

# The Influence of Different Production Processes on the Aromatic Composition of Peruvian Pisco

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## Abstract

This work evaluates, from chemical and sensory points of view, the impact of certain production process variations on the aromatic profiles of the Quebranta and Italia varieties of pisco. It studies the influence of distilling must that has been fermented completely or incompletely (green must); of carrying out distillation in a *falca* or in an alembic; and of varying the scale of production (industrial scale or artisan small scale).

The chemical differences observed between piscos of complete fermentation and incomplete fermentation (green musts) was more marked in the Quebranta variety. These differences were also noted in the sensory evaluation, with the representative green must sample being defined as the most terpenic, and as having a greater aromatic intensity.

In general, it was seen that distillation in a *falca* gives slightly higher levels of the majority of the compounds in both Quebranta and Italia piscos.

For their part, the different scales of production appear to have a greater impact on the Quebranta variety, introducing significant differences in 16 compounds, while the Italia variety showed significant differences in only three compounds. In general, the majority of the analysed compounds showed higher levels in the industrial-scale piscos.

**Keywords:** Pisco; Distillation; Quebranta; Italia; Aroma; Falca; Alembic; Green must

## Introduction

The alcoholic beverage “pisco” is produced in Peru by distilling wine made from several varieties of grapes. Depending on the grape variety and the processes of fermentation and distillation used, both the chemical and the sensory characteristics can undergo changes that make it possible to obtain piscos of differing aromatic character. At the present time, the production conditions and equipment used are described in the *Norma Técnica Peruana (NTP 211.001)* [1]. According to the regulation governing pisco’s Denomination of Origin, water cannot be added to the wine or the liquor, nor is double distillation allowed. These two aspects distinguish pisco from other spirits produced in the Southern Cone. Following this regulation, the distillation of alcoholic must can be carried out at the end of fermentation, or with fermentation incomplete. In the latter case, the final product is called green must pisco. The producers who make these green must piscos depend on personal empirical knowledge acquired over time. Ordinarily, fermentation is stopped when a level of about 25 grams of sugar per litre is reached; this usually occurs between the fourth and the tenth day of fermentation.

Regarding the distillation process, the regulation stipulates that pisco production must be done exclusively by direct, discontinuous distillation, separating the head and tail so that only the middle part of the product is selected. The equipment permitted for this operation is the *falca*, the alembic, or the alembic with a wine heater. Both types of alembic should be made of copper or tin, whereas the *falca* is made of bricks and clay with the walls covered in a mix of lime and cement. It consists of a basin in which the most is heated over a wood fire. It is important to keep in mind that the form or design of the distillation equipment can also affect the end characteristics of the distillate. The *falca*, which is a much more rustic piece of equipment than the

alembic, permits a lower level of condensation of alcoholic fumes, and more easily allows a greater amount of impurities to pass.

Lastly, it is worth noting that, in present-day Peru, the greater percentage of pisco is produced by artisan methods that follow traditional customs acquired by the producers, and a lower proportion is produced on an industrial scale, although this situation has been changing in recent years. The difference in production scale could perhaps be reflected in the aromatic composition of the end product.

Until now, there have been no studies of the influence of certain production processes on the aromatic composition of Peruvian piscos: namely, the point in time when distillation takes place, the distillation equipment, and the scale of production. Nevertheless, studies previously published by Cacho et al. [2,3] give detailed information about the aromatic profile of artisan piscos from the most representative varieties (the Quebranta as a non-aromatic pisco and the Italia as an aromatic pisco). The present work has used part of that information as a reference for the purpose of comparing the effect of the different production processes on the aromatic composition of these distillates. Other authors, such as Agosin et al. [4], Lillo et al. [5], Herraiz et al. [6] and Bordeu et al. [7] have focused their attention on

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the quantitative and sensory analysis of Chilean pisco. There are also many studies of the aromatic composition of wine distillates similar to pisco, like orujo, grappa, and calvados [8-14], but none allows for evaluation of the production process.

The principal objective of the present work is to test whether there are significant differences in the aromatic composition of the Quebranta and Italia varieties of pisco when comparing:

- 1) piscos obtained from completely fermented musts and incompletely fermented musts (green musts);
- 2) piscos produced by distillation in a *falca* and an alembic;
- 3) piscos produced on an artisan scale and an industrial scale.

The final part of the study is a sensory evaluation of the effect of the technological variations on the pisco's final aroma.

## Materials and Methods

### Pisco samples

Fifty-one samples of pisco were analysed: 28 of the Italia variety and 23 of the Quebranta variety. Nine of the Quebranta piscos were produced from completely fermented musts using artisan methods and alembic distillation. The rest of the analysed Quebranta piscos were obtained by varying one of the three production stages. Specifically, six were obtained from incompletely fermented musts, eight by a large-scale (industrial) process, and three were distilled in a *falca* rather than an alembic. Of the Italia samples, nine were obtained under what we considered to be reference conditions (complete fermentation, artisan production, alembic distillation). Of the rest, seven came from incomplete fermentation of the must, five were obtained on an industrial scale, and seven were distilled in a *falca*. Table 1 provides information about all the groups of samples analysed.

### Reagents and standards

Dichloromethane and methanol of LiChrosolv quality were supplied by Merck (Darmstadt, Germany), and absolute ethanol by Panreac (Barcelona, Spain), all of ARG quality. Pure water was obtained from a Milli-Q purification system (Millipore, Bedford, MA). Semi-automated solid-phase extraction (SPE) was carried out with a VAC ELUT 20 station supplied by Varian (Walnut Creek, CA). The LiChrolut EN resins and polypropylene cartridges were obtained from Merck (Darmstadt, Germany).

The chemical standards used for identifications were supplied by Aldrich (Steinheim, Germany), Fluka (Buchs, Switzerland), Poly-Science (Niles, USA), Lancaster (Strasbourg, France), and Alfa Aesar (Karlsruhe, Germany). An alkane solution (C8–C28), 20 mg L<sup>-1</sup> in dichloromethane, was employed to calculate the linear retention index (LRI) of each analyte.

### Chemical quantitative analyses by direct injection of the distillate in a gas chromatograph (GC-FID)

This analysis was carried out following the procedure proposed by

López-Vázquez et al. [12]. This method consists of direct injection of the sample in the chromatographic system after the addition of certain internal standards (4-methyl-2-pentanol and 4-decanol). In this way, 18 majority volatile compounds were able to be quantified in the pisco samples. The calibration method is described in detail in a previous paper [15].

### Chemical quantitative analyses by solid phase extraction (SPE) followed by injection in a gas chromatograph (GC-Ion Trap-MS)

This analysis was carried out using the method proposed and validated by López et al. [16] with some modifications. This method consists of obtaining a representative extract of the sample via a solid phase extraction process (SPE), to which a known quantity of internal standards (4-hydroxy-4-methyl-2-pentanone and 2-octanol), is added, and afterward is analysed by GC-MS. This method allowed for the quantification of 45 volatile compounds, present in lower concentrations, which were not able to be quantified by the GC-FID method. Calibration information about all the quantified compounds is described in detail in two previous papers [15,16].

### Sensory analysis

**Sensory panel:** The sensory panel was formed by twelve judges aged 23-40. All the judges had some previous experience in sensory analysis.

**Triangle tests:** For the purpose of comparing, from a sensory point of view, the different sample groups analysed in this study, and evaluating whether the chemical changes resulting from the distinct production processes affected the final aroma of the piscos, different triangle tests were carried out comparing representative samples from each group according to the Spanish Norm AENOR 87-006-92 [17]. For this purpose, eight representative samples were prepared (four of each variety), mixing equal portions of piscos produced under the same conditions.

For this test, the panellists were presented with three coded samples, two of which were identical, and the other, different. The panellists were asked to identify the odd sample of the three. This sensory test is employed frequently for detecting the existence of small differences amongst samples. Once the test was concluded, the results obtained were interpreted, for which a significance level of 95% was required in all cases.

### Statistical analysis

For the data obtained from the chemical quantitative analysis, three different one-factor analysis of variance (ANOVA) tests were carried out to look for discriminant odorants. Also, Discriminant Analysis was carried out using SPSS software (version 15.0) from SPSS Inc. (Chicago).

## Results and Discussion

### Chemical differences

Tables 2 and 3 show the results obtained from quantitative analysis

Fermentation process	ITALIA				QUEBRANTA			
	Complete	Incomplete	Complete	Complete	Complete	Incomplete	Complete	Complete
Distillation instrument	Alembic	Alembic	Alembic	Falca	Alembic	Alembic	Alembic	Falca
Production scale	Artisan	Artisan	Industrial	Artisan	Artisan	Artisan	Industrial	Artisan
Number of samples	9	7	5	7	6	6	8	3
Abbreviation	I-C-A-C	I. Green Must	I. Industrial	I. Falca	Q-C-A-C	Q.Green Must	Q. Industrial	Q. Falca

**Table 1:** Groups of pisco samples analysed in this study according to their variety, fermentation process, distillation instrument and production scale.

of 64 volatile compounds in 23 and 28 pisco of the Quebranta and Italia varieties, respectively, produced using differing procedures. In both tables the samples have been collected into four groups. The first was considered to be the reference group, containing the pisco resulting from the distillation of fresh must with complete fermentation, distilled

in an alembic using artisan methods; these being the ones most often found on the market.

The second group includes green must pisco (with incomplete fermentation), distilled in an alembic using artisan methods. Comparing this group with the reference group, evaluation was made

QUEBRANTA mg/L	Q-C-A-C* (n=6)			Green Must (incomplete fermentation) (n=6)			Industrial (n=8)			Falca (n=3)		
	min	max	av	min	max	av	min	max	av	min	max	av
Acetaldehyde <sup>a, b</sup>	21.0	69.0	37.3	30.5	169	90.9	35.7	88.1	57.6	5.8	34.1	22.3
Isobutanol	1.5	2.4	2.0	1.4	3.2	2.2	1.7	4.5	2.5	1.3	2.1	1.7
Methyl acetate	2.9	5.4	4.2	3.3	6.9	5.3	3.4	7.6	5.3	3.6	4.3	4.0
Ethyl acetate	33.1	116	63.8	54.7	129	82.4	79.2	618	218	2.6	95.1	63.3
Propanol	19.7	52.2	34.5	26.1	77.6	43.1	31.0	163	66.2	22.4	57.8	42.6
Isobutanol	89.4	206	136	39.3	204	133	25.8	188	83.7	93.3	211	159
1-Butanol	2.1	8.2	4.2	2.7	7.8	4.1	1.9	5.8	3.9	7.5	65.7	31.2
2-Methyl-1-butanol <sup>b</sup>	71.7	121	104	48.7	127	102	34.5	120	66.1	70.6	132	110
3-Methyl-1-butanol <sup>b</sup>	328	598	497	207	658	516	132	556	326	378	619	533
3-Hydroxy-2-butanone	9.8	12.3	11.2	10.3	16.1	12.5	10.8	31.7	14.6	10.0	11.5	10.8
Ethyl lactate	11.2	41.8	24.1	11.1	36.4	22.2	10.6	343	72.8	22.4	33.4	27.8
1-Hexanol	1.7	3.9	2.4	1.7	5.2	3.5	1.3	3.4	2.4	1.6	4.7	3.4
Ethyl octanoate	0.5	1.3	0.8	0.7	3.9	1.4	0.4	5.6	1.8	0.1	1.2	0.6
Furfural	1.3	5.1	3.5	1.6	6.9	3.6	1.5	24.4	8.8	1.2	3.7	2.4
Acetic acid	15.6	124	84.1	39.7	141	93.3	38.6	319	139	89.1	173	140
2,3-Butanediol	11.7	32.2	22.1	14.4	22.3	19.1	10.5	31.7	21.9	10.7	23.5	18.8
Diethyl succinate	0.6	2.9	2.1	0.5	2.0	1.3	0.5	4.3	2.1	0.9	2.6	1.9
β-Phenylethanol <sup>b</sup>	16.6	48.8	37.5	6.4	53.1	33.8	5.2	37.8	17.2	26.3	48.3	39.0
<b>µg/L</b>												
Isobutyl acetate	117	281	171	103	316	209	72.1	407	227	19.4	187	95.0
Ethyl butyrate <sup>b</sup>	89.3	206	147	110	460	203	65.6	588	343	135	380	255
Butyl acetate <sup>a, b, c</sup>	0.8	5.7	2.0	3.6	18.8	9.5	6.7	31.3	15.6	11.9	13.1	12.4
Ethyl 2-methylbutyrate	8.9	126	54.0	8.8	84.4	39.6	14.2	248	71.0	8.5	34.5	20.5
Ethyl isovalerate	19.5	252	102	24.9	199	93.6	52.7	524	169	22.1	69.1	41.2
Isoamyl acetate	176	1593	502	447	2300	973	142	3422	918	29.6	388	199
Ethyl hexanoate <sup>a</sup>	69.9	142	100	113	179	137	78.3	244	157	11.5	230	94.0
l-Limonene oxide	1.9	7.8	4.0	2.5	29.0	11.6	1.2	61.3	14.9	2.4	154	67.2
c-3-Hexenol	49.8	177	105	82.7	412	155	59.7	556	209	108	397	230
c-Linalool oxide	33.4	185	82.8	49.5	574	221	18.1	2387	462	78.1	759	305
l-Linalool oxide	22.5	119	56.9	30.6	318	137	12.6	1347	249	33.1	737	273
α-Terpinolene <sup>c</sup>	1.5	3.7	2.8	2.8	39.6	15.4	2.6	79.1	15.9	4.5	21.1	13.5
Benzaldehyde	40.2	102	74.1	51.5	262	124	66.8	556	201	55.7	159	110
Linalool	99.2	548	272	90.8	2198	832	47.2	5562	810	193	425	286
Ethyl furoate	18.2	35.7	26.1	13.3	56.6	34.5	21.2	128	53.3	10.3	32.9	23.5
Phenylacetaldehyde <sup>b</sup>	1.8	5.8	3.8	0.7	13.1	7.2	6.3	16.5	10.5	2.4	106	44.0
Ethyl decanoate	660	1612	1099	518	11541	3495	81.8	9800	1957	37.6	2487	888
α-Terpineol	41.0	211	107	38.1	1072	324	17.7	4539	692	52.2	262	136
Neryl acetate <sup>b, c</sup>	2.5	2.7	2.6	0.9	3.3	2.2	<0.2	1.6	0.9	0.7	1.8	1.4
β-Citronellol	29.6	62.3	43.3	22.9	313	129	9.4	662	106	45.8	110	71.6
Nerol	14.5	71.1	37.2	26.1	256	107	9.8	837	123	39.5	71.2	51.3
β-Damascenone <sup>a</sup>	6.1	31.9	20.6	38.7	90.5	63.0	8.8	66.2	28.9	16.9	58.9	32.5
β-Phenylethyl acetate	603	5885	2711	1059	9865	4311	120	1948	881	917	3743	2350
Geraniol	24.1	82.2	51.6	31.4	486	186	14.7	1108	158	43.3	100	66.6
Guaiacol <sup>b</sup>	8.5	15.2	12.5	0.1	54.4	15.1	<0.1	2.5	0.5b	7.9	18.5	12.6
Benzyl alcohol	303	932	456	150	525	319	106	2514	628	385	4054	1664
Ethyl dihydrocinnamate <sup>c</sup>	1.1	4.0	2.2	1.4	19.7	6.4	1.0	12.7	3.5	3.0	41.1	22.1
c-Whiskylactone		<0.5		<0.5	7.2	1.6	<0.5	15.5	5.5	0.5	0.5	0.5
o-Cresol <sup>c</sup>	4.7	6.4	5.5	3.0	13.6	6.1	2.6	9.8	6.1	8.1	20.4	12.5
γ-Nonalactone <sup>a</sup>	18.1	44.2	29.0	30.1	98.9	55.5	9.5	110	48.2	32.0	121	64.0
4-Ethylguaiacol	3.2	35.1	14.4	3.7	309	57.9	<0.1	23.3	5.6	3.5	26.8	11.7
m-Cresol <sup>b</sup>	2.3	5.3	4.0	0.1	5.3	3.2	<0.1	5.0	2.3	4.3	10.7	7.0
4-Propylguaiacol	0.1	0.8	0.3	<0.02	1.5	0.4	<0.02	0.7	0.2	0.5	3.4	1.5

Ethyl cinnamate <sup>b</sup>	0.4	1.7	1.1	0.8	54.7	10.4	0.8	4.5	2.6	0.9	16.5	6.3
γ-Decalactone	2.0	6.3	3.9	2.5	6.0	4.6	1.5	8.3	3.9	2.2	4.2	3.5
4-Ethylphenol	5.2	48.6	21.1	8.9	207	71.9	0.2	261	80.1	10.6	39.4	22.9
δ-Decalactone <sup>c</sup>		<0.1		<0.1	10.5	2.4	<0.1	6.2	1.5	1.0	2.2	1.7
4-Vinylguaiacol	3.3	21.0	9.1	4.0	32.7	11.6	0.3	22.3	8.0	1.6	4.5	3.4
2,6-Dimethoxyphenol		<0.1			<0.1			<0.1			<0.1	
Farnesol <sup>b</sup>	242	1015	473	405	912	623	15.8	188	63.1	121	356	277
4-Vinylphenol <sup>b,c</sup>	437	777	552	65.4	1320	520	18.6	291	108	32.5	97.2	67.8
Vanilline <sup>b</sup>	0.5	2.4	1.3	0.8	5.0	2.4	<0.03	1.0	0.1	0.2	8.2	3.1
Methyl vanillate		<0.04		<0.04	2.0	0.4		<0.04		<0.04	121	40.7
Ethyl vanillate	0.3	1.4	0.9	<0.05	9.7	4.1		<0.05b		<0.05	92.0	31.4
Acetovanillone		<0.03			<0.03		<0.03	0.09	0.04		<0.03	
Syringaldehyde		<0.02			<0.02			<0.02		<0.02	64.2	21.4

\* pisco samples of Quebranta variety produced by complete fermentation of must, distilled in alembic, and produced on an artisan scale.

<sup>a</sup> Statistically significant differences measured by ANOVA at  $p < 0.05$  according to fermentation process, <sup>b</sup> scale of the process, and <sup>c</sup> distillation instrument.

av: average concentration, min: minimum concentration, max: maximum concentration, n: number of samples analysed.

The identification of all these compounds was based on the coincidence of gas chromatographic retention index in DB-WAX column and mass spectrum with those pure compounds.

**Table 2:** Compounds analyzed by GC-FID and GC-MS in Quebranta pisco samples produced by complete and incomplete fermentation of wine, on an industrial or non-industrial scale, and distilled in alembic and falca.

ITALIA	I-C-A-C* (n=9)			Green Must (incomplete fermentation) (n=7)			Industrial (n=5)			Falca (n=7)		
	min	max	av	min	max	av	min	max	av	min	max	av
Acetaldehyde	12.1	90.5	41.1	32.1	618	157	20.2	59.4	47.1	17.4	280	57.7
Isobutanol	1.5	3.1	2.1	1.5	2.1	1.8	2.0	3.8	2.8	1.7	2.5	2.1
Methyl acetate <sup>c</sup>	3.4	5.7	4.2	3.6	5.6	4.2	3.5	10.5	5.7	4.4	7.0	6.1
Ethyl acetate	76.1	455	235	49.0	260	141	66.8	172	121	67.0	547	162
Propanol	43.4	108	64.1	36.7	153	83.5	50.5	194	94.9	37.9	80.9	47.2
Isobutanol	63.1	235	139	68.3	152	106	27.7	138	91.8	99.4	172	117
1-Butanol	2.0	59.8	10.2	1.9	17.6	5.9	2.7	4.4	3.6	4.6	70.2	15.7
2-Methyl-1-butanol	38.6	87.8	63.7	44.0	92.6	61.6	33.9	79.2	60.6	59.8	74.4	65.4
3-Methyl-1-butanol	182	434	301	218	565	345	220	390	292	298	371	330
3-Hydroxy-2-butanone	9.7	40.0	21.4	9.9	45.2	18.1	10.3	22.5	13.0	9.7	15.7	11.2
Ethyl lactate	22.4	72.9	44.5	11.0	67.7	32.1	12.8	355	125	32.5	91.9	47.4
1-Hexanol	1.9	7.3	3.5	1.1	9.6	3.5	1.1	3.4	2.1	1.6	4.5	2.8
Ethyl octanoate	0.3	0.7	0.5	0.5	3.1	1.1	0.4	2.4	1.0	0.3	0.9	0.6
Furfural	1.4	9.2	5.3	0.6	16.0	6.4	4.4	38.7	14.4	0.7	7.1	4.7
Acetic acid	102	512	206	8.0	338	148	7.7	180	81.3	30.4	316	145
2,3-Butanediol <sup>a,c</sup>	17.3	32.9	26.4	6.5	23.7	15.1	13.1	28.5	19.3	15.1	20.2	17.2
Diethyl succinate	0.7	4.4	1.4	0.5	1.9	0.8	0.6	1.9	1.3	0.6	2.5	1.9
β-Phenylethanol	8.0	30.9	17.9	6.4	26.2	14.0	6.8	26.9	13.0	13.6	28.9	22.5
<b>µg/L</b>												
Isobutyl acetate	125	920	304	112	457	189	98.2	247	156	121	979	268
Ethyl butyrate	62.2	1141	244	56.7	391	139	121	427	217	152	2681	563
Butyl acetate	9.1	78.1	20.6	<0.8	12.5	7.8	6.4	27.2	12.7	8.5	204	38.7
Ethyl 2-methylbutyrate	8.0	58.4	25.5	5.4	53.4	18.4	9.4	76.1	28.7	6.3	329	79.5
Ethyl isovalerate	21.5	168	68.6	15.1	82.7	43.8	37.7	187	80.9	17.3	716	183
Isoamyl acetate	170	535	315	164	1727	579	84.5	2770	789	154	589	329
Ethyl hexanoate	48.9	140	83.6	76.7	135	103	68.0	202	127	58.1	154	102
l-Limonene oxide <sup>c</sup>	44.6	113	71.6	30.0	101	62.2	40.9	272	120	73.9	118	106
c-3-Hexenol	288	1499	845	78.9	935	477	342	928	543	451	946	684
c-Linalool oxide <sup>c</sup>	1115	2492	1701	754	2112	1489	1127	3329	2111	1842	2449	2245
l-Linalool oxide <sup>c</sup>	548	1105	758	442	1742	797	446	880	665	935	1354	1129
α-Terpinolene	44.3	293	122	51.9	209	119	35.7	69.0	54.1	39.1	220	109
Benzaldehyde	29.3	543	186	51.3	205	122	139	809	418	102	257	148
Linalool	3766	8780	5851	3086	14965	8425	3078	6852	4899	4502	8047	5816
Ethyl furoate	10.6	39.3	24.8	11.0	46.9	24.0	18.8	61.8	41.4	12.4	46.9	34.3
Phenylacetaldehyde <sup>a</sup>	2.1	10.1	4.9	1.4	3.5	2.5	1.6	7.1	4.2	3.3	8.3	5.0
Ethyl decanoate	252	1274	510	222	9473	2062	69.8	2070	629	250	1254	654
α-Terpineol	2312	5597	3814	1695	5727	3710	2428	6803	4660	3117	5301	4086
Neryl acetate		<0.2		<0.2	3.8	1.1	<0.2	1.4	0.7	<0.2	1.6	0.5

β-Citronellol	383	1307	821	196	1212	770	132	828	551	490	1188	733
Nerol <sup>a</sup>	282	1227	804	425	2050	1324	253	1222	639	623	1227	788
β-Damascenone	60.2	90.8	76.1	55.4	368	138	43.9	119	85.8	31.8	143	101
β-Phenylethyl acetate	250	3262	982	341	2157	1143	121	1768	790	80.4	4137	1456
Geraniol	554	2053	1424	737	3922	2276	402	2249	1096	1041	2461	1413
Guaiacol <sup>b,c</sup>	3.0	7.6	5.0	2.4	11.2	5.7	5.5	9.2	7.5	5.1	17.2	11.6
Benzyl alcohol	139	856	355	78.5	398	254	44.0	1394	580	188	846	581
Ethyl dihydrocinnamate <sup>b</sup>	0.1	8.6	2.7	<0.1	18.8	6.0	1.9	18.5	9.2	1.7	62.0	11.0
c-Whiskylactone	5.9	13.3	8.0	<0.5	17.8	7.8	<0.5	11.6	5.2	<0.5	20.1	9.7
o-Cresol <sup>c</sup>	3.0	6.9	4.7	2.7	10.9	5.9	3.4	10.2	6.8	4.7	16.2	8.4
γ-Nonalactone	18.5	536	126	15.6	160	96.6	53.9	182	106	50.6	67.9	58.5
4-Ethylguaiacol	1.5	486	65.4	<0.1	192	36.2	<0.1	97.5	47.7	5.6	97.3	52.5
m-Cresol <sup>c</sup>	<0.1	4.1	2.0	1.5	4.0	2.3	<0.1	5.7	3.3	3.8	10.6	5.9
4-Propylguaiacol	<0.02	0.7	0.1	<0.02	1.0	0.2	<0.02	1.6	0.5	<0.02	0.5	0.3
Ethyl cinnamate <sup>a,b</sup>	<0.1	3.7	0.8	0.5	9.2	3.5	<0.1	7.1	4.0	<0.1	3.1	2.1
γ-Decalactone	1.6	25.7	7.9	1.5	16.3	6.9	5.2	10.7	7.7	3.4	7.4	5.0
4-Ethylphenol	2.0	317	47.1	<0.2	1913	361	0.2	470	184	11.6	404	70.1
δ-Decalactone <sup>c</sup>	<0.1	15.9	5.6	3.3	9.9	6.2	5.4	31.3	15.6	6.2	21.2	12.6
4-Vinylguaiacol	2.7	62.0	12.7	12.6	50.3	29.2	0.3	50.5	15.9	0.3	51.2	23.1
2,6-Dimethoxyphenol		<0.1			<0.1			<0.1			<0.1	
Farnesol <sup>a</sup>	90.9	375	259	140	743	457	13.1	489	138	101	619	295
4-Vinylphenol	32.7	272	117	51.1	747	305	95.1	306	195	19.2	1354	308
Vanilline	0.3	3.4	1.2	1.0	7.4	2.4	0.6	12.6	3.7	<0.03	4.4	2.2
Methyl vanillate		<0.04			<0.04			<0.04			<0.04	
Ethyl vanillate		<0.05		<0.05	4.1	1.1	<0.05	10.6	3.0		<0.05	
Acetovanillone <sup>a</sup>		<0.03		<0.03	1.00	0.42	<0.03	1.2	0.4		<0.03	
Syringaldehyde		<0.02			<0.02			<0.02			<0.02	

\* pisco samples of Italia variety produced by complete fermentation of must, distilled in alembic, and produced on an artisan scale.

<sup>a</sup> Statistically significant differences measured by ANOVA at  $p < 0.05$  according to fermentation process, <sup>b</sup> scale process, and <sup>c</sup> distillation instrument.

av: average concentration, min: minimum concentration, max: maximum concentration, n: number of samples analysed.

The identification of all these compounds was based on the coincidence of gas chromatographic retention index in DB-WAX column and mass spectrum with those pure compounds.

**Table 3:** Compounds analysed by GC-FID and GC-MS in Italia pisco samples produced by complete and incomplete fermentation of must, on an industrial or artisan scale, and distilled in alembic and *falca*.

of the effect on the volatile composition of these piscos obtained from most of complete fermentation or incomplete fermentation (green must).

The third group consists of piscos of complete fermentation, distilled in an alembic and produced on an industrial scale. Thus, this group served for evaluating the effect of different production scales.

Finally, the fourth group includes piscos of complete fermentation, distilled in a *falca* using artisan methods. Thus, the samples of this last group served for evaluating the influence of distillation in an alembic or a *falca*.

These two tables show the minimum, maximum, and average levels for each of the four sample groups. Different analyses of the variance of a single factor (ANOVA) were carried out in order to evaluate: 1) the "time point of distillation" factor, 2) the "production scale" factor, and 3) the "distillation equipment" factor. The compounds that showed significant differences ( $p < 0.05$ ) in these ANOVA analyses are shown with distinct letters (a,b,c).

**Time point of must distillation:** As can be seen in Table 2, few significant differences were found between the Quebranta pisco samples resulting from completely or incompletely fermented must (comparison of the first two columns of the table). In fact, only five of the 64 analysed compounds showed significant differences via ANOVA. These are acetaldehyde, butyl acetate, ethyl hexanoate, β-damascenone, and γ-nonalactone. In every case, the highest levels corresponded to the green must pisco samples. It is notable that both the ethyl hexanoate

and the acetaldehyde were found in concentrations greater than the olfactory threshold, in both the green musts and the piscos with complete fermentation, as explained below, which leads us to think there is a possible sensory effect from both aromatic compounds in the characteristic aroma of the Quebranta variety. Furthermore, with most of the studied families (esters, acetates, terpenes, cinnamates, volatile phenols, and lactones), slightly higher levels were observed in the samples of green must pisco.

With respect to the Italia variety (Table 3), the differences that were found to relate to complete versus incomplete fermentation corresponded to six compounds. Nerol, ethyl cinnamate, farnesol, and acetovanillone (detected in low concentrations) were shown to be significantly higher in the samples that were produced from green must; while other compounds like 2,3-butanediol and phenyl acetaldehyde showed the lowest levels when fermentation was incomplete. It is worth noting that the majority of these discriminant compounds were found in low concentrations, much lower than their corresponding olfactory thresholds, as will be presented below, which could be an indicator of the low aromatic potential of these compounds on an individual level. Nevertheless, it is possible to state that some important terpenes, such as linalool, nerol, and geraniol, were at slightly higher levels in the green musts. Linalool was found in concentrations of up to 14.96 mg L<sup>-1</sup> in the green must samples (nearly double the maximum value found in the samples produced from completely fermented musts). Nevertheless, for this pisco variety, higher concentrations of the majority of analysed volatiles were not so clearly observed in the green musts.

**Distillation equipment:** Some of the compounds in the Quebranta variety, such as neryl acetate and 4-vinylphenol, were shown to be significantly higher in the piscos distilled in an alembic, whereas other compounds, such as butyl acetate,  $\alpha$ -terpinolene, ethyl dihydrocinnamate, o-cresol, and  $\delta$ -decalactone, had significantly greater values in samples distilled in a *falca*.

In general terms, the samples distilled in a *falca* were characterized by higher concentrations of terpenes, cinnamates, vanillines, and, to a lesser extent, of higher alcohols. Especially noteworthy was the presence of methyl vanillate and ethyl vanillate in concentrations reaching  $121 \mu\text{g L}^{-1}$  and  $92 \mu\text{g L}^{-1}$ , respectively, in some samples.

The samples distilled in an alembic showed higher levels of the ester family and also of some volatile phenols, such as 4-vinylphenol, whose average values using an alembic were higher than those of the *falca* by a factor of eight.

With respect to the Italia variety, eight of the nine differentiating compounds showed significantly higher values in the samples distilled in a *falca*, of which three were terpenes (t-limonene oxide, c- and t-linalool oxide) and three were volatile phenols (guaiacol, o-cresol and m-cresol). On the other hand, the samples distilled in an alembic showed significantly higher values only of 2,3-butanediol. In general terms, once again there were slightly higher observed levels of analysed volatiles in the *falca*-distilled samples.

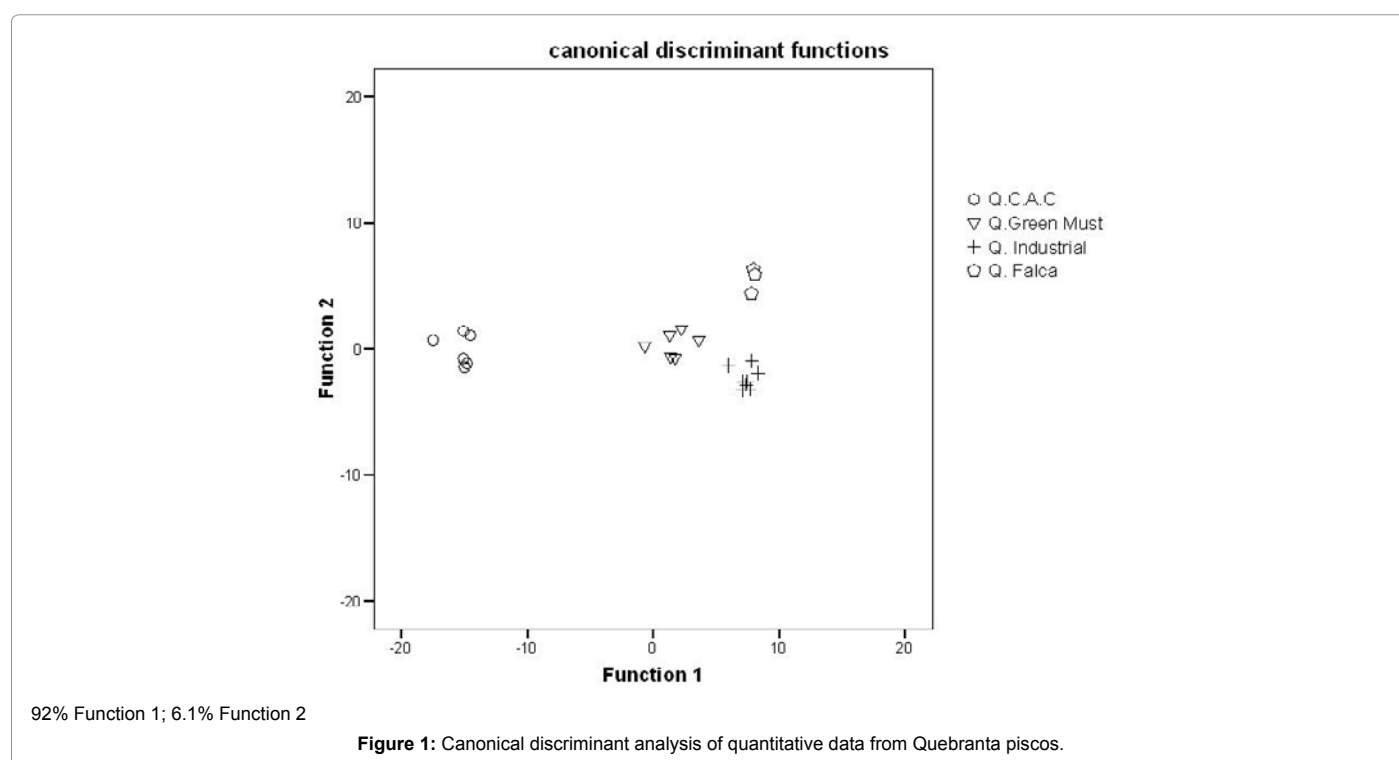
**Production scale:** In samples of the Quebranta variety of pisco, 16 of the 64 analysed compounds showed significant differences when a comparison of production scale, either industrial or artisan, was made (Table 2). Samples produced by artisan methods showed significantly higher contents of some alcohols, such as 2- and 3-methyl-1-butanol and  $\beta$ -phenylethanol, some volatile phenols (guaiacol, m-cresol and 4-vinylphenol), and some acetates and vanillins. Nevertheless, the samples from industrial-scale production were generally richer in nearly all the analysed compounds. Of these, it is especially noteworthy

that the observed levels of acetaldehyde, phenyl acetaldehyde, of some esters, and of ethyl cinnamate were significantly higher than those found in the artisan piscos.

The samples of the Italia variety showed fewer important differences than those observed in the Quebranta piscos (as can be seen in Table 3). In fact, only three compounds allowed for a statistical differentiation of the two production processes. These were guaiacol and the cinnamates (ethyl cinnamate and ethyl dihydrocinnamate), which showed higher values in samples produced on an industrial scale. In general, it can be concluded that, for the Italia variety, the aromatic profiles observed in the two types of processes were very similar, according to the quantitative data. In fact, the compounds that showed significant differences were found in low concentrations, such that, on first impression, they do not appear sufficient to allow for sensory differentiation.

**Graphic representation of quantitative results:** The main conclusion from these quantitative results is that the variations attributed to the evaluated processes (the point in time when distillation takes place, the distillation equipment, and the scale of production) depend mostly on the grape variety, since the differentiating compounds are not the same in the two varieties, except for o-cresol and  $\delta$ -decalactone, which showed higher levels in the *falca*-distilled samples in both the Quebranta and the Italia varieties; and ethyl cinnamate, which showed higher concentrations in samples produced on an industrial scale in both varieties.

In order to see graphically the differences amongst the samples produced by the distinct processes, a discriminant analysis was carried out on all the quantitative data, grouping the samples according to production process. Figure 1 shows a graphic representation of Quebranta variety samples, located in the space defined by the two obtained canonical functions, of which canonical function 1 explained 92 % of the variance and allowed for a clear separation of three sample



groups. This graphic clearly shows that what is taken as the reference group (piscos with complete fermentation, alembic distillation, and artisan production) is well removed from the rest of the groups, all of which underwent some type of technical variation in the production process. Therefore, it is clear that each of the evaluated parameters causes differences in the volatile composition of the resulting piscos. Figure 1 allows for the conclusion that the greatest differences in the volatile composition of the Quebranta piscos were obtained when changing either the distillation equipment (*falca*/alembic) or the production scale (artisan/industrial), while the differences were less marked between green must and completely-fermented must samples.

In the same way, Figure 2 shows the distribution resulting from a discriminant analysis applied to samples of the Italia variety. Function 1, which was able to explain 84.5% of the total variance, showed in this case a more clear separation between the four sample groups. Here, the greatest distance appeared between the reference samples and those produced on an industrial scale.

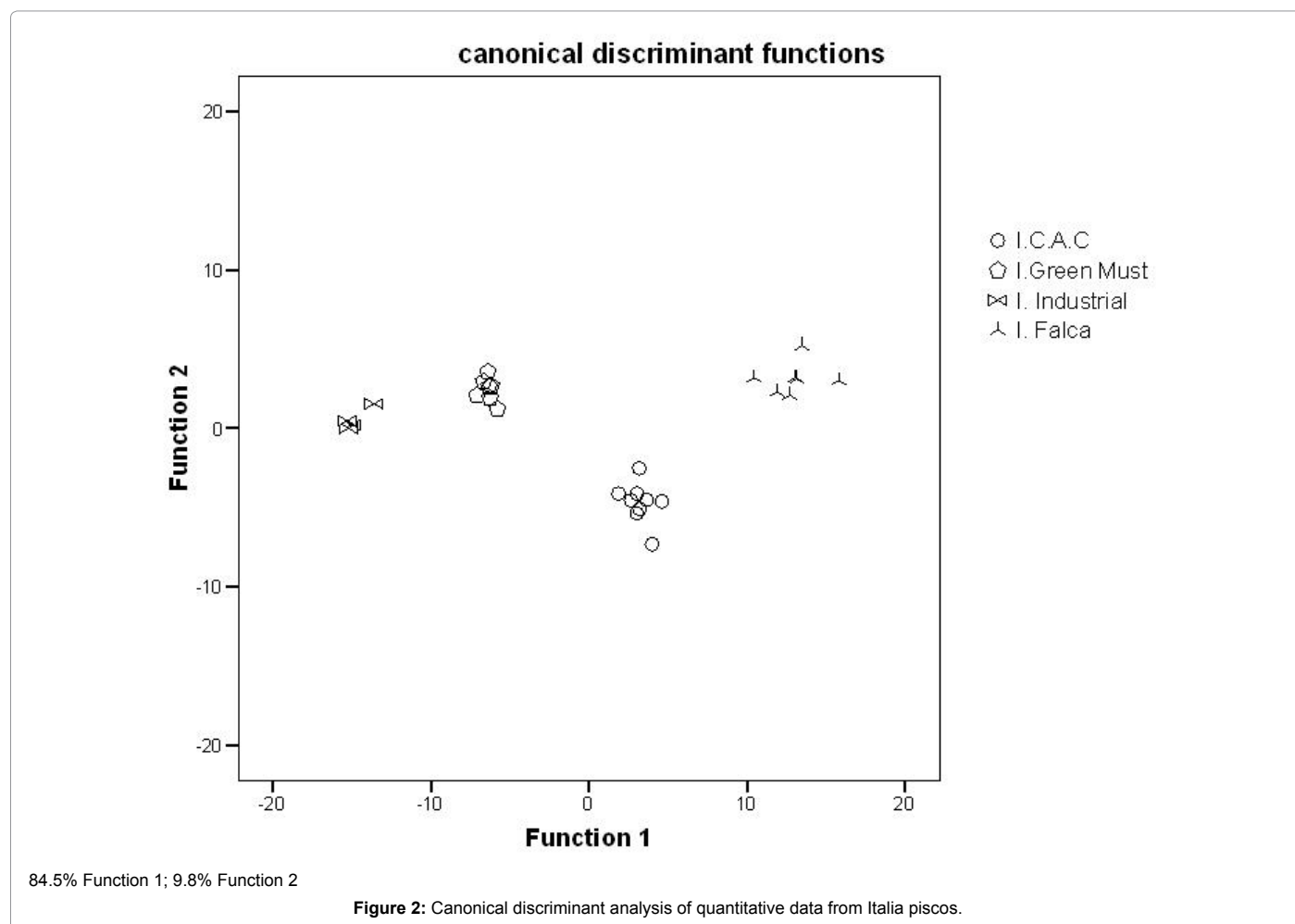
### Sensory analysis

**Sensory contribution of individual aromatic compounds:** Tables 4 and 5 show the thresholds of individual olfaction for 35 selected compounds, representing each of the principal chemical families which were determined in a previous work [18]. Using these thresholds, aroma values were estimated for each of the compounds in the Quebranta and Italia piscos, respectively.

Based on these results, the compound with the highest aromatic relevance, showing the maximum OAV in both varieties, was ethyl isovalerate. Levels of this ethyl ester were on average 34 and 37 times higher than the threshold in the industrially-produced Quebranta samples and in the *falca*-distilled Italia samples, respectively. This fact would explain the greater aromatic intensity attributed to these Quebranta samples during sensory analysis, when compared with the artisan-produced samples. Nevertheless, this fact would not explain observations made with regard to the Italia variety, where no significant differences were seen when either a *falca* or an alembic were used for distillation (Table 6).

Another distinctive compound of the Italia variety is linalool. Table 5 shows how the average concentrations are eight times greater than the olfactory threshold in samples produced from green must, when compared with those produced by complete fermentation. Another outstanding compound is 3-methyl-1-butanol, with median OAV of between 3 and 5 in both varieties, but with the highest obtained values in the *falca*-distilled Quebranta variety. Finally, ethyl hexanoate also stands out, showing OAV >1 in 48 of the 51 analysed samples, with median OAV of between 1.7 and 3.1 in both varieties, with the highest value being found in the industrial-scale Quebranta.

The OAV of the volatile phenols, lactones, some terpenes, acetates, alcohols, acetic acid,  $\beta$ -damascenone, farnesol, and phenyl acetaldehyde were less than 0.1 units in all the analysed samples, which suggests, at



	Odour Threshold*	Q-C-A-C (n=6)		Green Must (n=6)		Industrial (n=8)		Falca (n=3)		
		OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)	
<b>QUEBRANTA</b>	<b>mg L<sup>-1</sup></b>									
<b>Esters</b>										
Ethyl butyrate	0.25	0.6	0	0.8	1	1.4	5	1.0	2	
Ethyl 2-methylbutyrate	0.1	0.5	1	0.4	0	0.7	2	0.2	0	
Ethyl isovalerate	0.005	20	6	19	6	34	8	8.2	3	
Ethyl hexanoate	0.05	2.0	6	2.7	6	3.1	8	1.8	1	
<b>Acetates</b>										
Methyl acetate	> 500	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
Butyl acetate	10	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
β-Phenylethyl acetate	2.5	1.1	3	1.7	3	0.4	0	0.9	1	
<b>Alcohols</b>										
1-Butanol	> 1000	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
2-Methyl-1-butanol	75	1.4	5	1.4	5	0.9	2	1.5	2	
3-Methyl-1-butanol	100	5.0	6	5.2	6	3.3	8	5.3	3	
2,3-Butanediol	250	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
β-Phenylethanol	20	1.9	5	1.7	5	0.9	2	1.9	3	
<b>Terpenes</b>										
t-Limonene oxide	5	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
c-Linalool oxide	5	<0.1	0	<0.1	0	0.09	0	<0.1	0	
t-Linalool oxide	5	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
α-Terpinolene	2.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
Linalool	1	0.3	0	0.8	2	0.8	1	0.3	0	
α-Terpineol	300	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
β-Citronellol	1	<0.1	0	0.1	0	0.1	0	<0.1	0	
Nerol	40	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
Geraniol	3	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
<b>Sesquiterpene</b>										
Farnesol	> 1000	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
<b>Aldehydes</b>										
Acetaldehyde	25	1.5	4	3.6	6	2.3	8	0.9	2	
Phenylacetaldehyde	0.3	<0.1	0	<0.1	0	<0.1	0	0.2	0	
<b>Volatile phenols</b>										
Guaiacol	0.1	0.12	0	0.15	0	<0.1	0	0.1	0	
o-Cresol	0.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
m-Cresol	0.1	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
4-Vinylguaiacol	2.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
4-Vinylphenol	> 100	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
<b>Norisoprenoide</b>										
β-Damascenone	10	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
<b>Cinnamates</b>										
Ethyl cinnamate	> 1	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
Ethyl dihydrocinnamate	0.03	<0.1	0	0.2	0	0.1	0	0.7	1	
<b>Lactones</b>										
γ-Nonalactone	0.625	<0.1	0	<0.1	0	<0.1	0	0.1	0	
δ-Decalactone	2	<0.1	0	<0.1	0	<0.1	0	<0.1	0	
<b>Acids</b>										
Acetic acid	600	0.1	0	0.2	0	0.2	0	0.2	0	

Q-C-A-C: pisco samples of Quebranta variety produced by complete fermentation of must, distilled in alembic, and produced on an artisan scale.

av: average, n (OAV>1): number of samples exceeding the threshold value. \*Odour threshold values (in ethanol/water solution 40% (v/v)) were estimated by Cacho et al. [1].

**Table 4:** Individual odour thresholds (mg L<sup>-1</sup>) and average odour activity values (OAV<sub>av</sub>) of different compounds obtained in Quebranta pisco samples produced by different processes.

least on the surface, that these compounds do not individually have any great sensory impact.

**Sensory differences (triangle tests):** The aromatic changes introduced by the different production processes were evaluated by means of triangle tests on each variety, following the previously-described procedure (2.5.2.). The results are shown in Table 6.

As can be seen, the samples produced from green must were significantly different, from a sensory point of view, from the samples produced using completely fermented must; this was true for both grape varieties. According to the panel of tasters, the Quebranta green must pisco presented a sweeter and more terpenic aroma, with greater aromatic intensity, which may be explained by the higher observed levels of terpenes and acetates. The Italia green must pisco, on the other



	Odour Threshold*	I-C-A-C (n=9)		Green Must (n=7)		Industrial (n=5)		Falca (n=7)	
		OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)	OAV <sub>av</sub>	n (OAV>1)
<b>ITALIA</b>	<b>mg L<sup>-1</sup></b>								
<b>Esters</b>									
Ethyl butyrate	0.25	1.0	2	0.6	1	0.9	1	2.3	3
Ethyl 2-methylbutyrate	0.1	0.3	0	0.2	0	0.3	0	0.8	1
Ethyl isovalerate	0.005	14	9	8.8	7	16	5	37	7
Ethyl hexanoate	0.05	1.7	8	2.1	7	2.5	5	2.0	7
<b>Acetates</b>									
Methyl acetate	> 500	<0.1	0	<0.1	0	<0.1	0	<0.1	0
Butyl acetate	10	<0.1	0	<0.1	0	<0.1	0	<0.1	0
β-Phenylethyl acetate	2.5	0.4	1	0.5	0	0.3	0	0.6	1
<b>Alcohols</b>									
1-Butanol	> 1000	<0.1	0	<0.1	0	<0.1	0	<0.1	0
2-Methyl-1-butanol	75	0.8	3	0.8	1	0.8	2	0.9	0
3-Methyl-1-butanol	100	3.0	9	3.5	7	2.9	5	3.3	7
2,3-Butanediol	250	0.1	0	<0.1	0	<0.1	0	<0.1	0
β-Phenylethanol	20	0.9	3	0.7	1	0.6	1	1.1	5
<b>Terpenes</b>									
t-Limonene oxide	5	<0.1	0	<0.1	0	<0.1	0	<0.1	0
c-Linalool oxide	5	0.3	0	0.3	0	0.4	0	0.4	0
t-Linalool oxide	5	0.1	0	0.2	0	0.1	0	0.2	0
α-Terpinolene	2.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0
Linalool	1	5.8	9	8	7	4.9	5	5.8	7
α-Terpineol	300	<0.1	0	<0.1	0	<0.1	0	<0.1	0
β-Citronellol	1	0.8	3	0.8	1	0.6	0	0.73	2
Nerol	40	<0.1	0	<0.1	0	<0.1	0	<0.1	0
Geraniol	3	0.5	0	0.8	2	0.4	0	0.47	0
<b>Sesquiterpene</b>									
Farnesol	> 1000	<0.1	0	<0.1	0	<0.1	0	<0.1	0
<b>Aldehydes</b>									
Acetaldehyde	25	1.6	7	6	7	1.9	4	2.3	2
Phenylacetaldehyde	0.3	<0.1	0	<0.1	0	<0.1	0	<0.1	0
<b>Volatile phenols</b>									
Guaiacol	0.1	<0.1	0	<0.1	0	<0.1	0	0.1	0
o-Cresol	0.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0
m-Cresol	0.1	<0.1	0	<0.1	0	<0.1	0	<0.1	0
4-Vinyguaiacol	2.5	<0.1	0	<0.1	0	<0.1	0	<0.1	0
4-Vinylphenol	> 100	<0.1	0	<0.1	0	<0.1	0	<0.1	0
<b>Norisoprenoide</b>									
β-Damascenone	10	<0.1	0	<0.1	0	<0.1	0	<0.1	0
<b>Cinnamates</b>									
Ethyl cinnamate	> 1	<0.1	0	<0.1	0	<0.1	0	<0.1	0
Ethyl dihydrocinnamate	<0.1	0.1	0	0.2	0	0.3	0	0.4	1
<b>Lactones</b>									
γ-Nonalactone	0.625	0.2	0	0.2	0	0.2	0	<0.1	0
δ-Decalactone	2	<0.1	0	<0.1	0	<0.1	0	<0.1	0
<b>Acids</b>									
Acetic acid	600	0.3	0	0.2	0	0.1	0	0.2	0

I-C-A-C: pisco samples of Italia variety produced by complete fermentation of must, distilled in alembic, and small scale produced av: average., n (OAV>1): number of samples exceeding the threshold value. \* Odour threshold values (in ethanol/water solution 40% (v/v)) were estimated by Cacho et al. [1].

**Table 5:** Individual odour thresholds (mg L<sup>-1</sup>) and average odour activity value (OAV<sub>av</sub>) of different compounds obtained in Italia pisco samples produced by different process.

hand, was defined as having a more floral and raisin aroma. This may be partially related to the higher (though not significantly higher) levels of compounds like β-damascenone, of acetates such as isoamyl acetate and β-phenylethyl acetate, linear esters, and terpenes like linalool, known to give sweet and floral aromas.

Regarding the samples distilled in a *falca* or an alembic, a sensory

distinction between these two methods could be made only for the Quebranta variety: the *falca*-distilled Quebranta samples were said to have a more intense aroma, which some judges describes as an aroma of nuts. There were no significant sensory differences noted in the Italia variety samples, as related to the distillation equipment; this corroborates the few quantitative differences observed. Finally, the Italia variety was found to have more significant sensory

		Correct responses/ total responses	p
"Time point of distillation" effect	Quebranta	18/24	< 0.001
	Italia	16/24	0.001
"Distillation equipment" effect	Quebranta	15/24	0.01
	Italia	11/24	NS
"Production scale" effect	Quebranta	13/24	0.05
	Italia	17/24	< 0.001

NS: No significant difference

**Table 6:** Triangle test results.

differences ( $p < 0.001$ ), when industrial and artisan production scales were compared. The artisan production sample was described as being sweeter and with a greater aromatic intensity. This result was concordant with the distribution obtained in Figure 2, although the ANOVA did not show great quantitative differences. Nevertheless, it is possible that the higher level of some terpenes, such as linalool and geraniol, in the artisan samples contributed to this aromatic intensity.

In the case of the Quebranta variety, the industrial production samples showed a greater aromatic intensity, which could be attributable to the higher levels of esters and terpenes found in the samples produced on an industrial scale.

## Conclusions

It is possible to deduce from the results obtained in this study that chemical differences do indeed result from modifications in the production processes of pisco. Green must piscos can be seen to have slightly higher levels of most analysed volatiles when compared with piscos made from completely fermented musts, especially in the case of Quebranta piscos. On the other hand, *falca*-distilled piscos show slightly higher levels of volatiles as compared to those distilled in an alembic; the production scale also affects the chemical profile of both varieties of pisco. In any case, when the individual behaviour of the compounds in both grape varieties is evaluated, distinct behaviours were observed.

Nearly all the studied technological variations had a sensory effect, with the exception of the change in distillation equipment in the Italia variety. Only a few compounds showed the same behaviour in the distillates of the studied grape varieties. These were *o*-cresol and  $\delta$ -decalactone, which showed higher levels after *falca* distillation in both varieties, and ethyl cinnamate, which had significantly higher levels when made on an industrial scale in both the Quebranta and the Italia varieties.

Lastly, an evaluation was made of the individual aromatic effect of 35 odorants in these analysed pisco samples, in terms of their aroma values (OAV). Especially noteworthy is ethyl isovalerate, which reached aroma values of up to 34 aroma units in the industrial-scale Quebranta, while reaching only 20 aroma units in the artisan samples. In the Italia variety piscos, this ester reached its highest aroma value in the *falca* distillates (37 aroma units), while reaching only 14 aroma units in the alembic distillates. Other compounds that showed aroma values higher than 1 were: 2-methyl-1-butanol, 3-methyl-1-butanol,  $\beta$ -phenylethanol, and acetaldehyde in Quebranta piscos, and 2-methyl-1-butanol, linalool, and acetaldehyde in the Italia variety. Therefore, all these aromatic compounds must be kept in view as having a possible effect on the overall aroma of these pisco varieties.

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