, amounts of the three different distillation fractions were collected and analyzed by the same procedure followed for all other samples.

### 2.2. Reagents

Standards of analytical quality were supplied by Merck (Darmstadt, Germany) and Sigma-Aldrich (Buchs, Switzerland).

### 2.3. Gas chromatographic (GC) analysis

Samples from each distillate were analyzed by direct injection into a gas chromatograph, using the internal standard method. 2-Octanol was used as internal standard. The GC unit used was a Fisons 9000 series gas chromatograph equipped with a FID detector. The separation column was a 30 m long  $\times$  0.32 mm internal diameter fused silica capillary DB-Wax with a film thickness of 0.25  $\mu$ m. The following GC parameters were kept constant: detector temperature, 240 °C; injector temperature, 200 °C; injection mode: split with split ratio ca. 50 ml/min; injection volume, 1  $\mu$ l. Column temperature program: 40 °C (7 min), from 40 °C at a rate of 15 °C/min to 160 °C (1 min), from 160 °C at a rate of 30 °C/min to 230 °C (5 min). Carrier gas: He, at a flow rate of 1.45 ml/min. Analyses were carried out in triplicate and their average were calculated.

### 2.4. GC/MS analysis

The preparation of tsipouro samples for the identification of the volatile compounds includes liquid/liquid extraction with  $\text{CH}_2\text{Cl}_2$ . The extracts were obtained using the following procedure: tsipouro (30 ml),  $\text{CH}_2\text{Cl}_2$  (2 ml) as extracting agent, 0.4 ml of 2-octanol 72.04 mg/l in  $\text{CH}_2\text{Cl}_2$  as internal standard, and sodium chloride (1 g), were transferred in a 50 ml Erlenmeyer flask cooled with melting crushed ice and magnetically stirred at 200 rpm for 2 h. The tsipouro/ $\text{CH}_2\text{Cl}_2$  emulsion formed during stirring was frozen at –20 °C for 30 min and then separated from the aqueous layer. Some anhydrous  $\text{Na}_2\text{SO}_4$  was added after extraction to eliminate residual water. The  $\text{CH}_2\text{Cl}_2$  extracts were transferred without concentration into a screw-cup vial and subjected to GC/MS analysis.

The GC/MS analysis was performed on a HP-6890 gas chromatograph coupled to an HP-5973 mass selective detector. The separation column, the GC parameters and the column temperature program were the same to those used for GC analysis. Carrier gas: He, at a flow rate of 1.5 ml/min. The temperature of the transfer line to the mass spectrometer was 260 °C and the source temperature 230 °C. All mass spectra were acquired in the electron impact (EI) mode at 70 eV, using full scan with a mass acquisition range of 28–550 amu. The identification of compounds from their mass spectra was carried out using computerized library search facilities (Wiley 275 L).

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