

Comparison of a Novel Distillation Method versus a Traditional Distillation Method in a Model Gin System Using Liquid/Liquid Extraction

DEREK GREER,^{*,†} LES PFAHL,[†] JIM RIECK,[‡] TIM DANIELS,[†] AND OSCAR GARZA[†]

Bacardi-Martini Product Development, 12200 North Main Street, Jacksonville, Florida 32218, and
Clemson University, F148 Poole Hall, Clemson, South Carolina 29634

This research studied a novel form of distillation (high vacuum distillation) as a method for preserving volatile aroma chemicals important to the organoleptic attributes of a four botanical model gin as well as the degradation products generated during the heating required in traditional methods of gin distillation. A 2⁵ factorial experiment was conducted in a partially confounded incomplete block design and analyzed using the PROC MIXED procedure from SAS. A model gin was made of dried juniper berries (*Juniperus communis*), coriander seed (*Coriandrum sativum*), angelica root (*Angelica archangelica*), and dry lemon peel (*Citrus limonum*). This was distilled on a traditional still utilizing atmospheric pressure and a heating mantel to initiate phase separation as well as a novel still (high vacuum) utilizing high vacuum pressures below 0.1 mmHg and temperatures below -15 °C to initiate phase separation. The degradation products (α -pinene, α -phellandrene, *E*-caryophyllene, and β -myrcene) were present at greater levels (~10 times) in the traditional still-made gin as compared to the novel gin.

KEYWORDS: Gin; liquid extraction; vacuum distillation; *Juniperus communis*; *Coriandrum sativum*; *Angelica archangelica*; *Citrus limonum*

INTRODUCTION

This research demonstrates the benefit of distilling botanical extracts (particularly for the manufacture of gin) at temperatures below 0 °C, which retains the natural flavor of the botanicals better than under traditional conditions, thereby producing a superior gin. Gin is a distilled spirit that is colorless and is at least 37.5% alcohol by volume (ABV) in the European Union (EU) to at least 40% ABV in the United States (1). The distillation normally takes place around 70–80 °C. Some makers will apply a slight vacuum to get the distillation to take place at or around 60 °C. Gin was invented by a Dutch physician and professor named Franciscus de la Boe at the University of Leiden in 1650 (2). de la Boe was looking for an inexpensive medication to treat kidney disorders by developing unique ways to deliver juniper oil, a known diuretic, into the body (3). The solvents available to de la Boe were fruit brandies and raw neutral grain spirits. The grain neutral spirits (GNS) were inexpensive and readily available; however, they were harsh and less palatable (3). The addition of juniper oil improved the taste of the GNS (3). He named the medicine “Genever” from the French word “genievre”, which means juniper (2).

Juniperus communis is a member of the Cupressaceae family (4). An evergreen shrub, commonly found in Europe and North

America, it grows well in mild to cool climates and produces small dark blue/purple berries. The berries (*Fructus juniperi*) are used to make essential oils and are used as a diuretic and to flavor gin (5).

According to 27 CFR, in the United States, there are three classes of gin: distilled gin, redistilled gin, and compound gin. Distilled gin is made by original distillation of the mash in the presence of juniper berries (and other botanicals). Redistilled gin is made by using GNS and juniper berries or essential oils of juniper berries. Both of these can be labeled “distilled” in the United States. Compound gin is made by adding natural flavors directly to GNS without a subsequent distillation. This type of gin must be labeled “compound gin”.

Vacuum distillation is not a new idea. The application of a vacuum to improve distillation characteristics is widely used in the petroleum and perfume industry. It is also used in the concentration and dehydration of foods and food ingredients. The benefit of applying a vacuum to distill essential oils has been widely explored. Babu et al. (6) found lower yields but higher quality chemical compositions of *Curcuma longa* L. leaf oil when distilled under vacuum. Lopes et al. (7) reported on the chemical composition of *Citrus sinensis* oil distilled at 80 °C under 10–30 mbar (7.5–22.5 mmHg) finding that while their research used a stronger vacuum than previous experiments, it confirmed previous conclusions finding increases in oxygenated terpenes and a reduction in monoterpenes. This is significant since lower levels of monoterpenes mean greater stability. Fang

* To whom correspondence should be addressed. Tel: 317-484-4091.
E-mail: derek.greer@sensient-tech.com.

[†] Bacardi-Martini Product Development.

[‡] Clemson University.

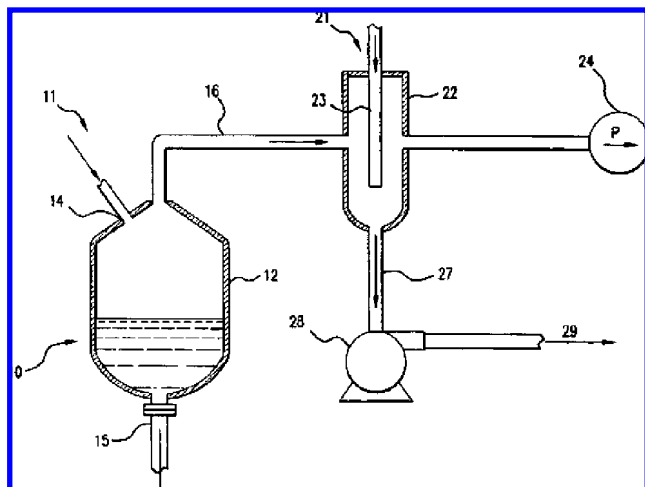


Figure 1. Schematic of the still (17).

et al. (8) compared supercritical CO₂ extraction and vacuum distillation of bergamot oil. They found that vacuum distillation was more effective at removing reactive monoterpenes but had lower yields and inconsistent results. Bagger-Jørgensen et al. (9) studied how effective vacuum distillation can be at stripping black currant juice aroma. They found that the recovery of volatiles was between 68–83%. They further found that the lower the temperature was, the better the recovery was. The technical literature from the past 15–20 years has shown that vacuum distillation effectively circumvents high temperatures and reduces monoterpene formation in the final product.

In recent years, there has been a resurgence of gin in the marketplace, with attempts to make “super-premium” gins using different botanical ingredients. This research studies a new process of distilling gin where, in addition to premium ingredients, the process of distillation does not begin until the temperature of the solution is below 0 °C and the temperature of the solution of the run is maintained at around –15 °C. This novel process was then compared to the traditional distillation by statistical comparison of flavor components between the two products.

MATERIALS AND METHODS

Sourcing and Preparation of Botanicals. All botanicals were obtained from Tradall S.A. (Meryin, Switzerland). Using a digital balance, 37.2 g of juniper berries (*J. communis*), 52.8 g of coriander seeds (*Coriandrum sativum*), 18.0 g of angelica root (*Angelica archangelica*), 10.8 g of dry lemon peel (*Citrus limonum*), 4270 g of 95% ABV GNS, and 687 g of deionized water were weighed and placed into a stainless steel pot and allowed to steep at room temperature (23 °C) for 24 h.

Traditional Still Construction and Distillation Process. A still was constructed with glass purchased from Ace Glass (Vineland, NJ). The set up consisted of one 20 L round-bottom flask; one corresponding top with a 25/35 socket adapter; one glass distillation adapter with 10/30 temperature orifice, a 24 in. arm, and 25/35 ball adapters at each end; and one 24 in. Graham Coil Condenser.

The contents of the pot were added to the 20 L round-bottom flask, and the flask was placed onto a heating mantle (purchased from Ace Glass) and distilled until the temperature of the distillate vapor passing over the inserted thermometer reached 95 °C. This insured that most of the alcohol had distilled at that temperature point.

Novel/Vacuum Still Construction and Distillation Process. The novel still, also known as a “vacuum still”, was designed and assembled by Myers Vacuum (Kittanning, PA). It consisted of one glass 20 L cylinder (Figure 1, 12) with stainless steel plates for a top and bottom (called the gin kettle); one stainless steel 90° elbow (Figure 1, 16), which bolted to the gin kettle; a glass 2 L collecting reservoir (Figure

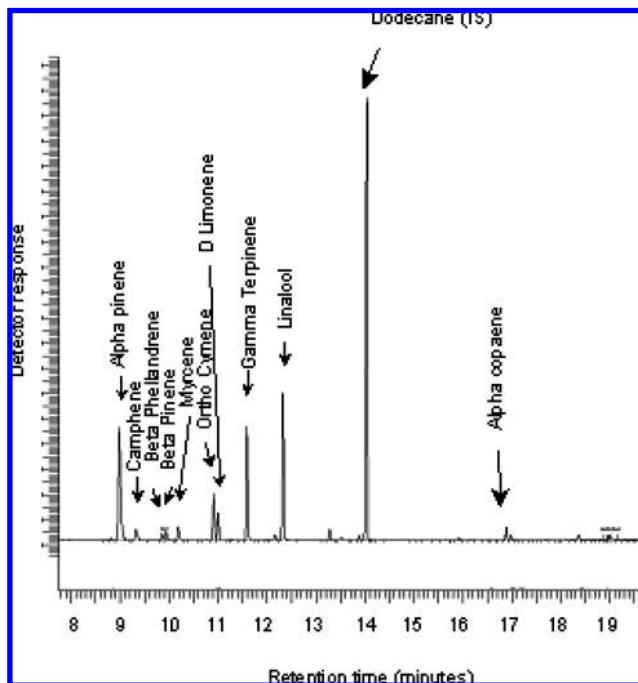


Figure 2. Chromatogram of coriander seeds after distillation on the traditional still.

1, 22) with a stainless steel coldfinger at the top (Figure 1, 23); one vacuum pump capable of pulling a vacuum of 10⁻⁶ mmHg (Figure 1, 24); and a positive displacement pump (Figure 1, 28) with a vacuum check valve.

The contents of the pot were added to the kettle on the vacuum still. The lid was secured, and the vacuum pump was turned “on”. The coldfinger condenser was filled with liquid nitrogen via a dewar (Praxair, Jacksonville, FL). A needle valve (used to throttle the vacuum) attached to the vacuum still was slowly opened to gradually reduce the pressure in the vacuum chamber. The operating conditions of the still were controlled by the amount of liquid nitrogen in the coldfinger and the vacuum needle valve. The distillation continued until the contents of the kettle froze, signifying that the majority of ethanol had been distilled.

The distillate (referred to henceforth as “gin”) was stored in an amber glass bottle with a silicon-sealed cap. All samples were appropriately marked and stored in a corrugated box in a dark freezer at –20 °C until extraction.

Extraction. The alcohol content of the gin was analyzed on an Anton-Paar densitometer and then brought to 40% ABV. Thirty-eight milliliters of the 40% ABV gin was added to an extraction tube. Then, 2 mL of a 50/50 solution (volume/volume) of hexane (CAS #110-54-3) and dichloromethane (CAS #75-09-2) with an internal standard (IS) of 100 ppm dodecane (CAS #112-40-3) was added. All chemicals were obtained from Fisher Scientific (Pittsburgh, PA). Salt (NaCl) was added to the tubes in excess. The tubes were placed in a Glas-Col tube rotating mixer (Fisher Scientific, Pittsburgh, PA; part 099ARD4512), set on 30% of full speed and mixed for 30 min, and allowed to rest and separate for 5 min, and the top layer was pipetted into a 2 mL glass vial with a screw cap and silicon septa.

Gas Chromatography (GC). A Hewlett-Packard 6890 GC with mass spectrometer (MS) (Agilent Technologies, Palo Alto, CA) was used for identification of volatile components. Additional replicate samples were analyzed on a Hewlett-Packard 5890 GC with a flame ionization detector (FID) (Agilent Technologies) and a Kovats alkane index (C5–C30) obtained from Restek (Bellefonte, PA). The analytical conditions were as follows: A 30 m DB5 column with 5 m silicon guard column from Restek (part #10223-124) was used on both gas chromatographs. The internal diameter of the column was 0.25 mm, and the film thickness was 0.25 μm. The injector temperature was 225 °C, the detector temperature was 225 °C, and the flow rate was 4 mL/min with split less injection. The oven temperature profile was 60 °C with 10 min hold time followed by a linear 4 °C/min ramp to 225 °C

Table 1. SAS Output (ANOVA) of α -Pinene Levels and Interactions (A = Juniper, B = Coriander, C = Angelica, D = Lemon, and E = Still Type)

covariance parameter estimates				
cov parm	group	estimate		
time		0		
residual	vacuum	0.006396		
residual	traditional	0.3018		

type 3 tests of fixed effects				
effect	no. DF	den DF	F value	Pr > F
A	1	16.7	590.74	<.0001
B	1	16.7	11.58	0.0035
A × B	1	16.7	2.38	0.1418
C	1	16.7	0.08	0.7811
A × C	1	16.7	0.00	0.9667
B × C	1	16.7	0.00	0.9712
A × B × C	1	16.7	0.00	0.9667
D	1	16.7	0.08	0.7753
A × D	1	16.7	0.03	0.8545
B × D	1	16.7	0.06	0.8110
A × B × D	1	16.7	0.20	0.6631
C × D	1	16.7	2.01	0.1743
A × C × D	1	16.7	2.26	0.1516
B × C × D	1	16.7	2.02	0.1741
A × B × C × D	1	16.7	1.70	0.2106
E	1	16.7	257.97	<.0001
E × A	1	16.7	203.05	<.0001
E × B	1	16.7	6.59	0.0202
E × A × B	1	16.7	0.87	0.3640
E × C	1	16.7	0.01	0.9049
E × A × C	1	16.7	0.00	0.9892
E × B × C	1	16.7	0.07	0.8008
E × A × B × C	1	16.7	0.04	0.8510
E × D	1	16.7	0.34	0.5685
E × A × D	1	16.7	0.29	0.5995
E × B × D	1	16.7	0.16	0.6899
E × A × B × D	1	16.7	0.12	0.7341
E × C × D	1	16.7	4.10	0.0591
E × A × C × D	1	16.7	4.19	0.0568
E × B × C × D	1	16.7	1.10	0.3091
E × A × B × C × D	1	16.7	1.59	0.2246

Table 2. Average Amount of α -Pinene and Average Difference for Vacuum (Novel) Still and Traditional Still^a

α -pinene in botanicals				distillation method comparison		
juniper berries	coriander seed	angelica root	lemon peel	vacuum	traditional	difference
present	absent	absent	absent	0.9390	4.1300	-3.1910
present	absent	absent	present	0.9550	2.9650	-2.0100
present	absent	present	absent	1.0050	3.0045	-1.9995
present	absent	present	present	0.8760	4.2000	-3.3240
present	present	absent	absent	0.9950	4.3600	-3.3650
present	present	absent	present	1.0790	4.4650	-3.3860
present	present	present	absent	1.2450	4.1500	-2.9050
present	present	present	present	1.0225	4.6400	-3.6175

^a Standard errors for vacuum (0.05655), traditional (0.3885), and difference (0.3926).

with a final 30 min hold time at 225 °C. The 6890 MS used Hewlett-Packard ChemStation (Agilent Technologies) software for integration. The 5890 GC used Perkin-Elmer TurboChrom (Perkin-Elmer, Welle-sley, MA) software for integration.

Experimental Design and Aliasing Structure. A 2⁵ factorial experiment was conducted in a partially confounded incomplete block design. Four of the factors examined in this study were ingredients (A = juniper berries, B = coriander seed, C = angelica root, and D = lemon peel). The levels for these factors were the absence or presence of the ingredient. The fifth factor (E) was the type of still (traditional or vacuum). The balanced incomplete block design used time as the

Table 3. SAS Output (ANOVA) of β -Myrcene Levels and Interactions (A = Juniper, B = Coriander, C = Angelica, D = Lemon, and E = Still Type)

covariance parameter estimates				
cov parm	group	estimate		
time		0		
residual	vacuum	0.002658		
residual	traditional	0.01362		

type 3 tests of fixed effects				
effect	no. DF	den DF	F value	Pr > F
A	1	22	901.89	<.0001
B	1	22	23.17	<.0001
A × B	1	22	20.19	0.0002
C	1	22	1.68	0.2087
A × C	1	22	0.73	0.4014
B × C	1	22	0.71	0.4094
A × B × C	1	22	0.26	0.6139
D	1	22	0.05	0.8254
A × D	1	22	0.28	0.6012
B × D	1	22	0.19	0.6684
A × B × D	1	22	0.86	0.3629
C × D	1	22	4.59	0.0434
A × C × D	1	22	3.60	0.0710
B × C × D	1	22	1.79	0.1941
A × B × C × D	1	22	1.17	0.2908
E	1	22	417.70	<.0001
E × A	1	22	407.41	<.0001
E × B	1	22	6.72	0.0166
E × A × B	1	22	5.25	0.0319
E × C	1	22	0.54	0.4697
E × A × C	1	22	0.48	0.4946
E × B × C	1	22	3.73	0.0663
E × A × B × C	1	22	3.66	0.0687
E × D	1	22	0.60	0.4450
E × A × D	1	22	1.32	0.2622
E × B × D	1	22	0.09	0.7687
E × A × B × D	1	22	0.31	0.5838
E × C × D	1	22	4.30	0.0499
E × A × C × D	1	22	4.31	0.0497
E × B × C × D	1	22	0.18	0.6722
E × A × B × C × D	1	22	0.31	0.5862

block factor and was constructed using the four-way interaction of juniper × coriander × angelica × lemon) as the design generator. The replicate used the three-factor interaction coriander × angelica × lemon as the design generator so that all effects could be analyzed. The data were subjected to analysis of variance (ANOVA) using the PROC MIXED procedure from SAS. Tests for heterogeneous variance between the two types of stills, main effects, and interactions were examined using $\alpha = 0.05$.

RESULTS AND DISCUSSION

Comparison of the predistilled and postdistilled model gins (with the four botanicals combined) as well as pre- and postdistillation for each of the four botanicals individually are outlined below. The compounds were identified using a GC/MS with a commercially available library and then run on a GC with FID and alkane indices. These two methods were compared to verify the compound.

Juniper Berries. Juniper berries are the largest contributor to the overall flavor of gin and statistically significant on both stills. The extraction and concentration of compounds found in juniper alone, as well as the degradation products derived from the monoterpenes found in juniper, contribute significantly to the flavor and aroma of gin. However, the composition of monoterpenes can vary greatly with origin. Chatzopoulos and

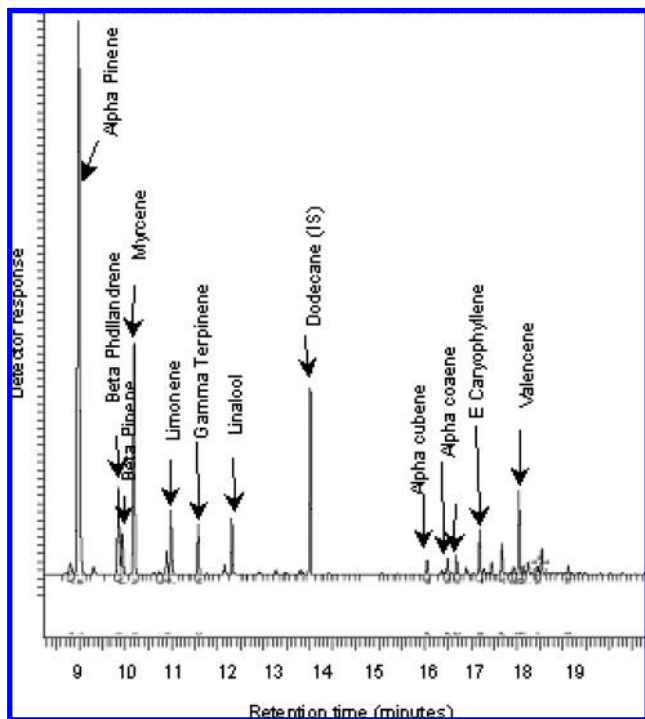


Figure 3. Chromatogram of coriander seeds and juniper berries after distillation on the traditional still.

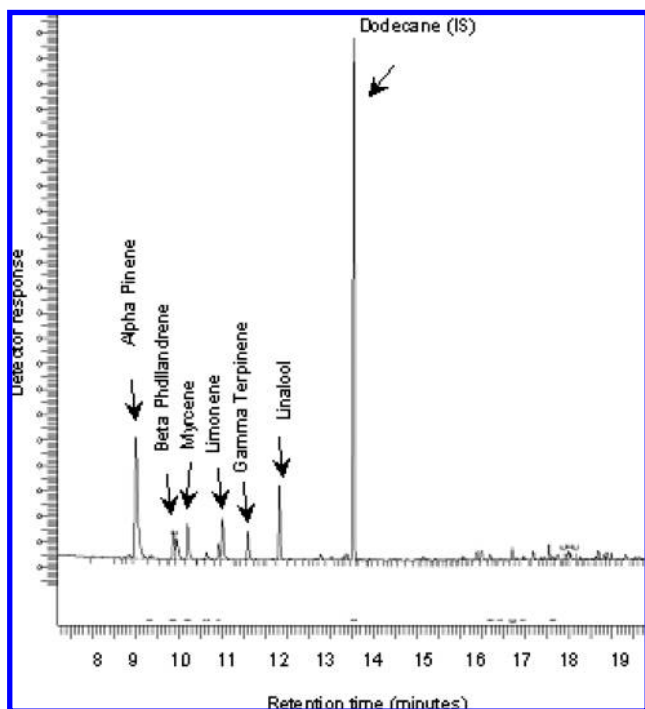


Figure 4. Chromatogram of predistillation (four botanicals) after steeping for 24 h.

Katsiotis (10) found the amounts of monoterpenes at 27, 10, and 10%, respectively. The berries were sourced from the Olympus Mountains in Greece. Ochocka et al. (11) sourced berries from different regions of Poland and found that α -pinene varied from 27 to 80%, depending on the region where they were harvested. The authors also reported a wide range of myrcene (3–42%) and sabinene (0.5–42.5%). When pulling berries from Sardinia, Italy, Angioni et al. (4) found levels of the three compounds at 49, 2.65, and 0.21%, respectively. Clearly, there is wide variation in the levels of chemicals present.

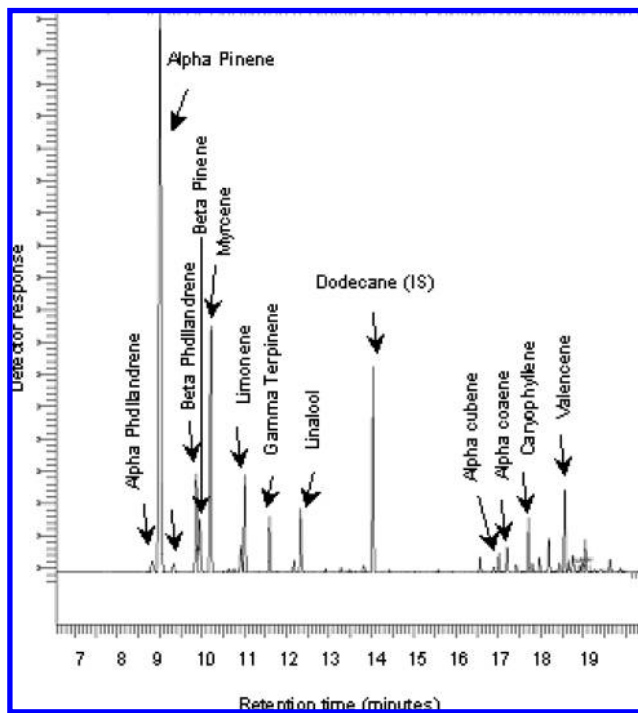


Figure 5. Chromatogram of modelgin (four botanicals) after distillation on a traditional still.

This is to be expected in a natural product exposed to different climates, soil, predation, and other environmental factors that affect composition. In all samples regardless of location, α -pinene was the predominant chemical on a percent basis in *F. juniperi*.

A patent by Dyr and Uher (12) in the Czech republic for the manufacture of gin from steam distillates of juniper berries (*J. communis*) elucidates some of the compounds found in gin that are derived from juniper. The authors explain that steam distilling *J. communis* results in a mixture of α -pinene, camphene, cadinene, junipene, borneol, and terpineol. The residue of *J. communis* is then used to make gin. They also explain that these compounds are responsible for the “pungent taste in the distillate”. All of these compounds are monoterpenes, which is important considering that Nykanen, Nykanen, and Alkio (13) found that juniper berries were approximately 54% monoterpenes and 25% sesquiterpenes.

Villalon et al. (14) studied juniper extracts that are used to make gin and also found that α -pinene, limonene, terpinolene, and α -terpineol were all present in gin made from juniper berries. The generalized composition of the berries includes several monoterpenes; the three most prominent compounds in the juniper oil are α -pinene, myrcene, and sabinene. Examination of juniper berries prior to distillation and after distillation in our research concurs with the Villalon et al. conclusion. The compounds in the predistillation samples had a slight increase in concentration; the largest was β -phellandrene with a 3.2 magnitude increase. α -Pinene and D-limonene had increases that were below the concentration effect due to volume. The increase in monoterpenes is clearly seen when comparing the concentrations of the aroma-active chemicals manufactured in a traditional fashion and one that uses high vacuum and no heat. We found a 1.2 magnitude increase in α -pinene, a 3.2 magnitude increase in β -phellandrene, and a 0.81 magnitude increase in D-limonene and decreases in β -myrcene, *E*-caryophyllene, linalool, and valencene.

In fact, the increases seen in the postdistillation samples from the novel (vacuum) still can be explained by the concentration

Table 4. R_t , Area, and Relative Concentration As Compared to IS for Predistillation and Postdistillation Model Gin (Novel and Traditional Methods)

	predistillation			postdistillation—traditional			postdistillation—vacuum		
	R_t	area	ratio (relative to IS)	R_t	area	ratio (relative to IS)	R_t	area	ratio (relative to IS)
α -phellandrene	8.82	9904.04	0.01	8.83	65968.54	0.10	8.83	16810.09	0.02
α -pinene	9.02	419469.26	0.47	9.03	2959383.00	4.66	8.99	691891.52	1.02
camphene	9.35	8630.00	0.01	9.33	38126.25	0.06	9.33	8531.93	0.01
β -phellandrene	9.88	58438.86	0.07	9.87	394377.17	0.62	9.87	105958.88	0.16
β -pinene	9.96	71186.59	0.08	9.95	234315.79	0.37	9.95	77497.40	0.11
β -myrcene	10.21	86781.21	0.10	10.21	914960.53	1.44	10.20	147930.03	0.22
3-carene	10.64	16603.21	0.02	10.64	11990.61	0.02	10.63	17512.48	0.03
δ -2-carene	10.78	505.28	0.00	10.76	12854.90	0.02	10.76	0.00	0.00
<i>ortho</i> -cyeme	10.93	31863.08	0.04	10.92	91422.05	0.14	10.92	20561.10	0.03
<i>D</i> -limonene	11.00	97854.15	0.11	11.01	337324.03	0.53	11.01	110490.95	0.16
γ -terpinene	11.60	57020.84	0.06	11.60	169060.03	0.27	11.59	14914.81	0.02
(+)-4-carene	12.17	3709.29	0.00	12.17	38457.45	0.06	12.16	7245.81	0.01
linalool	12.36	126843.02	0.14	12.33	177682.11	0.28	12.32	13100.55	0.02
dodecane (IS)	14.04	895279.52	1.00	14.04	635731.61	1.00	14.03	675365.17	1.00
α -cubene	16.56	8784.45	0.01	16.56	46805.10	0.07	16.55	12746.64	0.02
α -copaene	16.98	22399.94	0.03	17.01	62483.01	0.10	16.99	21067.56	0.03
β -elemene	17.19	8120.24	0.01	17.20	70602.26	0.11	17.19	13252.50	0.02
isodene	17.41	3101.31	0.00	17.41	25532.53	0.04	17.40	6443.23	0.01
<i>E</i> -caryophyllene	17.69	17553.55	0.02	17.70	154636.31	0.24	17.69	34467.23	0.05

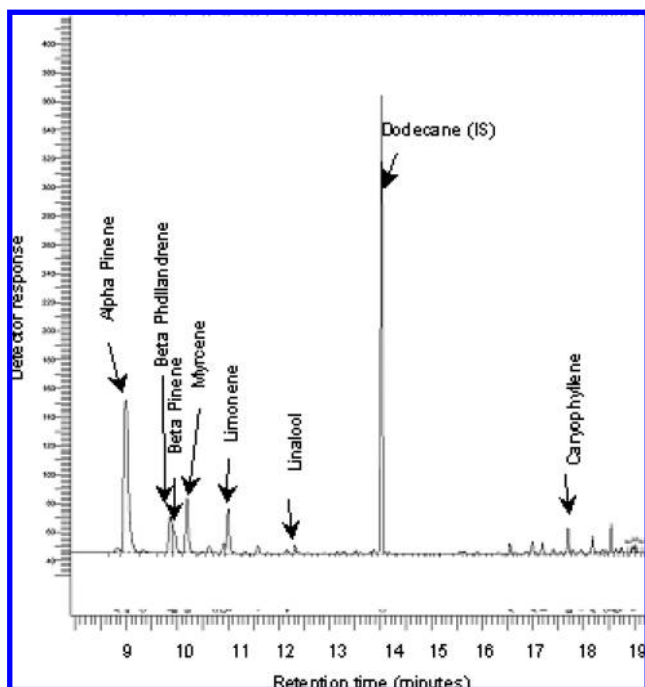
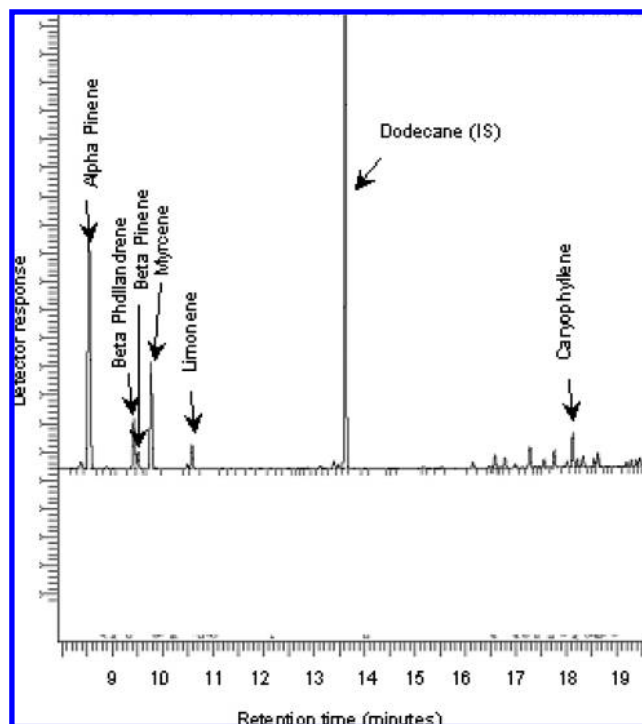
effect due to change in volume (ΔV) from predistillation to postdistillation. The initial volume was 5882 mL at 85% ABV (5000 mL of absolute alcohol). The volume recovered from the novel still after a full run was 3000 mL at 90% ABV (2700 mL of absolute alcohol). This is a 1.96 increase in concentration. The low recovery is due to several factors that include absorption of fluid by the botanicals, material left in the gin kettle (approximately 1000–1500 mL) and “blow-by”, or vaporized alcohol that failed to be recondensed by the coldfinger and was passed out of the system through the vacuum pump.

Other compounds either remained the same or decreased in concentration. The traditional method reflects a general concentration increase in all compounds varying from an 8.0 magnitude increase (seen in α -pinene) to a 1.2 magnitude increase (γ -muurolene).

Juniper Berries and Coriander Seeds. Coriander seeds (which are actually fruits) are normally the largest ingredient

by weight in most gins. Distillation of coriander alone on the traditional still revealed the largest quantity of linalool found in all of the botanicals. **Figure 2** illustrates the significant concentration in γ -terpinene, α -pinene, β -myrcene, and linalool. A substantial amount of the linalool present in the model gin was probably contributed by the coriander seed, since the chemical was low in concentration in all other botanicals, a conclusion supported in the literature by Kerrola et al. (15). Analysis of juniper berries and coriander seeds distilled individually and combined showed the main effects (juniper and coriander) as well as their two factor interaction ($p < 0.05$) to be significant for α -pinene and β -myrcene when juniper is present (**Tables 1–3**). This interaction is reflected by the overall increase in α -pinene and β -myrcene (**Figure 3**).

Traditional Distillation: Pre- and Postdistillation Comparison. Comparison of compounds present in the predistillation

**Figure 6.** Chromatogram model gin (four botanicals) after distillation on a novel (vacuum) still.**Figure 7.** Chromatogram of predistillation of juniper berries after steeping for 24 h.

model gin (**Figure 4**) and postdistillation of the model gin (**Figure 5**) distilled using traditional methods showed that in all 30 trials on the traditional still, the compounds present in postdistillation samples increased in concentration as compared to the predistillation samples. The compound with the greatest increase relative to the IS was δ -2-carene with a 36 magnitude increase; however, its overall concentration remained relatively small (**Table 4**). The compounds with the largest increases and greater impact in the model gin produced using traditional methods were α -pinene, α -phellandrene, *E*-caryophyllene, β -myrcene, and valencene, all with a 10-magnitude increase from predistillation samples. Camphene showed a 6-magnitude increase in concentration, while terpinene and D-limonene showed a 4-fold increase in magnitude. Linalool doubled in concentration.

Novel/Vacuum Still Pre- and Postdistillation Comparison.

Comparing the predistillation chromatogram of the model gin (**Figure 4**) with the postdistillation chromatogram using the vacuum still (**Figure 6**), it is evident from observation that the majority of botanicals were concentrated by vacuum distillation (**Table 4**). α -Phellandrene, α -pinene, *E*-caryophyllene, and β -myrcene showed a two magnitude increase vs predistillation samples. α -Copaene had a 1.4 increase in magnitude, while valencene showed no change at all in concentration. Terpinene and linalool (in post distillation samples) both showed an overall decrease in concentration as compared to predistillation samples. The overall chromatogram of the model gin produced using the novel method (**Figure 6**) is similar to the chromatogram of juniper berries distilled alone on the novel still (**Figure 7**). Earlier work has shown that juniper is a significant contributor to the aroma-active chemicals in gin (*16*).

Novel/Vacuum and Traditional Gin Distillation Comparison. Overall botanical aromatic compounds extracted in the largest concentrations using the vacuum still were α -phellandrene, α -pinene, *E*-caryophyllene, and β -myrcene with a 2-fold increase. The difference between the traditional method and the vacuum method on the concentration of α -pinene, α -phellandrene, *E*-caryophyllene, and β -myrcene cannot be overstated. The traditional method of distillation (applied heat) had a 5-magnitude greater concentration of these four monoterpenes (**Table 4**). Bauba and Kaula (*17*) found similar results when they distilled essential oils both in vacuum and under conventional methods, which had lower and higher levels of monoterpenes, respectively. The literature has shown that monoterpenes generation is stimulated by the application of heat (*18*). The process by which they are generated is theorized to be the mevalonic pathway (*19*). An intermediary of isopentenyl pyrophosphate is generated via the mevalonic pathway in which mevalonic acid is phosphorylated to generate isopentenyl pyrophosphate. A racemic mixture of isopentenyl pyrophosphate and $\gamma\gamma$ -dimethylallyl pyrophosphate occurs. Both isomers are partially electrophilic and partial nucleophilic (*20*). This allows for them to join forming an isoprene intermediary, geranyl pyrophosphate. Geranyl pyrophosphate can readily undergo dimerization (*20*) to become many different monoterpenes. The mevalonic pathway is influenced by temperature; thus, an increase in monoterpenes, like α -pinene and camphene, is proportional to the amount of heat in the system.

This is further illustrated in looking at the concentrations of α -pinene, camphene, β -pinene, and terpinolene, which were all found in gin samples distilled solely from juniper berries (*16*). Comparison of **Figures 4** (predistillation gin novel/vacuum after steeping) and **6** (postdistillation gin novel/vacuum) shows very little difference in the number of overall peaks.

A likelihood ratio test for heterogeneous variances for two types revealed that the variances were different (<0.0001) for α -pinene and β -myrcene. For α -pinene, the variance from the vacuum still (0.006396) was less than the variance from the traditional still (0.3018). For β -myrcene, the variance from the vacuum still (0.002658) was less than the variance from the traditional still (0.01362). Therefore, the ANOVA incorporated different variance components for the two types of stills. If juniper berries are not added, there is no difference in the average amount of α -pinene and β -myrcene for the vacuum and traditional stills. If juniper berries are added (which is required for gin), then the average difference in the amount of α -pinene and β -myrcene between the vacuum and the traditional stills depends on the other ingredients. However, regardless of the combination of ingredients, the average amount of α -pinene and β -myrcene for the vacuum still is always smaller than for the traditional still.

The distillation of botanical extracts for the manufacture of gin using the novel (high vacuum) still at reduced temperatures greatly reduces the amount of monoterpenes present in the finished gin. Greer et al. (*21*) found that a trained panel, in a blind test, was able to distinguish the vacuum-distilled gin as a product with "less nasal pungency, more floral, less spicy aroma". This was further confirmed in Bauba and Kaula (*17*) who found that essential oil distilled in vacuum had a more pleasant aroma as compared with oil distilled under traditional conditions. We further confirmed that α -pinene and β -myrcene had a statistically significant two-factor interaction with the traditional still and not with the novel still.

The vacuum distillation process produces a gin with much less monoterpenes present. These monoterpenes can be precursors to off-flavors in finished gins. These findings demonstrate the importance of avoiding excess heat during the manufacture of "super-premium" gins that contain the natural flavor profile of the botanicals with minimal off-flavors. Further process details as well as other specific applications for this still/process (rum, vodka, etc.) are outlined in our 2006 U.S. Patent filing (*21*). The industrial scale up of this technology required overcoming some technical challenges, most notably, what to use as the condenser refrigerant. Use of LN2 commercially would be cost prohibitive. These technical challenges have been resolved, and proof of concept testing on a commercial scale is ongoing.

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