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(54) Title: PROCESS FOR ALCOHOL PRODUCTION

(57) Abstract: In one aspect, the present invention provides a method of increasing the alcohol concentration of a wine product, the method comprising the step of treating the wine product so as to reduce the amount of material from the wine product to produce a treated wine product. In a preferred embodiment, the treating step comprises subjecting the wine product to membrane filtration such as nanofiltration. Further processing steps may include ion exchange, sterile filtration and carbon filtration. Methods are also disclosed for further increasing the alcohol content in the treated wine product. The present invention also extends to a treated wine product having substantially no colour, flavour or odour yet having an alcohol concentration of at least about 4% (v/v) in addition to a treated wine product produced according to the method of the invention. The present invention further extends to an apparatus and its use in the production of a treated wine product.



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### "Process for alcohol production"

### Cross-Reference to Related Applications

The present application claims priority from Australian Provisional Patent Application No 2005901855 filed on 13 April 2005, the content of which is incorporated herein by reference.

### Field of the Invention

The present invention relates to spirit production and in particular, production of a novel wine product for use as a component of high quality spirits and other products having an alcohol component, for example, products such as perfumes, deodorants and the like. The present invention further relates to an apparatus for making the novel wine product.

### Background of the Invention

Traditionally, high quality spirits such as, for example, liqueurs, schnapps, whiskey, sherry and brandy containing high alcohol concentrations (usually above 22% v/v), are produced by either one of two methods: (i) distillation of a fermented product such as wine so as to increase their alcohol concentration, or (ii) fortification of an low alcohol concentration product (such as wine) with a high alcohol concentration spirit to increase their alcohol concentration.

In fortification, it is desirable that the fortifying alcohol be substantially pure or of a high quality, so that a high quality fortified product is obtained having the characteristics of good colour, clarity, taste and odour. As such, fortified alcohol products such as liqueurs, schnapps and brandy are produced by the fortification of wine with a high quality spirit which is expensive and therefore undesirable. Although low quality spirits are inexpensive and therefore present possible fortifying alcohols, they tend to be overly sweet, have a thick or "syrupy" consistency, and have low clarity (i.e., are cloudy). Such characteristics result in a lower quality fortified product and is therefore undesirable.

While high quality fortified beverages such as liqueurs and cream liqueurs tend to be produced through fortification, high quality spirits such as rum, vodka, whiskey are normally produced through distillation. Distillation, however, is quite an expensive process and therefore undesirable for use. In addition, many undesirable by-products are produced as part of the distillation process and so the distilled product often requires further processing so as to remove various dissolved solids that impart

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undesirable odours, flavours and sometimes colour to the spirit. This further processing adds to the expense and is therefore also undesirable.

There exists the need, therefore, for an alternative, high quality and high alcohol concentration source of alcohol that may be readily used for fortification of low alcohol concentration wines so as to produce high quality fortified products such as liqueurs, cream liqueurs, brandy and spirits.

### Summary of the Invention

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The present invention provides a method of increasing the alcohol concentration of a wine product, the method comprising the step of treating the wine product so as to reduce the amount of material from the wine product to produce a treated wine product.

Preferably the treated wine product has an alcohol concentration of above 4% (v/v), preferably between about 4% (v/v) to about 23% (v/v), more preferably between about 8% (v/v) to about 23% (v/v).

Preferably the material that is removed is in the size range of 100 daltons to 1600 daltons. In this way, materials such as dissolved solids, suspended solids, acids, tannin and microbes are substantially removed, yet the alcohol concentration is substantially unchanged with minimal, or no, removal of ethanol (alcohol).

A number of separation techniques may be utilised to remove the material which may be applied as a single treatment step or in combination with two or more treatment steps. Such techniques include, but are not limited to, membrane filtration including nanofiltration, ultrafiltration or microfiltration, carbon filtration, frame filtration, sterile filtration and ion exchange. The wine product may be subjected to a number of different treatment steps or the same treatment step more than once. For instance, the wine product may be subjected to nanofiltration through a plurality of nanofiltration units followed by further treatment such as ion exchange.

The treated wine product after at least one treatment step is separated into a alcohol-containing stream and a waste stream, wherein the alcohol-containing stream comprises most, or substantially all, of the alcohol from the wine product. Preferably, the alcohol concentration in the alcohol-containing stream increases with each treatment step, wherein only the alcohol-containing stream is subjected to further treatment after one or more treatment steps, while the waste-containing stream is discarded. In this way, by maintaining the alcohol concentration at a maximum within the alcohol-containing stream throughout the one or more treatment steps, recycling of the waste stream in order to recover alcohol is avoided. This results in a simple and efficient process.

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In one embodiment, the present invention provides a method of increasing the alcohol concentration of a wine product, the method comprising the step of treating the wine product with membrane filtration. Preferably, the membrane filtration is nanofiltration wherein one or more nanofiltration units are employed. More preferably, nanofiltration is used to remove material having a size of about 400 daltons to about 1600 daltons. In this way, material such as suspended and dissolved solids are removed yet none, or a minimal amount, of the alcohol is removed.

It will be appreciated that the nanofiltration units may be arranged in series or parallel or in two or more banks comprising a plurality of nanofiltration units. As such, each bank may be brought on line as required, depending on the wine product to be treated.

In accordance with another embodiment of the invention, the plurality of nanofiltration units may be arranged in banks wherein each bank comprises a two or more nanofiltration units arranged in series with the two or more banks arranged in parallel such that the permeate exiting each bank is combined and comprises an increased concentration of alcohol, yet is substantially devoid of other materials such as acids and suspended or dissolved solids.

In another embodiment of the invention, the wine product is passed through a plurality of nanofiltration units arranged in series wherein the retentate from each nanofiltration unit is discarded while the permeate from each nanofiltration unit is combined and comprises an increased concentration of alcohol, yet is substantially devoid of other materials such as acids and suspended or dissolved solids.

In another embodiment of the invention, the wine product is passed through one or more nanofiltration units, the plurality of units arranged in series, wherein the permeate from the one or more nanofiltration units comprises an increased concentration of alcohol, yet is substantially devoid of other materials such as acids and suspended solids. Unlike the previous embodiment, however, rather than discarding the retentate, this is recycled through the nanofiltration unit and subjected to diafiltration with a condensate. Preferably, the condensate is a condensate from the condensation of a wine product selected from any one of grape juice, low-alcohol wine or waste wine, or a combination of any thereof.

In yet a further embodiment of the invention, a wine product comprising high quality wine is passed through one or more nanofiltration units wherein the permeate from one or more nanofiltration units comprises an increased amount of alcohol. It will be appreciated that in previous embodiments low quality or waste wine is typically used such that the major stream (on a percentage volume basis) is typically the

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permeate stream which is to be used as the final product. The retentate stream is the minor stream (on a percentage volume basis) and normally discarded or subjected to further processing. By way of example, when the wine product to be treated comprises low quality or waste wine, the nanofilter may be configured to yield a permeate stream comprising 90% (v/v) of the inlet wine product and a retentate stream comprising 10 % (v/v) of the inlet wine product. In this further embodiment, however, since high quality wine is employed, the nanofilter is configured so that the major stream (on a percentage volume basis) is the retentate which will have an increased amount of flavour, colour and odour. The minor (on a percentage volume basis) permeate stream may be used as the final treated wine product or may be subjected to further processing so as to further increase its alcohol concentration.

The pH of the treated wine product following membrane filtration will typically be in the range of about 3.7 to about 4.3 and so buffering may be required to raise the pH to a level of about 6.0 to about 7.2, preferably about 6.5 to about 7.0, more preferably, about 7.0. Preferably also, the treated wine product after membrane filtration will have a titratable acidity of less than about 1.0 %, more preferably less than about 0.5 %. Preferably also, the treated wine product will be a clear liquid having an absorbance of 0.05 atomic units at about 430 nm.

In another embodiment of the invention, step of removing material so as to increase the alcohol concentration of a wine product achieved through one or more membrane filtration steps is followed by ion exchange. Preferably the wine product is deionised during the ion exchange treatment step, wherein material containing mono-, di-, or multi-valent ions is primarily removed, or substantially reduced. Following deionisation, the pH of the treated wine product will typically be in the range of about 6.5 to about 7.0, and so buffering may not be required. Preferably also, the treated wine product after membrane filtration and/or ion exchange will have a titratable acidity of less than about 1.0 %, more preferably less than about 0.05 %.

In yet another embodiment, the treated wine product following membrane filtration and/or ion exchange, is subjected to a sterile filtration step. Preferably the sterile filtration step follows the ion exchange treatment step. The wine product may also be subjected to a further fortification step, wherein fortification preferably occurs after the sterile filtration step. If the wine product has any residual colour, flavour or odour following membrane filtration or any subsequent treatment steps, it may be subjected to a further carbon filtration step.

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In a preferred embodiment, the step of treating the wine product comprises a nanofiltration step followed by ion exchange. Preferably, the wine product is deionised during the ion exchange step in this embodiment.

In another embodiment, the treated wine product may be used to as a base to produce a sweet wine such as, for example, late harvest Riesling, sweet liqueurs, port, sauternes and, sherry. In this embodiment, the wine product is first subjected to membrane filtration, preferably nanofiltration, followed by the addition of sugar.

In a particularly preferred embodiment of the invention, the present invention provides a method of increasing the alcohol concentration of a wine product by removing material from the wine product yet retaining all, or substantially all of the alcohol from the wine product, the method comprising subjecting the wine product to:

- (a) nanofiltration to remove or reduce the level of material having a size range of about 400 daltons to about 1600 daltons;
- (b) ion exchange to substantially remove or reduce the level of dissolved or 15 suspended material;
  - (c) sterile filtration to substantially remove any yeast, bacteria or moulds;
  - (d) optional fortification with a high alcohol-concentration spirit to increase the alcohol concentration of the treated wine product to at least about 20 %(v/v);
- (d) carbon filtration to substantially remove any residual colour bodies, flavour 20 and/or odour, and
  - (d) an optional polish filtration step through a filter having a pore size of about 1.0  $\mu m$  to about 1.5  $\mu m$ .

Although the above steps are preferably performed in the above order, a person skilled in the art will understand that two or more steps may be interchanged without departing from the invention.

The present invention also provides a method of increasing the concentration of alcohol in a wine product, the method comprising:

- (a) subjecting the wine product to at least one membrane filtration step so as to yield a permeate stream comprising a treated wine product; and a retentate stream; and
  - (b) subjecting the retentate stream to diafiltration with a condensate.

In a preferred embodiment of the invention, the wine product is subjected in step (a) to nanofiltration and the condensate in step (b) is a condensate from the condensation of a wine product selected from any one of grape juice, low-alcohol wine or waste wine, or a combination of any thereof.

In another embodiment of the invention, steps (a) and (b) may be repeated until the desired concentration of alcohol in the treated wine product is achieved.

The present invention also provides a treated wine product produced in accordance with the method of the invention.

The present invention also provides a treated wine product having substantially no colour, flavour or odour yet having an alcohol concentration of at least about 4% 5 (v/v), preferably of about 4% to about 23% (v/v), more preferably about 4% to about 20% (v/v), even more preferably about 8% to about 17% (v/v).

The present invention also provides a treated wine product having a composition comprising:

about 4%(v/v) to about 23% (v/v) ethanol;

10 less than about 1% (v/v) glycerol;

less than about 1% (v/v) acetic acid; and

the balance water.

The present invention also provides a treated wine product having a composition comprising:

about 4%(v/v) to about 23%(v/v) ethanol;

less than about 1%(v/v) glycerol;

less than about 0.2%(v/v) acetic acid; and

the balance water.

The present invention also provides an alcohol containing beverage comprising 20 the treated wine product produced in accordance with the method of the present invention.

The present invention also provides an alcohol containing beverage with the following components:

- (a) a treated wine product produced in accordance with the method of the invention and optionally,
  - (b) one or more components that alter the colour, and/or clarity and/or flavour, and/or viscosity of the alcohol containing beverage.

The present invention also provides a treated wine product having the following composition:

- 30 about 20% (v/v) to about 23% (v/v) alcohol;
  - less than about 0.02 g/l of one or more organic acids; and
  - an absorbance at about 280 nanometres of about 0.135 150 absorbance units.

The present invention also provides a treated wine product having the following o composition:

about 20 to about 23% (v/v) alcohol;

less than about 0.02 g/l of one or more organic acids; and an absorbance at about 420 nanometres or more of less than about 0.005 absorbance units.

Preferably the organic acid is selected from lactic acid or citric acid.

The present invention also provides an apparatus for producing a treated wine product, the apparatus comprising:

- (a) a membrane filtration unit for removing material having a size range of about 1600 daltons to about 100 daltons;
- (b) an ion exchange unit for removing dissolved or suspended material, the ion exchange unit comprising a second inlet and a second outlet, wherein the second inlet is coupled to the first outlet,
  - (c) a sterile filtration unit for substantially removing any yeast and/or bacteria, the second filtration unit comprising a third inlet and a third outlet, wherein the third inlet is coupled to the second outlet;
- (d) a carbon filtration unit for substantially removing or reducing the level of any residual odour and/or flavour, the third filtration unit comprising a fourth inlet and a fourth outlet, wherein the fourth inlet is coupled to the third outlet; and
  - (e) a means for driving the bulk wine through the apparatus which enters the first inlet and exits the fourth outlet as a treated wine product.

The resulting wine product may then be stored, bottled or blended upon exiting the fourth outlet means.

Preferably the means for driving the bulk wine through the apparatus is a pump. Persons skilled in the art will understand that the type and size of pump required is dependent on the volumes of wine product to be processed.

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#### Definitions

As used herein, the term "treated wine product" is an alcohol product having substantially no taste, colour or odour and an alcohol concentration of about 4% to about 23% (v/v). In the context of the invention, the treated wine product may be used as a base for producing an alcohol containing beverage. The treated wine product may also be used in other products such as, for example, perfumes and deodorants or any product in which alcohol is a component or is used as a base component.

As used herein, the term "wine product" is any wine, wine derivative or wine waste product produced from, for example, the fermentation of grape, plum, kiwi fruit, mango, grain, potato, agave, corn, wheat, rye, sugar cane, molasses or any other fermentable substrate. In particular, excess wine produced in a winery or poor (lower

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quality) wine may be used as a source of wine product. Another source of wine product may be the waste product of a alcohol fermentation process; that is, the product of a fermentation process that produces poorer quality or an nonpotable wine. Such wine products contain materials such as dissolved or suspended solids, sugars, acids, tannin, microbes which all contribute to, or affect the taste of, the colour, taste (flavour), clarity and odour of the wine product.

As used herein, the term "material", includes materials such as dissolved or suspended solids, sugars, tannin, acids, microbes but does not include alcohol (ethanol) or water.

As used herein, the term "alcohol containing beverage" encompasses any beverage that contains alcohol such as wines, beer, fortified wines and spirits including, but not limited to, Brandy (Cognac, Amagnac), Schnapps, liqueurs (cream, coffee, chocolate, fruit, herbal), Vodka, Rum, Whiskey (Scotch, Irish, Bourbon, Rye), Tequila and Gin.

As used herein, the term "sterile" is intended to convey that the treated wine product is substantially free of any microbes such as yeast, bacteria, fungi and moulds, however, the total elimination of such microbes is not required to achieve a "sterile" product. It is desirable to remove all, or nearly all, of the microbes as these may grow and cause unwanted flavours through further fermentation of any residual sugar and cloud the spirit thereby affecting its flavour, colour, clarity and odour.

#### Detailed description of the Invention

In accordance with a preferred embodiment of the invention, the wine product is treated to remove material so as to produce a treated wine product having substantially no colour, taste or odour, yet retain or have an increased alcohol concentration following treatment, even in the absence of fortification. This is achieved, in one embodiment, through membrane filtration.

Typical membrane filtration systems include microfiltration, ultrafiltration and nanofiltration. Microfiltration typically removes material in the size range of approximately 0.1 to 5  $\mu$ m, while ultrafiltration removes material in the size range of approximately 0.005 to 0.5  $\mu$ m, and nanofiltration removes material in the size range of approximately 0.0005  $\mu$ m to 0.01  $\mu$ m.

Microfiltration is a low pressure (10 to 100 psig) process and is typically used for separating larger sized solutes from aqueous solutions by means of a semi-permeable membrane. Microfiltration typically retains large suspended solids, such as bacteria, and passes some suspended solids and all dissolved material such as water,

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monovalent ions, multivalent ions and viruses. Microfiltration is typically performed by flowing the process solution along the membrane surface under pressure. Such configurations are typically referred to as cross-flow separation. In this way, a portion of the process flow passes through the membrane while the other portion continues to 5 flow across the membrane so that any retained material is swept away with the process flow. This assists in avoiding accumulation on the membrane surface, and therefore blockage thereof, by the retained material. The portion of the process stream that passes through the membrane is referred to as the permeate while the portion that is retained and usually contains the rejected materials is called the concentrate or 10 retentate.

Ultrafiltration is a low pressure (5 to 150 psig) process for separating larger sized solutes from aqueous solutions by means of a semi-permeable membrane. Ultrafiltration typically retains particulate matter, bacteria, viruses, suspended solids, large macromolecules and proteins, but passes material such as acid, water, monovalent 15 and multivalent ions. Like microfiltration, ultrafiltration may also be configured as a cross-flow filtration system.

Nanofiltration is a low to moderately high pressure (typically 50-450 psig) process which typically retains divalent salts and organics, yet allows monovalent ions, water and acid to pass through. Nanofiltration may also remove colour bodies in the 20 wine product. Like microfiltration and ultrafiltration, nanofiltration may also be configured as a cross-flow filtration system. Nanofiltration serves to remove a wide range of suspended and dissolved material from the wine product, thereby removing most, if not all, of the colour, taste and/or odour from the wine product. Nanofiltration also, however, retains all, or substantially all of the alcohol, and therefore, represents a particularly preferred form of membrane filtration for use in accordance with the method of the present invention. Persons skilled in the art will appreciate, however, that other modes of filtration may be utilised in order to produce a treated wine product substantially devoid of colour, taste and/or odour, yet have an alcohol concentration of at least about 4 %(v/v), preferably, about 4%(v/v) to about 23%(v/v).

If required, the wine product may be subjected to a pre-treatment step (prior to, for example, membrane filtration) to remove solid materials (solid-liquid separation). This pre-treatment step mainly removes suspended solid materials or sediment, primarily to avoid blocking of the membranes during subsequent filtration steps. Such suspended solids include, for example, grape skins and other types of sediment. 35 Various types of solid-liquid filtration methods are suitable including traditional clarification techniques such as settling (sedimentation), centrifugation and cross-flow

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or membrane filtration such as microfiltration or ultrafiltration. Persons skilled in the art will understand that the choice of filtration system depends on the nature of the suspended solids that are to be separated from the bulk wine.

Following membrane filtration, the pH of the treated wine product will typically be around 3.5 - 4.5 and buffering is therefore required alter the pH of the treated product to about 6.5 to 7.0.

In another embodiment of the invention, the membrane filtration step is followed by an ion exchange step. Ion exchange involves replacing positive and negative ions in the process stream with sodium and chloride ions and works on the principle of using ion exchange resins coated with replacement ions such as sodium (Na<sup>+</sup>) and chloride (Cl<sup>-</sup>) which serve to replace charged species such as cations (e.g., magnesium and sodium) and acids in the process stream. A particular form of ion exchange is deionisation, wherein the resins are coated with hydrogen (H<sup>+</sup>) and hydroxide (OH<sup>-</sup>) ions as replacement ions.

Preferably the wine product is deionised during ion exchange, wherein positive and negative ions in the wine product are replaced with hydroxide (OH) and (H<sup>†</sup>) ions which can combine to form water. In this way, materials such as dissolved mineral salts, tannins and any charged species may be removed from the wine product. The deionisation step may achieve the separation of almost any type of charged molecule such as, for example, saccharides, proteins, nucleotides and amino acids.

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The two common types of deionisation configuration are two-bed deionisers or mixed-bed deionisers. In the two-bed system, separate tanks are used each containing a cation and anion resin. In the tank containing that cation resin, cations such as magnesium, calcium or sodium are removed and replaced with hydrogen ions. In the tank containing the anion resin, acids are absorbed and replaced with hydroxide ions. The hydrogen and hydroxide ions in the wine product may then combine to form water.

In the mixed bed system, the cation and anion resins are thoroughly mixed in a single tank and act as a series of alternating cation and anion exchange units to produce a high quality product substantially devoid of charged species. In particular, the deionisation step removes, for example, flavour components such as small peptides or nucleosides, organic acid fractions so as to reduce acidity, or "nitrogen components" so as to reduce colour from aging.

Following deionisation, the pH of the treated wine product will typically be around neutral (pH 7), however, if the pH is below about 6.2, buffering may be required to alter the pH of the treated product to about 6.5 to 7.0.

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In another embodiment, the wine product following membrane filtration, ion exchange, or both, is subjected to a further "sterile" filtration step in which all, or substantially all, or the microbes such as yeast, bacteria, fungi and moulds are removed. While the term "sterile" is used throughout the specification, it will be understood that the total elimination of such microbes is not required to achieve a "sterile" product. It is desirable, however, to remove all, or nearly all, of the microbes as these may grow and cause unwanted flavours through further fermentation of any residual sugar and cloud the spirit thereby affecting its flavour, colour, clarity.

Sterile filtration is particularly preferred when the treated wine product is to be 10 used as a base for producing a sweet wine such as, for example, a port or sherry. For this purpose, sugar is desirably added to the treated wine product, preferably after membrane filtration. The residual sugar, however, serves as a carbon source on which microbes such as yeast and bacteria are able to grow and as such, produce a cloud or haze in the wine product. Such a haze is able to be removed through a sterile filtration step.

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Sterile filtration may be achieved by membrane filtration such as ultrafiltration or microfiltration. Sterile filtration may also be achieved by other means such as centrifugation, irradiation.. In a particularly preferred embodiment, sterile filtration is achieved by membrane filtration using microfiltration, ultrafiltration or nanofiltration in combination or alone.

It will be understood that not only microbes such as yeast and bacteria may be removed during the sterile filtration step, but other particles besides microbes may also be removed during the sterile filtration step.

In some instances, there may be some residual colour, flavour or odour in the treated wine product following membrane filtration and/or ion exchange and/or sterile filtration. Removal of any residual colour, flavour and/or odour is desirable as the quality of the treated wine product increases with decreasing colour, flavour and/or odour, thereby resulting in a higher quality alcohol containing beverage.

A preferred method of achieving colour, flavour and/or odour removal is by 30 carbon filtration, preferably using activated carbon. Other methods of achieving colour removal include the use of diatomaceous earth, however, these are less desirable as they do not also remove any residual flavour, and primarily remove colour.

If carbon filtration is used, a further polishing filtration step may be required especially if there has been any "breakthrough" of the carbon filter. Any residual 35 carbon may be removed by plate and frame filtration using a filter having a size range of about 1.0 µm to about 1.5 µm.

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The resulting treated wine product is a clear liquid substantially devoid of residual colour, flavour and odour so as to provide an alcohol base that is suitable for the production of alcohol containing beverages selected from any one of schnapps, liqueurs, cream liqueurs, brandy, whiskey, rum, gin, vodka or tequila, sherry, bourbon, 5 cocktails etc. Alternatively, the treated wine product may be used as a component in. or as a base component of, any product comprising an alcohol, such as, for example, deodorants, performes and the like.

In some instances, it is desirable to further increase the alcohol concentration of the treated wine product. This may be achieved by fortification and this optional step 10 typically involves the addition of a high alcohol-concentration product such as 99% (v/v) spirit, to the treated wine product. Fortification typically increases the alcohol concentration of the treated wine product from about 4 - 23 % (v/v), more preferably about 8 - 20 % (v/v).

Alternatively, the alcohol concentration of treated wine product may be 15 increased by freeze concentration. Freeze concentration involves removing heat from the treated wine product by subjecting it to a temperature drop, at or below 0 °C, so as to freeze the water component which has a higher freezing temperature than alcohol. Upon freezing, the water may be removed thereby increasing the concentration of alcohol in the wine.

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More specifically, freeze concentration involves passing the treated wine product through a heat exchanger of which the external surface is cooled by a refrigerant. This causes tiny ice (water) crystals to form at this cold surface leaving a concentrated wine product in the form of a liquid within the heat exchanger. Once the crystals are formed, they enter a mixed vessel called a recrystallizer wherein most of 25 the small crystals melt allowing larger crystals to grow. This crystal nucleation process is termed "ripening". As it is necessary to have a continuous flow of small crystals in the recrystallizer, liquid from the recrystallizer is extracted and circulated through the heat exchanger and then returned back to the recrystallizer. Once there are sufficient crystals in the recrystallizer, they are separated from the concentrated liquid using a 30 wash column wherein the crystals are separated and washed to remove any remaining concentrate, melted and discharged as water. Fresh feed, in this case, treated wine product, replaces the water that is removed from the system which is mixed with the recirculation stream entering the heat exchanger. Once the concentrated product reaches the desired concentration, it can then be discharged from the liquid 35 recirculation line after exiting from the filter in the recrystallizer.

Another method of increasing the concentration of the alcohol in the treated wine product, if desired, is to use condensate from the condensation of, for example, a wine product such as grape juice, waste wine or a low-alcohol wine to diafilter the retentate from membrane filtration. For instance, in one embodiment of the invention, the retentate from the nanofiltration unit may be mixed with a condensate, such as grape juice condensate, and returned to the nanofiltration unit for further separation of the alcohol from the retentate.

In addition to the use of a condensate from the condensation of grape juice, other condensates include evaporation of wine products such as low-alcohol wine or waste wine or a combination of any such wine products.

In accordance with another embodiment of the invention, the diafiltered product may be passed through any one of the described process steps described herein, such as for example, nanofiltration, and then subjected to diafiltration again. This process may be repeated as many times as desired until the desired alcohol concentration in the wine treated product is achieved.

Any discussion of documents, acts, materials, devices, articles or the like which has been included in the present specification is solely for the purpose of providing a context for the present invention. It is not to be taken as an admission that any or all of these matters form part of the prior art base or were common general knowledge in the field relevant to the present invention as it existed before the priority date of each claim of this application.

Throughout this specification the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as shown in the specific embodiments without departing from the spirit or scope of the invention as broadly described. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.

### Brief Description of the Figures

Figure 1 is a block flow diagram of the process in accordance with a preferred embodiment of the invention.

Figure 2 is a chromatograph and results from HPLC analysis of a treated wine product in accordance with the present invention.

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Figure 3 is a chromatograph of a GCMS analysis of a treated wine product in accordance with the present invention.

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# Example 1: Process steps involved in treating a wine product so as to increase its alcohol concentration.

Referring to figure 1, a wine product 1 is pumped, at ambient temperature throughout, by pump 2 through a nanofiltration unit 3 under a pressure of 60 to 100 bar.

The retentate 5 is discarded. In the permeate 7, less than 1.0% of the suspended and dissolved material remains with substantially all of the alcohol in the initial wine product 1 having been retained. The pH of the permeate 7 exiting the nanofiltration unit 3 is about 3.5 to 4.5.

The permeate 7 from the nanofiltration unit 3 is then pumped through an ion exchange column 9 which serves to remove dissolved and suspended solids, charged species and colour bodies leaving a product stream 11 having an alcohol concentration of about 4% (v/v) to about 20% (v/v). The product stream 11 also has a pH of about 6.5 to about 7.0 and a titratable acidity of less than 1.0%.

The product stream 11 is then pumped through a sterile filtration unit 13 with a 20 0.5 µm cuno filter. Microbes such as yeast and bacteria are substantially removed during the sterile filtration step leaving a sterile product 15 which exits the sterile filtration unit 15.

The sterile product 15 then enters a fortification tank 16 in which wine spirit 17 having an alcohol concentration of 99% (v/v) is mixed with the sterile product 15 so as to raise the concentration of the wine product to about 22% (v/v) resulting in a fortified wine product 19.

The fortified wine product 19 is then pumped through a carbon filtration unit 21 with a 1 hour contact (residence) time. Following carbon filtration, the filtered product 23 is subjected to a final polishing step in the polishing filtration unit 25 having a filter size of about 1.0 mm to about 1.5 mm. The treated wine product 27 is then stored in storage tanks 29 ready for sale, blending etc.

Example 2: Composition analysis of a treated wine product in accordance with the method of the present invention.

A composition analysis of a sample of a treated wine product produced in accordance with the method of the present invention follows:

Сотролепт		Amount/comment			
Alcohol		21.5-22.5% (v/v)			
Acidity		Less than 0.5% titratable acidity			
pН		6.5-7.2			
Glucose and Fructose		Less than 0.05 g/l			
Heat stability		Passes			
Lactic acid		Less than 0.01 g/l			
Citric acid		Less than 0.01 g/l			
Absorbance:	at 280 nm	0.135-150 absorbance units			
	at 420 nm	Less than 0.005 absorbance units			
	at 520 nm	Less than 0.005 absorbance units			
	at 720 nm	Less than 0.005 absorbance units			
Flavour		Negligible to slight alcohol taste			
Odour		Negligible - no odour			
Transparency		No observable particulate matter when			
•		observed in a darkened room with a thin			
		beam of light			

# 10 Example 3: Composition of a treated wine product in accordance with the method of the invention as analysed by HPLC.

A treated wine product treated in accordance with a method of the present invention was subjected to an analysis by HPLC. The HPLC profile and results are shown as Figure 2.

A tabular summary of the component analysis as ascertained by HPLC of the treated wine product follows:

Analysed Component	Amount (g/L)*
Citric acid	-
Glucose	-
Malic Acid	
Fructose	-
Succinic Acid	_
Lactic Acid	_
Tartaric Acid	
Glycerol	. 0.953894
Acetic Acid	0.188073
Ethanol	21.69037

<sup>\*</sup> a nil result indicates the absence of a component or a component amount below the detectable level.

The balance of the composition is water.

# Example 4: Composition of a treated wine product in accordance with the method of the invention as analysed by GCMS.

A treated wine product treated in accordance with a method of the present invention was subjected to an analysis by GCMS. The GCMS profile is shown as Figure 3.

10 A tabular summary of a qualitative analysis of the GCMS follows:

Compound Retention Time (mins)	Compound	Match Quality (%)		
1.44	acetaldehyde	86		
2.28	acetic acid	72		
2.96	ethanol	91 91		
8.43	3-methyl-1-butanol (impure)			
10.09	2-hydroxypropanoic acid	56		
13.09	butanedioic acid	64		
14,93	benzencethanol	90		
<u> 17.75</u>	1,2,3-propanetriol (glycerol)	83		

### CLAIMS:

1. A method of increasing the alcohol concentration of a wine product, the method comprising the step of treating the wine product so as to reduce the amount of material from the wine product to produce a treated wine product.

- 2. A method according to claim 1 wherein the treated wine product has an alcohol concentration of at least about 4% (v/v).
- 3. A method according to claim 1 or claim 2 wherein the treated wine product has an alcohol concentration of at least about 4% (v/v) to about 23% (v/v).
  - 3. A method according to any one of the preceding claims wherein the treated wine product has an alcohol concentration of between about 8% (v/v) to about 23% (v/v).
- 15 4. A method according to any one of the preceding claims wherein the material that is reduced in amount is in the size range of about 100 daltons to about 1600 daltons.
- 5. A method of increasing the alcohol concentration of a wine product, the method comprising the step of treating the wine product so as to reduce the amount of material from the wine product to produce a treated wine product, wherein the treating step comprises subjecting the wine product to membrane filtration.
- 6. A method according to claim 5 wherein the membrane filtration is 25 nanofiltration.
  - 7. A method according to claim 5 wherein the material that is reduced in amount is in the size range of about 400 daltons to about 1600 daltons.
- 30 8. A method according to any one of the preceding claims wherein the treated wine product has a titratable acidity of less than about 1.0 %
  - 9. A method according to any one of the preceding claims wherein the treated wine product has a titratable acidity of less than about 0.5 %.

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- 10. A method according to any one of the preceding claims wherein the treated wine product is a clear liquid having an absorbance of 0.05 atomic units at about 430 nm.
- 11. A method according to any one of claims 5 to 10 wherein the step of removing material so as to increase the alcohol concentration of a wine product is achieved through one or more membrane filtration steps is followed by ion exchange.
- 12. A method according to any one of claims 5 to 11 wherein the wine product is deionised during the ion exchange treatment step, wherein material containing mono-,
   10 di-, or multi-valent ions is primarily removed, or substantially reduced.
  - 13. A method according to any one of claims 5 to 12 wherein the treated wine product after membrane filtration and/or ion exchange has a titratable acidity of less than about 1.0 %

- 14. A method according to any one of claims 5 to 13 wherein the treated wine product after membrane filtration and/or ion exchange has a titratable acidity of less than about 0.05 %.
- 20 15. A method according to any one of claims 5 to 14 wherein the treated wine product following membrane filtration and/or ion exchange, is subjected to a sterile filtration step.
- 16. A method according to claim 15 wherein the sterile filtration step follows the 25 ion exchange treatment step.
  - 17. A method according to any one of the preceding claims wherein the wine product is subjected to a further fortification step
- 30 18. A method according to any one of claims 15 to 17 wherein the fortification step occurs after the sterile filtration step.
  - 19. A method according to any one of the preceding claims wherein the wine product is subject to a further carbon filtration step.

- 20. A method according to any one of the preceding claims wherein the step of treating the wine product comprises a nanofiltration step followed by ion exchange.
- 21. A method according to claim 20 wherein the wine product is deionised during 5 the ion exchange step.
  - 22. A method according to any one of the preceding claims wherein the treated wine product is used as a base to produce a sweet wine.
- 10 23. A method according to claim 22 wherein the wine product is produced according to the method of any one of the preceding claims followed by the addition of sugar.
- 24. A method according to claim 22 wherein the wine product is treated by 15 membrane filtration.
  - 25. A method according to claim 24 wherein the membrane filtration is nanofiltration.
- 20 26. A method of increasing the alcohol concentration of a wine product by removing material from the wine product yet retaining all, or substantially all of the alcohol from the wine product, the method comprising subjecting the wine product to:
  - (a) nanofiltration to remove or reduce the level of material having a size range of about 400 daltons to 1600 daltons:
- 25 (b) ion exchange to substantially remove or reduce the level of dissolved or suspended material;
  - (c) sterile filtration to substantially remove any yeast, bacteria or moulds;
  - (d) optional fortification with a high alcohol-concentration spirit to increase the alcohol concentration of the treated wine product to at least about 20 %(v/v);
- 30 (d) carbon filtration to substantially remove any residual colour bodies, flavour and/or odour; and
  - (d) an optional polish filtration step through a filter having a pore size of about 1.0  $\mu m$  to about 1.5  $\mu m$ .

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- 27. A method of increasing the concentration of alcohol in a wine product, the method comprising:
- (a) subjecting the wine product to at least one membrane filtration step so as to yield a permeate stream comprising a treated wine product; and a retentate stream; and
- 5 (b) subjecting the retentate stream to diafiltration with a condensate.
- 28. A method according to claim 27 wherein the wine product is subjected in step
  (a) to nanofiltration and the condensate step (b) is a condensate from the condensation
  of any one of grape juice, waste wine, low-alcohol wine or a combination of any
  10 thereof.
  - 29. A method according to claim 27 or claim 28 wherein steps (a) and (b) are repeated until the desired concentration of alcohol in the treated wine product is achieved.

30. A treated wine product produced in accordance with the method of any one of the preceding claims.

- 31. A treated wine product having substantially no colour, flavour or odour yet 20 having an alcohol concentration of at least about 4% (v/v).
  - 32. A treated wine product according to claim 31 wherein the alcohol concentration is about 4% to about 23% (v/v)
- 25 33. A treated wine product according to claim 31 or 32 wherein the alcohol concentration is about 4% to about 20% (v/v).
  - 34. A treated wine product according to any one of claims 31 to 33 wherein the alcohol concentration is about 8% to about 17% (v/v).
  - 35. A treated wine product having a composition comprising: about 4%(v/v) to about 23% (v/v) ethanol; less than about 1% (v/v) glycerol; less than about 1% (v/v) acetic acid; and
- 35 the balance water.

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36. The present invention also provides a treated wine product having a composition comprising:

about 4%(v/v) to about 23%(v/v) ethanol;

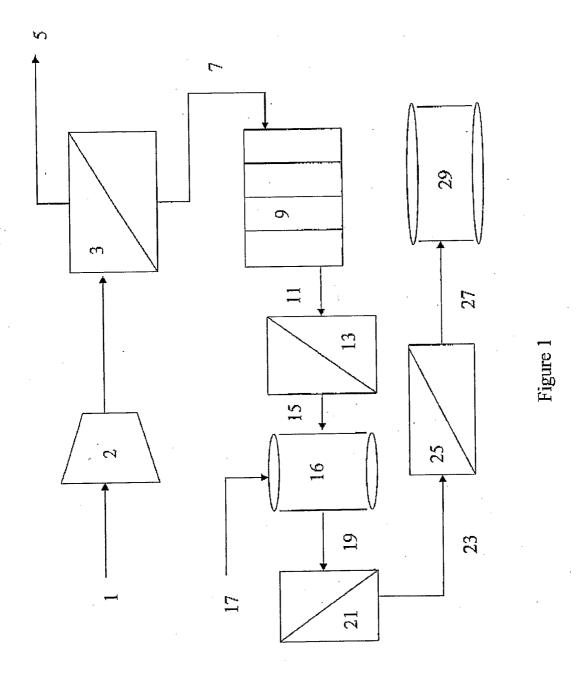
less than about 1%(v/v) glycerol;

- 5 less than about 0.2%(v/v) acetic acid; and the balance water.
  - 37. An alcohol containing beverage comprising a treated wine product produced in accordance with the method of any one of claims 1 to 29.

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- 38. An alcohol containing beverage with the following components:
- (a) a treated wine product produced in accordance with the method of any one of claims 1 to 29 and optionally,
- (b) one or more components that after the colour, and/or clarity and/or flavour, and/or viscosity of the alcohol containing beverage.
  - 39. A treated wine product having the following composition:
    - about 20% (v/v) to about 23% (v/v) alcohol;
    - less than about 0.02 g/l of one or more organic acids; and
- 20 an absorbance at about 280 nanometres of about 0.135 150 absorbance units.
  - 40. A treated wine product having the following composition:
    - about 20 to about 23% (v/v) alcohol;
    - less than about 0.02 g/l of one or more organic acids; and
  - an absorbance at about 420 nanometres or more of less than about 0.005 absorbance units.
- 41. A treated wine product according to claim 40 wherein the organic acid is lactic acid or citric acid.
  - 42. An apparatus for producing a treated wine product, the apparatus comprising:
  - (a) a membrane filtration unit for removing material having a size range of about 100 daltons to about 1600 daltons;

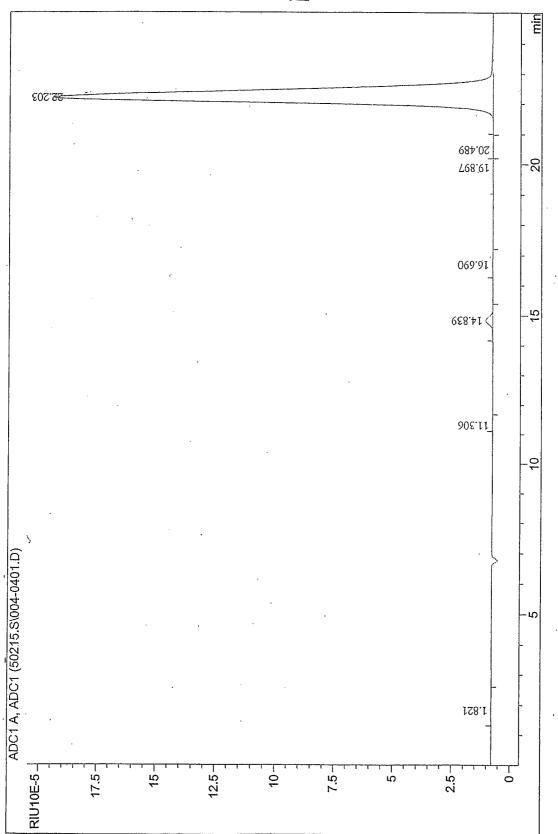
- (b) an ion exchange unit for removing dissolved or suspended material, the ion exchange unit comprising a second inlet and a second outlet, wherein the second inlet is coupled to the first outlet,
- (c) a sterile filtration unit for substantially removing any yeast and/or bacteria,
   the second filtration unit comprising a third inlet and a third outlet, wherein the third inlet is coupled to the second outlet;
  - (d) a carbon filtration unit for substantially removing or reducing the level of any residual odour and/or flavour, the third filtration unit comprising a fourth inlet and a fourth outlet, wherein the fourth inlet is coupled to the third outlet; and
- 10 (e) a means for driving the bulk wine through the apparatus which enters the first inlet and exits the fourth outlet as a treated wine product.



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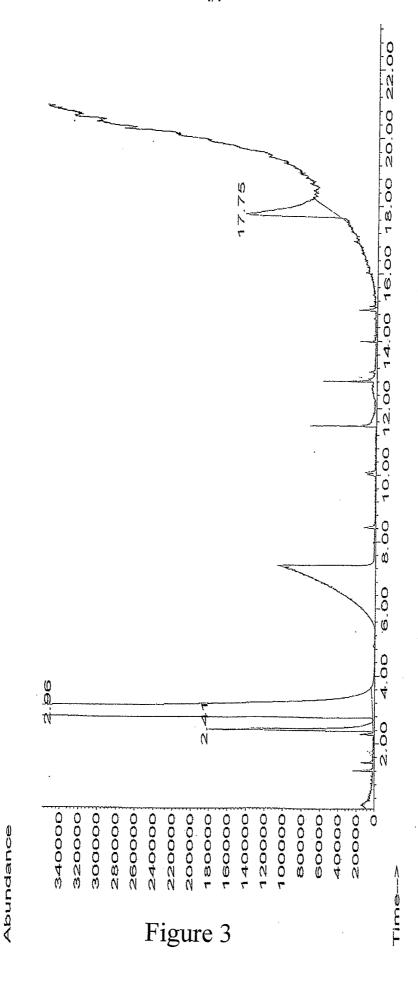
2

Figure 2



RetTime Type [min]	Area RIU10E-5*	Amt/Area	Amount [g/L]	Grp Name	
9.388 9.979 10.600 10.766 13.665		-	-	Citric Acid ( Glucose  Malic Acid Fructose Succinic Acid	
RetTime Type [min]	Area RIU10E-5*	`Amt/Area	Amount [g/L]	Grp Name	
14.100 14.839 BP 16.690 MM 22.203 MM	6.09609e-1	8.45094e-2 1.55710e-1 2.05343e-2		Lactic Acid Glycerol Acid Acetic Acid	i
Totals:			2285989		,
RetTime Type [min] -	Area	Amt/Area	Amoùnt [g/L]	Grp Name	
9.030 9.581 13.258 14.242 15.841		-	-	Citric Acid Tartaric Acid Succinnic Acid Lactic Acid Acetic Acid	
Totals :			0.00000		

Figure 2 cont.



International application No.

PCT/AU2006/000500

			PCT/AU2006/000500	
A.	CLASSIFICATION OF SUBJECT MATTER			
Int. Cl.		·		
C12G 1/00 (2 B01D 15/04 ( B01D 25/02 ( B01D 61/00 (	(2006.01)	C12H 1/00 (2006.01) C12H 1/04 (2006.01) C12H 1/07 (2006.01)		
According to	International Patent Classification (IPC) or to b	oth national classification and IPC		
В.	FIELDS SEARCHED			
Minimum docu	umentation searched (classification system followed by	by classification symbols)		
Documentation	n searched other than minimum documentation to the	extent that such documents are included	in the fields searched	
WPIDS; CAP fortif?, concertransparent?,	base consulted during the international search (name PLUS; FSTA; MEDLINE; JAPIO: wine?, grape ntrat?, acid?, absorban?, optic? densit?, transmis colourless?, odourless?, tasteless?, glycerol?, ac	?, alcohol?, filt?, ion(w)exchange?, no ssion?, wavelength?, nanometer?, no ssion?, wavelength?, nanometer?, no ssion?, wavelength?, nanometer?, no ssion?	anofilt?, nano(w)filt?, membran?	
Category*	Citation of document, with indication, where	appropriate, of the relevant passages	Relevant to claim No.	
Х	Derwent Abstract Accession No. 2005-568363/58, Class D16 KR 2005022796 A (DOOSAN CORP) 08 March 2005 Abstract			
X	WO 2005/014771 A (GRAIN PROCESSING Abstract; Paragraphs [10], [15]-[18]; claims;	G CORP) 17 February 2005	. 1-3, 19, 30, 31 36, 39-41	
X	WO 2001/078881 A (TUDHOPE B, R) 25 O Abstract	ectober 2001	1, 5, 6, 20, 30, 31-36, 42	
X, P	US 2005 249851 A (WOLLAN, D) 10 Novement; Table 3	mber 2005	1, 5-6, 19- 20,30, 31- 36,39, 42	
X F	Further documents are listed in the continua	tion of Box C X See pat	ent family annex	
"A" documer not cons "E" earlier a internati	categories of cited documents; nt defining the general state of the art which is sidered to be of particular relevance  pplication or patent but published on or after the ional filing date  "X"	later document published after the internatic conflict with the application but cited to ununderlying the invention document of particular relevance; the claim or cannot be considered to involve an invealone	derstand the principle or theory  led invention cannot be considered novel  intive step when the document is taken	
or which another "O" document or other		document of particular relevance; the claim involve an inventive step when the docume such documents, such combination being o document member of the same patent familiary.	nt is combined with one or more other by by by to a person skilled in the art	
but later	nt published prior to the international filing date than the priority date claimed			
Date of the actu 09 June 2000	ual completion of the international search	Date of mailing of the internationa	•	
	ing address of the ISA/AU	Authorized officer	) ,	
AUSTRALIAN PO BOX 200, '	N PATENT OFFICE WODEN ACT 2606, AUSTRALIA	GAYE HOROBIN	·	
	: pct@ipaustralia.gov.au (02) 6285 3929	Telephone No: (02) 6283 2069	)	

International application No. PCT/AU2006/000500

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT Relevant to Category\* Citation of document, with indication, where appropriate, of the relevant passages claim No. Derwent Abstract Accession No. 2004-725538/71, Class D16 1, 5, 6, 11, 19, KR 2004053813 A (KIM Y G) 24 June 2004 20, 30, 31-36, X, Y Abstract 42, Derwent Abstract Accession No. 2005-036446/04, Class D16, E14, J01 NZ 532913 A (WINE NETWORK TECHNOLOGY PTY LTD) 29 October 2004 X, Y Abstract 1, 5, 6, 19, 20, 30 1, 4, 5, 6, 11, EP 1146115 A (ENOLOGICA VASON SRL) 17 October 2001 20, 30, 31-36, X, Y Whole document 39-41, 42 WO 1993/023151 A (SMITH, C. R.) 25 November 1993 X Whole document; abstract and page 3, paragraph 3; 1-4, 8, 9, 30, 42 Derwent Abstract Accession No. 2004-285187/27, Class D16 EP 1403362 A2 (INDAGRO GASQUET SA) 31 March 2004 1, 5, 6, 8, 9, 30, 31-36, 39-41 X Abstract Derwent Abstract Accession No. 2005-599197/62, Class D16 EP 1571200 A (WARSTEINER BRAUEREI CRAMER GMBH & CO KG) 07 September 2005 1-3, 5, 6, Y, P Abstract Derwent Abstract Accession No. 2006-092360/10, Class D16 EP 1611940 A (WARSTEINER BRAUEREI CRAMER GMBH & CO KG) 04 January 2006 1-3, 5, 6, Y, P Abstract WO 1992/008783 A (DOW DANMARK AS) 29 May 1992 1, 5, 6, 31-36, Y Abstract 39-41, 42 "Food Technology in Australia", (1981), 33 (1) 12-13, 2 ref (WILLIAMS, P.J.; STRAUSS, C. R.; KLINGNER, K. E.; OBST, S. R.; ANDERSON G. L. G.): "Development of a process for the deodorisation of spirit recovered from 31-36, 39-41 Y grape marc."

International application No. PCT/AU2006/000500

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet) This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons: 1. Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely: ٠2. Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically: 3. Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a) Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet) This International Searching Authority found multiple inventions in this international application, as follows: (See supplemental box) As all required additional search fees were timely paid by the applicant, this international search report covers all 1. searchable claims. As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite 2. payment of additional fees. As only some of the required additional search fees were timely paid by the applicant, this international search report 3. covers only those claims for which fees were paid, specifically claims Nos.; No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: Remark on Protest The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee. The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation. No protest accompanied the payment of additional search fees.

International application No.

PCT/AU2006/000500

Supplemental Box

(To be used when the space in any of Boxes I to VIII is not sufficient)

### Continuation of Box No: III (Unity of Invention)

It is considered that the application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept. In coming to this conclusion the International Search Authority has found that there are **three** different inventions as follows:

- (a) Claims 1-30, and 37-38, which are directed to a method of increasing the alcohol concentration of a wine product by reducing the amount of material (or removing material) from the wine product to produce a treated wine product; to a treated wine product produced by the claimed method (claim 30), as well as to an alcohol containing beverage comprising the treated wine product produced by the claimed method (claims 37-38). It is considered that the method of increasing the alcohol concentration of a wine product by reducing the amount of material (or removing material) from the wine product to produce a treated wine product comprises a first special technical feature;
- (b) Claims 31-36, and 39-41, which are directed to a treated wine product having an alcohol concentration of at least about 4% (v/v). It is considered that the treated wine product having an alcohol concentration of at least about 4% (v/v) comprises a second technical feature.
- (c) <u>Claim 42</u>, which is directed to an apparatus for producing a treated wine product, the apparatus comprising a membrane filtration unit, an ion exchange unit, a sterile filtration unit, a carbon filtration unit, and a means for driving the bulk wine through the apparatus. It is considered that the apparatus comprising these recited features comprises a third special technical feature.

However, the sets of claims specified above each have a different special technical feature: the claims of each of set (b) and set (c) above are not limited to the method recited in the claims of either set (a) or of each other: these sets of claims do not share a common general inventive concept with any of the other sets of claims recited in the application. Thus, the application does not comply with the requirements of unity of invention (Rules 13.1, 13.2 and 13.3) as it relates to three different general inventive concepts.

International application No.

Information on patent family members

PCT/AU2006/000500

This Annex lists the known "A" publication level patent family members relating to the patent documents eited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report			Patent Family Member				
KR	2005022796	······································					
WO	2005014771	CA	2506621	US	2005064066		
WO	0178881	AU	40977/01	EP	1276550	US	2003075506
		US	2006073248	ZA	200209138		
US	2005249851	AU	2004201949				
WO	9323151	AU	42319/93	BG	99165	CA	2135305
		EP	0639105	HU	70805	US	5480665
		ZA	9303213		<u> </u>		
KR	2004053813		-		·		
NZ	532913						
EP	1146115	HU	0101535	US	7022366	· US	2001031293
EP	1403362	AU	2003244567	CA	2441013	FR	2845096
		US	2004067280				
EP	1571200	DE	1020040288				
EP	1611940	DE	1020040287				
WO	9208783	AU	88584/91	BR	9106981	CA	2095917
		CZ	9300887	EP	0557325	HU	67497

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX