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**FEASIBILITY TESTING OF CHILL FILTRATION OF BROWN SPIRITS TO
INCREASE PRODUCT STABILITY**

By

Juan Cristobal Merizalde Carrillo
B.S. ChE., University of Louisville, May 2009

A Thesis
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for the Professional Degree

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Department of Chemical Engineering

May 2015

**FEASIBILITY TESTING OF CHILL FILTRATION OF BROWN SPIRITS TO
INCREASE PRODUCT STABILITY**

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A Thesis Approved On

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ABSTRACT

This thesis summarizes a feasibility study focused on filtering American whiskey, matured in new toasted and charred casks, at 25 °F in order to remove haze which forms when product is diluted from cask strength (62% ABV) to bottling strength (40 – 50% ABV). Chill filtration is a process already implemented in the brown spirits industry in order to guarantee the stability of products at different ethyl alcohol concentrations. The filtration trials and cellulose-pad performance were evaluated in terms of economics, turbidity reduction, test time, throughput, color reduction, and shelf life stability. Those trials with satisfactory overall color and stability results were tested to determine the best performing filtration system.

Chill filtration showed better product stability when compared with the current filtration system (consisting of carbon treatment and overnight hold). Two filtration pads were initially tested: a cellulose/diatomaceous earth (DE) pad and a cellulose-only pad. Although the cellulose/DE pad provided excellent stability results, it exhibited low product throughput and excessive color removal. The cellulose-only pads provided overall acceptable product stability, low color removal, and adequate throughput.

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I. INTRODUCTION

Chill filtration is a standard operation of the Scotch Whiskey production process. However, its implementation for American brown spirits is relatively new. Each spirits type and brand needs to be explored separately considering the distinction of flavor and aroma complexity of each product. The purpose of the present research was to demonstrate the feasibility of chill filtration of a specific brown spirit to increase the product stability. The author was contracted by Brown Forman Corporation to perform this study with the understanding that the results would be used as the basis of his MEng thesis. All work was conducted under a confidentiality agreement between Brown Forman, the University of Louisville, the author, and his thesis director. Because of the proprietary nature of the components studied, this report refers to the product as “whiskey A”. In a similar manner, filter membranes are referenced only by their generic properties and compositions.

The stability of whiskey A at different bottling strengths is a critical step in the company’s main production process since a stable final product is required to maintain that product’s reputation. Currently this product achieves stability by overnight treatment with activated carbon; however, this process does not provide consistent stability and reduces production flexibility. The filtration processing also represents a significant usage of scarce real estate because of the equipment’s large footprint.

The objective of this project is to develop and implement a chill filtration system to add flexibility for future growth and increase product stability while maintaining other quality criteria within specification.

This work focuses on the use of cellulose and diatomaceous earth filter membranes that are readily available on the market for the filtration of whiskey A. The work included designing and setting up an experimental testing unit. The discussion that follows includes a review of the theory associated with whiskey stability, a description of the experimental apparatus and procedure used in the feasibility study, and conclusion that may be drawn from this work.

This thesis also gives a review of relevant literature including a general review of filter membrane selection. Experimental equipment and procedure are then discussed. Finally, results are discussed and appropriate conclusions are presented. Data and calculation are found in the attached appendixes.

II. BACKGROUND

A. Wood and Distilled Spirits

Distilled spirits are all alcoholic beverages (such as brandy, whiskey, or rum). The high concentration of ethyl alcohol is obtained by distilling fermented mixtures that are usually obtained from fermentable fruits or from a starchy material (such as various grains) that have been previously brewed. Usually the freshly distilled spirits are colorless and harsh in taste possessing a strong alcoholic flavor. If these products are allowed to mature (age) by storing them in sealed wood containers (barrels), the liquid gains a yellow to crisp dark-brown color, its taste becomes smooth, and the sensory notes (flavor and aroma) become complex and pleasing.

While allowing distilled spirits to age, biological reactions do not occur due to the high concentration of alcohol. On the other hand, chemical reactions are the major path for changes in the barrel and also some physical changes such as evaporation. The most commonly used type of wood for barrels is oak because of its natural sealing properties but the role of the wood during the maturation process can be widely extended by using other types of wood (maple) or by using fire-charred barrels.

1. *Bourbon Whiskey*

Bourbon's legal definition varies somewhat from country to country, but according to the US Federal Standards of Identity for Distilled Spirits, "a bourbon must be produced in the

United States, made from a grain mixture that is at least 51% corn, aged in new, charred-oak barrels, distilled to no more than 80% alcohol by volume (160 proof), entered into the barrel for aging at no more than 62.5% alcohol by volume (125 proof), bottled at 40% alcohol by volume (80 proof) or more, and state its age of maturation if it was in the barrel for less than four years” (ECFR, 2013).

2. *American White Oak*

Brown spirits, specifically whiskeys, are matured in charred American white oak barrels. According to “*Chemistry of Winemaking*” by Singleton, the particular features of the oak wood species that make them suitable for this process are their hardness, dimensional stability, relative impermeability, rays (the radial ribbons extending vertically through the tree), and tyloses (response from decay in heartwood). The relative impermeability and porosity of American white oak allows fluid to be retained in the cask. The movement of fluid is given by the vessels that are basically porous vertical elements (Parham and Gray, 1984). The wood matrix makes challenging for liquid to move from the inside to the outside of the cask. In regards to the structure, clearly the composition of oak is somewhat variable. It is important to divide the chemical constituents of oak wood into two major groups: cell wall components and extractives.

TABLE II-I presents the cell wall components of heartwood American white oak typically used for cooperage. (Singleton, 1974). The high lignin and tannin concentrations contribute to the flavor properties of alcoholic beverages (Maga, 2009). Maturation of spirits comes about from three different processes: some evaporation of alcohol and water through the sides of the wooden barrel, introduction of oxygen into the container, and extraction of substances from the wood into the beverage.

A simplified explanation is that cellulose forms the basic skeletal structure, with hemicelluloses serving as a matrix. Lignin acts as an encrusting material, permeating the cell walls and intercellular regions, allowing the wood to be rigid and able to withstand mechanical stress.

TABLE II-I
COMPOSITION OF HEARTWOOD AMERICAN WHITE OAK
(Singleton, 1974)

Material / Substance	Percentage, %
Cellulose	50
Hemicellulose	22
Lignin	32
Acetyl Groups	2.8
Hot-water extractables	5 – 10

Other substances are not integral parts of the cell structure and therefore are easily removed by solvents without affecting the physical properties and strength of the wood. H. Brown states in the *Textbook of Wood Technology*, that wood contains a wide range of extractable substances including volatile oils, fats, resins, tannins, carbohydrates, sterols, and inorganic salts, particularly calcium oxalate. It is these compounds that impart characteristic odors (Brown, et. al., 1949). The group of extractable compounds of particular importance in oak wood are the tannins, and their importance to matured flavor cannot be underestimated.

Tannins are polyphenols and are classified as either condensed or hydrolyzable (Brown, et. al., 1949). The hydrolyzable tannins are esters of a sugar, usually glucose, with one or more polyphenolic moieties. These two types of hydrolyzable tannins have been found in cooperage

oak but their distribution is extremely variable; this means that extractable tannins may vary greatly in casks or barrels from similar sources. As it is stated in *Chemistry of Winemaking*:

“Tannins are present in the wood rays and are therefore fairly accessible to maturing spirit; they have germicidal properties, contribute to the coloring of the wood, and are considered to protect the tree from attack or decay” (Singleton, 1974).

Other studies presented by C. Chen in 1970, show that heartwood American oak contains a number of sterols that can be related to whiskey stability. Chen identified β -sitosterol, stigmasterol, campesterol, and traces of β -dihydrosterol (See FIGURE 2.1).

FIGURE 2.1. Atomic Composition of: (a) Stigmasterol, and (b) β -Sitosterol.
(Maga, 2009)

Despite their low levels, their presence can cause a permanent floc or haziness in bottled whiskies. A problem can occur when American whiskey that has been stored in a cask or barrel is diluted with water to achieve common bottling strengths. These organic sterols are not soluble in water though they are soluble in most organic solvents since, as seen on FIGURE 2.1, they contain one alcohol functional group.

Other compounds that can cause floc or haziness in bottled whiskies are long chain ethyl esters such as ethyl palmitate and ethyl myristate. These esters are common to most whiskies but not all whiskies have the same concentration. These fatty esters behave as surfactants because

they have long carbon chains that are hydrophobic that prevent them from mixing with water as well as a hydrophilic group (See FIGURE 2.2).

FIGURE 2.2. Atomic Composition of: (a) Ethyl Palmitate, and (b) Ethyl Myristate.
(Sigma-Aldrich, 2015)

Under non-mixing conditions these fatty esters behave as micelles. Micelles are spherical clumps of lipid molecules where the hydrophobic carbon tail points toward the center, away from the water (See FIGURE 2.3). Their contribution to cloudiness makes the reduction of these compounds a factor to take into consideration in the search of stability.

FIGURE 2.3. Spherical Micelle Structure Layout.
(Rusano – Glossary of Nanotechnology and Related Terms, 2011)

B. Maturation of Bourbon

The changes taking places in maturing bourbon involve the extraction of wood components from the barrel and interaction of ethanol from the unaged distillate and the barrel wood. The distillate is usually stored at high proofs or high alcohol content since the organic compounds in the oak wood have more affinity for ethanol than for water. Parham and Gray (1984) state that optimum extraction strength is 110 proof and that tannins account for around two-thirds of the extract, with lignin making up most of the remainder.

Table II-II indicates the results of Gas Chromatography (GC) and High Pressure Liquid Chromatography (HPLC) analysis of a sample of standard American whiskey A presented by Brown-Forman Process, Research, and Development Laboratories. The results clearly show that the whiskey extracted volatile aroma compounds such as hydrocarbons, acids, esters, phenols, alcohols, and terpenes.

TABLE II-II

**HIGH PERFORMANCE LIQUID CHROMOTOGRAPHY (HPLC) AND GAS
CHROMATOGRAPHY OF STANDARD AMERICAN WHISKEY A**
(Brown Forman Laboratory Database, 2008)

GC Compound	C (ppm)	HPLC Compound	C (ppm)
Ethyl Propionate	1.3	Glucose	52.9
Acetal	19.8	Fructose	173.8
n-Pentyl Alcohol	0.8	Arabinose	59.2
Isobutyl Acetate	2.0	Acetic Acid	253.2
Ethyl Butyrate	1.0	Gallic Acid	6.4
Ethyl Lactate	2.9	HMF	7.2
n-Hexyl Alcohol	1.6	Furfural	17.9
Isoamyl Acetate	9.7	Syringic Acid	4.9
5-Methyl-Furfural	2.3	Vanillin	5.2
Ethyl Caproate	1.1	Syringaldehyde	16.0
n-Octanol	0.6	Coniferaldehyde	10.6
Phenethyl Alcohol	19.9	Ellagic Acid	14.9
Trans-2-Nonenal	0.4	Maltose	0.0
Diethyl Succinate	0.3	Sucrose	0.0
Ethyl Caprylate	1.9		
Phenethyl Acetate	1.8		
Oak Lactone	5.3		
Eugenol	2.1		
Ethyl Caprate	2.1		
Ethyl Laurate	1.4		
Ethyl Myristate	2.3		
Ethyl Palmitate	6.3		

It is important to mention that charred-oak barrels contain much higher amounts of aromatic aldehydes than uncharred ones. According to the studies presented by K. Nishimura in

1983, heating wood appears to break down lignin and facilitate aromatic aldehyde formation (See TABLE II-III).

TABLE II-III
AROMATIC ALDEHYDE CONTENT (PPM) OF CHARRED AND UNCHARRED OAK
CHIPS IN 60% ETHANOL
(Nishimura, 1983)

Compound	Charred	Uncharred
Vanillin	6.25	0.14
Acetovanillone	0.37	0.02
Syringaldehyde	12.40	0.27
Acetosyringone	0.66	-
Coniferaldehyde	8.40	-
Vanillic Acid	2.63	0.35
Sinapaldehyde	5.40	0.04

On the other hand, matured bourbons contain sugars although their concentrations are too low to impart any significant sweetening effect. Also oak lactone (β -methyl- γ -octalactone) characterizes matured bourbons. This compound has a coconutlike aroma and can contribute to a mature flavor according to sensory panels.

Besides the chemistry involved in the maturation process, the influence of warehousing conditions on alcohol losses and flavor production could affect the sensory profile of the spirit. During the initial weeks after filling into barrels there is a settling-in period during which alcohol and water may be lost from the bulk within the barrel in variable amounts while the inner surface of the wood is permeated and leaks sealed. Alcohol and water will continue diffusing through the barrel staves and evaporate from the wood surface over the course of maturation. This being the

case, environmental features within the warehouse such as temperature, humidity, and ventilation are related to the relative losses of alcohol and water and to flavor development.

Taking into account liquid-vapor equilibrium and the physical properties of alcohol and water, if warehouse humidity is low, the rate of loss of water is greater than that of alcohol, whereas diffusion of water is depressed when humidity is high and the alcoholic strength of the contents of the barrel steadily declines with age. Nevertheless, there is an overall loss of alcohol in all locations during maturation. According to professional experience by Brown-Forman Engineer Joe Zimlich and Chemist Charles Geisler, an average of 3% per year of alcohol at casking is lost in barrels (Private Communication, 2008).

As mentioned, the temperature of maturation would be expected to influence the extractions already discussed. Swan cites Reazin in a study presented in 1983:

“Reazin studied the effect of average temperatures ranging from 18.1°C to 23°C upon flavor component development. In this study acetaldehyde and nonvolatile acids increased by up to 48%, being the greatest difference found, while oak lactone and furfural did not appear to change at all. The majority of components increased at 4% per degree Celsius. Reazin pointed out, however, that although the reactions could be increased with temperature there does exist an optimum temperature to produce the desired product quality. it should also be noted that the changing alcoholic strength will influence the time in the maturation cycle at which the optimum reaction rates relative to the alcoholic strength will occur” (Swan, 1986).

Up to this point it has been shown that the maturation of bourbon whiskey in oak wood is extremely complex, and the practice varies from one spirit-producing region to another as well as with the humidity, temperature, and ventilation conditions. It has also been stated that organic compounds such as stigmasterol, β -sitosterol, and others have been proven to alter the stability of final bottled bourbon.

C. Filtration of Matured Bourbon

Filtration is the removal of suspended particles from a fluid by a filter medium. The objectives for filtration operations fall into one of the following: clarification for liquid purification, separation for solid recovery, separation for both liquid and solid recovery, or separation aimed at facilitating or improving other plant operations. In the food and beverage industry, clarifying filtration is often performed in equipment containing deep packed beds, membrane or cake filtration. Stability of the final product relies on the clarification of spirits by removing relatively small amounts of suspended solids from suspension.

Spirits filtration presents many challenging tasks in beverage filtration. The filtered final product must be free from visible particles and have a clear and bright appearance. Whiskeys are filtered to remove haze which forms on dilution from cask strength (110 to 130 proof) to bottling strength (80 – 100 proof). In the industry, whiskeys are treated with granulated activated carbon and filtered after a given carbon retention time or hold time.

1. Chill Filtration as a Solution to Haze Formation

Chill filtration is a commercial process performed by the majority of spirits producers to ensure the quality of their products. During this process, a haze forms in the beverage (whiskey, sherry, brandy, cognac, rum, certain white and red wines, liquors and cordials) at temperatures below about 45° F (Ichem Food and Drink Subject Group, 2000). Haze precursors apparently cause the "haze" or “cloudiness” to form a separate phase which can be then removed using standard techniques such as filtration (Wisniewski, 2009). In many cases, this chilling treatment is not completely effective and several chilling and precipitation treatments may prove necessary.

As stated before, the experiments completed by Reazin (1983) show that temperature changes directly have an effect on the concentration of aldehydes and esters. Research results and studies presented by the Process, Research, and Development Department of Brown-Forman have also shown that chill filtration removes polyphenols such as esters and aldehydes which account for the aromatic characteristics of whiskey. The GC and HPLC analysis for these experiments are held confidential by Brown-Forman and are not presented. The summary of results in TABLE II-IV clearly shows that chill filtration is a promising process for haze reduction since the chill filtration process reduces turbidity to much less than that achieved in the standard process.

TABLE II-IV
ANALYTICAL RESULTS OF UNFILTERED WHISKEY VERSUS WHISKEY
FILTERED UNDER CHILL FILTRATION CONDITIONS AND CURRENT IN-PLANT
PROCESS
(Brown Forman Laboratory Database, 2008)

	Proof	Color	Ambient	Chill	Optek	Optek 11
				4hr	90	
	°	LB	NTU	NTU	NTU	ppm
Untreated Whis. A	79.5	12	2.5 – 1.5	> 55	2.0	0.905
Chill Trial #1	79.96	9.11	0.269	2.74	0.207	0.176
Chill Trial #2	79.87	9.32	0.235	2.13	0.130	0.161
Chill Trial #3	79.97	9.16	0.203	2.01	0.133	0.159
Standard Process	79.4	9.40	< 1.000	< 15.0	< 0.800	< 0.300

Where:

Proof = A measure of how much alcohol is contained in the beverage. It is defined as twice the percentage of alcohol by volume (% abv).

Color = The light absorbance of the sample, as measured by spectrometer, is correlated to a Lovibond scale (LB) in order to characterize the color of the whiskey. The spectrometer reading must be taken at 430nm for whiskey analysis considering good whiskey color is reached at that wavelength peak.

Ambient = A measurement of cloudiness or haziness of the fluid caused by individual particles, measured using a Nephelometer. The units are Nephelometric Turbidity Units (NTU). Ambient and Optek 90 take this reading at ambient temperature; Chill 4hr is a measurement of the same property at -5°C.

Optek 11 = A reading equivalent to that given by Optek 90 at room temperature, but expressed in concentration units (ppm); both readings are taken using the same piece of equipment using different procedures.

Examination of the data presented in Table II-IV clearly shows that the analytical quality control results obtained from a chill filtration process are satisfactory. Obtaining Chill 4hr results less than 15 NTU guarantees a clean and clear product with no floc or haze due to changes in temperature. This separation process allows the production of a fine whiskey but with slight changes in its sensory profile.

From a thermodynamics perspective, the chilling process clearly changes the solid-liquid equilibrium of the mixture. The solubility of a given solute (in this case the organic compounds) in a given solvent (here, ethanol/water) typically depends on temperature (Prausnitz, J., Lichtenthaler, R., and Azavedo, E., 1999). Thus, in a 40% ethanol/ 60% water system (80 proof product), the organic compounds are nearly always more soluble at higher temperatures than at lower ones. If the temperature of the 40% ethanol/ 60% water system decreases, the polarity of the solvent becomes too high and the organic compounds become barely soluble, instead forming

clusters that separate themselves from the solution. A mixture at these conditions can therefore be easily separated by filtration.

While chill filtration produces a clear product it has the disadvantages of significant energy consumption in reducing the temperature of the liquid, and slight modifications to the sensory profile of the product. The benefits of a more economical filtration system that yields similar or better results with less processing time could be quite appealing to the industry.

Background supports the stability of the product, but the following pages will allow to determine what filtration material is more feasible to optimize this process.

III. INSTRUMENTATION AND EQUIPMENT

Chill filtration trials were performed using a lab scale unit designed and set up at the Brown Forman Processing Research and Development laboratory in Louisville, KY. All the trials were run using the same system with no major modifications required between runs. Figure 3.1 shows a schematic of the experimental chill filtration system.

This system consists of a 3 liter feed tank containing unfiltered whiskey from the plant, mixed with a predetermined dose of activated carbon, a pump to displace the liquid, stainless steel coil line inside the chill liquid connected to the filter unit, a chill unit, a temperature transmitter, and a product receiving flask. Table III-I is a complete list of the equipment used.

TABLE III-I
EQUIPMENT LIST

1. Stirring plate:	Corning PC-210 Magnetic Stirrer Ceramic Top
2. Flexible tubing:	Masterflex® PFA Teflon tubing ¼" ID
3. Feeding flask:	500 mL PYREX® Erlenmeyer flask
4. Receiving flask:	100mL PYREX® Erlenmeyer flask
5. Whiskey pump:	Cole-Parmer® Variable-flow reversible pump 115VAC
6. Whiskey Filter:	Ertel Alsop® Vertical Filter housing 2" diameter
7. Cooling Coils:	Stainless steel 316 tubing
8. Chill Unit:	Cole-Parmer® Polystat Recirculator 500W (-10°C/80 °C)
9. Chiller Liquid:	70/30 solution Propylene Glycol/Water
10. Temperature meter:	Oakton Acorn Thermocouple Thermometer (T probe)
11. Pressure gauge:	Liquid-filled gauge, plastic case, ¼" NPT bottom
12. Valves (V-#):	Parker® two-way SS straight ball valves, 1/8" NPT
13. Agitator:	Stand-mount variable speed electric mixer, 1/40 HP
14. Spectrometer:	Thermo Genesys® 10UV Vis Spectrophotometer
15. Density meter:	Anton Paar® DMA 4500 M
16. Turbidity meter:	Hach® 2100AN Laboratory Turbidimeter, EPA, 115VAC

FIGURE 3.1. Chill Filtration System Schematic

IV. PROCEDURE

The experimental procedure has been divided into sections as a standard operating procedure (SOP) to perform the chill filtration trials was developed. Due attention must be paid to safe operating conditions at all times. The steps in the SOP are as follows:

A. Safety Key Points

1. Wear the proper personal protective equipment (PPE) at all times: lab coat and safety glasses.
2. Ensure the working area is clean at all times. The liquids used in this experiment are flammable.

B. Filter Mounting

1. This filter has three main parts: bottom flange made of steel, middle piece made of glass, and top flange made of steel.
2. Ensure that the filter housing is clean and free of dirt and has no residue remaining from previous runs.
3. The housing accepts 2 inch diameter pads. Use only a new purchased or cut pad that has the correct diameter.
4. Place the porous sintered stainless steel support in the filter housing flange.

5. Place the filter pad on top of the stainless screen support.
6. Place the sealing gasket on top of the pad ensuring it is centered in the device.
7. Place the center piece of the filter (sight glass) on top of the sealing gasket. Make sure the piece is centered and aligned with the gasket.
8. Set the top filter housing flange, topside down on the working-bench and place the top sealing gasket on it. Make sure the gasket is properly sealed to the flange.
9. Place the top flange, with the gasket facing down, on top of the glass sitting on the bottom flange support ensuring proper alignment of the bolt fittings.
10. Insert the three securing bolts through the filter fittings and finger tighten them. Then use an Allen wrench to evenly tighten all bolts. Do not over-tighten as this could cause sealing issues.

C. Equipment Drying

The flow lines must then be dried to remove any residual water that could affect the alcohol strength in the sample.

1. Connect all clean hoses together to the lab air inlet source. Dry with lab air for 5 minutes.
2. Connect the clean metal coil to the lab air inlet source and air dry for 5 minutes.
3. Verify that all fittings, pump, valves, and flasks are clean and dried and dry further with lab air if needed.

D. Equipment preparation

1. Place the holding flask on top of the stirring plate. Carefully, place a stirring bar in the flask. Turn on the stirring plate to verify functionality. Turn off after being tested.
2. Place one end of the hose in the holding flask and connect the other to the inlet of the pump.
3. Using another hose, connect the outlet of the pump to the metal coils inlet.
4. Connect the other end of the metal coils to the inlet of the filter housing.
5. Connect the PG-1 straight into the filter unit.
6. Connect valve V-1 to the inlet of the filter housing.
7. Connect the outlet of the filter housing to the 3-way fitting that holds pressure gauge PG-2 and valve V-2.
8. Connect outlet of valve V-2 to the in-line temperature probe TP-1.
9. Extend a hose connection from the temperature probe TP-1 fitting to the inlet of the receiving flask.
10. Ensure the operation of chill liquid agitator. Turn off after test.
11. Place the connected coils and the filter housing inside the chiller tank.

E. Whiskey Preparation

In order to avoid extended holding times of whiskey with activated carbon, it was decided to prepare several smaller samples instead of one large batch.

1. Take a 200mL sample of the preliminary liquid and run the following test:
 - a. Initial Turbidity
 - b. Initial Color

- c. Initial proof
- 2. Pour 500 mL of whiskey into the holding flask.
- 3. Weigh the following amounts by the given ratios for the 500mL whiskey sample:
 - a. 1oz/100proof gallons (PG) of activated carbon
 - b. 8oz/100PG of bodyfeed (diatomaceous earth)

F. Chill Filtration Test

1. Turn on chiller unit and set the temperature to 25°F. Wait until temperature gauge TG-1 displays the desired temperature.
2. Turn on chiller agitator.
3. Place the 500mL flask of whiskey on the stirring plate and begin agitating it.
4. Pour the measured amount of activated carbon into the 500mL whiskey sample.
Immediately after, pour the measured amount of bodyfeed into the same sample flask.
Stir for 1 minute.
5. Verify that valves V-1 and V-2 are open.
6. Start pumping the whiskey from the 500mL flasks through the chill system and keep agitation of the liquid in this sample flask.
 - a. Target flow rate is 21mL/min.
7. Collect 100mL samples of filtrate during the filtration process.
8. For every sample, record on the lab notebook:
 - a. Whiskey outlet filtration temperature (target 25 °F)
 - b. Whiskey Flow rate (mL/min)
 - c. Filtration time (minutes, seconds)

9. After collecting the fourth sample, prepare another 500mL flask of whiskey and add the required amounts of activated carbon and bodyfeed. Right after the first flask has been filtered, start the next flask.
10. Continue the filtration process under constant flow conditions until the inlet filter pressure reaches 45 psig. After this pressure is reached, change operations to constant pressure filtration mode by decreasing the feed flow rate in order to keep a constant 45 psig pressure on the filter.
11. Continue the filtration process, replacing the feed flask as needed, until it takes more than 10 minutes to collect a 100 mL sample of filtrate.

G. Shut Down and Waste Disposal

1. When it is determined that the filtration run has ended, stop the peristaltic pump.
2. Turn off chiller unit, chill agitator, and stir plate.
3. Remove the metal coils and the filter from the chiller unit.
4. Slowly remove the tubing from the filter unit to release the pressure in the system.
Collect all liquid in the container.
5. After pressure has been released completely, start removing all housing fittings.
6. Drain all hoses and systems into a plastic waste container.
7. Take the filter housing apart and properly dispose of the filter pad.
8. After draining the system, collect all liquid (mostly whiskey) and pour it into a container appropriate for disposal of waste flammable liquids.
9. Take all system parts to the laboratory drain and rinse thoroughly. If possible connect the water hose and rinse each part for at least 30 seconds.

H. Sample Analysis

1. Pour the filtrate samples into 100mL bottles to prevent evaporation. Allow them to reach room temperature.
2. Samples are then analyzed for:
 - a. Ambient turbidity
 - b. Color
 - c. Alcohol concentration using a density meter
 - d. HPLC

V. RESULTS AND DISCUSSION OF RESULTS

A. Preliminary Screening

Chill filtration may be described as the process whereby a brown spirit (whiskey) is filtered at temperatures ranging from 15°F to 40°F to remove its haze potential at bottling strength. Chill filtration prevents potential hazing formation if the final product is stored at a low temperature prior to or after purchase by the consumer. The current research examines the impact of each individual filtration parameters (filtration temperature, holding time, filter medium, etc.) to identify how each one affects the removal of haze components to ensure the product remains stable. Finding the optimum filtration conditions for each specific product is highly beneficial to the industry because it could cut production cost while maintaining or improving product quality.

The Louisville Brown-Forman plant currently uses an over-night carbon treatment filtration process with a specific filtration membrane. Extensive internal preliminary testing, using one strength of whiskey, determined the optimum temperature and holding time for transfer of this product to a chill filtration process, the need to continue using activated carbon, and the holding time at low temperatures prior to filtration. From the results of that research it was determined to perform an instantaneous chill filtration at 25°F with no holding time for temperature equilibration and carbon treatment reduction. From the preliminary research it was determined that the filtration area needed to be increased from the current system to raise the amount of liquid per unit of surface area or flux ratio.

With an activated carbon treatment, filtration temperature, and no holding time now defined, it was necessary to identify suitable cellulose and DE filter pads as possible candidates for the filtration medium. This study examined the characteristics of two potential membrane candidates including cost, permeability, nominal retention, ash content, extractable minerals, recommended maximum flow rate, and recommended maximum differential pressure. Several pads were tested but only two candidates remained promising: pad A and pad B, described in the next section.

B. Feasibility Testing of Chill Filtration

1. Filter Pads Tested

The properties of the filter medium tested following the preliminary screening are shown in Table V-I. These membranes were commercially available at the time of this work and are commonly used in the beer, distilled spirits, gelatin, juice, and wine industries. Both filter membranes are specifically designed for polishing filtration applications in the food and beverage industries. Both filter membranes have a comparable retention grade as shown in Table V-I. The main difference between these two products is their constituent components. Pad A consists of a mixture of cellulose and diatomaceous earth (DE), while pad B is primarily a cellulose pad with a polymer insert that aids with the wet bursting strength of the sheet. Pad A is the pad used in the current filtration process. Ash content was determined by the Certificate of Analysis (CofA) provided by the manufacturer.

TABLE V-I**FILTER SHEETS EXAMINED FOR CHILL FILTRATION**

(Source: Data from pad manufacturers whose names are held confidential by Brown-Forman)

Parameter	Units	Pad A	Pad B
Nominal Retention Size	µm	0.8 - 1.0	0.7 - 1.0
Permeability	gpm/ft ²	5.03	3.93
Ash Content	%	60	< 1
Thickness	in.	0.17	0.15
Iron content	µg/g	2.2	-

2. Stability: Results and Discussion

Stability results data are presented in Appendix 2. These include production and trial samples randomly selected. The following definitions help explain the Rating category (“Rate”) assigned to a particular run in Appendix 2.

Rate 0: Clear product with no sign of haze potential.

Rate 1: Clear product with slight haze that eventually goes back into solution. Not detected by the consumer.

Rate 2: Clear product with haze that may go back into solution. Not detected by consumer.

Rate 3: Hazy product with heavy wisps that do not go back into solution. May be detected by the consumer.

Rate 4: Hazy product with heavy wisps. Haze will be detected by the consumer.

The stability study compared product stability from the currently practiced in-plant filtration process to the stability using chill filtration. For this analysis the results of filtrations with pad A and pad B are combined. Several trial and production samples were analyzed and stability was ranked or rated by testing panels within the technical services department of the company using the 0 to 4 rating scale described earlier.

FIGURE 5.1. Stability Rating of Randomly Collected Samples from the Current Filtration Process at Different Proofs (Stability rating from 0 no haze to 4 heavy wisps)

Figure 5.1 presents the ratings for all the tested production samples. No 80 or 86 proof samples were tested because no production runs of 80 or 86 proof were processed during the testing timeframe. Figure 5.1 shows that, when other filtration parameters are equal, there is a high correlation between the alcohol strength and the probability of obtaining a product that may need to be re-filtered (Ratings 3 or 4, orange and red bars in the figure). More than 80% of the 90 proof samples tested were completely outside of the acceptable quality standard obtaining Ratings 3 or 4. Only 30% of the 94 proof samples earned Ratings 3 or 4. Eighty percent of the 100 proof samples tested were within acceptable quality margins (Ratings of 0 and 1) and the other 20% merited a warning, getting a Rating of 2. Based on these results for the higher alcohol products it can be strongly expected that the standard lower proof products (80 and 86 proof) will

also be most likely in need of multiple reprocessing steps to achieve an acceptable stability rating.

The stability results clearly support the premise that the long chains of polyphenols such as esters are hydrophobic. Because of its short carbon chain, ethanol mixes easily with water at any bottling concentration. On the other hand, alcohols with longer chains than ethanol do not mix readily with water. A higher concentration of water, i.e. a lower proof whiskey, allows for more long carbon chain components such as fatty esters to come out of solution making the liquid more prone to haze and instability. The Chill filtration process, compared to the current standard process, removes a higher percentage of fatty esters across all proofs. The removal of these compounds allow for a more stable product (See TABLE V-II).

TABLE V-II
CONCENTRATION AND PERCENT REDUCTION OF FATTY ESTERS AFTER
CURRENT FILTRATION PROCESS VS. CHILL FILTRATION TRIALS (AVERAGE
ACROSS ALL PROOFS)

Compound	Initial Material Unfiltered Whiskey (ppm)	Current Process Filtered Whiskey (ppm - %Red.)	Chill Filtration Filtered Whiskey (ppm - %Red.)
Ethyl Caproate	5	1 (80%)	1 (80%)
Ethyl Caprylate	5	2 (60%)	1 (80%)
Ethyl Caprate	10	2 (80%)	2 (80%)
Ethyl Laurate	6	2 (33%)	1 (83%)
Ethyl Myristate	3	2 (33%)	0.3 (90%)
Ethyl Palmitate	10	6 (40%)	2.5 (75%)

FIGURE 5.2 .Stability Results of Chill Filtration Trials at Different Proofs (Stability rating from 0 no haze to 4 heavy wisps)

Figure 5.2 presents the stability test results for the samples tested using chill filtration using both pad A and B as filter media. Since the challenge of filtering low proof products is well known, chill filtration trials on 80 and 86 proof samples were performed in addition to testing higher proof samples. Following chill filtration trials no samples, at any of the whiskey alcohol concentrations, earned the failing rating of 4. No samples of 90 proof product were in the Rating 3 or 4 range, whereas 80% of such samples tested using the current in-plant filtration process were in that range. Only 10% of 94 proof samples were rated at 3 whereas 30% of those produced using the current process rated a 3 or 4. All chill filtered 100 proof samples were in spec with more than 80% achieving the desirable 0 rating with the remainder being rated 1.

While it is not possible to compare the 80 and 86 proof chill filtered results with production trial results, considering that only 3 samples at 86 proof were Rated 3 out of a total of

25 samples of 80 and 86 proof product provides a satisfactory level of comfort that chill filtration can provide a more stable product at all proof ranges compared to the regular process.

Further analysis was performed in order to understand the stability results of the Rated 3 86 proof samples. After reviewing records it was possible to determine that the three 86 proof samples with unsatisfactory stability rating belonged to Filtration Trials 15, 16, and 17. Data show a potential leak during the early stages of the filtration could have caused these results. Initial turbidity of samples in trial 16 and 17 were above 1 NTU. These numbers show that the filter unit was not properly sealed during the start of the filtration trial. The rest of samples within the trials show that the unit was properly sealed dropping the turbidity of the samples.

3. Filtration: Results and Discussion

Data collected from the filtration trials are presented in Appendix 1. These include all data taken about whiskey feed, flow rates, temperature, color loss, ambient turbidity are also shown in Appendix 1. The following definitions help explain the raw data.

Trial No.: Determines the trial run. A total of 38 filtration trials were completed.

Sample proof: Determines the alcohol concentration of the tested whiskey. Five whiskey concentrations were tested: 80, 86, 90, 94, and 100 proof. These concentrations match to all the products manufactured and sold for domestic and international markets.

Time: Determines the time elapsed between samples collected (min).

Pressure: Determines the differential pressure across the filter housing (psi).

Flow: Determines the average flow rate of the filtrate (ml/min).

Flux: Filtration rate expressed as gallons of filtrate per minute per square foot of filter sheet surface area (gpm/ft²).

Sample No.: Determines the number of samples passed through the filter per trial. Each sample is 100 milliliters of feed material.

Turbidity: This is a measure of attenuation of light as it passes through a sample column of filtered whiskey (Nephelometric Turbidity Units or NTU).

Color: this is a measure of absorbance at a wavelength of 430nm (dimensionless).

Color Loss: it is the percentage of tannins lost during the filtration process. It is calculated by dividing the difference of initial color minus color following filtration by the initial color.

Temperature: Temperature of the cooling source and of the filtered whiskey after filtration (°F).

Carbon treat: amount of activated carbon mixed with the whiskey for immediate treatment.

Amount is defined by the amount of alcohol in the sample (oz/100PG).

Bodyfeed: amount of filter aid (DE) added to a sample (g/L).

Two filter media were tested: pad A and pad B. A new pad was used on every single trial. Both pads were tested under the same conditions: filtration temperature, flow rate, carbon treatment, and bodyfeed load trying to maintain a constant flux ratio of 0.31 gpm/ft². Whiskeys at different alcohol concentrations were tested using each pad type.

FIGURE 5.3. Percent Color Loss of Whiskey at Different Proofs Using Filter Pad A and Filter Pad B

Figure 5.3 shows the percent color loss of whiskey samples of different proofs filtered using new pads A and B for each filtration. The data shows that at the beginning of the filtration while using a brand new pad A, regardless of its alcohol concentration, a 25 percent color loss was exceeded. In the brown spirits industry, it is not allowed to remove more than 25 percent of the initial color prior to bottling. Even though the product is stable, it would not be able to be used and needs to be discarded. After the first few 100 mL batches, the filtered liquids remain in the allowable range for color removal.

Using this criterion, Pad B qualified as the optimal filter sheet candidate to be used for chill filtration. All batches filtered through pad B were within the color specification and reaching the proper liquid stability.

FIGURE 5.4. Average Ambient Turbidity of Chill Filtration Samples

Ambient turbidity of a sample is the first and quickest indicator that fines were captured during filtration. The current specification for this whiskey is that turbidity be below 1.0 NTU. As shown in Figure 5.4 all filter tests using Pad A and Pad B achieved the desired turbidity specification for products of all proof tested with a tendency towards lower turbidity as proof increased.

FIGURE 5.5. Average Differential Pressure Across the Filter at Different Proofs

Trials at 86, 90, 94, and 100 proof used the same concentration of filter aid: 1 gram of filter aid per Liter of material). The samples at 80 proof were filtered with 3 times the amount of filter aid used for the other trials (3g filter aid/L). These values were obtained from current in-plant use considering the difficulty presented in filtering the lower proof product. Regardless of the pad type, trials at all proofs behaved similarly. Trials at different alcohol concentrations were expected to have more dramatic difference in differential pressure across the filter. Figure 5.5 shows that trials at 86, 90, 94, and 100 proof behaved similarly. Trials at 80 proof showed a reduced pressure drop across the trial considering 300% more of filter aid was used compared to the other proof trials. It was expected to observe an increase of differential pressure across the filter more rapidly by lowering the proof of the incoming material. It was expected to see the opposite correlation, the lower the proof, the sooner the pressure across the filter will increase.

This was expected considering the viscosity of whiskey at 32°F increases when the alcohol content is lower in the 80 – 100 proof range (Yusa, 1973) as shown in Figure 5.6.

FIGURE 5.6. Viscosity vs. Water/Ethanol Composition at 1atm at different temperatures 32°F, 70°F, and 122°F (Yusa, 1973).

Also, it was expected to remove more wood component with lower proofs impacting the pressure across the filter because of the low solubility of longer carbon chain components. The only factor that seems to explain the data is the amount of filter aid used during filtration: 80 proof used 3 times more filter aid compared to the other alcohol strength products.

Flow controls and temperature controls were not an issue during the trials. An average flow rate for all trials of 17 mL/min at a processing temperature of 25°F, was targeted for the length of the study.

Pad A and B provided the desired product stability proving that chill filtration is a feasible process for finishing this product. However, it was also determined that only pad B should be

considered as the potential candidate for chill filtration since it satisfied all testing parameters. The initial color removal by pad A shows the excessive impact of diatomaceous earth on color removal from brown spirits.

C. Problems Encountered During Testing

Unintentional dilution of the first sample was a major concern in this project. Considering the sample size collected was 100 milliliters, one milliliter of extraneous water collected throughout the system could dramatically reduce the proof of the sample to an undesirable alcohol concentration.

At the beginning of the experimentation, only the alcohol concentration of the unfiltered material was being tested, not the first filtrate sample. The alcohol concentration of the filtered samples was not being tested considering the equipment was assumed to be dry. During the first trials, pads A and B were providing filtered samples with high color losses in the first sample collected indicating that both pads were behaving similarly. Considering we started seeing color loss variability between the first and second filtered samples using pad A or pad B, the proof of each first filtered sample was then tested and showed the source of the issue. Figure 5.6 displays an example of how the problem was solved for pad B.

FIGURE 5.7. Difference Between Percent Color Loss at Sample Proof Versus Target Proof of First Filtered Sample

Initial color was normalized to remove the alcohol concentration factor. Color loss is usually calculated as the difference between final filtrate color and initial unfiltered color divided by the initial color of the sample. This formula is acceptable if no other variables change; basically, assuming proof remains the same. If proof of the sample changes, this formula is no longer valid and proof needs to be measured. Color and alcohol concentration of the low proof samples were measured with the absorbance meter and the density meter respectively. When alcohol dilution was no longer an issue, initial color loss was in range and showed less variability. The main cause of high variability in color loss was determined when a trial was being run with equipment that has been left to dry overnight as opposed to equipment that was cleaned, left to dry for a few hours and then used. For example, during the filtration trial of 80

proof material, a residual of 1 mL of wash water left in the apparatus is sufficient to reduce the proof of the first 100mL sample from 80 to 79 proof.

D. Economic Considerations

This chill filtration feasibility study used commercially available filter pads. The price per sheet is of the same order of magnitude with the cost of pad B being just 20% greater per square foot than pad A. Compared to the current ambient temperature process, Chill Filtration is more energy intensive, and requires more square footage of filter sheet per gallon of whiskey processed. However, Chill Filtration requires less foot print and 80% less activated carbon than the current process, and an automated system will require less manpower considering the chill system is expected to be more robust. Once the initial capital cost has been invested, Chill filtration is about 36% more cost efficient than the current process or an approximate savings of \$10 per barrel of product. (See TABLE V-III).

TABLE V-III

**OPERATION COST OF CURRENT FILTRATION PROCESS VS. CHILL
FILTRATION PER BARREL OF PRODUCT**

Parameter	Units per Barrel	Current Process	Chill Filtration Process
Preparation Time	min/barrel	720	12
Filtration time	min/barrel	12	5
Operators required	op/barrel	2	2
Operator Cost	\$/barrel	26.70	5.30
Filter Pad Cost	\$/barrel	0.45	1.30
Filter Aid Cost	\$/barrel	0.01	0.02
Carbon Cost	\$/barrel	0.47	0.10
Chiller Cost	\$/barrel	0	10.9
Total Cost	\$/barrel	27.63	17.62

VI. CONCLUSIONS

1. Chill filtration is definitely feasible for use as the final polishing step prior to bottling whiskey products. It provides a more stable product over the standard technique.
2. A filter pad with a cellulose filter medium, Pad B in this work, allows for stable filtration without removing unnecessary tannins from brown spirits, as compared to the currently used filter pad that also contains diatomaceous earth (Pad A in this work).
3. Use of the proper amount of bodyfeed (diatomaceous earth mixed into the feed whiskey as a filter aid) during filtration extends the life of the filter medium.
4. Whiskey stability is highly correlated with the alcohol concentration, or proof, of the bottling product. This work showed that higher alcohol content products at 90, 94 and 100 proof, were more stable than lower proof products of 80 and 86 proof.
5. Chill Filtration removes long chain fatty esters, such as ethyl palmitate, ethyl myristate and ethyl laurate from untreated whiskey, reducing their concentrations in the final product to levels that are 25% or less of those in the feed, compared to the typical 60% of original levels remaining after conventional filtration. Since these fatty acids are major contributors to haze formation in lower proof spirits, Chill Filtration is thus more effective than the current technique at reducing or preventing haze in these products.

VII. RECOMMENDATIONS

1. Continue same experimentation directed towards the optimization of bodyfeed requirements during the Chill Filtration process.
2. Conduct cost reduction projects after plant implementation of Chill Filtration.
3. Conduct a study to further understand color removal and stability of products during filtration.
4. Conduct further analysis of color removal and stability during Chill Filtration to optimize the amount of activated carbon required per proof.

APPENDIX 1: RAW DATA

TABLE A1-1

TRIAL 1 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
1	80	A	0:00	8	18	0.331					24.4	24.6
1	80	A	0:05	8	18	0.331	1	0.962	0.774	14%	24.4	24.6
1	80	A	0:10	8	17	0.313	2	0.952	0.791	13%	24.4	24.6
1	80	A	0:15	8	17	0.313	3	0.779	0.801	11%	24.4	24.4
1	80	A	0:20	8	17	0.313	4	0.763	0.806	11%	23.9	23.9
1	80	A	0:25	9	17	0.313	5	0.724	0.810	10%	23.9	23.9
1	80	A	0:30	10	17	0.313	6	0.704	0.815	10%	23.9	23.9
1	80	A	0:35	10	17	0.313	7	0.718	0.823	9%	24.3	24.3
1	80	A	0:40	10	17	0.313	8	0.704	0.831	8%	24.1	24.4
1	80	A	0:45	11	17	0.313	9	0.705	0.821	9%	23.7	24.1
1	80	A	0:50	11	17	0.304	10	0.665	0.818	10%	23.7	23.9
1	80	A	0:55	12	17	0.313	11	0.627	0.823	9%	24.3	24.3
1	80	A	1:00	12	17	0.313	12	0.632	0.826	9%	24.1	24.3
1	80	A	1:05	13	17	0.313	13	0.590	0.826	9%	24.1	24.3
1	80	A	1:10	14	17	0.313	14	0.549	0.829	8%	24.4	24.4
1	80	A	1:15	14	17	0.313	15	0.533	0.829	8%	24.3	24.4
1	80	A	1:20	15	17	0.313	16	0.483	0.830	8%	24.3	24.4
1	80	A	1:25	15	17	0.313	17	0.498	0.832	8%	24.3	24.4
1	80	A	1:30	16	17	0.313	18	0.504	0.832	8%	24.3	24.3
1	80	A	1:35	16	17	0.313	19	0.490	0.832	8%	24.4	24.4
1	80	A	1:40	16	17	0.304	20	0.725	0.832	8%	24.4	24.4
1	80	A	1:45	16	17	0.313	21	0.459	0.836	8%	24.4	24.4
1	80	A	1:51	17	15	0.276	22	0.723	0.835	8%	24.3	24.4
1	80	A	1:56	16	16	0.294	23	0.400	0.837	7%	24.3	24.3
1	80	A	2:02	17	16	0.294	24	0.406	0.839	7%	24.1	24.1
1	80	A	2:07	17	16	0.294	25	0.480	0.840	7%	24.1	24.1

TABLE A1-2

TRIAL 2 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
2	80	A	0:00	7	17	0.304					24.1	24.3
2	80	A	0:05	7	17	0.313	1	0.567	0.688	25%	24.1	24.3
2	80	A	0:10	7	17	0.313	2	0.631	0.749	19%	24.1	24.1
2	80	A	0:15	8	17	0.313	3	0.645	0.777	16%	24.1	24.1
2	80	A	0:20	8	17	0.313	4	0.646	0.795	14%	24.1	24.1
2	80	A	0:25	9	17	0.313	5	0.589	0.805	13%	24.1	24.1
2	80	A	0:30	9	17	0.313	6	0.599	0.815	11%	24.1	24.1
2	80	A	0:35	9	17	0.313	7	0.564	0.818	11%	23.9	23.9
2	80	A	0:40	10	17	0.313	8	0.583	0.827	10%	23.9	23.9
2	80	A	0:45	10	17	0.313	9	0.507	0.824	10%	24.1	24.1
2	80	A	0:50	11	17	0.313	10	0.535	0.827	10%	24.1	24.1
2	80	A	0:55	11	17	0.313	11	0.565	0.830	10%	24.1	24.1
2	80	A	1:00	12	17	0.313	12	0.476	0.834	9%	24.1	24.1
2	80	A	1:05	12	17	0.313	13	0.433	0.835	9%	24.1	24.1
2	80	A	1:10	13	17	0.313	14	0.444	0.837	9%	24.1	24.1
2	80	A	1:15	14	17	0.313	15	0.438	0.839	9%	24.1	24.1
2	80	A	1:20	14	17	0.313	16	0.435	0.829	10%	24.1	24.1
2	80	A	1:25	15	17	0.313	17	0.403	0.835	9%	24.1	24.1
2	80	A	1:30	15	17	0.313	18	0.406	0.837	9%	24.1	24.1
2	80	A	1:35	16	17	0.313	19	0.385	0.839	9%	24.1	24.1
2	80	A	1:40	16	17	0.313	20	0.399	0.838	9%	24.1	24.1
2	80	A	1:45	16	16	0.294	21	0.454	0.844	8%	24.1	24.1
2	80	A	1:50	17	16	0.294	22	0.401	0.844	8%	24.1	24.1
2	80	A	1:55	17	16	0.294	23	0.393	0.843	8%	24.1	24.1
2	80	A	2:00	17	17	0.304	24	0.376	0.818	11%	24.1	24.1
2	80	A	2:05	17	16	0.294	25	0.386	0.814	12%	24.1	24.1

TABLE A1-3

TRIAL 3 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
3	80	A	0:00	7	17	0.304					24.4	24.4
3	80	A	0:05	7	17	0.304	1	0.798	0.773	15%	24.6	24.6
3	80	A	0:10	8	18	0.322	2	0.711	0.793	12%	24.8	24.8
3	80	A	0:15	8	17	0.313	3	0.736	0.808	11%	24.6	24.6
3	80	A	0:20	8	17	0.313	4	0.732	0.811	10%	24.3	24.3
3	80	A	0:25	8	17	0.304	5	0.635	0.816	10%	24.4	24.4
3	80	A	0:30	9	17	0.304	6	0.653	0.821	9%	24.6	24.6
3	80	A	0:35	10	17	0.313	7	0.599	0.824	9%	24.4	24.4
3	80	A	0:40	10	17	0.313	8	0.542	0.828	9%	24.4	24.4
3	80	A	0:45	10	17	0.304	9	0.624	0.827	9%	24.3	24.3
3	80	A	0:50	11	17	0.304	10	0.539	0.829	9%	24.4	24.4
3	80	A	0:55	11	17	0.313	11	0.502	0.832	8%	24.4	24.4
3	80	A	1:00	12	17	0.313	12	0.492	0.835	8%	24.4	24.4
3	80	A	1:05	13	17	0.313	13	0.466	0.809	11%	24.4	24.4
3	80	A	1:10	13	17	0.313	14	0.490	0.811	10%	24.3	24.3
3	80	A	1:15	14	17	0.313	15	0.450	0.811	10%	24.4	24.4
3	80	A	1:20	14	17	0.313	16	0.450	0.812	10%	24.6	24.6
3	80	A	1:25	15	17	0.313	17	0.462	0.814	10%	24.4	24.4
3	80	A	1:30	16	17	0.313	18	0.438	0.814	10%	24.4	24.4
3	80	A	1:35	16	17	0.313	19	0.400	0.816	10%	24.4	24.4
3	80	A	1:40	16	17	0.304	20	0.401	0.816	10%	24.4	24.4
3	80	A	1:45	16	17	0.304	21	0.582	0.822	9%	24.4	24.4
3	80	A	1:50	18	17	0.313	22	0.414	0.823	9%	24.4	24.4
3	80	A	1:55	18	17	0.313	23	0.345	0.824	9%	24.4	24.4
3	80	A	2:00	18	17	0.313	24	0.406	0.821	9%	24.3	24.3
3	80	A	2:05	18	17	0.313	25	0.405	0.826	9%	24.3	24.3

TABLE A1-4

TRIAL 4 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
4	80	A	0:00	7	17	0.304					24.4	24.4
4	80	A	0:05	7	17	0.304	1	0.607	0.620	32%	24.6	24.6
4	80	A	0:10	8	18	0.322	2	0.669	0.710	22%	24.8	24.8
4	80	A	0:15	8	17	0.313	3	0.710	0.752	18%	24.6	24.6
4	80	A	0:20	8	17	0.313	4	0.694	0.789	14%	24.3	24.3
4	80	A	0:25	8	17	0.304	5	0.628	0.811	11%	24.4	24.4
4	80	A	0:30	9	17	0.304	6	0.628	0.812	11%	24.6	24.6
4	80	A	0:35	10	17	0.313	7	0.619	0.822	10%	24.4	24.4
4	80	A	0:40	10	17	0.313	8	0.590	0.823	10%	24.4	24.4
4	80	A	0:45	10	17	0.304	9	0.542	0.826	10%	16.7	24.3
4	80	A	0:50	11	17	0.304	10	0.557	0.829	9%	24.4	24.4
4	80	A	0:55	11	17	0.313	11	0.540	0.830	9%	24.4	24.4
4	80	A	1:00	12	17	0.313	12	0.630	0.834	9%	24.4	24.4
4	80	A	1:05	13	17	0.313	13	0.550	0.833	9%	24.4	24.4
4	80	A	1:10	13	17	0.313	14	0.527	0.836	9%	24.3	24.3
4	80	A	1:15	14	17	0.313	15	0.499	0.837	9%	24.4	24.4
4	80	A	1:20	14	17	0.313	16	0.506	0.838	8%	24.6	24.6
4	80	A	1:25	15	17	0.313	17	0.503	0.839	8%	24.4	24.4
4	80	A	1:30	16	17	0.313	18	0.498	0.842	8%	24.4	24.4
4	80	A	1:35	16	17	0.313	19	0.485	0.844	8%	24.4	24.4
4	80	A	1:40	16	17	0.304	20	0.438	0.843	8%	24.4	24.4
4	80	A	1:46	16	17	0.304	21	0.527	0.842	8%	24.4	24.4
4	80	A	1:50	18	17	0.313	22	0.671	0.846	8%	24.4	24.4
4	80	A	1:55	18	17	0.313	23	0.470	0.845	8%	24.4	24.4
4	80	A	2:00	18	17	0.313	24	0.445	0.845	8%	24.3	24.3
4	80	A	2:05	18	17	0.313	25	0.549	0.844	8%	24.3	24.3

TABLE A1-5

TRIAL 5 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
5	80	A	0:00	7	22	0.405					24.1	24.1
5	80	A	0:43	8	24	0.442	1	1.590	0.429	57%	24.8	24.8
5	80	A	0:48	8	19	0.350	2	0.700	0.720	28%	24.8	25.0
5	80	A	0:53	9	20	0.368	3	0.841	0.776	22%	23.7	24.1
5	80	A	0:58	11	20	0.368	4	0.745	0.795	21%	22.3	22.3
5	80	A	1:03	13	20	0.368	5	0.719	0.808	19%	21.7	22.5
5	80	A	1:08	15	21	0.386	6	0.699	0.810	19%	22.3	23.0
5	80	A	1:13	16	20	0.368	7	0.701	0.816	18%	21.6	22.1
5	80	A	1:18	13	20	0.368	8	0.731	0.824	18%	23.0	23.0
5	80	A	1:23	12	16	0.294	9	0.814	0.831	17%	24.4	24.4
5	80	A	1:26								32.0	32.0
5	80	A	1:28	12	15	0.276					23.7	23.7
5	80	A	1:33	13	16	0.294	10	0.720	0.829	9%	23.0	23.0
5	80	A	1:38	14	19	0.350					22.3	22.3
5	80	A	1:40				11	0.707	0.824	9%	32.0	32.0

TABLE A1-6

TRIAL 6 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
6	80	A	0:00	8	14	0.258					23.4	23.5
6	80	A	0:06	10	20	0.368	1	1.370	0.435	52%	24.4	24.6
6	80	A	0:11	10	21	0.386	2	0.739	0.723	20%	23.4	23.7
6	80	A	0:16	11	22	0.405	3	0.734	0.779	14%	22.6	22.8
6	80	A	0:21	12	22	0.405	4	0.673	0.803	12%	23.0	23.0
6	80	A	0:26	12	22	0.405	5	0.678	0.821	10%	24.1	24.1
6	80	A	0:31	13	22	0.405	6	0.653	0.827	9%	24.3	24.3
6	80	A	0:36	14	22	0.405	7	0.632	0.846	7%	24.3	24.4
6	80	A	0:41	14	22	0.405	8	0.627	0.834	8%	24.3	24.3
6	80	A	0:46	15	20	0.368	9	0.611	0.834	8%	23.9	23.9
6	80	A	0:51	16	20	0.368	10	0.616	0.832	8%	23.7	23.9
6	80	A	0:57	17	19	0.350	11	0.618	0.838	8%	24.4	24.6
6	80	A	1:03	17	18	0.331	12	0.586	0.858	6%	24.6	24.8
6	80	A	1:08	18	19	0.350	13	0.579	0.840	7%	23.5	23.9
6	80	A	1:14	18	19	0.350	14	0.587	0.836	8%	24.4	24.6
6	80	A	1:20	18	18	0.331	15	0.571	0.842	7%	23.7	23.7
6	80	A	1:26	19	18	0.331	16	0.536	0.840	7%	24.6	24.3
6	80	A	1:32	19	18	0.331	17	0.593	0.846	7%	24.4	24.4
6	80	A	1:39	23	18	0.331	18	0.789	0.845	7%	23.7	23.7
6	80	A	1:45	25	18	0.331	19	0.487	0.836	8%	23.4	24.4
6	80	A	1:51	25	18	0.331	20	0.503	0.849	7%	24.3	25.2

TABLE A1-7

TRIAL 7 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
7	80	B	0:00	10	21	0.386					23.5	31.1
7	80	B	0:05	10	20	0.368	1	0.352	0.777	14%	19.6	30.2
7	80	B	0:10	11	21	0.386	2	0.533	0.810	10%	18.5	30.7
7	80	B	0:15	11	20	0.368	3	0.474	0.812	10%	18.1	30.2
7	80	B	0:20	11	17	0.313	4	0.548	0.819	9%	17.1	29.7
7	80	B	0:25	11	18	0.331					16.3	28.8
7	80	B	0:27				5	0.552	0.817	9%	32.0	32.0
7	80	B	0:30	12	17	0.313					16.5	28.4
7	80	B	0:35	13	16	0.294	6	0.543	0.810	10%	22.6	37.0
7	80	B	0:40	13	16	0.294					26.4	37.2
7	80	B	0:43				7	0.571	0.830	8%	32.0	32.0
7	80	B	0:45	12	16	0.294					23.0	34.5
7	80	B	0:50	11	14	0.258					20.8	31.3
7	80	B	0:52				8	0.564	0.835	7%	32.0	32.0
7	80	B	0:55	14	12	0.221					19.0	30.9
7	80	B	1:00	14	5	0.092	9	0.586	0.821	9%	16.2	29.8

TABLE A1-8

TRIAL 8 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
8	80	B	0:00	13	24	0.442					19.6	31.6
8	80	B	0:05	12	22	0.405	1	0.440	0.728	20%	19.6	31.1
8	80	B	0:10	12	21	0.386	2	0.605	0.805	11%	16.5	29.1
8	80	B	0:15	14	22	0.405	3	0.505	0.822	9%	15.8	28.0
8	80	B	0:20	15	22	0.405	4	0.504	0.825	9%	15.8	26.6
8	80	B	0:25	19	22	0.405				100%	14.9	26.2
8	80	B	0:27				5	0.488	0.829	9%	32.0	32.0
8	80	B	0:30	21	21	0.386	6	0.470	0.833	8%	16.7	29.1

TABLE A1-9

TRIAL 9 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
9	80	B	0:00	6	7	0.129					23.7	23.5
9	80	B	0:04	12	12	0.221					23.5	23.5
9	80	B	0:07	11	16	0.294					23.5	23.5
9	80	B	0:09	11	15	0.276	1	1.080	0.649	29%	23.7	23.7
9	80	B	0:16	12	16	0.294	2	0.624	0.799	13%	23.9	24.1
9	80	B	0:23	13	16	0.294	3	0.608	0.825	10%	23.9	23.7
9	80	B	0:30	15	16	0.294	4	0.585	0.830	9%	23.9	23.9

TABLE A1-10

TRIAL 10 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
10	80	B	0:00	10	9	0.166					23.7	23.9
10	80	B	0:06	10	13	0.239					24.4	24.8
10	80	B	0:10	10	16	0.294	1	0.400	0.654	28%	24.3	25.0
10	80	B	0:17	10	16	0.294	2	0.616	0.802	12%	23.7	24.3
10	80	B	0:23	12	16	0.294	3	0.666	0.821	10%	23.7	24.1
10	80	B	0:30	12	16	0.294	4	0.651	0.830	9%	23.2	23.5
10	80	B	0:37	14	16	0.294	5	0.629	0.833	8%	23.7	23.9
10	80	B	0:44	15	16	0.294	6	0.637	0.838	8%	23.4	23.7
10	80	B	0:50	16	16	0.294	7	0.624	0.839	8%	23.7	23.7
10	80	B	0:57	17	16	0.294	8	0.621	0.843	7%	24.3	24.4
10	80	B	1:03	17	16	0.294	9	0.614	0.848	7%	24.4	24.6
10	80	B	1:09	18	16	0.294	10	0.567	0.844	7%	23.7	24.1
10	80	B	1:16	19	16	0.294	11	0.535	0.842	7%	23.5	23.9
10	80	B	1:22	21	16	0.294	12	0.546	0.844	7%	24.1	23.9
10	80	B	1:29	21	16	0.294	13	0.527	0.847	7%	24.3	24.4
10	80	B	1:35	23	16	0.294	14	0.515	0.848	7%	24.1	24.3
10	80	B	1:42	23	16	0.294	15	0.518	0.846	7%	24.8	24.8
10	80	B	1:48	24	15	0.276	16	0.478	0.847	7%	24.3	24.4
10	80	B	1:54	25	16	0.294	17	0.464	0.841	7%	23.9	23.9
10	80	B	2:02	26	13	0.230	18	0.470	0.841	7%	23.7	23.9
10	80	B	2:09	28	14	0.258	19	0.460	0.842	7%	23.9	23.9
10	80	B	2:13	28	14	0.258	20	0.482	0.842	7%	23.9	23.9

TABLE A1-11

TRIAL 11 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
11	86	B	0:00	5	11	0.202					24.8	25.2
11	86	B	0:06	10	18	0.331	1	0.823	0.779	21%	24.3	24.6
11	86	B	0:11	9	17	0.304	2	0.671	0.896	9%	24.3	24.3
11	86	B	0:16	9	17	0.313	3	0.547	0.910	8%	24.3	24.3
11	86	B	0:21	10	17	0.313	4	0.462	0.926	6%	24.4	24.4
11	86	B	0:26	10	17	0.313	5	0.404	0.930	6%	24.4	24.4
11	86	B	0:31	11	17	0.313	6	0.391	0.927	6%	24.3	24.3
11	86	B	0:36	11	17	0.313	7	0.368	0.923	6%	24.3	24.3
11	86	B	0:41	12	17	0.313	8	0.323	0.922	6%	24.3	24.3
11	86	B	0:46	13	17	0.313	9	0.321	0.922	6%	24.3	24.3
11	86	B	0:51	13	17	0.313	10	0.308	0.920	7%	24.4	24.4
11	86	B	0:56	14	17	0.313	11	0.319	0.923	6%	24.4	24.4
11	86	B	1:01	14	17	0.313	12	0.308	0.922	6%	24.4	24.3
11	86	B	1:06	15	17	0.313	13	0.243	0.921	7%	24.3	24.3
11	86	B	1:11	15	17	0.313	14	0.380	0.923	6%	24.3	24.3
11	86	B	1:16	15	17	0.313	15	0.328	0.917	7%	24.3	24.3
11	86	B	1:21	16	17	0.304	16	0.266	0.922	6%	24.3	24.3
11	86	B	1:26	16	17	0.304	17	0.272	0.921	7%	24.3	24.3
11	86	B	1:31	17	17	0.304	18	0.279	0.925	6%	24.3	24.3

TABLE A1-12

TRIAL 12 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
12	86	B	0:00	3	7	0.129					24.3	24.4
12	86	B	0:05	9	16	0.285	1	0.898	0.756	23%	39.9	24.3
12	86	B	0:10	9	18	0.331	2	0.336	0.888	10%	24.4	14.2
12	86	B	0:15	10	17	0.313	3	0.397	0.907	8%	24.1	24.4
12	86	B	0:20	11	17	0.313	4	0.376	0.916	7%	24.1	24.4
12	86	B	0:25	13	17	0.313	5	0.392	0.922	6%	24.1	24.3
12	86	B	0:30	14	17	0.313	6	0.348	0.924	6%	24.1	24.3
12	86	B	0:35	15	17	0.313	7	0.300	0.926	6%	24.1	24.3
12	86	B	0:40	16	17	0.313	8	0.298	0.926	6%	24.1	24.4
12	86	B	0:45	17	17	0.313	9	0.267	0.926	6%	24.1	24.3
12	86	B	0:50	19	17	0.313	10	0.328	0.927	6%	24.1	24.3
12	86	B	0:55	20	17	0.313	11	0.270	0.927	6%	24.1	24.3
12	86	B	1:00	22	17	0.313	12	0.444	0.925	6%	24.1	23.9
12	86	B	1:05	26	17	0.304	13	0.494	0.913	7%	24.3	24.3
12	86	B	1:10	27	17	0.304	14	0.240	0.932	5%	24.3	24.4
12	86	B	1:15	27	15	0.276	15	0.218	0.928	6%	24.1	24.1
12	86	B	1:21	30	16	0.294	16	0.245	0.926	6%	24.1	24.1
12	86	B	1:27	32	15	0.276	17	0.314	0.928	6%	23.9	24.1
12	86	B	1:32	33	14	0.258	18	0.310	0.926	6%	23.9	24.1

TABLE A1-13

TRIAL 13 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
13	86	B	0:00	2	14	0.248					24.8	25.3
13	86	B	0:05	7	18	0.331	1	0.845	0.793	19%	24.6	25.0
13	86	B	0:10	7	16	0.285	2	0.583	0.889	9%	24.4	24.4
13	86	B	0:15	8	18	0.322	3	0.509	0.906	8%	24.3	24.3
13	86	B	0:20	9	17	0.313	4	0.485	0.912	7%	24.4	24.4
13	86	B	0:25	9	17	0.304	5	0.469	0.914	7%	24.6	24.6
13	86	B	0:30	10	17	0.313	6	0.354	0.915	7%	24.6	24.6
13	86	B	0:35	10	17	0.313	7	0.372	0.915	7%	24.6	24.6
13	86	B	0:40	10	17	0.313	8	0.309	0.915	7%	24.8	24.8
13	86	B	0:45	11	17	0.313	9	0.290	0.915	7%	24.6	24.6
13	86	B	0:50	11	17	0.313	10	0.297	0.916	7%	24.6	24.6
13	86	B	0:55	12	17	0.313	11	0.296	0.920	6%	24.6	24.6
13	86	B	1:00	12	17	0.313	12	0.294	0.914	7%	24.6	24.6
13	86	B	1:05	12	17	0.313	13	0.261	0.913	7%	24.6	24.6
13	86	B	1:10	13	17	0.304	14	0.330	0.914	7%	24.6	24.6
13	86	B	1:15	15	18	0.322	15	0.365	0.915	7%	24.6	24.8
13	86	B	1:20	15	18	0.322	16	0.316	0.913	7%	24.8	24.6
13	86	B	1:25	15	18	0.322	17	0.294	0.913	7%	24.4	24.4
13	86	B	1:30	15	18	0.322	18	0.377	0.914	7%	24.4	24.4

TABLE A1-14

TRIAL 14 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
14	86	B	0:00	2	7	0.129					25.0	25.0
14	86	B	0:05	9	18	0.322	1	0.878	0.820	17%	25.0	25.7
14	86	B	0:10	9	16	0.294	2	0.739	0.922	7%	24.1	25.0
14	86	B	0:15	11	18	0.322	3	0.587	0.940	5%	23.7	24.4
14	86	B	0:20	12	18	0.322	4	0.541	0.949	4%	23.9	24.6
14	86	B	0:25	12	17	0.304	5	0.491	0.954	3%	24.1	24.6
14	86	B	0:30	13	16	0.294	6	0.471	0.954	3%	24.1	24.4
14	86	B	0:35	14	17	0.313	7	0.405	0.956	3%	24.1	24.6
14	86	B	0:40	16	16	0.294	8	0.410	0.958	3%	24.1	24.6
14	86	B	0:45	17	18	0.322	9	0.372	0.957	3%	24.1	24.6
14	86	B	0:50	18	17	0.304	10	0.344	0.958	3%	24.1	24.4
14	86	B	0:55	19	17	0.304	11	0.337	0.957	3%	24.1	24.6
14	86	B	1:00	20	16	0.294	12	0.352	0.959	3%	24.1	24.6
14	86	B	1:05	21	17	0.304	13	0.340	0.961	3%	24.1	24.6
14	86	B	1:10	23	17	0.304	14	0.322	0.956	3%	24.1	24.6
14	86	B	1:15	24	17	0.304	15	0.263	0.959	3%	24.1	24.6
14	86	B	1:20	25	17	0.304	16	0.304	0.959	3%	24.1	24.6
14	86	B	1:25	26	17	0.313	17	0.243	0.957	3%	24.1	24.6
14	86	B	1:30	27	17	0.304	18	0.451	0.960	3%	24.1	24.6
14	86	B	1:35	28	17	0.304	19	0.236	0.960	3%	24.1	24.6
14	86	B	1:40	30	17	0.313	20	0.230	0.956	3%	24.1	24.6
14	86	B	1:45	31	17	0.304	21	0.226	0.956	3%	24.1	24.6
14	86	B	1:50	33	17	0.304	22	0.255	0.957	3%	24.1	24.8
14	86	B	1:55	33	17	0.313	23	0.245	0.959	3%	24.1	24.8

TABLE A1-15

TRIAL 15 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
15	86	B	0:00	3	7	0.129					24.6	25.2
15	86	B	0:05	8	16	0.294	1	0.863	0.854	13%	24.4	24.4
15	86	B	0:10	11	17	0.313	2	0.541	0.932	5%	24.6	24.6
15	86	B	0:15	10	16	0.294	3	0.507	0.951	3%	24.6	24.6
15	86	B	0:20	11	17	0.313	4	0.442	0.956	2%	24.6	24.6
15	86	B	0:25	12	18	0.322	5	0.378	0.957	2%	24.8	24.8
15	86	B	0:30	13	18	0.322	6	0.339	0.960	2%	24.8	24.8
15	86	B	0:35	14	18	0.322	7	0.323	0.958	2%	24.8	24.8
15	86	B	0:40	15	18	0.322	8	0.301	0.958	2%	24.8	24.8
15	86	B	0:45	15	17	0.304	9	0.302	0.958	2%	24.8	24.8
15	86	B	0:50	17	17	0.313	10	0.261	0.959	2%	24.8	24.8
15	86	B	0:55	17	17	0.304	11	0.234	0.958	2%	24.8	24.8
15	86	B	1:00	18	17	0.313	12	0.220	0.957	2%	24.8	24.8
15	86	B	1:05	19	17	0.313	13	0.240	0.959	2%	24.8	24.8
15	86	B	1:10	20	17	0.313	14	0.211	0.959	2%	24.8	24.8
15	86	B	1:15	22	17	0.313	15	0.215	0.958	2%	24.8	24.8
15	86	B	1:20	23	17	0.313	16	0.188	0.958	2%	24.8	24.8
15	86	B	1:25	24	17	0.313	17	0.181	0.959	2%	24.8	24.8
15	86	B	1:30	25	17	0.313	18	0.205	0.958	2%	24.8	24.8
15	86	B	1:35	26	17	0.313	19	0.181	0.959	2%	24.8	24.8
15	86	B	1:40	27	17	0.313	20	0.201	0.960	2%	24.8	24.8
15	86	B	1:45	28	17	0.313	21	0.178	0.960	2%	24.8	24.8
15	86	B	1:50	29	17	0.313	22	0.187	0.960	2%	24.8	24.8
15	86	B	1:55	30	17	0.313	23	0.186	0.960	2%	24.8	24.8

TABLE A1-16

TRIAL 16 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
16	86	B	0:00	0							23.8	
16	86	B	0:24	5	14	0.258	1	4.520	1.251	15%	23.6	35.3
16	86	B	0:10	12	18	0.331	2	0.381	1.397	5%	23.7	35.3
16	86	B	0:05	14	16	0.294	3	0.330	1.386	5%	23.6	35.4
16	86	B	0:06	19	19	0.350	4	0.305	1.377	6%	23.7	35.2
16	86	B	0:06	25	17	0.313	5	0.200	1.372	6%	23.4	35.2
16	86	B	0:05	32	16	0.294	6	0.273	1.340	8%	23.7	35.3
16	86	B	0:07	41	16	0.294	7	0.160	1.344	8%	23.3	35.2
16	86	B	0:08	30	9	0.166	8	0.229	1.319	10%	23.5	34.8
16	86	B	0:17	30	8	0.147	9	0.208	1.299	11%	22.5	34.4
16	86	B	0:23	34	4	0.074	10	0.164	1.271	13%	23.1	34.2
16	86	B	0:32	39	5	0.092	11	0.302	1.265	14%	22.9	34.2

TABLE A1-17

TRIAL 17 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
17	86	B	0:00	0	0	0.000					23.2	
17	86	B	0:19	6	14	0.258	1	3.060	1.222	17%	23.3	36.5
17	86	B	0:07	14	16	0.294	2	0.239	1.373	6%	23.5	35.1
17	86	B	0:07	19	16	0.294	3	0.183	1.373	6%	23.6	35.1
17	86	B	0:06	27	16	0.294	4	0.196	1.343	8%	23.2	34.7
17	86	B	0:05	43	16	0.294	5	0.302	1.361	7%	23.0	34.7
17	86	B	0:08	47	14	0.258	6	0.358	1.345	8%	23.1	34.5
17	86	B	0:09	44	8	0.147	7	0.309	1.337	9%	22.9	34.5
17	86	B	0:16	44	8	0.138	8	0.177	1.324	10%	23.0	34.2
17	86	B	0:22	45	6	0.110	9	0.144	1.322	10%	22.9	34.4
17	86	B	0:29	46	4	0.074	10	0.323	1.353	8%	23.2	34.3

TABLE A1-18

TRIAL 18 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
18	86	B	0:00	0							22.8	
18	86	B	0:27	9	12	0.221	1	4.620	0.646	39%	23.5	35.2
18	86	B	0:08	22	18	0.331	2	0.467	0.988	7%	22.9	34.8
18	86	B	0:05	28	22	0.405	3	0.382	1.005	6%	23.3	34.5
18	86	B	0:05	26	17	0.313	4	0.283	1.001	6%	23.1	34.4
18	86	B	0:06	28	16	0.294	5	0.208	1.002	6%	23.2	34.3
18	86	B	0:07	35	14	0.258	6	0.285	1.005	6%	22.9	34.5
18	86	B	0:07	37	13	0.239	7	0.259	0.999	6%	23.1	34.7
18	86	B	0:09	39	12	0.221	8	0.233	0.993	7%	23.3	34.5
18	86	B	0:11	38	8	0.147	9	0.215	0.988	7%	23.2	34.5
18	86	B	0:14	41	8	0.147	10	0.143	1.008	6%	23.2	34.5

TABLE A1-19

TRIAL 19 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
19	86	B	0:00	20	30	0.552	1	0.239	1.219	20%	24.3	23.7
19	86	B	0:03	20	22	0.405	2	0.190	1.288	15%	24.3	23.7
19	86	B	0:07	16	16	0.294	3	0.320	1.310	14%	24.3	23.7
19	86	B	0:11	26	16	0.294	4	0.205	1.320	13%	24.3	23.7
19	86	B	0:16	28	16	0.294	5	0.171	1.333	12%	24.3	23.7
19	86	B	0:21	35	19	0.350	6	0.239	1.341	11%	24.3	23.7
19	86	B	0:26	38	16	0.294	7	0.184	1.334	12%	24.3	23.7
19	86	B	0:34	40	13	0.239	8	0.333	1.328	12%	24.3	23.7
19	86	B	0:44	42	10	0.184	9	0.327	1.320	13%	24.3	23.7
19	86	B	0:54	42	8	0.147	10	0.140	1.311	13%	24.3	23.7

TABLE A1-20

TRIAL 20 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
20	86	B	0:00	12	18	0.331	1	2.510	1.212	20%	23.7	24.0
20	86	B	0:05	14	18	0.331	2	0.178	1.416	6%	23.7	24.4
20	86	B	0:10	16	17	0.313	3	0.171	1.438	5%	23.7	24.4
20	86	B	0:15	18	17	0.313	4	0.134	1.441	4%	23.7	24.4
20	86	B	0:20	22	17	0.313	5	0.125	1.465	3%	23.7	24.4
20	86	B	0:25	25	17	0.313	6	0.117	1.470	3%	23.7	24.3
20	86	B	0:31	29	18	0.331	7	0.177	1.474	2%	23.7	24.3
20	86	B	0:36	33	18	0.331	8	0.152	1.476	2%	23.7	24.2
20	86	B	0:41	37	17	0.313	9	0.119	1.476	2%	23.7	24.1
20	86	B	0:46	38	17	0.313	10	0.135	1.477	2%	23.7	24.1
20	86	B	0:52	39	17	0.313	11	0.201	1.471	2%	23.7	24.1
20	86	B	0:58	39	15	0.276	12	0.182	1.469	3%	23.7	24.0
20	86	B	1:05	39	15	0.276	13	0.126	1.469	3%	23.7	24.0
20	86	B	1:12	45	15	0.276	14	0.161	1.469	3%	23.7	24.0
20	86	B	1:19	42	12	0.221	15	0.143	1.466	3%	23.7	24.0
20	86	B	1:28	42	12	0.221	16	0.155	1.464	3%	23.7	24.0
20	86	B	1:37	41	12	0.221	17	0.161	1.448	4%	23.7	23.6

TABLE A1-21

TRIAL 21 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
21	86	B	0:00	11	16	0.294	1	5.930	1.425	12%	23.7	24.0
21	86	B	0:07	14	18	0.331	2	2.490	1.575	3%	23.7	24.3
21	86	B	0:12	15	17	0.313	3	0.849	1.585	2%	23.7	24.2
21	86	B	0:17	17	17	0.313	4	0.549	1.591	2%	23.7	24.2
21	86	B	0:23	19	17	0.313	5	0.406	1.594	1%	23.7	24.1
21	86	B	0:28	22	17	0.313	6	0.331	1.596	1%	23.7	24.2
21	86	B	0:33	25	17	0.313	7	0.267	1.601	1%	23.7	24.2
21	86	B	0:39	29	17	0.313	8	0.277	1.603	1%	23.7	24.2
21	86	B	0:45	34	17	0.313	9	0.250	1.606	1%	23.7	24.1
21	86	B	0:50	36	17	0.313	10	0.212	1.607	1%	23.7	24.1
21	86	B	0:55	37	17	0.313	11	0.228	1.604	1%	23.7	24.1
21	86	B	1:01	39	15	0.276	12	0.213	1.601	1%	23.7	24.1
21	86	B	1:07	42	15	0.276	13	0.189	1.602	1%	23.7	24.2
21	86	B	1:14	45	16	0.294	14	0.233	1.604	1%	23.7	24.3
21	86	B	1:20	43	16	0.294	15	0.174	1.605	1%	23.7	24.2
21	86	B	1:26	40	16	0.294	16	0.182	1.599	1%	23.7	24.0
21	86	B	1:34	42	15	0.276	17	0.496	1.597	1%	23.7	23.8

TABLE A1-22

TRIAL 22 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
22	86	B	0:00	11	21	0.386	1	0.426	1.303	17%	23.9	24.5
22	86	B	0:04	11	18	0.331	2	0.171	1.438	8%	23.9	24.6
22	86	B	0:09	13	18	0.331	3	0.159	1.449	7%	23.9	24.7
22	86	B	0:15	16	17	0.313	4	0.160	1.448	7%	23.9	24.7
22	86	B	0:20	20	17	0.313	5	0.143	1.447	8%	23.9	24.6
22	86	B	0:25	24	17	0.313	6	0.148	1.444	8%	23.9	24.6
22	86	B	0:30	30	18	0.331	7	0.121	1.443	8%	23.9	24.6
22	86	B	0:36	34	17	0.313	8	0.119	1.440	8%	23.9	24.6
22	86	B	0:41	38	17	0.313	9	0.113	1.435	8%	23.9	24.7
22	86	B	0:47	38	17	0.313	10	0.115	1.432	9%	23.9	24.5
22	86	B	0:54	36	13	0.239	11	0.131	1.430	9%	23.9	24.3
22	86	B	1:01	40	13	0.239	12	0.121	1.427	9%	23.9	24.3
22	86	B	1:10	42	12	0.221	13	0.118	1.419	9%	23.9	24.3
22	86	B	1:19	42	10	0.184	14	0.118	1.412	10%	23.9	24.2
22	86	B	1:30	42	9	0.166	15	0.134	1.406	10%	23.9	24.1
22	86	B	1:42	45	8	0.147	16	0.119	1.404	10%	23.9	24.1
22	86	B	1:56	45	7	0.129	17	0.121	1.405	10%	23.9	23.8

TABLE A1-23

TRIAL 23 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
23	86	B	0:00	11	17	0.313	1	3.500	1.295	17%	23.9	24.3
23	86	B	0:05	13	19	0.350	2	1.610	1.439	8%	23.9	24.3
23	86	B	0:11	14	17	0.313	3	0.695	1.433	8%	23.9	24.2
23	86	B	0:17	19	17	0.313	4	0.496	1.431	9%	23.9	24.3
23	86	B	0:22	24	16	0.294	5	0.418	1.431	9%	23.9	24.4
23	86	B	0:28	33	18	0.331	6	0.346	1.428	9%	23.9	24.4
23	86	B	0:33	32	17	0.313	7	0.239	1.422	9%	23.9	24.4
23	86	B	0:40	32	14	0.258	8	0.241	1.418	9%	23.9	24.3
23	86	B	0:47	40	14	0.258	9	0.243	1.417	9%	23.9	24.2
23	86	B	0:55	40	12	0.221	10	0.200	1.413	10%	23.9	24.2
23	86	B	1:05	40	10	0.184	11	0.237	1.407	10%	23.9	24.1
23	86	B	1:15	40	8	0.147	12	0.185	1.401	10%	23.9	24.1
23	86	B	1:27	45	9	0.166	13	0.180	1.399	11%	23.9	24.2
23	86	B	1:41	43	7	0.129	14	0.175	1.394	11%	23.9	24.0
23	86	B	1:58	43	5	0.092	15	0.177	1.391	11%	23.9	23.9
23	86	B	2:16	44	4	0.074	16	0.237	1.388	11%	23.9	23.7

TABLE A1-24

TRIAL 24 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
24	90	A	0:00	0	0	0.000	0	2.800	1.014	0%	23.9	23.7
24	90	A	0:05	12	24	0.380	1	0.259	0.537	47%	23.9	23.7
24	90	A	0:09	10	22	0.348	2	0.224	0.809	20%	23.9	23.7
24	90	A	0:14	11	23	0.364	3	0.186	0.875	14%	23.9	23.7
24	90	A	0:19	13	23	0.364	4	0.208	0.899	11%	23.9	23.7
24	90	A	0:24	15	22	0.348	5	0.166	0.915	10%	23.9	23.7
24	90	A	0:29	17	23	0.364	6	0.162	0.920	9%	23.9	23.7
24	90	A	0:34	15	15	0.237	7	0.155	0.920	9%	23.9	23.7
24	90	A	0:39	25	25	0.395	8	0.186	0.930	8%	23.9	23.7
24	90	A	0:43	20	18	0.285	9	0.152	0.926	9%	23.9	23.7
24	90	A	0:48	39	32	0.506	10	0.154	0.929	8%	23.9	23.7
24	90	A	0:51	35	28	0.443	11	1.590	0.932	8%	23.9	23.7
24	90	A	0:55	27	20	0.316	12	0.162	0.931	8%	23.9	23.7
24	90	A	1:01	35	23	0.364	13	0.140	0.934	8%	23.9	23.7
24	90	A	1:06	30	25	0.395	14	0.155	0.933	8%	23.9	23.7
24	90	A	1:11	44	26	0.411	15	0.159	0.936	8%	23.9	23.7
24	90	A	1:16	32	21	0.332	16	0.149	0.935	8%	23.9	23.7
24	90	A	1:20	38	24	0.380	17	0.161	0.932	8%	23.9	23.7
24	90	A	1:26	40	24	0.380	18	0.148	0.933	8%	23.9	23.7
24	90	A	1:31	38	20	0.316	19	0.140	0.934	8%	23.9	23.7
24	90	A	1:37	40	18	0.285	20	0.158	0.939	7%	23.9	23.7
24	90	A	1:44	43	18	0.285	21	0.158	0.944	7%	23.9	23.7
24	90	A	1:50	45	18	0.285	22	0.145	0.940	7%	23.9	23.7
24	90	A	1:56	44	17	0.269	23	0.139	0.940	7%	23.9	23.7
24	90	A	2:03	44	16	0.253	24	0.149	0.940	7%	23.9	23.7
24	90	A	2:10	45	14	0.221	25	0.138	0.934	8%	23.9	23.7
24	90	A	2:18	45	14	0.221	26	0.134	0.936	8%	23.9	23.7
24	90	A	2:26	45	12	0.190	27	0.143	0.936	8%	23.9	23.7

24	90	A	2:36	45	11	0.174	28	0.143	0.936	8%	23.9	23.7
24	90	A	2:45	42	11	0.174	29	0.201	0.933	8%	23.9	23.7
24	90	A	2:55	45	12	0.190	30	0.141	0.932	8%	23.9	23.7

TABLE A1-25

TRIAL 25 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
25	90	A	0:00	0	0	0.000	0	8.460	1.014	0%	24.1	23.9
25	90	A	0:05	9	22	0.348	1	0.317	0.541	47%	23.9	23.9
25	90	A	0:11	9	19	0.300	2	0.266	0.798	21%	23.9	23.9
25	90	A	0:16	16	20	0.316	3	0.219	0.878	13%	23.9	23.9
25	90	A	0:21	19	22	0.348	4	0.244	0.895	12%	23.9	23.9
25	90	A	0:26	18	18	0.285	5	0.223	0.904	11%	23.9	23.9
25	90	A	0:31	28	23	0.364	6	0.173	0.917	10%	23.9	23.9
25	90	A	0:36	25	21	0.332	7	0.194	0.919	9%	23.9	23.9
25	90	A	0:41	28	20	0.316	8	0.244	0.928	9%	23.9	23.9
25	90	A	0:46	32	20	0.316	9	0.149	0.925	9%	23.9	23.9
25	90	A	0:51	36	20	0.316	10	0.148	0.929	8%	23.9	23.9
25	90	A	0:56	39	20	0.316	11	0.349	0.919	9%	23.9	23.9
25	90	A	1:01	38	13	0.206	12	0.590	0.892	12%	23.9	23.9
25	90	A	1:13	41	15	0.237	13	0.133	0.934	8%	23.9	23.9
25	90	A	1:21	45	14	0.221	14	0.127	0.930	8%	23.9	23.9
25	90	A	1:30	45	11	0.174	15	0.135	0.923	8%	23.9	23.9
25	90	A	1:41	45	9	0.142	16	0.149	0.915	9%	23.9	23.9

TABLE A1-26

TRIAL 26 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
26	90	B	0:00	11	14	0.258					23.9	24.3
26	90	B	0:04	13	16	0.294					23.7	24.3
26	90	B	0:05	12	17	0.313	1	0.761	0.774	24%	23.7	24.1
26	90	B	0:10	12	18	0.331	2	0.424	0.919	10%	23.7	23.9
26	90	B	0:15	12	18	0.322	3	0.442	0.945	8%	23.9	23.9
26	90	B	0:20	14	17	0.313	4	0.384	0.952	7%	23.9	23.9
26	90	B	0:25	15	17	0.313	5	0.356	0.959	6%	23.9	24.1
26	90	B	0:30	15	18	0.322	6	0.336	0.965	6%	23.9	23.9
26	90	B	0:35	15	17	0.304	7	0.359	0.965	6%	23.9	23.9
26	90	B	0:40	15	17	0.304	8	0.435	0.967	5%	23.9	23.9
26	90	B	0:45	16	17	0.304	9	0.265	0.966	6%	23.9	23.9
26	90	B	0:50	16	17	0.304	10	0.256	0.967	5%	23.9	23.9
26	90	B	0:55	17	17	0.304	11	0.274	0.965	6%	23.7	23.7
26	90	B	1:00	17	17	0.304	12	0.262	0.966	6%	23.7	23.7
26	90	B	1:05	18	16	0.294	13	0.215	0.968	5%	23.7	23.7
26	90	B	1:10	18	16	0.294	14	0.225	0.967	5%	23.9	23.9
26	90	B	1:15	19	16	0.294	15	0.212	0.968	5%	23.9	23.9
26	90	B	1:20	19	16	0.285	16	0.238	0.967	5%	23.9	23.9
26	90	B	1:25	20	16	0.294	17	0.218	0.968	5%	24.1	24.1
26	90	B	1:30	21	16	0.294	18	0.334	0.969	5%	23.9	23.9
26	90	B	1:35	22	17	0.304	19	0.250	0.971	5%	23.9	23.9
26	90	B	1:40	22	16	0.294	20	0.249	0.969	5%	23.9	23.9
26	90	B	1:45	21	14	0.258	21	0.167	0.974	5%	23.9	23.9
26	90	B	1:51	22	15	0.276	22	0.172	0.973	5%	23.7	23.7
26	90	B	1:56	23	16	0.285	23	0.171	0.975	5%	23.9	23.9
26	90	B	2:01	23	16	0.285	24	0.163	0.979	4%	23.7	23.7
26	90	B	2:06	23	15	0.276	25	0.171	0.974	5%	23.7	23.7

TABLE A1-27

TRIAL 27 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
27	90	B	0:00	14	19	0.350	1	2.100	1.010	36%	23.9	24.5
27	90	B	0:05	20	18	0.331	2	0.519	1.431	9%	23.9	24.5
27	90	B	0:10	22	17	0.313	3	0.321	1.463	7%	23.9	24.5
27	90	B	0:15	27	17	0.313	4	0.194	1.475	7%	23.9	24.4
27	90	B	0:21	34	16	0.294	5	0.209	1.484	6%	23.9	24.3
27	90	B	0:28	35	12	0.221	6	0.202	1.479	6%	23.9	24.2
27	90	B	0:37	40	10	0.184	7	0.162	1.475	7%	23.9	24.1
27	90	B	0:47	41	8	0.147	8	0.196	1.473	7%	23.9	24.0
27	90	B	0:59	42	8	0.147	9	0.132	1.498	5%	23.9	24.0

TABLE A1-28

TRIAL 28 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
28	90	B	0:00	11	18	0.331	1	0.910	1.456	12%	23.9	24.7
28	90	B	0:05	11	18	0.331	2	0.270	1.595	4%	23.9	24.8
28	90	B	0:11	11	15	0.276	3	0.195	1.627	2%	23.9	24.7
28	90	B	0:17	15	18	0.331	4	0.166	1.630	1%	23.9	24.8
28	90	B	0:22	17	19	0.350	5	0.167	1.639	1%	23.9	24.9
28	90	B	0:27	18	17	0.313	6	0.160	1.638	1%	23.9	25.0
28	90	B	0:32	20	17	0.313	7	0.202	1.645	1%	23.9	24.9

TABLE A1-29

TRIAL 29 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
29	90	B	0:00	11	18	0.331	1	2.080	1.464	11%	23.9	24.1
29	90	B	0:05	13	17	0.313	2	0.821	1.568	5%	23.9	24.4
29	90	B	0:10	15	17	0.313	3	0.438	1.577	5%	23.9	24.3
29	90	B	0:15	18	17	0.313	4	0.235	1.585	4%	23.9	24.4
29	90	B	0:21	21	17	0.313	5	0.233	1.587	4%	23.9	24.3
29	90	B	0:26	23	17	0.313	6	0.206	1.590	4%	23.9	24.3
29	90	B	0:31	27	16	0.294	7	0.234	1.590	4%	23.9	24.3

TABLE A1-30

TRIAL 30 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
30	94	A	0:00	0	0	0.000	0	1.040	1.064	0%	24.1	23.7
30	94	A	0:05	12	26	0.411	1	0.869	0.549	48%	24.1	23.7
30	94	A	0:09	10	20	0.316	2	0.155	0.844	21%	24.1	23.7
30	94	A	0:15	14	23	0.364	3	0.150	0.925	13%	24.1	23.7
30	94	A	0:20	15	22	0.348	4	0.155	0.950	11%	24.1	23.7
30	94	A	0:25	15	20	0.316	5	0.138	0.961	10%	24.1	23.7
30	94	A	0:30	17	20	0.316	6	0.136	0.972	9%	24.1	23.7
30	94	A	0:35	20	21	0.332	7	0.139	0.979	8%	24.1	23.7
30	94	A	0:40	24	23	0.364	8	0.133	0.983	8%	24.1	23.7
30	94	A	0:45	26	22	0.348	9	0.136	0.986	7%	24.1	23.7
30	94	A	0:50	25	20	0.316	10	0.147	0.986	7%	24.1	23.7
30	94	A	0:55	25	19	0.300	11	0.145	0.989	7%	24.1	23.7
30	94	A	1:00	33	23	0.364	12	0.143	0.991	7%	24.1	23.7
30	94	A	1:05	30	20	0.316	13	0.127	0.991	7%	23.9	23.7
30	94	A	1:10	34	21	0.332	14	0.134	0.993	7%	23.9	23.7
30	94	A	1:15	35	21	0.332	15	0.126	0.994	7%	23.9	23.7
30	94	A	1:20	33	18	0.285	16	0.134	0.995	7%	23.9	23.7
30	94	A	1:25	42	22	0.348	17	0.122	0.995	7%	23.9	23.7
30	94	A	1:31	44	20	0.316	18	0.130	0.998	7%	23.9	23.7
30	94	A	1:36	40	20	0.316	19	0.129	1.006	6%	23.9	23.7
30	94	A	1:42	36	17	0.269	20	0.136	1.006	6%	23.9	23.7
30	94	A	1:48	44	21	0.332	21	0.142	1.014	6%	23.9	23.7
30	94	A	1:54	41	19	0.300	22	0.153	1.014	5%	23.9	23.7
30	94	A	2:00	43	18	0.285	23	0.124	1.013	5%	23.9	23.7
30	94	A	2:06	45	18	0.285	24	0.125	1.014	5%	24.1	23.7
30	94	A	2:12	43	17	0.269	25	0.138	1.012	5%	24.1	23.7
30	94	A	2:19	45	17	0.269	26	0.127	1.013	5%	24.1	23.7
30	94	A	2:25	45	15	0.237	27	0.136	1.013	5%	24.1	23.7

30	94	A	2:35	45	12	0.190	28	0.144	1.011	5%	24.1	23.7
30	94	A	2:45	42	13	0.206	29	0.127	1.012	5%	24.1	23.7
30	94	A	2:55	40	13	0.206	30	0.132	1.011	5%	24.1	23.7
30	94	A	3:08	40	10	0.158	31	0.143	1.008	5%	24.1	23.7

TABLE A1-31

TRIAL 31 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
31	94	A	0:00	0	0	0.000	0	2.480	1.071	0%	23.9	23.7
31	94	A	0:05	10	22	0.348	1	1.030	0.604	44%	23.9	23.7
31	94	A	0:10	9	18	0.285	2	0.335	0.869	19%	23.9	23.7
31	94	A	0:15	13	21	0.332	3	0.282	0.950	11%	23.9	23.7
31	94	A	0:20	15	22	0.348	4	0.219	0.973	9%	23.9	23.7
31	94	A	0:25	16	20	0.316	5	0.229	0.983	8%	23.9	23.7
31	94	A	0:30	17	20	0.316	6	0.179	0.990	8%	23.9	23.7
31	94	A	0:35	20	21	0.332	7	0.167	0.995	7%	23.9	23.7
31	94	A	0:40	23	20	0.316	8	0.161	1.000	7%	23.9	23.7
31	94	A	0:45	25	20	0.316	9	0.155	1.005	6%	23.9	23.7
31	94	A	0:50	28	20	0.316	10	0.151	1.008	6%	23.9	23.7
31	94	A	0:55	30	20	0.316	11	0.153	1.010	6%	23.9	23.7
31	94	A	1:02	33	21	0.332	12	0.151	1.009	6%	23.9	23.7
31	94	A	1:07	36	21	0.332	13	0.149	1.012	6%	23.9	23.7
31	94	A	1:12	38	20	0.316	14	0.143	1.012	6%	23.9	23.7
31	94	A	1:17	41	20	0.316	15	0.143	1.014	6%	24.1	23.7
31	94	A	1:22	45	21	0.332	16	0.148	1.016	5%	24.1	23.7
31	94	A	1:27	38	16	0.253	17	0.143	1.012	5%	24.1	23.7
31	94	A	1:34	45	18	0.285	18	0.150	1.017	6%	24.1	23.7
31	94	A	1:40	37	16	0.253	19	0.153	1.019	5%	24.1	23.7
31	94	A	1:47	45	18	0.285	20	0.153	1.017	5%	24.1	23.7
31	94	A	1:54	43	15	0.237	21	0.135	1.016	5%	24.1	23.7
31	94	A	2:04	43	15	0.237	22	0.140	1.016	5%	24.1	23.7
31	94	A	2:14	45	15	0.237	23	0.137	1.015	5%	24.1	23.7
31	94	A	2:24	45	14	0.221	24	0.139	1.015	5%	24.1	23.7
31	94	A	2:33	45	13	0.206	25	0.155	1.012	5%	24.1	23.7
31	94	A	2:44	45	13	0.206	26	0.158	1.012	6%	24.1	23.7
31	94	A	2:54	45	12	0.190	27	0.154	1.010	6%	24.1	23.7

31	94	A	3:00	43	11	0.174	28	0.157	1.010	6%	24.1	23.7
31	94	A	3:15	45	11	0.174	29	0.163	1.010	6%	24.1	23.7
31	94	A	3:30	45	11	0.174	30	0.414	1.010	6%	24.1	23.7

TABLE A1-32

TRIAL 32 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
32	94	B	0:00	8	15	0.276	1	0.597	1.500	10%	23.9	24.3
32	94	B	0:06	11	18	0.331	2	0.310	1.628	2%	23.9	24.5
32	94	B	0:11	11	17	0.313	3	0.211	1.638	2%	23.9	24.4
32	94	B	0:16	13	17	0.313	4	0.165	1.621	3%	23.9	24.5
32	94	B	0:22	15	17	0.313	5	0.153	1.626	3%	23.9	24.5
32	94	B	0:27	19	18	0.331	6	0.151	1.626	3%	23.9	24.5
32	94	B	0:32	21	18	0.331	7	0.139	1.621	3%	23.9	24.4

TABLE A1-33

TRIAL 33 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
33	94	B	0:00	11	20	0.368	1	0.605	1.484	12%	24.3	24.5
33	94	B	0:05	10	17	0.313	2	0.221	1.628	3%	24.3	24.5
33	94	B	0:10	11	18	0.331	3	0.151	1.670	1%	24.3	24.4
33	94	B	0:16	12	17	0.313	4	0.132	1.687	0%	24.3	24.3
33	94	B	0:21	14	17	0.313	5	0.129	1.696	-1%	24.3	24.4
33	94	B	0:27	15	17	0.313	6	0.130	1.707	-1%	24.3	24.4
33	94	B	0:32	16	17	0.313	7	0.134	1.724	-2%	24.3	24.4

TABLE A1-34

TRIAL 34 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
34	94	B	0:00	10	16	0.294	1	2.100	1.574	6%	24.3	24.4
34	94	B	0:06	12	18	0.331	2	0.435	1.676	0%	24.3	24.4
34	94	B	0:11	12	18	0.331	3	0.248	1.684	0%	24.3	24.4
34	94	B	0:16	14	18	0.331	4	0.198	1.689	0%	24.3	24.4
34	94	B	0:21	17	17	0.313	5	0.170	1.687	0%	24.3	24.4
34	94	B	0:26	19	17	0.313	6	0.148	1.689	0%	24.3	24.4
34	94	B	0:31	20	17	0.313	7	0.171	1.697	-1%	24.3	24.4

TABLE A1-35

TRIAL 35 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
35	100	A	0:00	0	0	0.000	0	1.370	1.133	0%	23.9	23.7
35	100	A	0:05	10	21	0.332	1	1.370	1.133	42%	24.3	23.7
35	100	A	0:10	10	19	0.301	2	0.516	0.661	13%	24.3	23.7
35	100	A	0:15	11	20	0.316	3	0.227	0.985	7%	24.3	23.7
35	100	A	0:20	13	20	0.316	4	0.207	1.049	6%	24.3	23.7
35	100	A	0:25	15	20	0.316	5	0.173	1.064	5%	24.3	23.7
35	100	A	0:30	16	20	0.316	6	0.154	1.075	5%	24.3	23.7
35	100	A	0:35	18	20	0.316	7	0.144	1.082	4%	24.3	23.7
35	100	A	0:40	20	20	0.316	8	0.154	1.086	4%	24.3	23.7
35	100	A	0:45	22	20	0.316	9	0.147	1.085	4%	24.3	23.7
35	100	A	0:50	24	20	0.316	10	0.149	1.093	3%	24.1	23.7
35	100	A	0:55	26	21	0.324	11	0.142	1.098	3%	24.1	23.7
35	100	A	1:01	28	20	0.316	12	0.140	1.097	3%	24.1	23.7
35	100	A	1:05	30	20	0.316	13	0.144	1.097	3%	24.1	23.7
35	100	A	1:12	31	22	0.340	14	0.143	1.099	3%	24.1	23.7
35	100	A	1:18	29	19	0.301	15	0.110	1.101	3%	24.1	23.7
35	100	A	1:24	35	22	0.340	16	0.152	1.102	3%	24.1	23.7
35	100	A	1:28	33	20	0.308	17	0.138	1.101	3%	23.9	23.7
35	100	A	1:35	35	20	0.308	18	0.141	1.100	3%	23.9	23.7
35	100	A	1:40	37	20	0.316	19	0.142	1.102	3%	23.9	23.7
35	100	A	1:45	39	20	0.316	20	0.144	1.099	3%	23.9	23.7
35	100	A	1:44	41	20	0.316	21	0.161	1.104	3%	23.9	23.7
35	100	A	2:00	43	22	0.340	22	0.143	1.103	3%	23.9	23.7
35	100	A	2:09	41	20	0.308	23	0.145	1.103	2%	23.9	23.7
35	100	A	2:15	44	20	0.316	24	0.155	1.106	3%	23.9	23.7
35	100	A	2:25	44	18	0.285	25	0.156	1.102	2%	23.9	23.7
35	100	A	2:35	45	20	0.316	26	0.283	1.106	3%	23.9	23.7
35	100	A	2:45	45	20	0.308	27	0.156	1.103	3%	23.9	23.7

TABLE A1-36

TRIAL 36 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
36	100	A	0:00	0	0	0.000	0	1.370	1.133	0%	24.3	23.9
36	100	A	0:05	10	21	0.332	1	0.516	0.661	42%	24.3	23.9
36	100	A	0:10	10	19	0.301	2	0.227	0.985	13%	24.4	24.1
36	100	A	0:15	11	20	0.316	3	0.207	1.049	7%	24.4	24.1
36	100	A	0:20	13	20	0.316	4	0.173	1.064	6%	24.4	24.1
36	100	A	0:25	15	20	0.316	5	0.154	1.075	5%	24.4	24.1
36	100	A	0:30	16	20	0.316	6	0.144	1.082	5%	24.4	24.1
36	100	A	0:35	18	20	0.316	7	0.154	1.086	4%	24.4	24.1
36	100	A	0:40	20	20	0.316	8	0.147	1.085	4%	24.4	24.1
36	100	A	0:45	22	20	0.316	9	0.149	1.093	4%	24.4	24.1
36	100	A	0:51	24	20	0.316	10	0.142	1.098	3%	24.4	24.1
36	100	A	0:56	26	21	0.324	11	0.140	1.097	3%	24.4	24.1
36	100	A	1:02	28	20	0.316	12	0.144	1.097	3%	24.4	24.1
36	100	A	1:08	30	20	0.316	13	0.143	1.099	3%	24.4	24.1
36	100	A	1:13	31	22	0.340	14	0.110	1.101	3%	24.4	24.1
36	100	A	1:18	29	19	0.301	15	0.152	1.102	3%	24.4	24.1
36	100	A	1:23	35	22	0.340	16	0.138	1.101	3%	24.4	24.1
36	100	A	1:28	33	20	0.308	17	0.141	1.100	3%	24.4	24.1
36	100	A	1:33	35	20	0.308	18	0.142	1.102	3%	24.4	24.1
36	100	A	1:38	37	20	0.316	19	0.144	1.099	3%	24.4	24.1
36	100	A	1:43	39	20	0.316	20	0.161	1.104	3%	24.4	24.1
36	100	A	1:48	41	20	0.316	21	0.143	1.103	3%	24.4	24.1
36	100	A	1:53	43	22	0.340	22	0.145	1.103	3%	24.4	24.1
36	100	A	2:03	41	20	0.308	23	0.155	1.106	2%	24.4	24.1
36	100	A	2:13	44	20	0.316	24	0.156	1.102	3%	24.4	24.1
36	100	A	2:23	44	18	0.285	25	0.283	1.106	2%	24.4	24.1
36	100	A	2:33	45	20	0.316	26	0.156	1.103	3%	24.4	24.1
36	100	A	2:42	45	20	0.308	27	0.338	1.103	3%	24.4	24.1

TABLE A1-37

TRIAL 37 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
37	100	B	0:00	3	7	0.129					24.4	24.6
37	100	B	0:05	12	18	0.331	1	0.932	1.062	22%	24.6	24.6
37	100	B	0:10	10	16	0.294	2	0.607	1.276	7%	24.6	24.6
37	100	B	0:15	12	16	0.294	3	0.257	1.310	4%	24.6	24.6
37	100	B	0:20	13	17	0.304	4	0.180	1.330	3%	24.6	24.6
37	100	B	0:25	15	17	0.304	5	0.235	1.340	2%	24.6	24.6
37	100	B	0:30	16	17	0.313	6	0.185	1.348	2%	24.6	24.6
37	100	B	0:35	20	18	0.322	7	0.147	1.338	2%	24.6	24.6
37	100	B	0:40	21	18	0.331	8	0.142	1.338	2%	24.6	24.6
37	100	B	0:45	21	17	0.304	9	0.140	1.348	2%	24.6	24.6
37	100	B	0:50	23	17	0.304	10	0.138	1.348	2%	24.6	24.6
37	100	B	0:55	25	17	0.304	11	0.142	1.348	2%	24.6	24.6
37	100	B	1:00	27	18	0.322	12	0.138	1.348	2%	24.6	24.6
37	100	B	1:05	28	17	0.304	13	0.150	1.346	2%	24.6	24.6
37	100	B	1:10	30	17	0.304	14	0.137	1.346	2%	24.6	24.6
37	100	B	1:15	31	17	0.304	15	0.146	1.348	2%	24.6	24.6
37	100	B	1:20	33	17	0.304	16	0.161	1.346	2%	24.6	24.6

TABLE A1-38

TRIAL 38 - EXPERIMENTAL FILTRATION DATA FOR CHILL FILTRATION FEASIBILITY TESTING

Trial No.	Sample Proof	Pad	Time	Pressure, psig	Flow, mL/min	Flux, gpm/ft ²	Sample No.	Turbidity NTU	Color, Abs	Color Loss, %	Glycol T, °F	Whiskey T, °F
38	100	B	0:00	4	13	0.239					24.4	24.4
38	100	B	0:05	10	18	0.331	1	0.797	1.251	9%	24.4	24.4
38	100	B	0:10	9	17	0.313	2	0.337	1.358	2%	24.6	24.6
38	100	B	0:15	10	17	0.304	3	0.151	1.364	1%	24.6	24.6
38	100	B	0:20	11	17	0.304	4	0.156	1.373	1%	24.6	24.6
38	100	B	0:25	13	17	0.313	5	0.145	1.377	0%	24.6	24.6
38	100	B	0:30	14	17	0.313	6	0.189	1.382	0%	24.6	24.6
38	100	B	0:35	16	17	0.313	7	0.162	1.379	0%	24.6	24.6
38	100	B	0:40	17	17	0.313	8	0.230	1.380	0%	24.6	24.6
38	100	B	0:45	19	17	0.313	9	0.160	1.379	0%	24.6	24.6
38	100	B	0:50	20	17	0.313	10	0.135	1.374	0%	24.6	24.6
38	100	B	0:55	22	17	0.313	11	0.131	1.375	0%	24.6	24.6
38	100	B	1:00	23	17	0.313	12	0.134	1.378	0%	24.6	24.6
38	100	B	1:05	25	17	0.313	13	0.135	1.378	0%	24.6	24.6
38	100	B	1:10	27	17	0.313	14	0.133	1.378	0%	24.6	24.6
38	100	B	1:15	29	17	0.313	15	0.149	1.376	0%	24.6	24.6
38	100	B	1:20	31	18	0.322	16	0.160	1.377	0%	24.6	24.6
38	100	B	1:25	33	18	0.322	17	0.148	1.374	0%	24.6	24.6

APPENDIX 2: STABILITY DATA

TABLE A2-1

STABILITY REPORT OF CHILL FILTRATION TRIALS AT 80 PROOF
(n= 19 SAMPLES)

Trial	Batch	Proof	SE Initial	SE Final	Comments
1	2	80	1	2	
1	15	80	0	0	
2	2	80	0	2	
2	16	80	0	0	
3	3	80	0	1	
3	16	80	0	0	
4	1	80	1	2	
4	15	80	0	0	
5	3	80	0	1	
5	10	80	0	0	
6	3	80	0	1	
6	14	80	0	0	
7	2	80	0	1	
7	8	80	1	1	
8	3	80	0	1	
9	2	80	0	2	
10	5	80	0	1	

TABLE A2-2

STABILITY REPORT OF CHILL FILTRATION TRIALS AT 86 PROOF
(n= 6 SAMPLES)

Trial	Batch	Proof	SE Initial	SE Final	Comments
12	5	86	1	3	
14	6	86	0	2	
16	5	86	0	2	
18	4	86	0	3	
20	3	86	2	3	Heavy wisps
22	5	86	0	1	

TABLE A2-3

STABILITY REPORT OF CHILL FILTRATION TRIALS AT 90 PROOF
(n= 11 SAMPLES)

Trial	Batch	Proof	SE Initial	SE Final	Comments
24	3	90	0	2	
24	17	90	0	0	
25	2	90	1	1	
25	12	90	0	0	
26	2	90	0	2	
26	20	90	0	0	
27	3	90	0	1	
27	6	90	0	1	
28	4	90	0	1	
29	2	90	0	2	Light wisp
29	7	90	0	1	

TABLE A2-4

STABILITY REPORT OF CHILL FILTRATION TRIALS AT 94 PROOF
(n= 44 SAMPLES)

Trial	Batch	Proof	SE Initial	SE Final	Comments
30	2	94	0	2	
30	4	94	0	2	
30	6	94	0	2	
30	8	94	0	1	
30	10	94	0	1	
30	12	94	0	0	
30	14	94	0	0	
30	16	94	0	0	
30	18	94	0	0	
30	20	94	0	0	
30	22	94	0	0	
30	24	94	0	2	
30	26	94	0	0	
30	28	94	0	0	
30	29	94	0	0	
30	30	94	0	0	
31	1	94	1	3	Heavy wisp
31	3	94	0	2	
31	5	94	0	1	
31	7	94	0	0	
31	9	94	0	1	
31	11	94	0	0	
31	13	94	0	0	
31	15	94	0	0	
31	17	94	0	1	
31	19	94	0	1	
31	21	94	0	2	
31	23	94	0	0	
31	25	94	0	0	
31	27	94	0	1	
31	29	94	0	1	
32	2	94	0	1	
32	3	94	0	1	
32	4	94	0	1	
32	5	94	0	1	
33	2	94	0	3	
33	3	94	0	1	

33	4	94	0	0	
33	5	94	0	1	
33	6	94	0	1	
34	2	94	1	3	Heavy wisp
34	3	94	0	1	
34	4	94	0	2	
34	5	94	0	0	

TABLE A2-5

STABILITY REPORT OF CHILL FILTRATION TRIALS AT 100 PROOF
(n= 17 SAMPLES)

Trial	Batch	Proof	SE Initial	SE Final	Comments
35	2	100	0	1	
35	5	100	0	0	
35	8	100	0	0	
35	11	100	0	0	
36	2	100	0	0	
36	5	100	0	0	
36	8	100	0	0	
36	11	100	0	0	
37	2	100	0	1	
37	5	100	0	0	
37	8	100	0	0	
37	11	100	0	0	
38	2	100	0	1	
38	4	100	0	0	
38	6	100	0	0	
38	8	100	0	0	
38	10	100	0	0	

APPENDIX 3: SAMPLE CALCULATION

The following sample calculation is for determining carbon added to a sample:

Data Needed

Proof of Sample	94 proof
Volume of sample	100 mL
Carbon treatment required	1oz/100PG

Calculate Proof Gallons in Sample (PGs)

Use percent proof (Proof/100) and convert units to gallons:

$$\text{PG in Sample} = 100 \text{ mL} \times \frac{94 \text{ proof}}{100 \text{ gal}} \times \frac{1 \text{ gal}}{3785.41 \text{ mL}} = 0.025 \text{ PGs}$$

Calculate Amount of Activated Carbon Needed (grams)

Multiply proof gallons by treatment and convert to grams:

$$\text{Carbon}_{add} = 0.025 \text{ PGs} \times \frac{1 \text{ oz}}{100 \text{ PG}} \times \frac{28.3495 \text{ g}}{1 \text{ oz}} = 0.007 \text{ grams}$$

The following sample calculation is to determine color loss of a sample:

Data Needed

Initial Absorbance at 430nm	1.619 A
Absorbance at 430nm after Filtration	1.425 A

Calculate Percent Color Loss

Find Color difference between initial and final color:

$$\text{Color Difference} = 1.619 - 1.425 = 0.194$$

Divide color difference by initial color and multiply by 100:

$$\% \text{ Color Loss} = \frac{0.194}{1.619} \times 100 = 12 \%$$

The following sample calculation is to determine flux ratio:

	<u>Data Needed</u>
Radius of Filter	0.85 in
Instant Filtration flow	17 mL/min

Calculate Flux Ratio (gpm/ft²)

Calculate area of the filter (circular area) in ft²:

$$Filter\ Area = \pi \times \left(0.85\ in \times \frac{1\ ft}{12\ in}\right)^2 = 0.016\ ft^2$$

Convert flow to gal/min:

$$Flow = 17 \frac{mL}{min} \times \frac{1\ gal}{3785.41\ mL} = 0.0045\ gal/min$$

Divide flow rate by surface area:

$$Flux = \frac{0.0045\ gal/min}{0.016\ ft^2} = 0.285\ gpm/ft^2$$

REFERENCES

- Alexander, J. 1945. *Colloid Chemistry: Principles and Applications*. New York: D. Van Nostrand Company.
- Brown, H., Banshin, A., and Forsaith, C. 1949. *Textbook of Wood Technology*. New York: McGraw-Hill.
- Electronic Code of Federal Regulations (ECFR), "Title 27: Alcohol, Tobacco Product and Firearms, Part 5 – Labeling and Advertising of Distilled Spirits, Subpart C – Standards of Identity for Distilled Spirits", February 2015, available from <http://www.ecfr.gov/cgi-bin/text-idx?c=ecfr;sid=5ea7acdd54dff93dbce97e668013dfd8;rgn=div8;view=text;node=27%3A1.0.1.1.3.3.25.2;idno=27;cc=ecfr>; accessed January 15, 2015.
- Fairmount Minerals. "Wedron Silica Co: Tech Data Sheet." *Fairmount Minerals*. May 2004, available from <http://www.fairmountminerals.com>; accessed March 1, 2011.
- Fruton, J. Molecules and Life: Historical Essays on the Interplay of Chemistry and Biology. New York: Wiley-Interscience, 1972.
- Icheme Food and Drink Subject Group." Membrane Filtration of Whisky." Food and Drink 2000: Processing Solutions for Innovative Products. Jan. 2000: 9. London, 2000.
- Maga, Joseph. The Contribution of Wood to the Flavor of Alcoholic Beverages. Fort Collins: Taylor and Francis, 2009.
- McGuire, M., and Suffet, I., eds. Treatment of Water by Granular Activated Carbon. Washington D.C.: American Chemical Society, 1983.
- Merizalde Carrillo, J. 2008. B3 Pilot Plant Laboratory Notebook No. 212. Brown-Forman Corporation: Process, Research and Development Department.
- Nishimura, K., et. al. Flavors of Distilled Beverages. London: Horwood Ellis/SCI, 1983.
- Parham, R., and Gray, R. The Chemistry of Solid Wood. Washington D.C.: American Chemical Society, 1984.

Prausnitz, J., Lichtenthaler, R., and Azavedo, E. Molecular Thermodynamics of Fluid-Phase Equilibria. New Jersey: Prentice Hall PTR, 1999.

Singleton, V. Chemistry of Winemaking. Washington D.C.: American Chemical Society, 1974.

Swan, J. Maturation of Potable Spirits. Edinburgh: Pentlands Scotch Whisky Research Ltd., 1986.

Wisniewski, I. "The Chill Factor." Whisky Production Aug. 2003: 64. Whisky Magazine. Brown-Forman Library, Louisville, KY. 2 Dec. 2009 <<http://www.whiskymag.com/magazine/issue33/12005487.html>>.

Yusa, Makoto. A Study of the Density and Viscosity of Water and Ethanol Mixtures Under High Pressure. Ontario: University of Windsor, 1973.

Zimlich, Joseph. Personal Interview. 20 August 2008.

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