Analysis of Smoak Tannins

A Project Report for E&J Gallo Winery and Worcester Polytechnic Institute

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April 4, 2017

Table of Contents

List of Tables

List of Figures

List of Equations

Acknowledgements

Thank you to the two project advisors Nikolaos Kazantzis and David DiBiasio for their encouragement and feedback during this project. Another massive thank you to Alex Kopchik and Ravi Ponangi, the on-site project advisors for helping out with research, calculations, and brainstorming ideas. Finally, a massive thank you to each and every single one of the Process Technology employees at E&J Gallo for their willingness to help run experiments and show their support for this project.

Abstract

E&J Gallo (Gallo) currently has a large waste stream that is a by-product of their oak toasting process in Livingston, CA that is used for flavor production. These materials are currently being stored and contain components that are of interest to the company. Because the process comes from oak, Gallo would like to use this existing waste stream to deliver a new tannin product differentiation tool. The goal of this project was to develop various isolation and extraction processes to separate the smoky flavors in the waste stream. In order to do this, the concentrations were first increased through pressure cooking and then exposed to five different separation/extraction processes: liquid-liquid extraction, column chromatography, batch distillation, steam stripping, and evaporation. Once the ideal tannins were extracted, the final step was to dose and spike wines to come up with the right flavor profile. While there were some results that were not conclusive, there was a positive outcome from other trials that will allow the company to create new aroma notes that may increase commercial sales and save the company money by removing the need to process the waste stream.

Executive Summary

This report was a cumulative effort between Worcester Polytechnic Institute and E&J Gallo Wineries. This project examined the analysis of smoky oak tannins (“Smoak”) in a large waste stream. The purpose of this project was to use this waste stream to deliver a new smoke tannin product differentiation tool for the company. Analysis of different compounds of interest in each solution was conducted primarily through three processes: increasing the production of methoxyphenols through cooking, various extraction/separations techniques, and dosing the extracted tannins in wine to create new aroma notes. The extraction/separation techniques used in this project were liquid-liquid extraction (LLE), batch distillation, steam stripping, and column chromatography. The compounds of focus were guaiacol and syringol. Guaiaicol is known for its smoky aroma whereas syringol is known for its mouth-feel and taste. Vanillin and other tannins are also included in the data for Gallo’s future interest. Unfortunately, Gallo was unable to obtain the waste stream of interest for this project, the Extracted Livingston Tannin (ELT-03) stream and so commercial tannins were used instead.

The major focus of this project involved the analysis of methoxyphenol production through cooking. Two different types of trials were conducted: one where total suspended solids (TSS) were varied and one where potassium carbonate was used to adjust the pre-cook pH. In total, thirteen trials were conducted. The results with varying TSS show that the post-cook pH decreased more with increasing oak chips (the solids), however this trend was not necessarily true for syringol and guaiacol production. The ideal TSS was found to be five grams of oak. With the use of potassium carbonate as the alkaline base, the results showed that the methoxyphenol concentrations increased 3-fold, giving promising results for separation.

Another focus involved the separation and extraction of guaiacol and syringol. The methods used—batch distillation, steam stripping, and LLE—yielded mediocre separation results. An attempt was made to simulate the distillation and extraction by using AspenPlus, a simulation software, but due to the non-ideal nature of the solution, the system was not obtaining real world results. For batch distillation and steam stripping, the separation was as expected with guaiacol coming off the condensate, and syringol concentrating in the residual/bottoms. In order to improve separation, Gallo should look into a sequential distillation process that first utilizes steam stripping and then batch distillation. Because there was more guaiacol in the bottom during steam stripping, Gallo should attempt sequential distillation to see the separation potential. LLE results essentially showed the entire feed matrix coming out with the solvent stream, indicating that a five stage extraction may be too high. It would be interesting for Gallo to do a simple one stage extraction to get a better idea of the separation in the raffinate and extract streams.

Overall, the best results came from column chromatography elutions. With column chromatography, the second pass elution showed promising results, both aromatically and analytically. There reached a point where the aroma was predominately smoky and smelled like pure guaiacol, of which analytical results showed high concentrations. The highest concentration of guaiacol and syringol in both elutions occurred around 40-60% elution, which is an economic advantage for Gallo. This is because high proof ethanol is costly, so with the knowledge that elution results are optimal at 40-60%, higher concentrations of ethanol do not need to be used for elution – thus saving the company money. Though the elution results were positive, the column adsorption results were poor, but this may be due to the increased number of tannins present in the feed, thus leaving less room for the guaiacol and syringol to adsorb onto the FPX-66 resin.

The final component of this project examined spiking wine with the eluted samples from column chromatography. With wine dosing, the best aroma/flavor was achieved at approximately

43 ppb of guaiacol, which allows the company to dial back on their 75 ppb guaiacol goal in each sample. This is a promising result that can be further expanded through more taste and aroma trials that will allow Gallo to come up with new aroma notes.

Disclaimer: Due to the similarities with the parallel 2017 Gallo MQP, there are components of this report that are shared in both papers, predominantly in the sections regarding background and steam stripping/distillation. There are also similarities to the 2014 Gallo MQP in terms of background information for Gallo-specific processes.

Background

History of Winemaking

Wine has been a part of human history since before civilization. According to paleontologists, some form of wine has been around for more than 100,000 years. From Arabs in early Mesopotamia, Egyptian pharaohs, and the ancients Greeks, all the way to today's society, humans throughout history have been consuming wine (Vine et al., 2002). Despite its long history, Christianity is considered by some as the reason that wine has become the developed industry it is today. Christ offering his cup at the Last Supper motivated monks to plant vineyards wherever they settled during the Middle Ages and the Renaissance, and this movement lasted into the New World, with the development of wines in both New York and California, but it wasn't until 1860 with Pasteur's discoveries that people understood the process behind fermentation (Vine et al., 2002). However, the mid 1800s saw an infestation of Black Rot fungus, which caused a lot of discouragement to winemakers. To battle the fungus, a disease-resistant vine was produced in 1854, after 7 years of testing. The cost was $5 per vine, which was an incredibly high price at the time, but the disease-resistant vines brought back the enthusiasm of the winemakers after the fungus epidemic. At the same time, vines from American vineyards were taken to Europe to help combat the *Phylloxera* root louse that was destroying European grapevines. As a result, there are still classic vines in European countries, such as France, that are still grafted on the American roots (Vine et al., 2002).

In addition to the problems caused by rot and fungus, the prohibition movement, started in 1846 in Maine, caused significant damage to the wine industry, and resulted in the burning of much of the recorded history of American wine. The prohibitionists worked to strike the word wine from the Bible, claiming it was actually “grape juice,” and tried to ban Greek and Roman classic literature that mentioned wine. While some winemakers got around the Volstead Act, which approved prohibition across America, by marketing their wine as medicinal, sacramental, or as cooking wines, many others were forced to shut down their businesses (Vine et al., 2002). Despite prohibition, home winemaking became legal, and allowed each household to produce up to 200 gallons of wine per year for household consumption. Regardless, some winemakers chose to continue production illegally, and became bootleggers, which then became a huge industry for gangster networks, who could get around law enforcement. Finally, in December of 1933, the Repeal came into effect, and ended the continued destruction to the wine industry in America (Vine et al., 2002). Since the end of prohibition, the wine industry has boomed.

Today, it is estimated that approximately 1 in every 100 people works in the wine industry, whether at the vineyard, the winery, or in the wine marketing sector. Additionally, it is estimated that the French and the Italians consume about 16 gallons of wine per person per year, as opposed to the 2 gallons per person per year in the United States (Vine et al., 2002). As a whole, the wine trade has maintained a sense of stability and proved itself as a lasting industry.

Process of Winemaking

Wine is produced through a chemical reaction called fermentation, which uses the yeast on the grape skins and, if desired, added yeast to create an alcoholic solution from the grapes’ juices. The glucose from the grapes reacts with the yeast to form both ethanol and carbon dioxide, as shown in Equation 1 (Boulton et al., 2002).

Equation 1: Formation of Alcohol from Sugar and Yeast

$$C6H12O6 + \text{Yeast} \rightarrow 2C2H5OH + 2CO2$$

The process begins with collection during the harvest. Afterwards, the grapes are exposed to approximately 30-100 ppm sulfur dioxide to kill and prevent any further growth of unwanted bacteria on the grapes before undergoing fermentation (Boulton et al. 2002 and Vine et al., 2002). Dry wines can be protected from the bacteria with the low end of 30 ppm sulfur dioxide while wines that have residual fermentable sugar are protected by 100 ppm or more sulfur dioxide (Vine et al., 2002) Finally, the wine is clarified before it is distributed to customers (Boulton et al., 2002).

While the general fermentation processes between red and white wine are essentially the same, the initial process differs. Red wine gets its color from the tannins in grapes (tannins are a material naturally in plants that precipitate protein), so the juice is allowed to stay in contact with the skins during fermentation. White wines, on the other hand, are made by pressing the grapes as quickly as possible and removing the skins, thus reducing contact time. The lower contact time gives less time for the color and tannins to seep, giving a whiter color and different flavor. Additionally, red wines must mature, while maturation is less important for white wines (The Difference Between Red and White, n.d.).

One of the most important steps of the winemaking process involves the sensory component, generally completed during mixing. For example, red wines are aged in oak barrels so that the oak tannins can be extracted. The oak tannins create a smoky flavor in the wine (Kopchik, 2016 and Vine, 2002). Other factors that play into the overall flavor of the wine are sweetness, acidity, body, and fruit. Sweetness is controlled by how much sugar is left unfermented in the wine, while acidity is a measure of tartness. As a result, a balance between acidity and sweetness must be achieved to obtain an optimal flavor (Vine, 2002). The body of a wine is similar to the mouth feel, or how the wine feels in the mouth, whether heavy or light. Finally, the fruit flavor in the wine has a huge impact on how the wine is perceived by tasters. The intensity of the fruit flavor varies with the alcohol content; the pH; and the aging material, as some wines are aged in oak barrels while others are aged in steel containers (Vine, 2002).

Sensory Evaluation

While the sensory components of wine are adjusted during the winemaking process, there are several different aspects that affect the tasting process. Some of these aspects include the physiological standings of the taster—hunger, fatigue, ability to smell and taste, et cetera (LaMar, 2001). Others involve both the shape and size of the wine glass, as different sizes affect the collection of wine volatiles (Mansfield, 2010). The combination of these attributes, along with a tasting process, allow a person to thoroughly experience and taste a wine.

The tasting process includes four elements: smell, taste, feeling, and seeing. A person's sense of smell is about 1,000 times more sensitive than their sense of taste, and as a result, smell is one of the most important elements in wine tasting. In fact, the flavor a person tastes is greatly influenced by smell, with about 75% of the flavor coming from smell, or olfaction, and the other 25% coming from taste, or gustation. With these facts in mind, the first step of the tasting process is to swirl the wine in the glass and smell the aroma. Doing this can give the taster an indication of the alcohol level in the wine. If the smell is similar to rubbing alcohol, the alcohol content is too high for the type of wine. Additionally, if upon tasting, the wine as a hot sensation, it is possible again that the alcohol content is high. However, if the wine "feels thin and unsatisfying on the palate," then the alcohol content is on the low side (5 Key Wine Components, 2012).

The combination of smelling, tasting, feeling, and seeing lead to a taste process that is thorough and thoughtful. To start tasting, about one-third or less of a wine glass should be filled, as this allows the vapors to stay in the glass. The wine should then be swirled in the glass to bring some of the vapor molecules into the air and increase the surface area for vapor to move. Next, one should smell the wine to play to the olfactory senses. This prepares the brain for the flavor to come. After smelling the wine, a sip should be taken and swirled around the mouth. This allows the taster to experience the mouthfeel of the wine. Slurping while drinking aerates the wine, volatizing it, which increases the intensity of the smell and flavor (LaMar, 2001). The taste testing process is an important part of analyzing the wine flavors produced during ingredient development and can be used to help ensure that a wine will be liked by consumers.

Tannins Being Observed

E&J Gallo has two major tannin production processes: ELT and LLT. At Gallo, the tannins from these two processes are combined together and treated to produce a final product that can be added to wine.

The Extracted Livingston Tannin (ELT) is produced by toasting oak chips in a rotary toaster followed by three washes. The chips are placed in a 13% ethanol bath and then two separate water baths for 24 hours each. The bath solutions are then combined and evaporated so that the desired tannins are concentrated at the bottoms and the oak condensate can be collected separately. At this point, the condensate is subject to many pH shifts and put through evaporation processes to increase the concentration of the tannins. The pH is first increased to 10 to reduce the volatility of the acidic solution and allow the ring structures of the tannins to open before going through an evaporation process. The pH is then increased to 12 to further the reaction and again evaporated to increase tannin concentration. In order to stop the reaction, the ring structures must be closed, so the product from evaporation is treated with citric acid to bring the pH down to 3. The condensate solution is then subject to column chromatography where the the solution is washed with two 40% ethanol washes followed by two to three 80% ethanol washes to obtain the final product (Ames & Walker, 2014).

The Livingston Liquid Tannin (LLT) has a much simpler production process, as it is just a highly concentrated condensate product formed from the wood toasting process. This condensate is condensed and concentrated by a factor of ten and then purified through column chromatography. In the resin column, the solution is washed with two 40% ethanol washes and two 80% ethanol washes to give the final product (Ames & Walker, 2014).

Purpose

The main purpose of this project was to use the waste stream from the ELT process and separate the desired flavors. The ELT-03 waste stream was a by-product of E&J Gallo's primary oak extraction step involved in flavor production. This stream contained ethanol, water, and other organic compounds. These other organic compounds include guaiacol, syringol, and other methoxyphenol and non-methoxyphenol compounds. Each of these compounds had a different flavor profile, but the products with smoke flavor were of importance in this project. Unfortunately, due to the processing time of the oak chips, the ELT-03 waste stream was not available for testing. Instead, many stand-ins such as MF2 (Mocha Fume 2), lab-made oak slurries of various concentrations, and another waste streams (CT-04) were used. Using these stand-ins should, in theory, yield similar results to the waste stream except with cleaner results. Once these components were extracted, they were able to be added to unprocessed wine for flavoring.

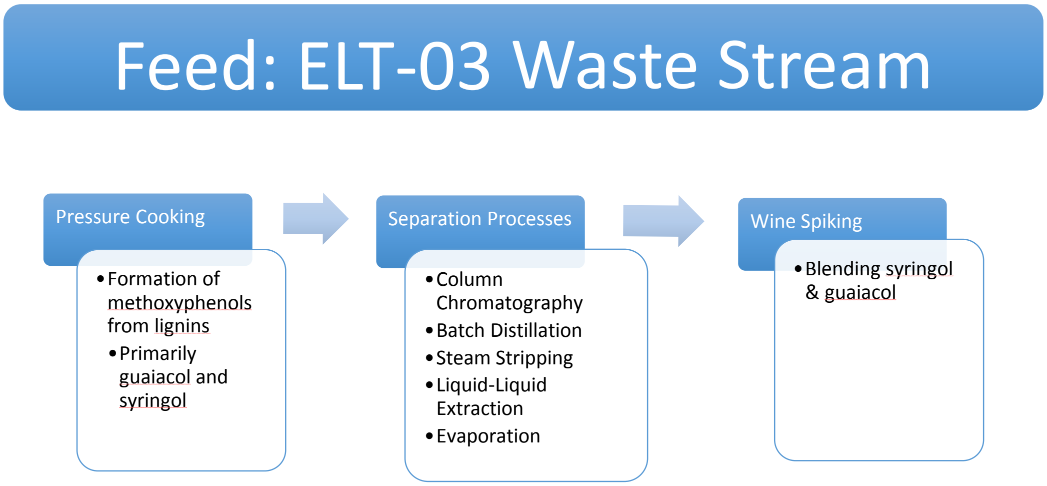
Expected Benefits

By recycling the ELT-03 waste stream, it is expected that Gallo will save money on treatment processes, as the amount of waste will be decreased. As an added bonus, creating a new smoky flavor may allow Gallo to start a new line of wines, thus increasing commercial sales. Because the process will not require the use of any new equipment, production costs should not be affected, and Gallo should instead see an increase in their revenue.

Experimental Background & Methodology

The two main smoky flavors of importance were predominantly guaiacol and syringol. These were found in very small concentrations in the waste stream (parts per million) and needed to be separated from the rest of the waste stream. Once the desired smoky flavors were separated, it was possible to add them to wines in different samples and conduct taste tests to determine optimal flavor. The final step was to use this concentration to estimate the feasibility of production as well as provide a case count based on the quantity of the flavor necessary for each bottle of wine. This process is illustrated in Figure 1 below.

Figure 1: Overall Project Methodology



There were five ideas for separation: distillation, steam stripping, column chromatography, evaporation, and liquid-liquid extraction, which are described in further details in the next section. Bench scale trials were conducted to find the ideal scheme for isolation/separation of guaiacol and syringol in the waste stream.

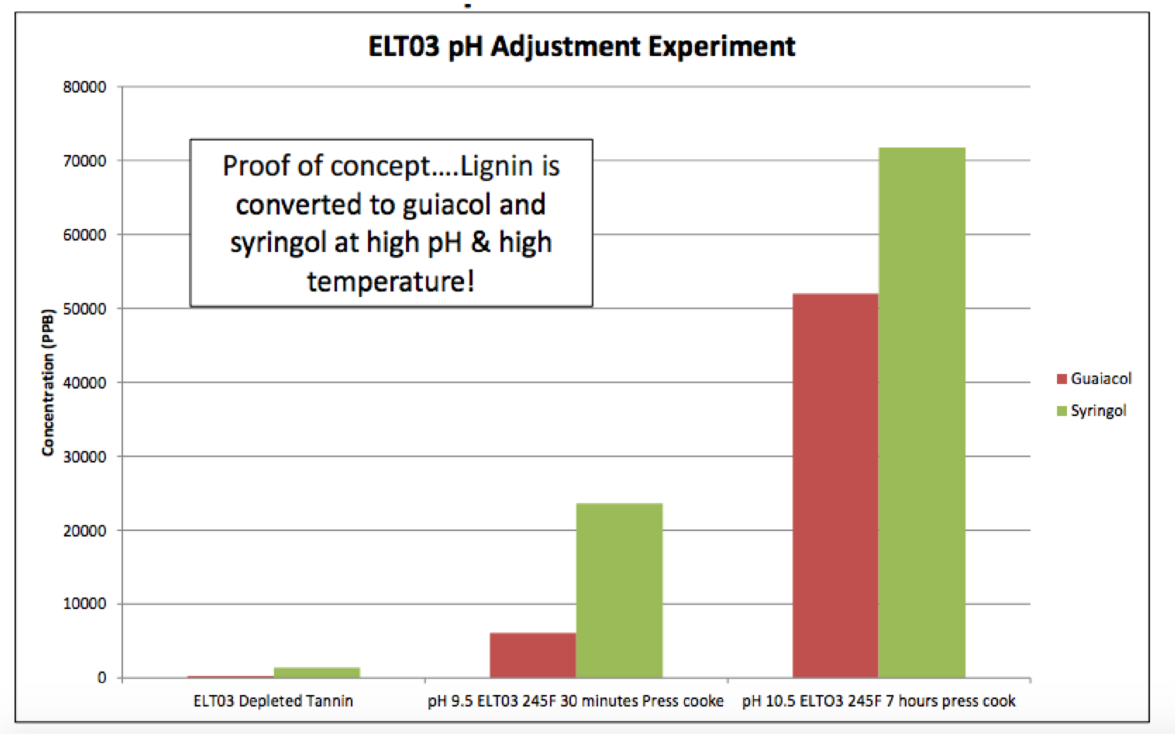
Separation and Extraction Techniques

Cooking Conditions

One way to increase the compounds of importance was to introduce the waste stream to different pressure, temperature, and pH ranges to try and increase the guaiacol and syringol concentrations in the stream. Gallo had already conducted proof of concept experiments in their lab using their small pressure-rated cooking vessel in the past to try to raise these concentrations. As seen in Figure 2, the waste stream lignins are converted to guaiacol and syringol at high temperatures, pH levels, and with more cooking time.

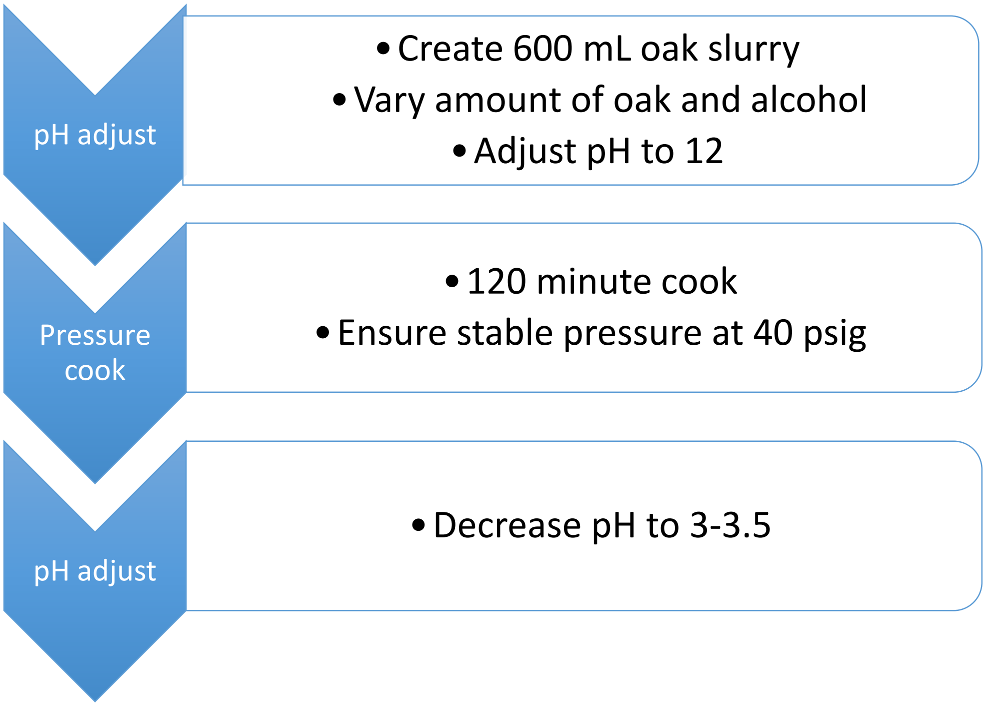
Figure 2: Various Pressure and Time Effects on Concentration of ELT Waste Stream

(provided by Gallo)



Based on past work conducted by intern Devin Ergler in 2014, optimal cooking parameters were found to be at a basic pH of 12, 40 psig maximum cooking pressure, and 120 minutes of cooking time. From this information, Gallo wanted to gain an understanding of the total suspended solids versus alcohol content in the cooked product. In order to understand this, various slurries of oak, water, and ethanol were mixed and cooked in a high pressure Soxhlet extractor, seen in Figure 4. The process is outlined below in Figure 3.

Figure 3: Outline of Cooking Process

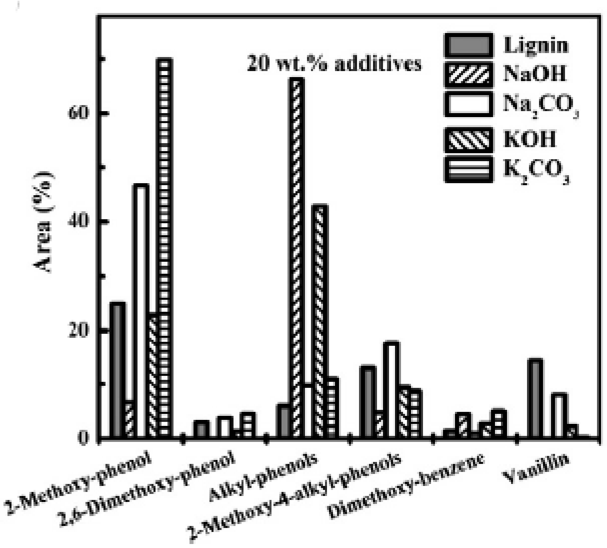


The pH was increased to 12 using potassium hydroxide in order to open the ring structures of the lactones and shift the reaction. After the cooking process, the pH was adjusted to 3-3.5 in order to increase the volatile aroma compounds and to re-close the ring structures. A more detailed procedure can be found in Appendix I.A. Once cooked, 50 mL of the sample was sent to the lab to test for aroma compounds and alcohol content. The rest of the samples were stored for further separations and extractions processing.

Figure 4: High Pressure Soxhlet Extractor (used for cooking)

Further research also indicated that methoxyphenol production is affected by the type of alkaline used to initially increase the pH of the slurry to 12. A study showed that “carbonates obviously promoted the production of methyoxyphenols while hydroxides greatly increased the alkyl-phenol production” (Peng, 2014). As seen in Figure 5, the 2-methoxyphenol production increases 3-fold when potassium carbonate (K_2CO_3) is used as the alkaline additive instead of potassium hydroxide (KOH). Once the optimal total suspended solid was determined with KOH as the alkaline, K_2CO_3 was also used to see the effects on guaiacol and syringol increase.

Figure 5: Results from Past Study on Methoxyphenol Production (Peng)

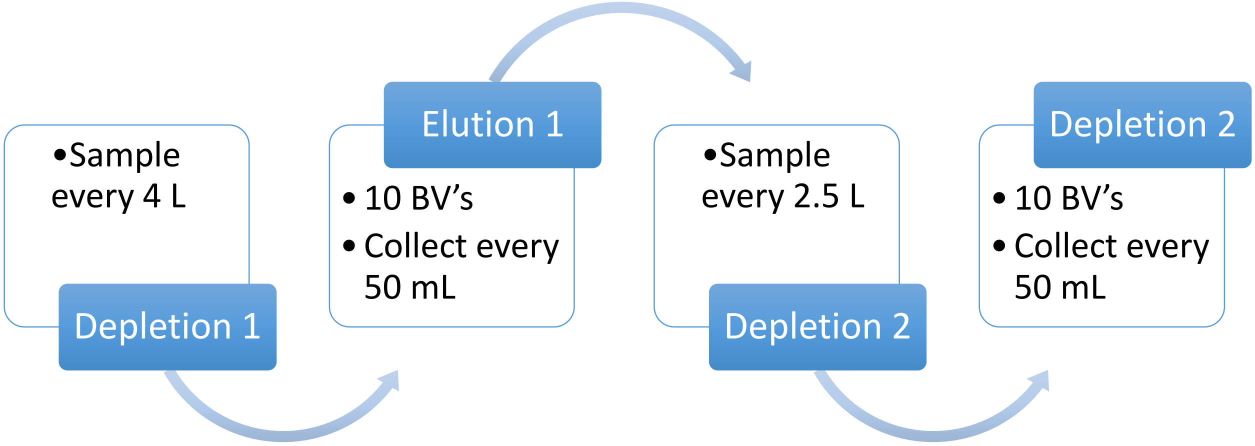


All in all, thirteen different trials were conducted where total suspended solids versus alcohol content and the effects of alkalinity were tested. Once the optimal cooking parameters were found and these concentrations were increased, the next step was to try to separate out the smoke components.

Column Chromatography

Chromatography, in general, is used to separate components. Column chromatography, specifically, also purifies components. These components are separated into two different phases: mobile phase and stationary phase (“CHE310: Column Chromatography”, n.d.). The stationary phase in column chromatography is the packed resin bed, a solid adsorbent placed in a glass column. The mobile phase is added to the top of the glass column and allowed to flow through the stationary phase using external pressure (a peristaltic pump). In theory, the mobile phase is the ELT-03 stream, but Mocha Fume 2 diluted with water and CT-04 was used instead. The column was packed at a 50 milliliter bed volume of FPX-66 resin, a food grade adsorbent resin used in Gallo's lab. The overall process of column chromatography is illustrated in Figure 6.

Figure 6: Column Chromatography Process



There were 20 liters of the feed added to the column where the flow rate was controlled with the pump around 8-10 milliliters/minute. The stream exiting the resin column contained the depleted tannins and samples were collected every 4 liters in order to map out how much of the tannins adsorbed onto the resin column. Once all 20 liters were depleted, the column was eluted with ten bed volumes of ethanol at various ethanol concentrations: 20%, 40%, 60%, 80%, and 95%. The depleted tannins were then re-fed through the resin column and eluted in order to further separate and purify the components in the feed stream.

Distillation

Distillation is a process of separation that can generally occur in either a batch process or a continuous process. Batch distillation tends to have a product with a lower alcohol content than with continuous distillation. With the production of alcoholic beverages, the distillation process separates alcohol and water. Heat is used to vaporize one of the components and isolate the alcohol and congeners within the feed (Total Wine & More, 2016 and How Distilling Works, n.d.). The first part of the alcohol mixture, known as the wash, is the heads. The heads contains the materials that have lower boiling points, such as ethanol and syringol. The seconds part of the wash to vaporize is the hearts. The hearts have the highest concentration of the desirable alcohols, such as the esters, which contribute fruity flavors. Finally, the last part of the wash to vaporize is tails. The tails has predominantly water. Like in non-alcohol based distillation, each stream will have different concentrations of each component than originally found in the feed (Total Wine & More, 2016 and How Distilling Works, n.d.).

The distillation process starts with a base containing sugars. The sugars are converted in the fermentation process into alcohol, which is then distilled. After distillation, flavoring can be adjusted to change the style and flavor template. In the wine process, the grape juice is fermented, and the product can then be distilled into brandy (Total Wine & More, 2016 and How Distillation Works, n.d.). Higher alcohol content in wine or wine products such as brandy require distillation because fermentation is not enough to get achieve the high alcohol content. Alcohol levels around 14-18% are toxic to the yeast in the fermentation process. As a result, another method must be used to get a more concentrated beverage (How Distillation Works, n.d.).

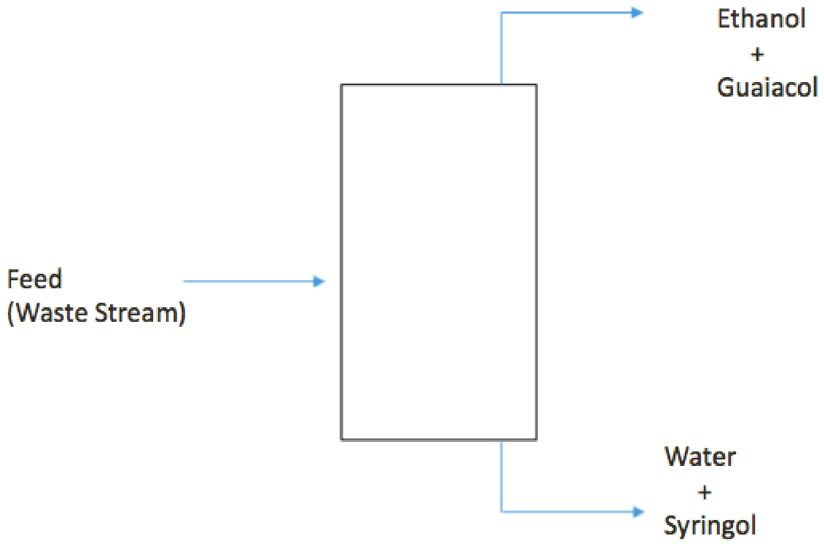
The main organic compounds of focus were syringol and guaiacol. As seen in Table 1, these have very different boiling points, so it would normally be very difficult to get the two organic compounds out together.

Table 1: List of Boiling Temperatures (Restek), Molecular Weight, Vapor Pressure, and Flavor/Smell Components

Component	Boiling Temperature (^oC)	Molecular Weight (g/mole)	Vapor Pressure (20-25 C)	Flavor/Smell
Ethanol	78.37	46.068	7.33 kPa	Sweet
Water	100	18.02	3.17 kPa	-
Guaiacol	204-206	124.14	0.0137 kPa	Roasted Coffee
Syringol	50-57	154.165	0.000823 kPa	Smokey

The idea for the distillation column was to have ethanol and guaiacol come off the top of the column and water and syringol off the bottoms stream, as seen in Figure 7. Once separated, the residual stream and condensate stream can be evaporated using a rotary evaporated, which will be described in a later section, to concentrate the guaiacol and syringol.

Figure 7: Distillation Column Separation



Three different distillation trials were conducted using the material from the cooking trials at three different alcohol levels and with different column types. The condensate was then collected in increments of 10% of the feed volume in order to map out separation of the important components. This set-up can be seen in Figure 8. These results were then compared with expected results from Aspen simulation software.

Figure 8: Distillation Column Set-up

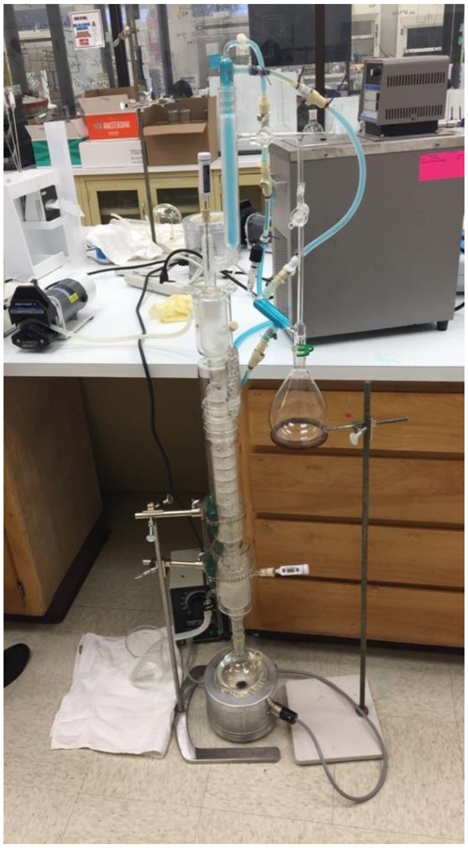
Steam Stripping

Steam stripping is a type of distillation that makes use of steam at the bottom of the column instead of using a reboiler. In steam stripping, water is the heavy component from which the other components, generally volatile organics, are separated (Steam Stripping, n.d.). There are several typical applications which stream stripping can be used for, including alcohol removal from water. The process will usually take place at temperatures close to 100^{o}C, the boiling point of water. While steam stripping requires lower operating temperatures than air stripping, another separation technique, steam stripping is more capital intensive (Steam Stripping, n.d.).

In steam stripping, steam is injected into the bottom of a tower as a means of heating the system and beginning vapor flow. The heat provided from the steam then brings the organic material, in this case, alcohol, into the vapor phase, where it can be carried out the top of the column. The stream is fed into the top of the column, and vaporization occurs as the feed falls (Steam Stripping, n.d.). The flow rate and pressure of the steam varies with the operating pressure, the material being separated, and the desired percentage of separation. The separation thermodynamics cannot be based on Henry's law because of the temperatures, interactions between materials, and varying concentrations, so the non-random two liquid (NRTL) activity coefficient model is used. It is recommended that laboratory tests be completed to determine the varying parameters, but usually vapor pressure data proves to be adequate (Steam Stripping, n.d.). This information was important while completing Aspen simulations.

The CT-04 waste stream was used as feed for steam stripping where 500 milliliters was fed to the system and completely stripped. The set up for this experiment can be seen in Figure 9. Various trials were conducted where the flowrate was adjusted and the feed was fortified. The condensate and bottoms were then sent for sample analysis.

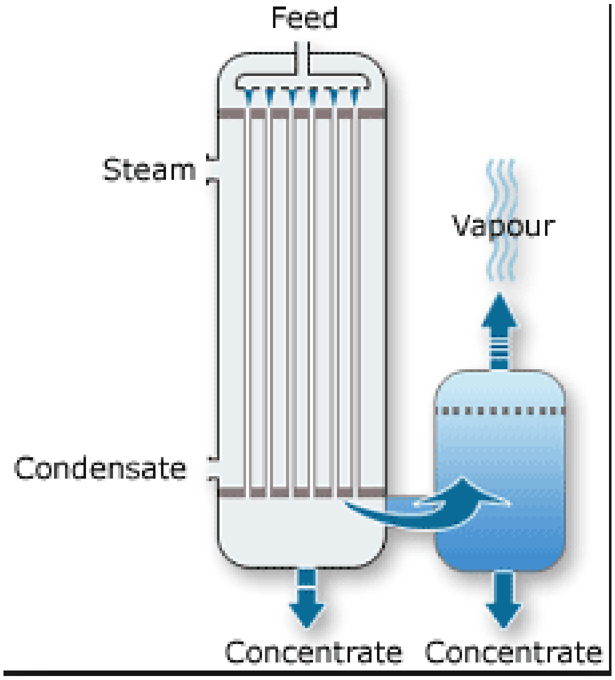
Figure 9: Steam Stripping Column Set-up



Falling Film Evaporator

The falling film evaporator works as a special type of heat exchanger and can be used to increase the concentration of certain components in the liquid. Here, the feed enters the top of the tubes and flows down, due to gravity, the evaporator. Steam is used as an external heating source, and two phases form inside the tubes and are separated by means of an external separator. As the waste stream flows down the tube, partial evaporation occurs, which can increase the composition of the desired guaiacol and syringol in the stream. This concept is demonstrated in Figure 10.

Figure 10: Falling Film Evaporator (Evaporation Technique, 2016)



Gallo has a LUWA-type falling film evaporator that separates light and heavy components using indirect heat transfer. However, the large scale evaporator was never used due to time constraints. Instead, smaller rotary evaporators were used for separation. The rotary evaporator, as seen in Figure 11, consists of a vacuum system, a condenser, and a motor unit that rotates the feed. The water bath was increased to about 70^{o}C, the chiller adjusted to -3^{o}C, and the feed was added to a round bottom flask and rotated. The vacuum was then turned down and the pressure was set to 300 milliPascals (mPa) until a desired ratio of condensate to residual was attained.

Figure 11: Bench-scale Rotary Evaporator Set-up

Liquid-Liquid Extraction

This separation technique is based on solubility. In theory, the waste stream would be combined with a solvent in order to separate the aqueous and non-aqueous phases to extract only the desired organic compounds (M., 2010). As seen in Figure 12, the feed and the solvent mix together to pull out the desired solute, in this case guaiacol and syringol, into the extract stream. Figure 13 shows the physical set-up of the liquid-liquid extraction process.

Figure 12: Liquid-Liquid Extraction Process

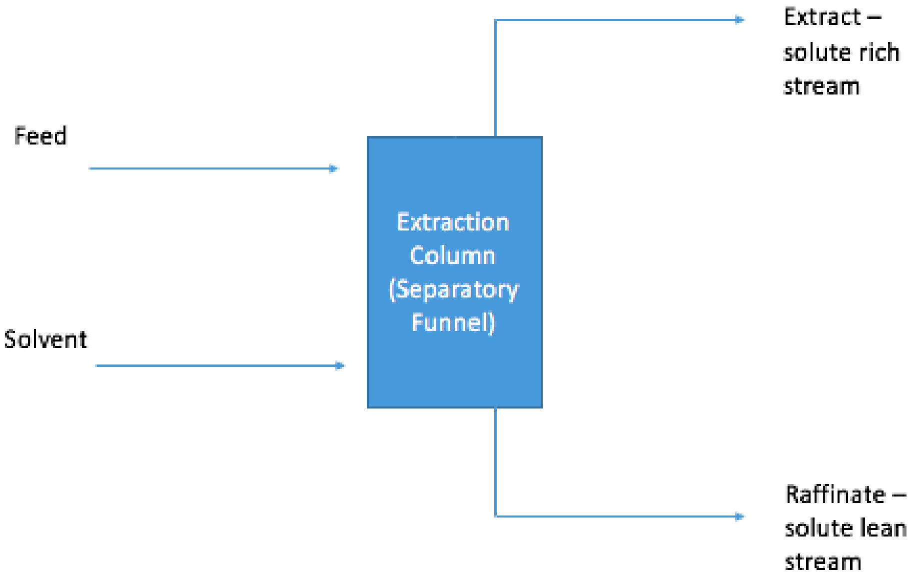
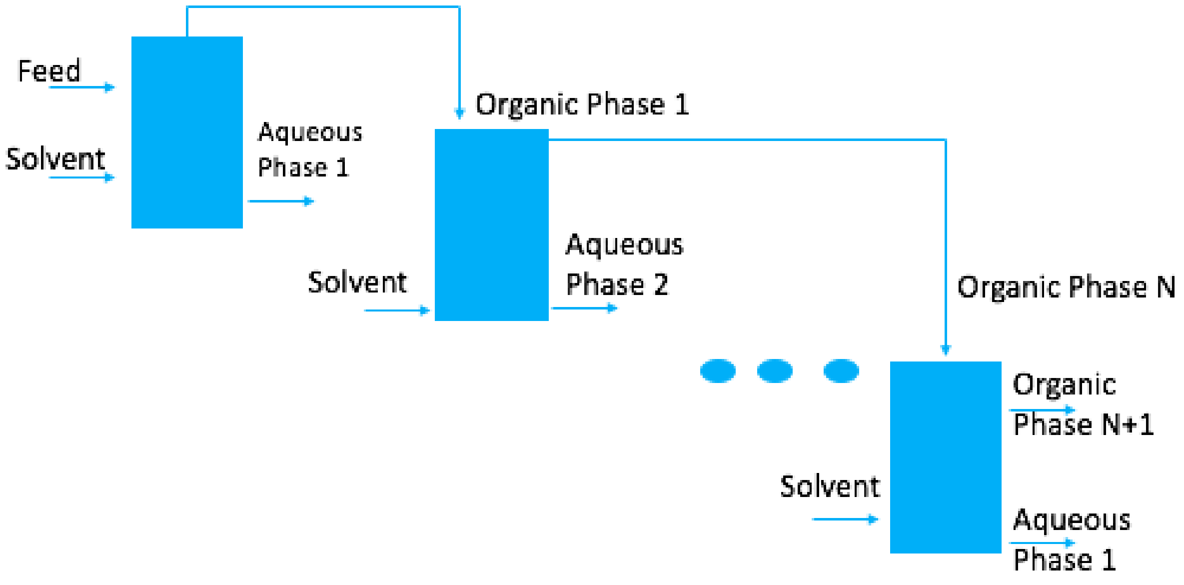


Figure 13: Liquid-Liquid Extraction Set-up

N-butanol was the solvent of choice as it ties to another project where n-butanol can be separated from the fusel oil side stream from brandy stills. Bench trial scales were conducted using a separatory funnel. Mocha Fume stand-ins were used as the feed and three trials were conducted. Two trials were conducted where the ratio of the feed to the solvent was maintained at a 1:1 ratio (50 mL each) and 1 trial was conducted where the the feed to solvent ratio was 1:2 (50 mL feed, 100 mL solvent). These trials mocked a five stage extraction where the organic phase was treated as the new feed and fresh solvent was added to the separatory funnel each time. This principle is illustrated in Figure 14.

Figure 14: Liquid-Liquid Extraction Lab Process (Multi-stage)



Lab Tests at Gallo

At Gallo, three analytical lab tests were run on all smoak samples submitted. These tests are described in Table 2.

Table 2: Analytical Tests at Gallo

Test Name	Test Purpose
MVAZ	-Enzymatic volatile aromas -Indicates amount of acetic acid
MALC	-Ethanol concentration
$MAROMOAK	-Aroma concentrations of of various compounds

Results

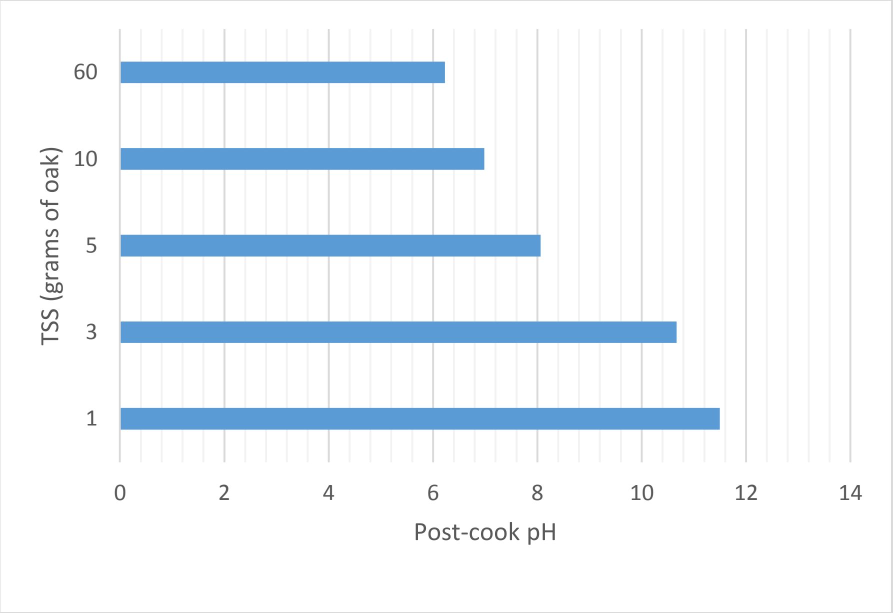
Cooking

As previously mentioned, the optimal cooking conditions had already been determined to be 40 psig for 120 minutes at a pH of 12. The main focus for cooking was to discover the effect of methoxyphenol production from the amount of total suspended solids (TSS) and alcohol content as well as the use of different bases. In total, thirteen trials were conducted.

Effect of Total Suspended Solids and Alcohol Content

The slurries were created with oak chips of various ethanol concentrations from 0-20% and the TSS varying from 1-60 gram(s). As seen in Figure 15, the post-cook pH, before it was re-adjusted back down to 3, decreased with increasing TSS. This was due to the breakdown of the lignins in the oak chips during the pressure cooking step. With more oak, there is more lignin breaking down and, as a result, more acetic acid production which reduces the final pH from the starting pH of 12.

Figure 15: TSS versus Post-Cook pH

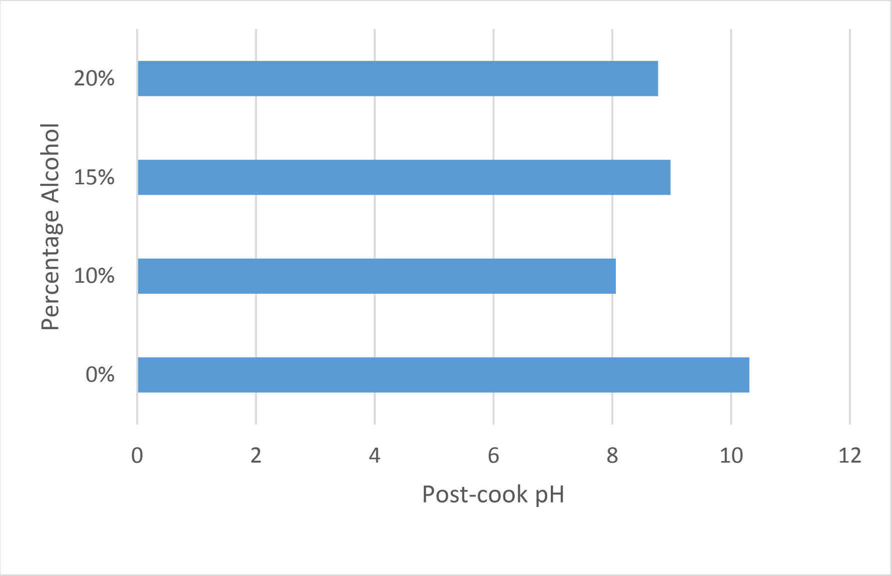


Acetic Acid Production

The amount of acetic acid produced for each trial was also calculated using the ICE method and the pKa values. The concentration of acetic acid produced was very low and these calculations can be found in Appendix II.C.

While there was a trend with TSS, varying the ethanol concentration did not have a large effect on post-cook pH. Figure 16 shows four different alcohol concentrations at a constant 5 grams of suspended solids in the slurry. In comparison to the 0% ethanol concentration, there is a pH decrease for the other samples in the chart. However, the trend is not consistent as the pH increases at 15% and 20% ethanol.

Figure 16: Alcohol Concentration versus Post-Cook pH with 5 grams of oak



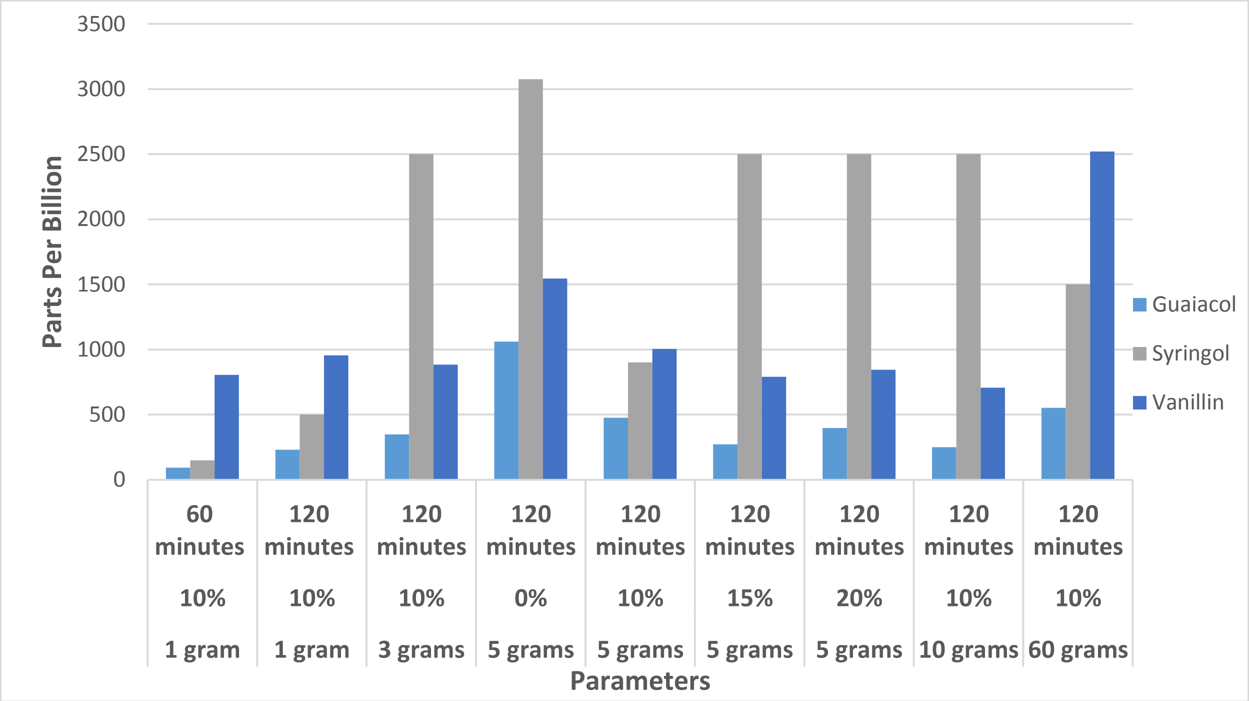
Sensory Testing

All samples were sent to the lab for analytical testing to get concentration profiles of the methoxyphenol production. However, lab results take about two weeks to process so sensory testing was performed at the end of the cooking trials to understand how to further experimental trials. The aroma for each specific trial can be found in Appendix V. Most samples had varying intensities of campfire smoke with notes of mocha and fall spices. In order to collect unbiased and well-rounded data, many employees at Gallo were asked to give their input. The sample with the most overall pleasing aroma was at 5 grams of oak and 10% ethanol with deep wood and pumpkin spices. It is important to note that the aroma of the cooked oak slurry varies significantly from the aroma of the ELT-03 waste stream. Devin Ergler's results in 2014 mentioned pungent notes of cumin and fish along with smoky aromas while the cooked oak aromas were much cleaner.

Analytical Lab Results

In terms of tannin production, there are three major tannins of focus for this MQP: syringol, guaiacol, and vanillin. Syringol and guaiacol are important due to their contribution of the smoky flavors and vanillin production is, in general, of interest for the company. There are other interesting trends in production of eugenol, cis-oak lactone, and trans-oak lactone that can be found in more detail in Appendix II. Figure 17 illustrates the different concentrations produced from cooking at various conditions.

Figure 17: All Cooking Trials (for TSS & Percentage Alcohol)



Looking at only guaiacol, there was a two-fold increase in concentration between the 1 gram oak and 60 grams of oak at 10% ethanol and 120 minutes of cooking. However, the difference in concentration between 5 grams and 60 grams at the same conditions were minute. Another interesting phenomenon was the spike in guaiacol and syringol concentration when there was no ethanol added to the 5 grams oak slurry, suggesting that the alcohol actually negatively impacted methoxyphenol production in the Soxhlet extractor. However, maintaining the pressure was a major challenge in this experiment, as the power had to be continually adjusted to keep a constant pressure. There were many instances when the pressure would overshoot to 50 psig or undershoot to 35 psig with a slight change in voltage. This instability affected the reaction mechanisms and therefore the methoxyphenol production, which likely is what resulted in inconsistent data. From the data collected, no definite conclusions can be made about the effect of alcohol on the methoxyphenol production until more trials are conducted for reproducibility.

Taking into account the multiple factors, it was decided that the trial with 5 grams of oak and 10% ethanol was the “optimal” condition for further testing at Gallo. While other trials had higher syringol and vanillin concentrations, this trial had the closest aroma to what was expected. The TSS in the slurry also closely mimicked the ELT-03 waste stream; only 1 gram of oak would have been too little in the waste stream and 60 grams would be oversaturated with oak chips.

Effects of Alkaline on Methoxyphenol Production

The past cooking trials were tested using KOH as the base to increase the pH before cooking. As mentioned before, past research indicates an increase in methoxyphenol production of K_2CO_3, as seen in Figure 5 in the Methodology. In order to test these results, 5 grams of oak with 10% ethanol slurries were used for consistency. Three trials were conducted: one where the pH was adjusted to 12 using only KOH, one with pH adjusted to 12 using only K_2CO_3, and one where a mixture of both bases were used. There were two major issues with the use of K_2CO_3 only. The first was trying the maintain pressure in the extractor and the second problem was with precipitation. K_2CO_3 increased the production of CO_2 in the extractor and the pressure would overshoot to 60 psig. Then, in order to maintain equilibrium, the pressure would then shoot back down to zero and the cooking process would have to be restarted. The other problem was foaming and solidification when citric acid was added to the cooked slurry. With each increment of citric acid added, bubbles formed due to the reaction of citric acid with K_2CO_3, as seen in Figure 18. There reached a point around a pH of 4 where the slurry completely solidified into a peanut butter-like texture. The solid was vigorously agitated and left sitting for a few hours and separation between the liquid and solid phase was noticed. Once there was known separation, the solidified slurry was centrifuged had approximately a 54% yield. Since the liquid recovery was so low, it was decided that a combination of KOH and K_2CO_3 should be used to increase the pH before cooking. After about a pH of 10, K_2CO_3 reaches its equivalence point in the slurry, and it takes almost twice as much base to increase the pH. This may have oversaturated the slurry, thus causing solidification during the post-cook pH adjustment. To prevent this, the pH was increased to about 9.5 with K_2CO_3 and then fully increased to 12 using KOH.

Figure 18: Precipitation During pH Decrease Step

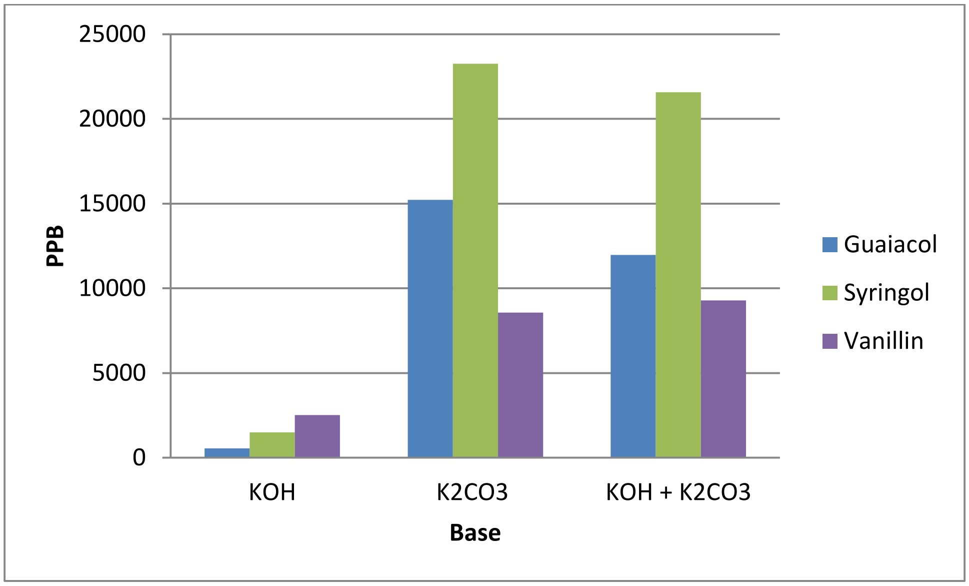
Foaming

Precipitation/solidification around pH pf 4.03

Centrifuged: approx 54% yield

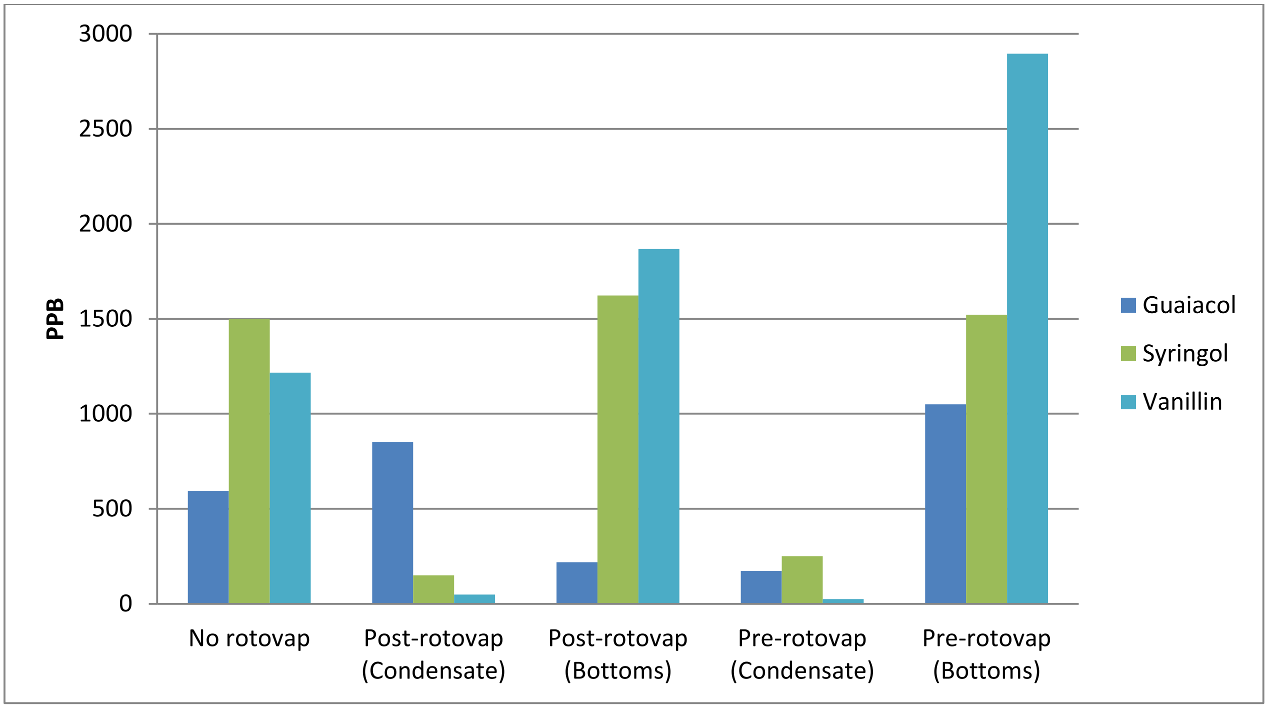
As seen in Figure 19, the concentrations of guaiacol and syringol increased by over 50 times when K_2CO_3 was used to increase the pH to 12 instead of KOH. When using a combination of the two bases, the guaiacol and syringol concentrations were a little smaller, but the vanillin concentration increased. Although the concentrations of the smoky tannins are lower, there was no solidification of the slurry and therefore no centrifuging step necessary. The tradeoff then became liquid yield versus increased concentration. Based on the results, it was recommended that the last two data sets (on the right) would be great concentrations for Gallo to work with. For example, the ratio of K_2CO_3 to KOH can be further examined and developed to see if a higher concentration of guaiacol and syringol can be obtained.

Figure 19: Effect of Base on Methoxyphenol Production



When performing sensory analysis of these samples, it was noted that with K_2CO_3, the smoky aroma was not pertinent. Instead, there was a much stronger starchy/pretzel aroma. With the pH decrease, the aroma became sweeter with a slight licorice/root beer undertone. In order to try to get the targeted aroma and see the effects of concentration, two samples of 5 grams oak, 10% ethanol, and pH adjusted with only K_2CO_3 were evaporated using a rotary evaporator after cooking. The cooked slurry was evaporated until about 60% condensate was produced. One sample was evaporated after the pH was adjusted back down to 3-3.5 (referred to as “post-rotovap”) and another was adjusted back up to a pH of 12 after cooking and then evaporated and re-adjusted back down to 3-3.5 (referred to as “pre-rotovap”). After evaporation, the bottoms had a slight wet-wood aroma, more so in the pre-rotovap sample and these sensory results follow the analytics. As seen in Figure 20, the guaiacol and syringol concentrations increased in the pre-rotovap bottoms sample. However, in the post-rotovap sample, there is a higher guaiacol concentration in the condensate. This may be due to the lower pH during evaporation. The tannins and aroma compounds were more volatile at a lower pH so guaiacol was more likely to come off as the condensate since it was the lighter key.

Figure 20: Evaporated K2CO3 Samples



Liquid-Liquid Extraction (LLE)

The purpose of LLE was to see the effect of separation using n-butanol as a solvent. Two trials were conducted using diluted Mocha Fumes (MF2) as the feed in a 1:1 ratio (1 part feed to 1 part solvent). One trial was conducted using MF2 as the feed in a 1:2 ratio (1 part feed to 2 parts butanol). Tables 3 and 4 describe the aroma compounds during the five stage extraction.

Table 3: MF2 1:1 Extraction Components

"Stage"	Aroma (Organic)	Color (Organic)	Color (Aqueous)	Volume (Aqueous) mL
1	BBQ Sauce	Dark Brown	Dark/Medium Brown	50 mL
2	Honey BBQ Sauce/Bacon	Dark Brown	Medium Brown	40 mL
3	BBQ Sauce	Dark Brown	Honey	35 mL
4	Stronger bacon notes/hickory	Darker Brown	Light Honey	~22 mL
5	Undertones lost	Dark Brown	Light Honey	18 mL
				Final Volume: 15 mL

Table 4: MF2 1:2 Aroma and Volume Results

"Stage"	Aroma (Organic)	Color (Organic)	Color (Aqueous)	Volume (Aqueous) mL
1	Very strong butanol/light notes of BBQ sauce	Dark Brown	Dark/Medium Brown	50
2	Honey BBQ sauce, notes of bacon	Dark Brown	Medium Brown	40
3	BBQ Sauce	Dark Brown	Honey	35
4	Notes of bacon, strong butanol	Dark Brown	Light Honey	22
5	Very much butanol - undertones of bacon lost	Dark Brown	Light Honey	15
				Final Volume: 15 mL (70% loss)

When n-butanol was added, the mixture separated into a dark brown, viscous layer and a lighter brown, nonviscous layer. Using aroma and visual senses, it was assumed that the organic layer (the darker, viscous layer) was the extract and the lighter layer was the raffinate. However, when the lab results came back, the opposite was true. The aqueous, nonorganic phase was determined to be the true extract phase, as the important solutes from the feed came out in this phase (recall that the extract phase is solute-rich and the raffinate phase is solute lean). As seen in Figures 21 and 22, nothing came out in the raffinate stream (the viscous stream that was assumed to be the organic phase). This is perplexing because the smoke and bacon aromas were present in the raffinate stream. It is possible the entire feed matric was extracted along with the n-butanol and the smoke aroma molecules stayed behind and concentrated with the rest of the feed. This would explain why the darker, viscous stream lost volume over time; the solutes were pulled out and the aroma molecules stayed intact.

Figure 21: MF2 1:1 Extraction Components

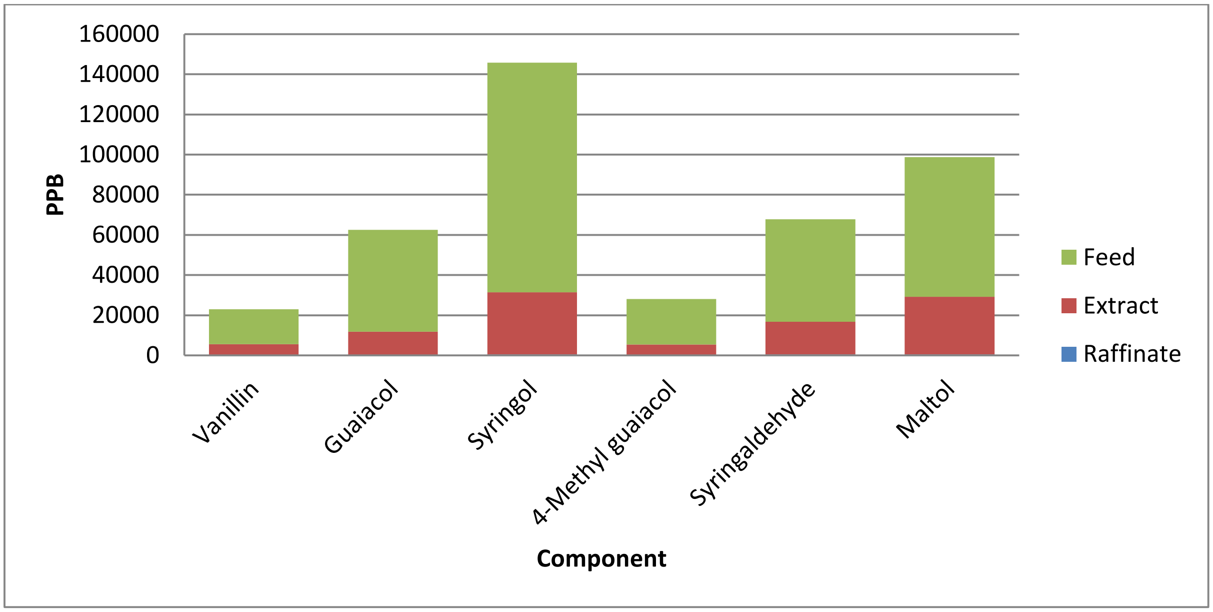
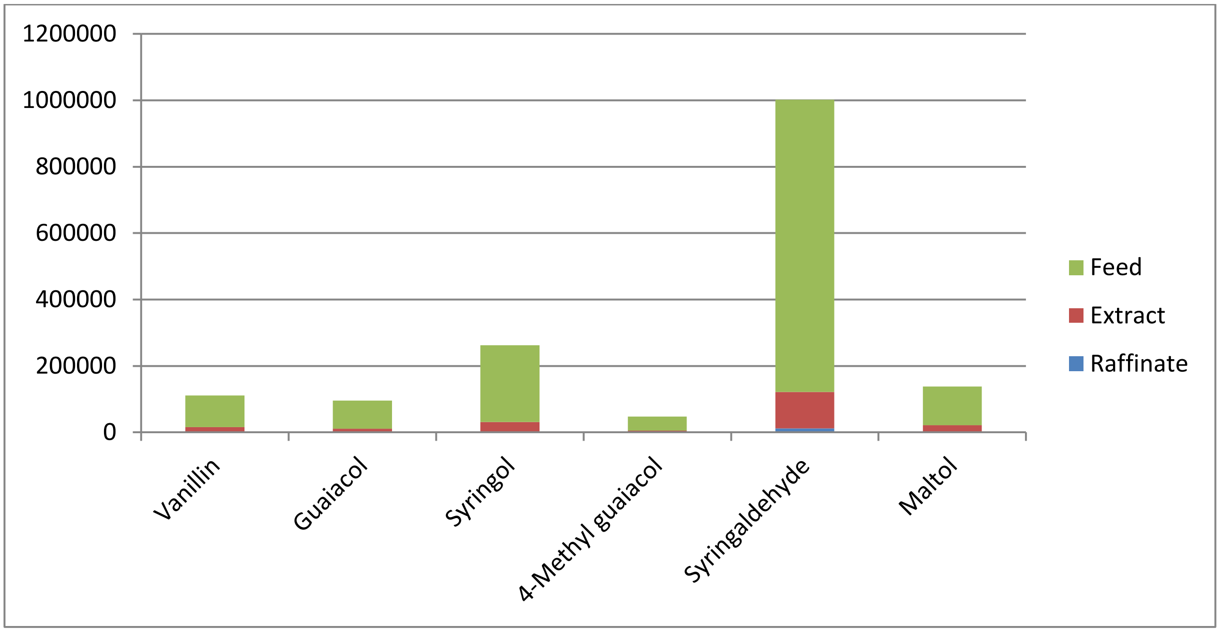


Figure 22: *MF2 1:2 Extraction Components*



Scale-Up

In order to scale up the LLE results from the two conducted trials in the separatory funnel, the Kremser equation was used to translate the results to a continuous counter-flow extraction. The Kremser equation (Equation 2) gives the number of theoretical stages from the following equation:

Equation 2: The Kremser Equation

$$N_{\text{Theoretical}} = \frac{\ln[\left(\frac{1}{E}\right)+(\frac{E-1}{E*x})}{\ln(E)}$$

where E is the stripping factor which indicates the ratio of the solute being stripped from the extract. In the Equation 3, V is defined as the solute in the extract and L is defined as the solute in the raffinate.

Equation 3: Stripping Factor

$$E\text{=Stripping Factor} = \frac{V*k}{L}$$

The stripping factor uses a distribution factor, K, which is the concentration of the solute in the extract phase (y) divided by the concentration in the raffinate phase (y), as seen in Equation 4 (Wankat).

Equation 4: Distribution Factor

$$\textbf{K=Distribution Factor}=\frac{y}{x}$$

The results of this scale-up can be seen in Tables 5 and 6. The value for the stripping factor and distribution factor are extremely high in both cases, and the theoretical number of stages is approximately -1, which cannot occur in practice. The high factor values indicate that the solute in the feed has completely come out in the extract phase, as suggested in Figures 21 and 22. The nonsensical negative number of stages is an indication that there is actually over extraction in the five stages, meaning that one stage may be sufficient for extraction. These calculations can be found in Appendix II.

Table 5: Scale-Up Results for MF2 1:1 Extraction

MF2 (1:1) **Solute**	**K**	**E**	**Number of Theoretical Stages**
Guaiacol	236.35	2718.02	***-0.973829761***
Syringol	61.76	710.24	***-0.996541394***

Table 6: Scale-Up Results for MF2 1:2 Extraction

MF2 (1:2) **Solute**	**K**	**E**	**Number of Theoretical Stages**
Guaiacol	72.36	1133.78	***-0.9859***
Syringol	17.40	272.67	***-0.9984***

The poor results from the scale-up and from the lack of separation may be due to assumptions made in these experiments. Kremser assumes that the diluent (water) and solvent (n-butanol) are completely immiscible. However, the water from the MF2 feed and the n-butanol are actually miscible up to approximately 20% by volume, which would have skewed the results. Also, the Kremser equation is used for counter-current extraction, but these experiments were run using five stages of co-current extraction. It is recommended that Gallo look into the results from a quick one stage extraction and compare the separation to these five stage separation results. It may also be interesting to look at the effects of feed temperature and pH on separation.

Batch Distillation

Three distillation trials with the 5 grams cooked oak slurry at different ethanol concentrations were combined and used as the feed. The first trial was only 10% ethanol, and as Figure 23 shows, a vigreux column was used with 3 valve trays. In order to maintain constant concentration of the condensate, the aim was to begin collection at a steady temperature of 78-80^{o}C. However, during the actual run, the temperature would spike well above 90^{o}C every time the reflux valve was opened to let out the condensate. It was also noticed that the liquid never went down the vigreux column, yet there was quite a bit of vapor going up. This was most likely an indicator that the system reached a pinch point, or that there was no liquid hold up in the column and the vigreux column was experiencing bad column performance.

Figure 23: Distillation Column Set-Up for 10% Ethanol Trial

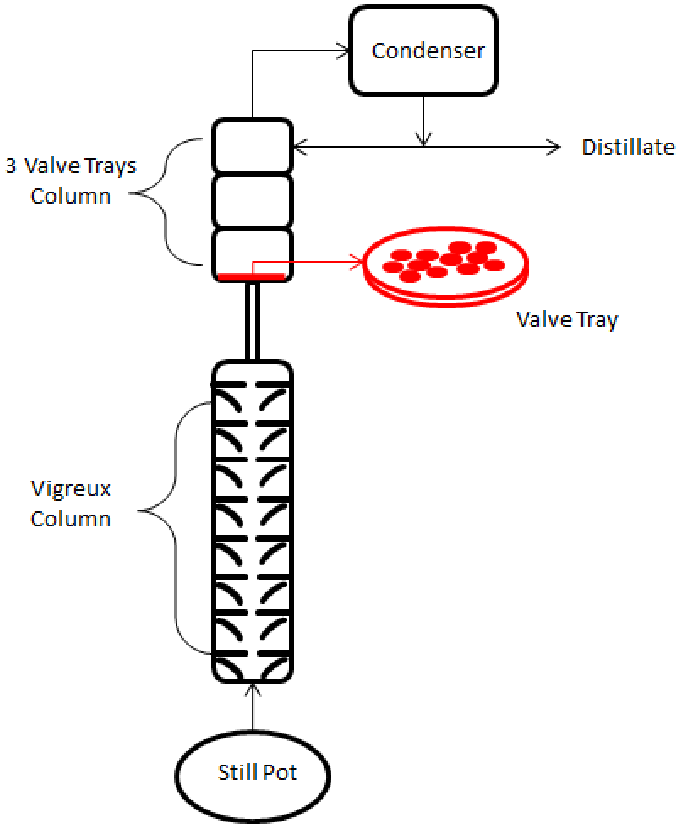


Figure 24 shows the results of this separation. All the alcohol (MALC) was distilled within the first two collections, leaving almost none in the residual. The trend in alcohol concentration closely follows the trend in pH spike, as shown in Figure 25. With increasing ethanol in the sample, the pH became more basic. In terms of component separation, the syringol stayed in the bottoms whereas most of the guaiacol was present in the condensate, as expected.

Figure 24: Distillation Results (Absolute) of 5 Gram Oak, 10% Ethanol

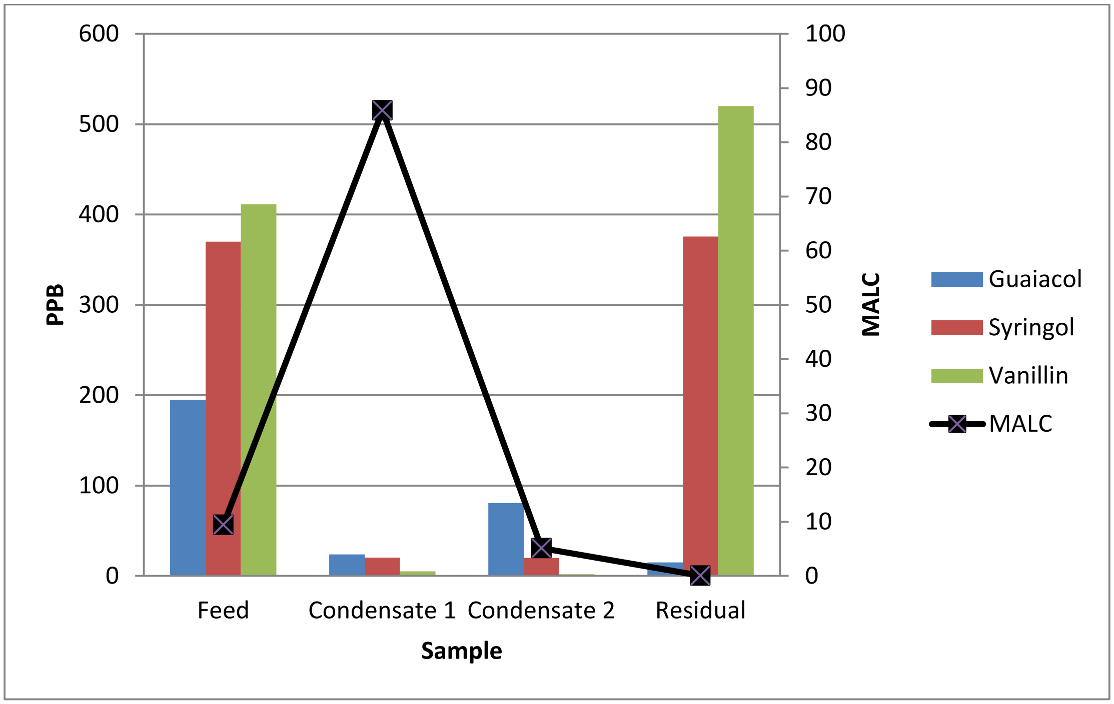
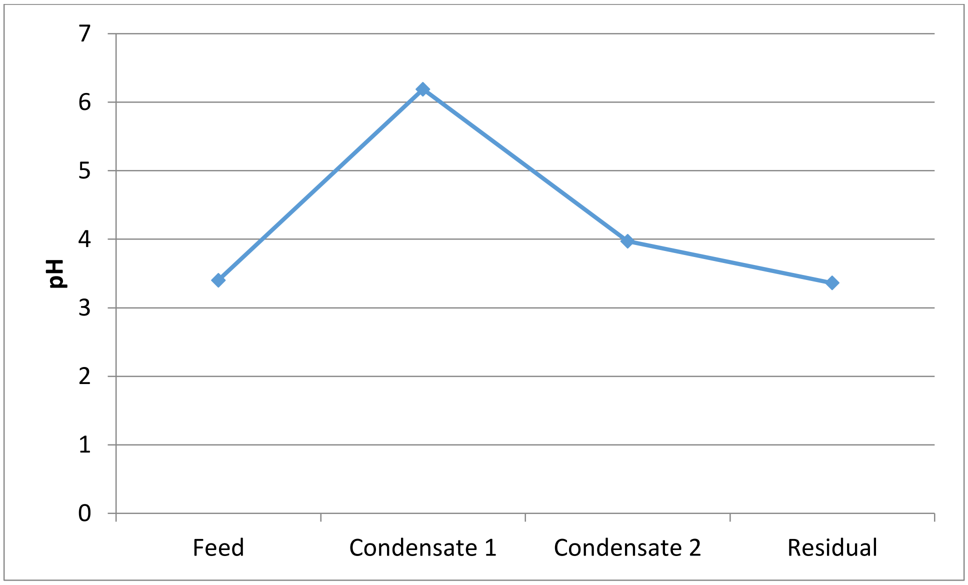
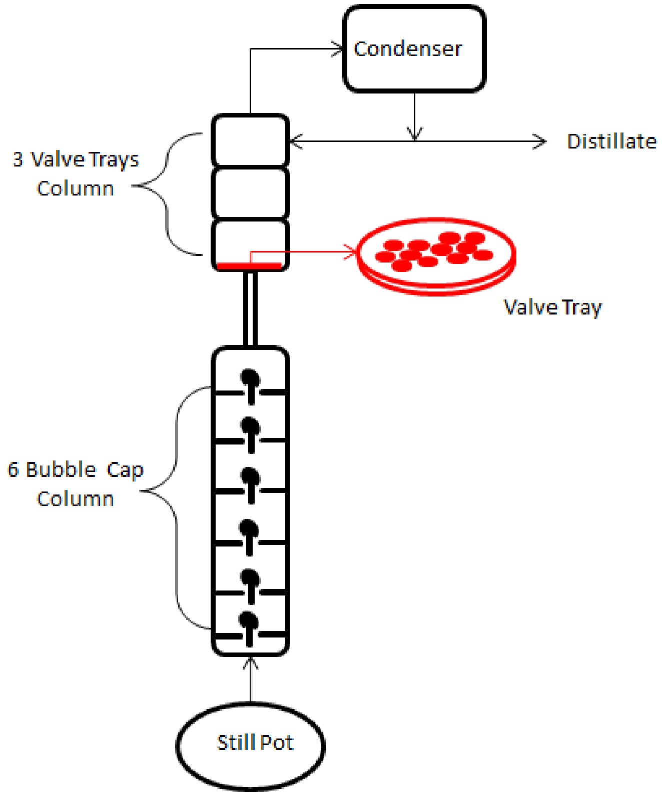


Figure 25: pH Trend for 5 Gram Oak, 10% Ethanol Distillation Trial



The next trial was the 5 grams oak sample, except with 20% ethanol. This time, six bubble cap columns were used in place of the vigreux column, as seen in Figure 26. The first two collections had notes of sweet potatoes and vanilla but the third collection had a salty and unpleasant odor. At this point, it was noticed that there was more vapor and less liquid condensing in the column. This trial also ran into a similar problem with maintaining constant temperature as in the previous trial. The condensate temperature kept spiking to 90^{o}C and then undershooting below 80^{o}C when the reflux valve was closed. With the thought that the ambient temperature changes were causing these problems (opening and closing doors to let in cool air), the column was then insulated for the rest of the trial. The temperature then started to stabilize and the condensate was collected.

Figure 26: *Distillation Column Set-Up for 20% Ethanol Trial*



The results of this trial are shown in Figure 27 and Figure 28. As seen from the first trial, the ethanol concentration decreases with each condensate collection, and all the guaiacol is evaporated when all the ethanol has been distilled. The separation with 20% ethanol shows more promising separation that the 10% ethanol, as there is a high concentration of syringol in the bottoms and very little guaiacol. This suggests that the light key (guaiacol) is more likely to distill off with higher alcohol content.

Figure 27: Distillation Results (Absolute) of 5 Gram Oak, 20% Ethanol

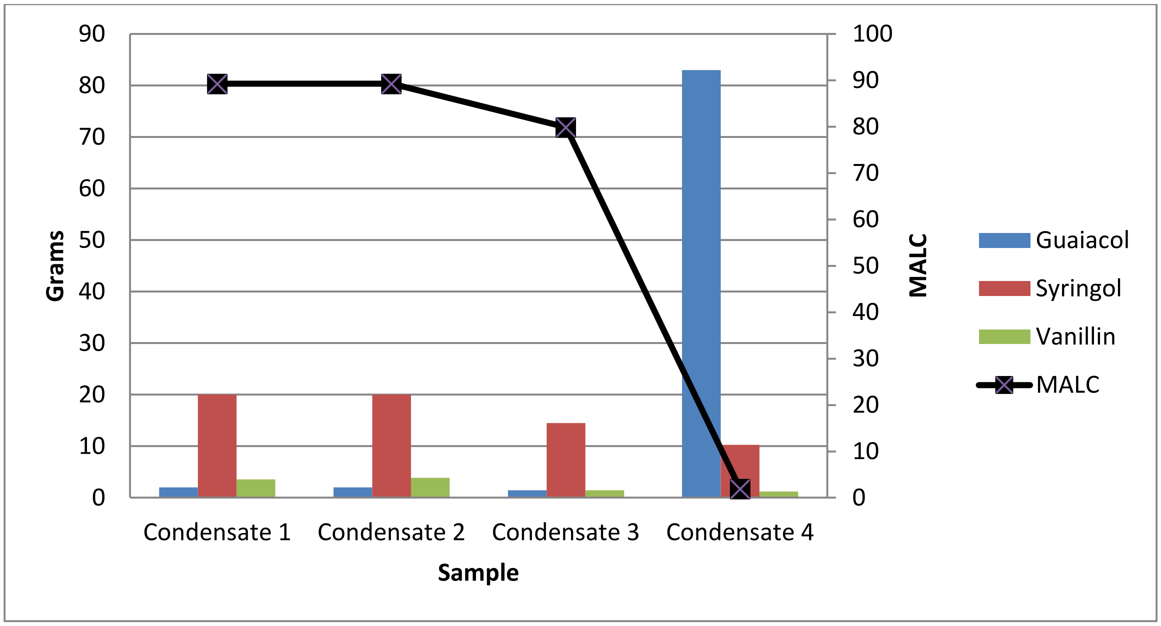
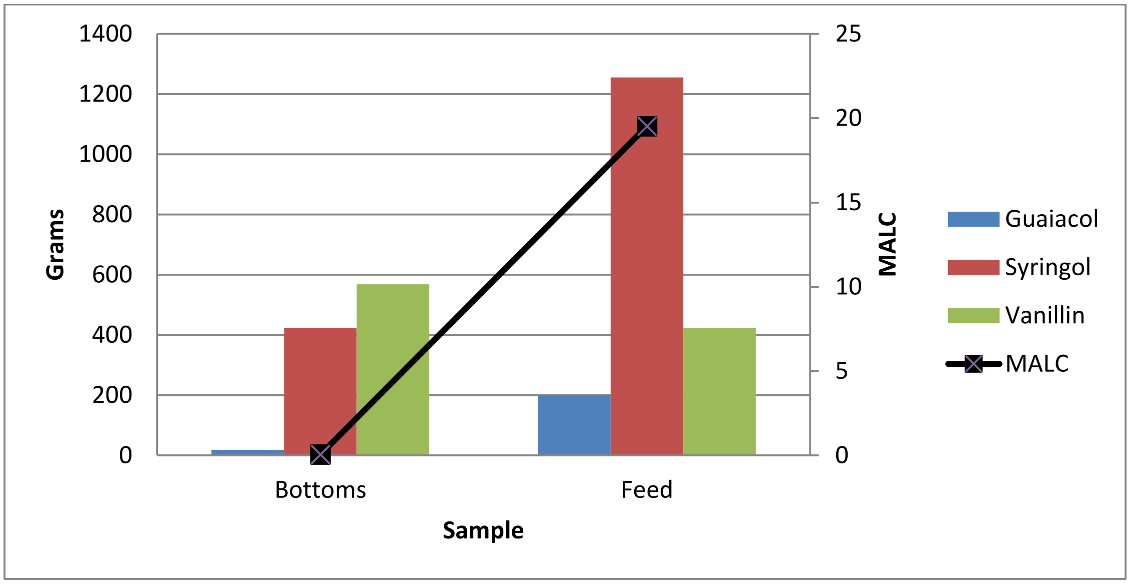
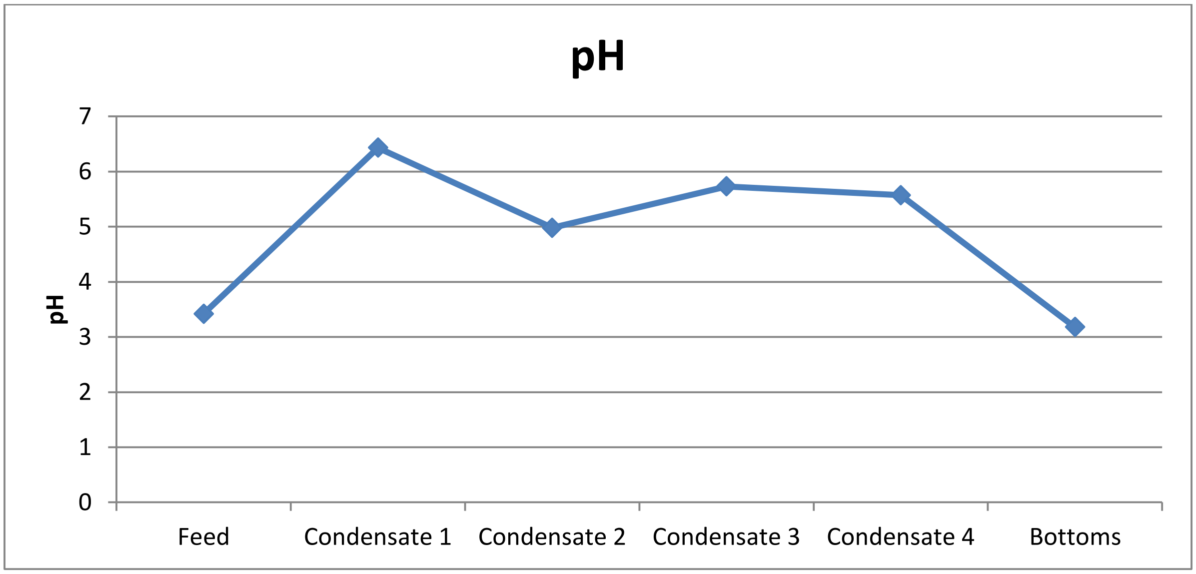


Figure 28: Distillation Results (Absolute) of 5 Gram Oak, 20% Ethanol



The pH trend for the 20% ethanol, as seen in Figure 29, is slightly different from the 10% ethanol trend. The pH of condensate 2 decreased and then increased in the next collection before stabilizing. It was expected that the pH would continually decrease as guaiacol is removed from the feed, as the volatile components are increasing in the condensate. This slight change in trend may be due to the temperature inconsistency when collecting the condensate.

Figure 29: pH Trend for 5 Gram Oak, 20% Ethanol Distillation Trial



In order to map out better separation, the 15% ethanol trial was run with only 3 valve trays, as seen in Figure 30. The first few collections smelled of sweet caramel and then the smell of ethanol started to overpower the condensate collections. The last collection had a sour note and the bottoms had a woodsy aroma. With this column, the temperature managed to stay constant until the last two collections. At these two collections, the guaiacol concentration is highest, as seen in Figure 32. MALC results were not given back from the lab in time for this project report, but it is expected based on the last two trials that the ethanol concentration was lowest at these two collections.

Figure 30: Distillation Column Set-Up for 15% Ethanol Trial

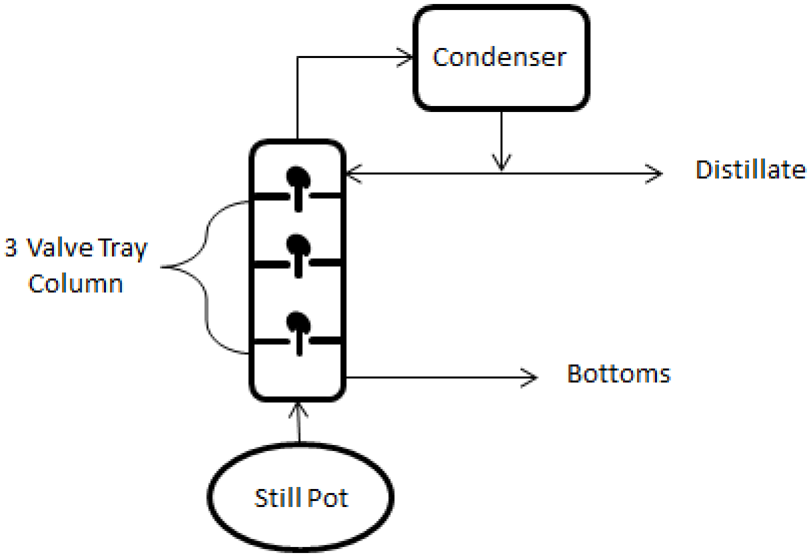


Figure 31:Distillation Results (Absolute) of 5 Gram Oak, 15% Ethanol

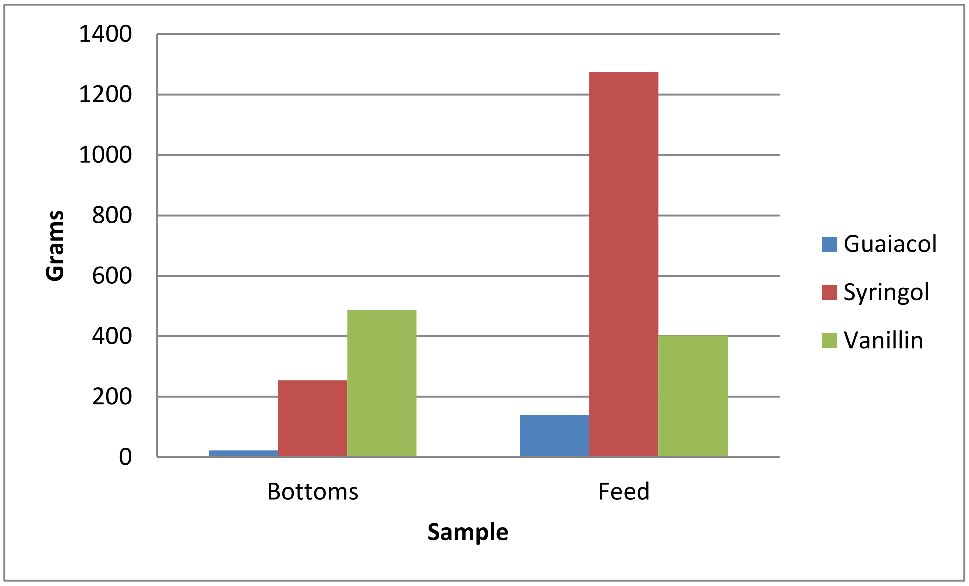
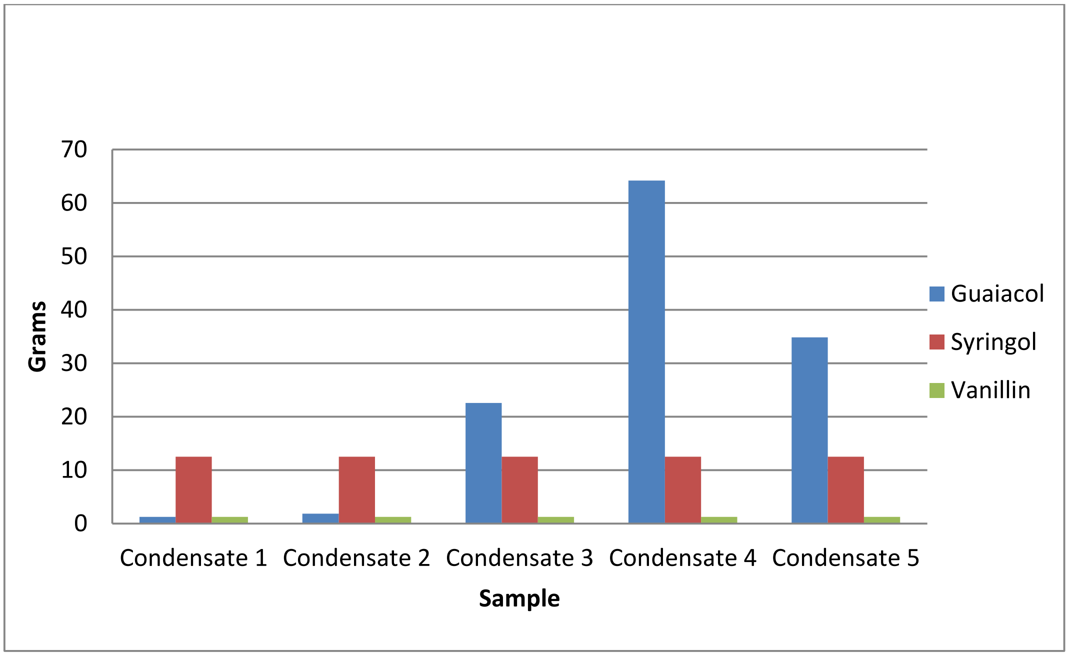
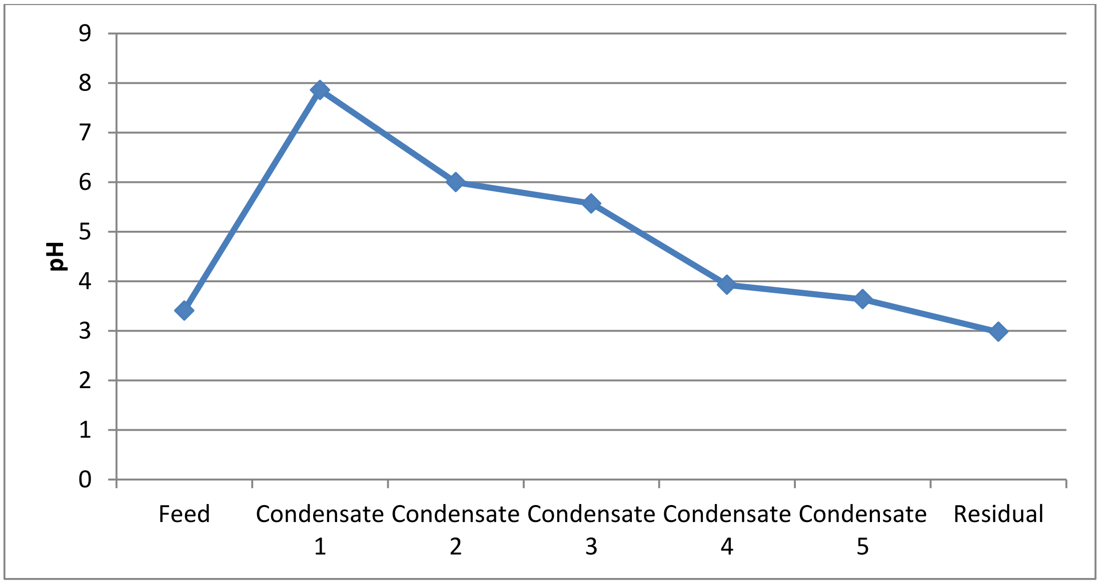


Figure 32: Distillation Results (Absolute) of 5 Gram Oak, 15% Ethanol



As seen in Figure 33, the pH trend decreases with each condensate, as it did for the 10% ethanol trial. This is an indication that more volatile components come out with more collections, with Condensate 4 and Condensate 5 having the largest amount of volatile components. This is in line with the information given in Figure 32 where the concentration of guaiacol is the highest at both of these collections.

Figure 33: pH Trend for 5 Gram Oak, 15% Ethanol Distillation Trial



Steam Stripping

The steam stripper was run using CT-04, an unprocessed waste stream containing smoak components. The smoak feed stream was initially transparent, so Valuable Dark Red (VDR), a grape component, was used to color the feed to a dark shade of pink. VDR is used in winemaking as a coloring agent, so it was expected to have negligible impact on the separation results and as such still comply with the component stipulations of wine. However, it was discovered that the VDR left solids in the CT-04 stream, and the impact of these solids is unknown. It was initially believed that the solids were oak particles, but after looking at the solid found in the bottoms and at oak solids, it was clear that this was not the case. These images can be seen in Appendix II.D. Several trials were completed using the waste stream as the feed. Variations were made in the feed flow rate, which ranged from 6 rotations per minute (rpm) to 10 rpm, while all other parameters were kept constant. Samples from both the distillate and bottoms were collected and sent out to be analyzed. The results of the analyzation are shown in Appendix II.D.

Figure 34 and Figure 35 show the concentration of the bottoms and distillate at two different flow rates: 8 rpm and 6 rpm. These results show that the higher flow rate yielded better separation of guaiacol in the distillate. As expected, guaiacol was pulled out in the light phase with the alcohol.

Figure 34: Steam Stripping at Feed Flow Rate of 8 RPM

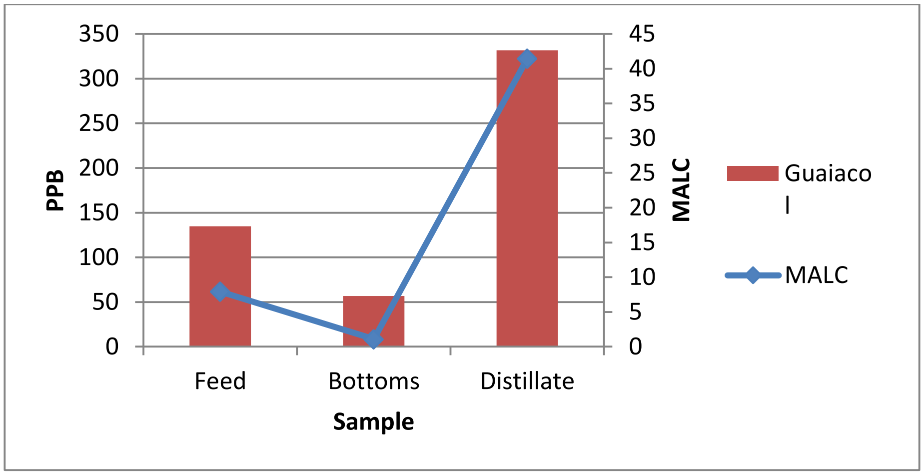
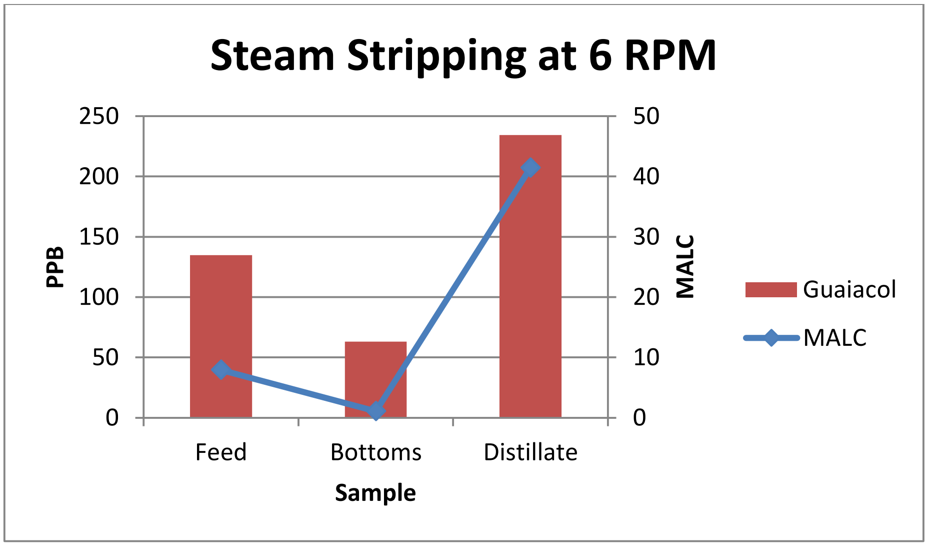


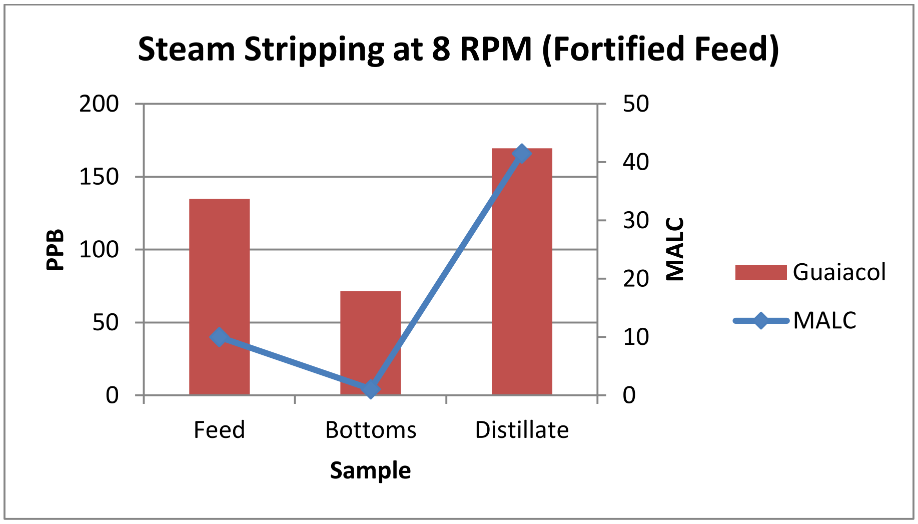
Figure 35: Steam Stripping at Feed Flow Rate of 6 RPM



Aroma testing was completed on both the bottoms and distillate samples to complete a sensory analysis. The samples taken from the distillate had a stronger alcohol smell, as was expected, because alcohol is more volatile than water, meaning it was more likely to come off the top of the column. This was corroborated by the lab results, which showed that most of the distillate samples contained predominantly furfural and furfural alcohol. The distillate was also clear, as the color components in the VDR were too heavy to transform into the vapor phase. Additionally, the samples collected from the bottoms had a significantly weaker smell for the same amount of volume. However, the smell among each distillate sample and among each bottoms sample were very similar. The smells were reminiscent of oak, which was supported by the fact that the smoak material gets its scent from oak tannins.

After completing several trials of steam stripping, the initial feed was fortified, taking the ethanol content from about 7% to about 10% by volume. This result can be seen in Figure 36. These variations with the feed were intended to see how the different levels of ethanol affected the overall separation.

Figure 36: Steam Stripping Fortified Feed at Flow Rate of 8 RPM



All in all, there were four key results from steam stripping the CT-04 stream:

1. There was 7.5 times the amount of guaiacol in the distillate than bottoms.
2. The non-fortified feed had little to no recognizable guaiacol in neither the bottoms nor distillate.
3. Fortifying the feed increased percentage of guaiacol in the distillate
4. Increasing feed flow-rate increased guaiacol production in distillate.

Sequential Distillation

Steam stripping and batch distillation showed inadequate separation results, with high amount of guaiacol still in the bottoms for steam stripping. It would be interesting for Gallo to look into sequential distillation to see how much further the waste stream components can be separated. It is recommended that Gallo steam strip the waste stream first and then batch distills in order to achieve further separation.

Column Chromatography

One full set of column chromatography was done with the diluted MF2 substituting as the waste stream. This feed was diluted to about 10 ppm guaiacol in order to mimic the ELT-03 waste stream. There was 20L of the diluted MF2 run through 50 mL of FPX-66 resin in order to compare results collected by Devin Ergler in 2014. He used the ELT-03 waste stream and his results can be found in Figure 37. The results from the experiment completed using the diluted MF2 can be found in Figure 38 and follow a similar trend, decreasing adsorption over time.

Figure 37: Column Depletion Results Using ELT-03 as Feed (Taken from Devin Ergler)

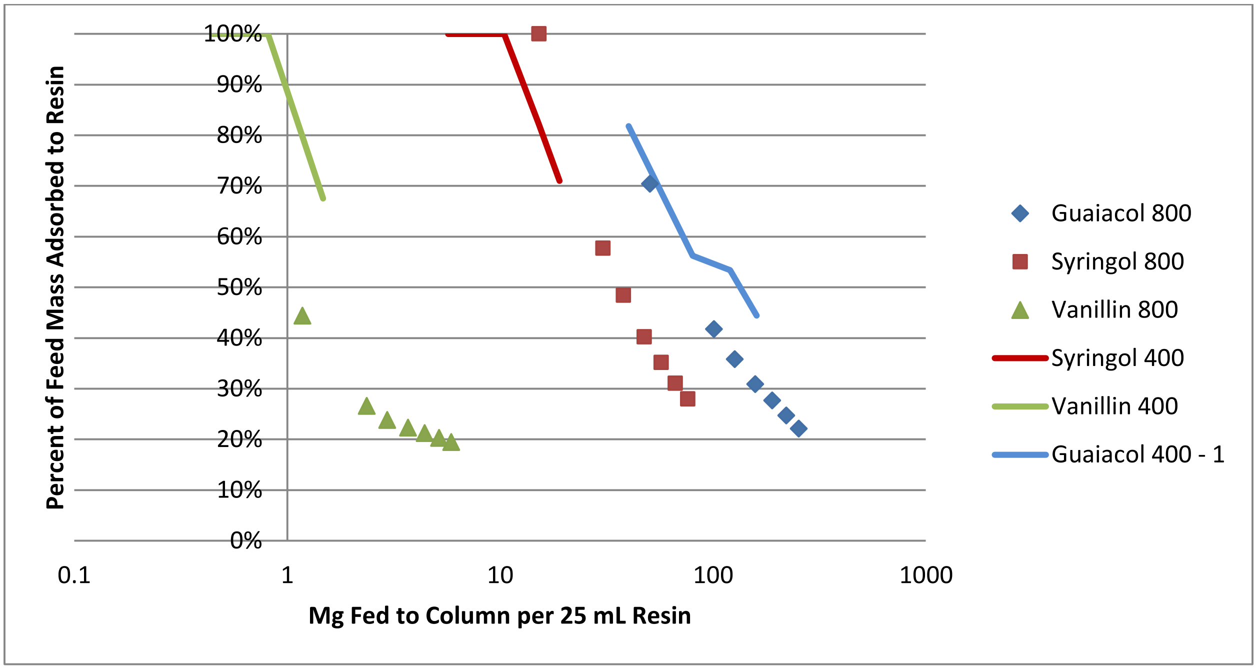
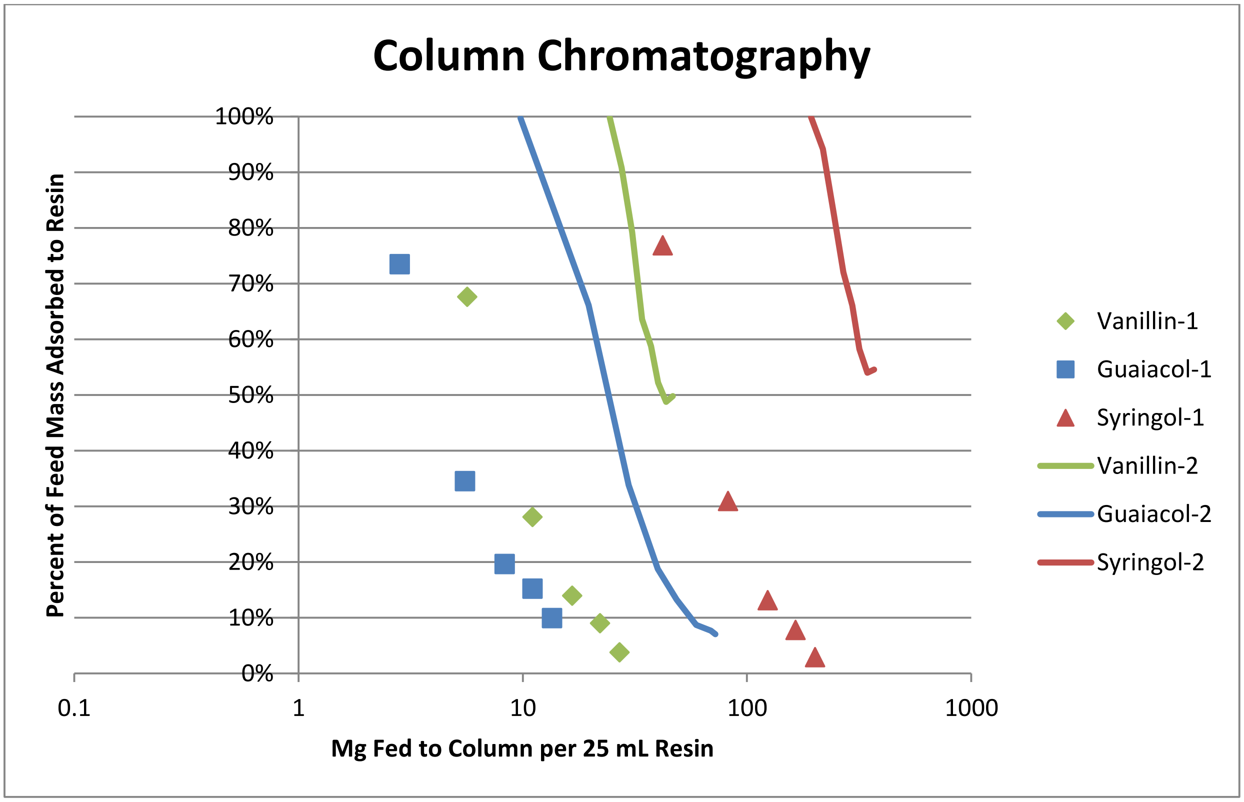


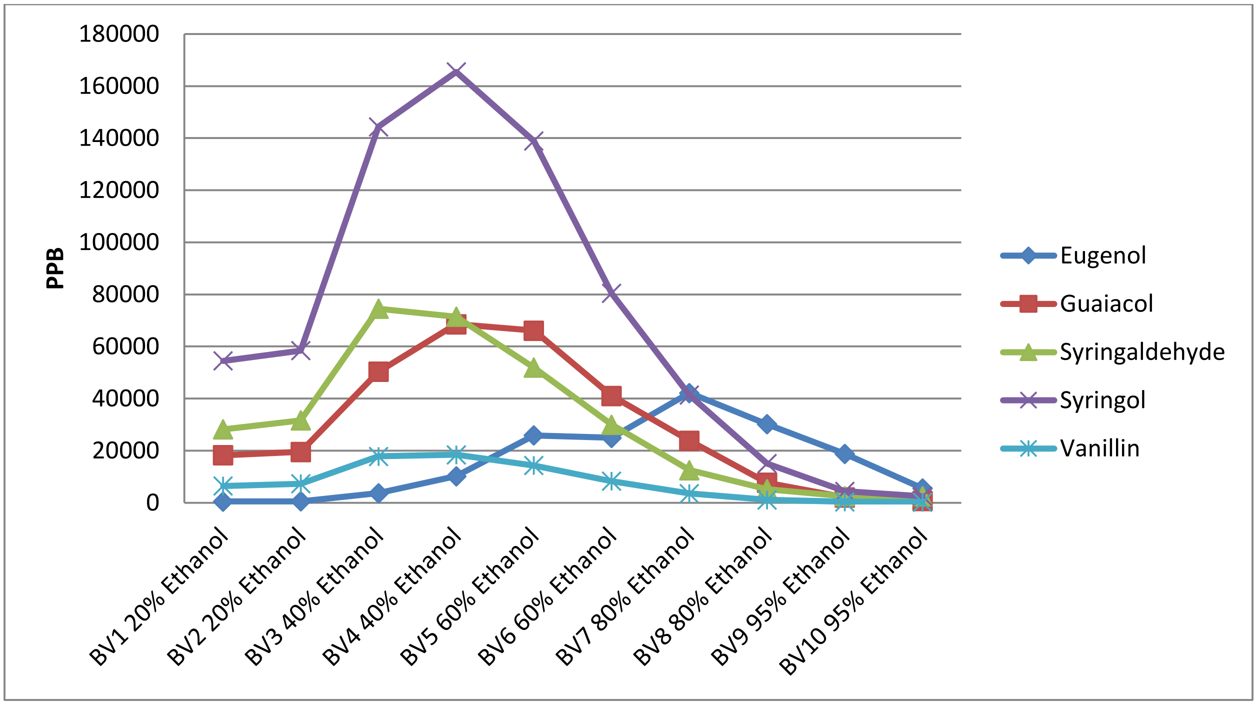
Figure 38: Column Depletion Results Using MF2 as Feed



The data for this experiment was normalized so that it could be compared to Devin's bed volume of 25 mL. In Figure 38, "1" refers to the first pass column chromatography results and "2" refers to the results from the second pass, when the depleted tannins from the first pass were re-used as feed. In Figure 37 (Devin's data), "400" refers to the first pass and "800" refers to the second pass. The results from using the MF2 had a much lower yield in terms of the guaiacol, syringol, and vanillin adsorbing to the column. This was due to the fact that the MF2 stream had more components in the stream than the waste stream did, making it much "busier." Since there were many concentrations in the MF2 feed, there was most likely other high affinity components that displaced the guaiacol in the resin, which is why the percent adsorption drops very quickly.

After the feed was run through the column both times, the column was eluted with ten bed volumes at five ethanol concentrations: 20%, 40%, 60%, 80%, and 95%. The concentration spikes can be seen for both elutions in Figure 39 and 41.

Figure 39:First Pass Column Elution Results



In the first pass elution, the concentration (especially of syringol) increased around bed volume 4, reached its peak around bed volumes 5 and 6, and rapidly decreased afterwards. This reduction in volatile components affected the pH trend of each elution and the pH slightly increased as the tannin concentrations decreased, as shown in Figure 40. Aroma tests were also conducted for each elution, as seen in Table 7. The more pleasant aromas were present at bed volumes 4-6, which is also where the tannin concentrations were highest.

Figure 40:pH Trend First Pass Column Elution

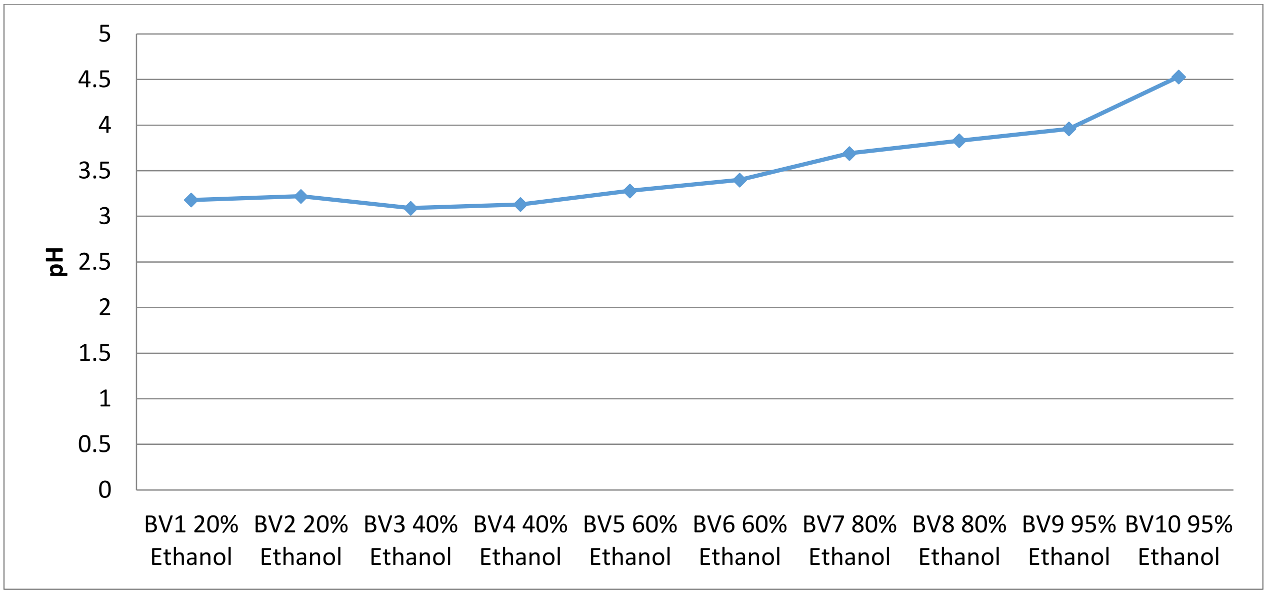


Table 7: Sensory Results from First Pass Elutions

Bed Volume	Percentage Ethanol	Aroma
1	20%	Barbeque sauce, hint of brown sugar
2	20%	Concentrated barbeque sauce, bacon
3	40%	Campfire smoke, hint of barbeque
4	40%	Barbeque, smoke
5	60%	Firewood
6	60%	Firewood smoke
7	80%	Cedar, cigars
8	80%	Hint of bacon, pipe tobacco
9	95%	Cigar, leather
10	95%	Very faint tobacco

In the second pass elution, the concentration of the tannins increased around bed volume 4, reached its peak around bed volumes 5-7, and rapidly decreased afterwards, as shown in Figure 41. As in elution 1, the pH trend, displayed in Figure 42, increased after bed volume 7, indicating that there were less volatile components detaching from the resin. The aromas for each bed volume are described in Table 8, and it was noticed that there was no complexity in the aroma from bed volumes 5-7. In fact, these aromas smelled predominantly of pure guaiacol.

Figure 41: Second Pass Column Elution Results

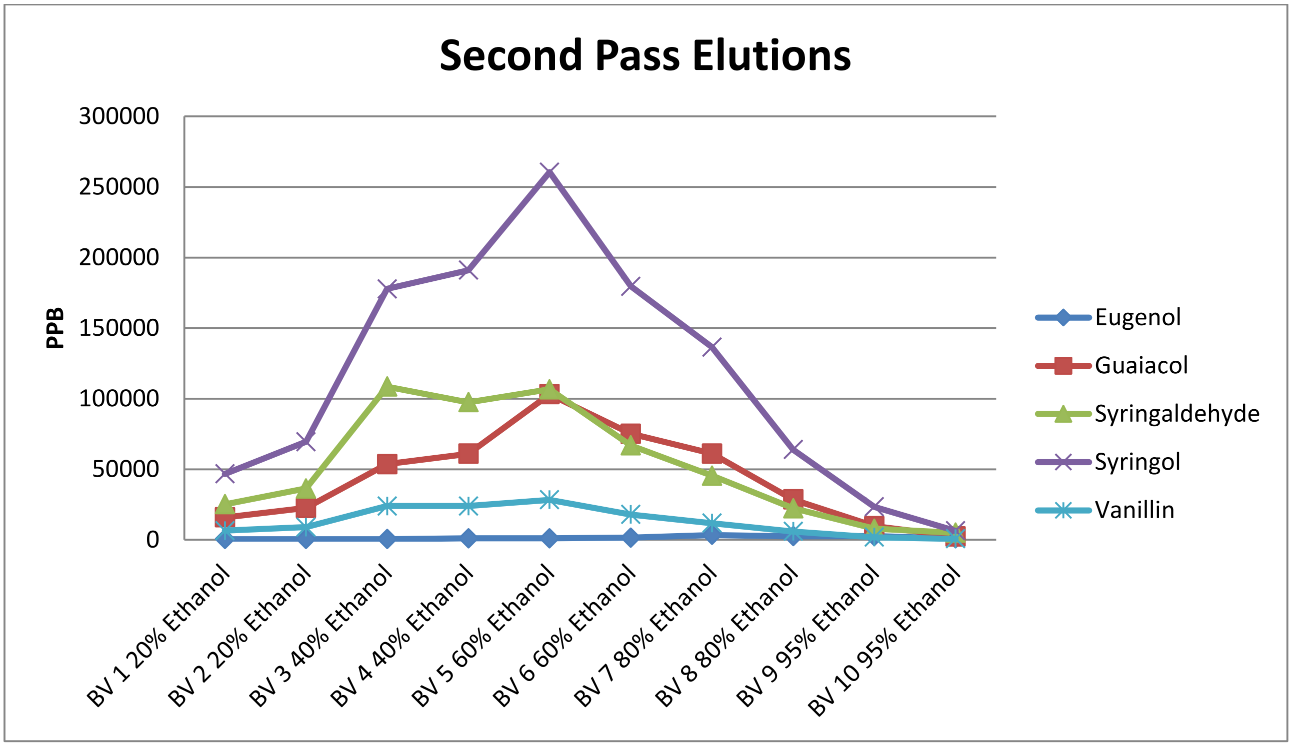


Figure 42: pH Trend Second Pass Column Elution

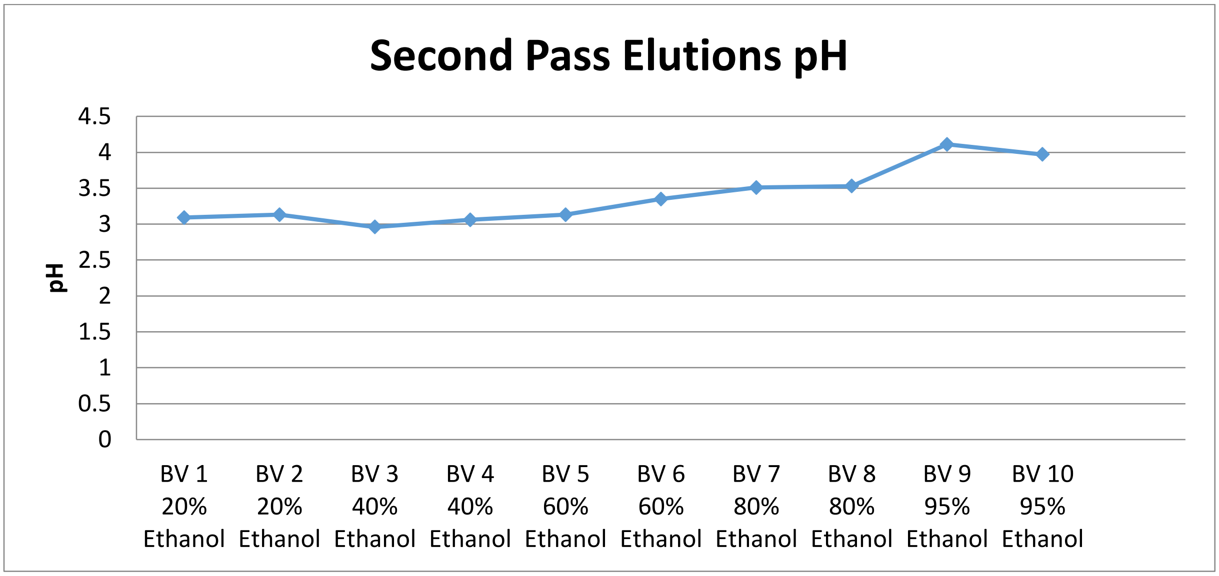


Table 8: Sensory Results from Second Pass Elutions

Bed Volume	Percentage Ethanol	Aroma
1	20%	Smokey, hint of crème brulee and bacon
2	20%	Smokey, roasted coffee, bacon
3	40%	Smokey, vanilla, cake-icing
4	40%	Spice + coconut
5	60%	Guaiacol
6	60%	Guaiacol, less flavor complexity
7	80%	Guaiacol, less flavor complexity
8	80%	Guaiacol, fainter aroma
9	95%	Guaiacol, fainter aroma
10	95%	Very faint smokey smell

Wine Dosing

The final step in this project was to dose wines using the extracted tannins from one of the separation techniques. Pinot Noir was chosen as the wine of choice for dosing because it pairs well with the smokiness in guaiacol and syringol. The elutions from the first pass column chromatography were used for the dosing. Bed volumes 4-6 were chosen based on the pleasant aroma results, and their high tannin concentrations. Bed volume 6 was dosed straight into the wine, whereas bed volumes 4 and 5 were evaporated using the rotary evaporator to concentrate guaiacol in the condensate and syringol in the residual. Past results suggested that the best way to concentrate guaiacol and syringol was to dilute the elution to 16% alcohol by volume and then rotovap the samples to achieve 68% of the feed volume in the condensate.

The concentrations of the tannins as well as the amount of tannins dosed in unprocessed Pinot Noir can be seen in Tables 9, 10, and 11.

Table 9: Bed Volume 4 Dosing Results

Bed Volume 4	**(Rotovap)**	
Guaiacol	21226.1	PPB
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**
0.5	5	**106.1305**
0.25	2.5	**53.06525**
Syringol	94148	PPB
Volume syringol added (mL) in 100 mL wine	Volume syringol in 1L	**PPB Syringol**
1.4	14	**1318.072**
0.7	7	**659.036**

Table 10: Bed Volume 4 Dosing Results

Bed Volume 5	**(Rotovap)**	
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**
0.3	3	**86.4363**
0.15	1.5	**43.21815**
Syringol	144965	PPB
Volume syringol added (mL) in 100 mL wine	Volume syringol in 1L	**PPB Syringol**
1.3	13	**1884.545**
0.65	6.5	**942.2725**

Table 11: Bed Volume 6 Dosing Results

Bed Volume 6		
Guaiacol	41034.4	PPB
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**
0.18	1.8	**73.86192**
0.09	0.9	**36.93096**

The original goal was to aim for 75 ppm of guaiacol in the pinot noir. However, when the trial was conducted using 75 ppm of guaiacol with bed volume 7, it was found that the flavor profile was too overwhelming and did not provide the right mouth-feel. Instead, the wines were dosed based on aroma until a hint of smoke was detected in the wine and then allowed to sit to let the flavors marry into the wine before tasting. The concentrated syringol and guaiacol from evaporation were dosed separately. These concentrations were also cut in half for another set of tastings, providing ten different dosings.

The full analysis of the tastings can be found in Appendix V, but overall it was decided that the best result was the guaiacol at bed volume 5 where the flavor was described as “dark and jammy.” This occurred at 43 ppb (see Table 10) which is much lower than Gallo’s target at 75 ppb. A lower dosing allows Gallo to dial back on the amount of product to add to the wine, thus making it more economically favorable.

There was unfortunately no time combine the syringol and guaiacol. It is recommended that Gallo try different blend ratios of the two in order to attain more favorable flavors. Another recommendation is to dose the second pass column chromatography trials. With the knowledge that the concentrations of guaiacol and syringol were higher in these trials, Gallo should specifically look into dosing bed volumes 5-7 in either Pinot Noir or Chardonnay.

Aspen Simulation Results

Distillation

For simulation purposes, Aspen Plus was used to try to obtain results for a continuous system. The column was designed with 40 stages operating at 25^{o}C and 15 psi. The total waste stream entering was input at 100 kmol/hour consisting of 15% guaiacol, 35% syringol, 25% ethanol, and 25% water, with the side draw stream coming out at 15 kmol/hour. These concentrations were chosen at random since the exact composition of the waste stream is unknown. There were three varied parameters of focus in the simulation: side draw stage, reboiler duty, and bottoms flow. As seen in Appendix III.A, the side draw stage was varied from stage 10 to stage 15 and showed the largest concentration of guaiacol coming out at 74%. Increasing the reboiler heat duty increased the syringol concentration coming out of the bottoms but decreased the guaiacol concentration coming out the distillate. As seen in Appendix III.A, a high reboiler heat duty of 10.75 shows that the system produces 100% syringol at the bottoms and 0% guaiacol at the top. Finally, the higher bottoms flowrate shows and increase of guaiacol concentration at the bottoms, as seen in Appendix III.A.

Next, a rotary evaporator would have been used to evaporate the ethanol and water to obtain pure guaiacol and syringol. A side stream was used to evaluate intermediate compositions in the column. When doing Aspen simulations, the side-draw was used to find the stage and temperature at which the guaiacol concentration was largest. These exact conditions were not replicable at Gallo and bench scale trials did not include reflux changes, reboiler heat duty changes, or the use of a side draw.

Falling Film

Ideally, the falling film evaporator can be simulated as a distillation column with no rectifying section. However, when consulting WPI faculty and AspenTech, it was determined that Aspen Shell & Tube Exchanger program would yield more accurate results, where the film would be on the tube side and the evaporator would be a vertical heat exchanger. This program is not available on the WPI servers and a consultation with the engineers at Gallo determined that software simulation would not be accurate. Gallo has a LUWA-type film evaporator with a 400 gallon capacity, but the temperature and pressure ranges are fixed. The only conditions available for change are to choose between vacuum, steam, or cooling water. While there was no opportunity to use the LUWA while at Gallo, evaporation was conducted at the bench scale level using a rotary evaporator to evaporate alcohol from experimental samples before sending them in for trials. The chiller operated at -3^{o}C and the vacuum pressure was set to about 200 mPa.

Liquid-Liquid Extraction

For simulation purposes, Aspen Plus was used to obtain separation results with the extraction column. In LLE, the ELT-03 waste stream enters in at the top of the column and the solvent enters at the bottom of the column. The waste stream consisted of water, ethanol, and guaiacol and n-butanol was tested as the solvent. Ideally, n-butanol should exhibit preferential affinity towards selected components in the feed (in this case, the guaiacol in the waste steam). As a result, two streams should exit—the extract, which contains the desired components extracted from the feed and the raffinate, which contains residual feed solution and solvent.

In Aspen, various pressures, temperatures, and number of stages were tested. For simulation purposes, it was assumed that the feed solution came in at 100 kmol/hour with 20% ethanol, 70% water, and 10% guaiacol and that the n-butanol solvent stream entered at the very last stage of the column at 200 kmol/hour. At various pressures, there was no change noted with the amount of extraction so other trials assumed the pressure of the column to be at 1 atm (atmospheric pressure). As seen in Appendix III.B, the separation increased at high temperatures and more stages. However, Aspen was not extracting all the water from the feed stream, and it instead came out with the rest of the extract. This does not represent the bench-scale trials at Gallo since pressure or temperature were not changed in the separatory funnel. It is recommended that with further LLE trials, Gallo looks into simulating a settler-mixer instead of an extraction column. This may give more accurate separation results.

Conclusions and Recommendations

In essence, the completion of this project was a successful continuation of Devin Ergler's work and the work performed by WPI students in 2014. Both proof of concept and novel experimental trials were conducted. However, there are still many directions that this project can take. Due to the short seven-week window, most trials were not able to be repeated for reproducibility, so it is highly possible that there may be data that needs to be re-evaluated. It is also important to note that the ELT-03 waste stream was never in use during this project, so the expected results will not be an exact match in terms of aroma, methoxyphenol concentrations, etc.

Overall, the best results came from pressure cooking, column chromatography elutions, and dosing. With pressure cooking trials, the ideal slurry parameter was determined to be at 5 grams of oak and 10% ethanol and there was indeed a significant methoxyphenol production with the use of K_2CO_3 as the base. It is recommended that the ratios of KOH and K_2CO_3 are varied to see the effects on the tannin production while decreasing the likelihood of foaming reaction. With column chromatography, the second pass elution showed promising results, both aromatically and analytically. There reached a point where the aroma was predominately smoky and smelled like pure guaiacol, of which analytical results showed high concentrations. The highest concentration of guaiacol and syringol in both elutions occurred around 40-60% ethanol, which is an economic advantage for Gallo. This is because high proof ethanol is costly, so with the knowledge that elution results are optimal at 40-60%, higher concentrations of ethanol do not need to be used for elution, saving the company money. Though the elution results were positive, the column adsorption results were poor. This may be due to the increased number of tannins present in the feed, which left less room for the guaiacol and syringol to adsorb onto the FPX-66 resin. It would be interesting for Gallo to try and neutralize or change the pH of the feed (which started at a pH of approximately 3) to see how it would affect the affinity of the tannins on the resin column.

With wine dosing, the best aroma/flavor was achieved at approximately 43 ppb of guaiacol which allows the company toe dial back on their 75 ppb guaiacol goal in each sample. This promising result should be further expanded by conducting more tasting and spikings by blending the syringol and guaiacol. Gallo should also spike the second pass elutions as those samples have a higher concentration in guaiacol and syringol and may lead to even more successful results.

Batch distillation, steam stripping, and LLE yielded mediocre separation results. For batch distillation and steam stripping, the separation was as expected with guaiacol coming off the condensate and syringol concentrating in the residual/bottoms. In order to improve separation, Gallo should look into sequential distillation. Since there was more guaiacol in the bottom during steam stripping, Gallo should try to first steam strip the waste stream and then batch distill the bottoms in order to see the separation. LLE results essentially showed the entire feed matrix coming out with the solvent stream, indicating that a five stage extraction may be too high. It would be interesting for Gallo to do a simple one stage extraction to get a better idea of the separation in the raffinate and extract streams. These trials were also run at standard temperature and pressure. While there would be no way to change the pressure in a separatory funnel, it would be interesting to see the separation that would occur when the feed is subject to pH and temperature changes.

Over the course of this project, there were some drawbacks that prevented extensive data analysis. First of all, there were many limitations with the bench scale equipment. The pressure cooker occasionally had pressure leaks coming out the top, making it difficult to maintain steady pressure. With steam stripping and batch distillation, the limitations hindered efforts to change parameters for separation. There were several limitations on the equipment: lack of ability to adjust pressure, lack of ability to adjust or completely control temperature, lack of ability to fully control reflux ratio on the distillation column, trouble getting steam to rise in the steam stripper, heating capability of the stills, and steam production. All of these limitations made it incredibly difficult to vary the separation parameters and find the best option available to separate the tannins. Should these limitations be addressed, then more thorough research and separation trials could be completed. A huge challenge in this project was conducting six different types of experiments, while running multiple pieces of equipment simultaneously. It was very difficult to monitor multiple experiments alone, as the equipment would occasionally malfunction, thus shifting focus to one experiment. However, while on the other side of the lab, another piece of equipment would also malfunction, causing a vicious cycle that hindered not only the precision of lab data, but also lab analysis. There was not much downtime in the lab for data analysis due to the demand of watching multiple pieces of equipment. This problem, along with lack of lab accessibility (interns were not allowed to be in the lab without other employees present) meant that there was a very small time frame left for data analysis. This project was heavily dependent on past research which Gallo would not distribute prior to the start of the project due to confidentiality restraints. This made days at the lab very strenuous with research and experimental trials, leaving less time for calculations and data analysis. Effective work could have been conducted if increased time was spent on analysis and reproducibility. It is heavily recommended that should this be a future project in a seven week time span, it should be a group effort, not a solo project. Planning ahead is another important recommendation, as the ELT-03 stream was not available for use during the project. As a result, different commercial tannins were used. However, the use of these stand-in tannins were not consistent, as different ones were used for different experimental trials. Each of these tannins had various starting concentrations of guaiacol and syringol, so these results may not be true when reproduced when using the ELT-03 waste stream as the feed. These recommendations should help in developing future projects related to tannin extraction and separation.

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Appendices

Appendix I: Experimental Procedures

I.A Cooking:

1. Increase the pH of using KOH oak slurry to desired starting pH.
2. Load the slurry into the Soxhlet high pressure extractor.
3. Add the top and use an allen wrench to tighten the screws.
4. Put the extractor in the insulated woven jacket and place on top of heating mantle.
5. Turn the heating mantle on high and increase the powerstat to 90-100 Volts.
6. Once the pressure gage reaches 10 degrees less than desired pressure, turn off heating mantle and powerstat.
7. Once pressure stabilizes, turn the powerstat to 70-75.
8. Start cooking timing when desired pressure is reached.
9. Adjust the power as needed to keep pressure constant and prevent overshoot.
10. Once cooking has finished, turn off the powerstat and take the extractor out of the jacket.
11. Place the extractor in an ice bath until the pressure decreases to 0.
12. Decrease the pH of the cooked product to 3-3.5 using citric acid or potassium carbonate and store in 750 mL bottles for further processing.

I.B Liquid-Liquid Extraction:

1. Place measured amount of solute and solvent in the separatory funnel.
2. Put a stopper on the funnel and slowly mix the contents of the funnel.
3. Once mixed, allow for the particles to settle and two distinct phases will appear: the hevy phase and the light phase. The heavy phase will be the solute (containing organics) and the lighter phase will be the solvent with extracted components.
4. Pour out the two phases into separate beakers.
5. If further separation is necessary, use the already extracted solute with fresh solvent and repeat steps 1-4.

I.C Column Chromatography:

1. Set up the chromatography column and cover the filter with a small piece of steel wool.
2. Obtain a 50 mL FPX-66 resin, mix with a small amount of DI water to create a slurry and pour into the column to pack it.
    a. Tap the bed to get rid of air bubbles.
3. Turn the pump on and run DI water slowly through the column to clean the resin.
4. Load feed through the column, collecting 4L fragments and submitting samples for analysis.
5. Once the feed runs through, the desired aromas should be held tightly by the resin.

6. Regenerate the column by eluting it with 2 bed volumes (100 mL total) of various ethanol solutions, starting with the most diluted concentration.

I.D Rotary Evaporator (Rotovap):

1. Set chiller temperature to -3 degrees Celsius.
2. Set water bath temperature to 75-80 degrees Celsius.
3. Grease round bottom flasks and clip the collection and sample flasks onto the apparatus.
4. Close the air valve to pressurize the system.
5. Lower the round bottom flask (with the feed inside) into the water bath and start the motor to start rotating the flask.
6. Start the pressure at 350 mPa and slowly decrease to avoid dumping.
7. Allow evaporation to occur until desired ratio of condensate to residual is attained.

I.E Liquid-Liquid Extraction

1. Place desired amount of feed in funnel, ensuring that the stopcock is in close position.
2. Add solvent to the funnel and close the funnel with a stopper.
3. Gently mix the two liquids by slowly flipping the funnel back and forth a few times.
4. Place the funnel back on the ring stand and remove the stopper and allow the phases to separate.
5. Collect the different phases separately.

I.F Steam Stripping

1. Obtain:
    a. One heating mantle with controller
    b. One hot plate/stirrer with stir bar
    c. One one-liter double-necked round-bottom flask with plug
    d. Two beakers or Erlenmeyer flasks for hold unstripped and stripped liquid
    e. Any number trays worth of oldershaw columns
    f. One distillation column middle-stage liquid collector
    g. One distillation column middle-stage liquid injector
    h. One distillation reflux/condenser head
    i. Two 24/40 to 29/42 GGJ adapters
    j. Boiling stones
    k. Ring stands for securing glassware
    l. Flask for collecting distillate
    m. One digital readout peristaltic pump with 24-gauge masterflex tubing
    n. One glass elbow with ball-in-socket GGJ for collecting stripped bottoms
    o. High-temperature tubing for collecting stripped bottoms

2. Fill three-quarters of one round bottom flask with a saturated salt solution (note: salt is optional but will create hotter steam temperatures) and drop in a few boiling stones. Keep more pure water reserved for refilling.

3. Put water-filled flask in heating mantle and turn on the heat to 80-100.

4. Stack the glassware in the following order. Please apply a small amount of grease on all GGJ's:
    a. Liquid collector (must be adapted from 24/40 to 29/42)
        i. Attach glass elbow to ball-in-socket GGJ with clip
        ii. Attach high-temp hose to glass elbow
        iii. Put end of hose in clean beaker to collect water during heat-up
        iv. Plug open port with stopper or thermometer
    b. Oldershaw column
    c. Liquid injector
        i. Attach masterflex tubing to injection port
        ii. Plug open port with stopper
    d. Condenser/reflux head (must be adapted from 29/42 to 24/40)
    e. Attach collection flask to reflux head using keck clip
    f. Insulate areas of single-walled glass using aluminum foil or padded insulation.

5. Put the liquid to be stripped on the hot plate/stirrer. Heat and stir, but monitor the temperature closely. Do not allow this liquid to boil or evaporate significantly unless a reflux is set up.
    a. Note: pre-heating the liquid will improve the efficiency of the stripper, but over-heating the liquid with cause the masterflex hose to break down as it is not rated for high temperatures. Significant evaporation of this liquid will cause fractionation before the stripper and this will skew results.

6. Allow the glass to heat up. Watch the distillate collection for condensate dripping and watch for the thermometer on the top of the reflux head to reach over 98 C. Once this happens, turn the heat up to max. This might take quite a while, an hour or more. Collect the condensed water in the collection flask, but remember to switch this flask out before collecting product

7. Slowly introduce the liquid to be stripped to the top of the column by turning on the peristaltic pump.
    a. Start with a low setting, around 10, and ramp up slowly. Monitor the vapor temperature at the top of the column and make sure it is stable. Make sure there is vapor reaching the condenser. Ideally the liquid would be loaded as quickly as possible without
    b. Monitor the color of the stripped bottoms and when the bottoms begin to change color, begin collecting in an empty, clean beaker or flask.
    c. If the top stages of the column cool down too much and vapor fails to reach the condenser, pause the pump and close the distillate collection valve to allow vapor to reflux back into the top of the column. Then restart the column following steps 7a and 7b.
    d. Monitor the level of the water at the base of the column. If the level drops below the top of the mantle, pause the column following step 7c and refill the flask with more pure water, preheated would be best.

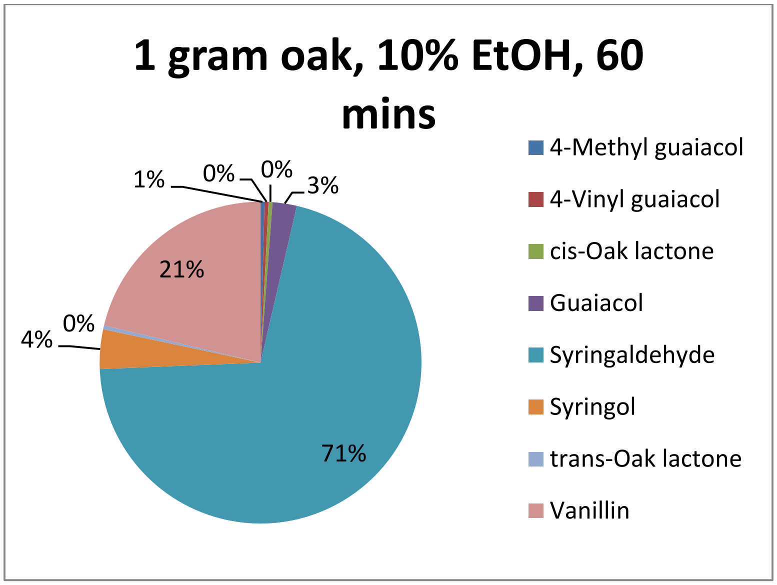
8. Once the stripping liquid is gone and the bottoms turns back to water, pause the column following step 7c. Empty the distillate collection flask and decide whether or not to do another pass on the stripped material.
    a. If so, restart the stripping process with the collected bottoms.

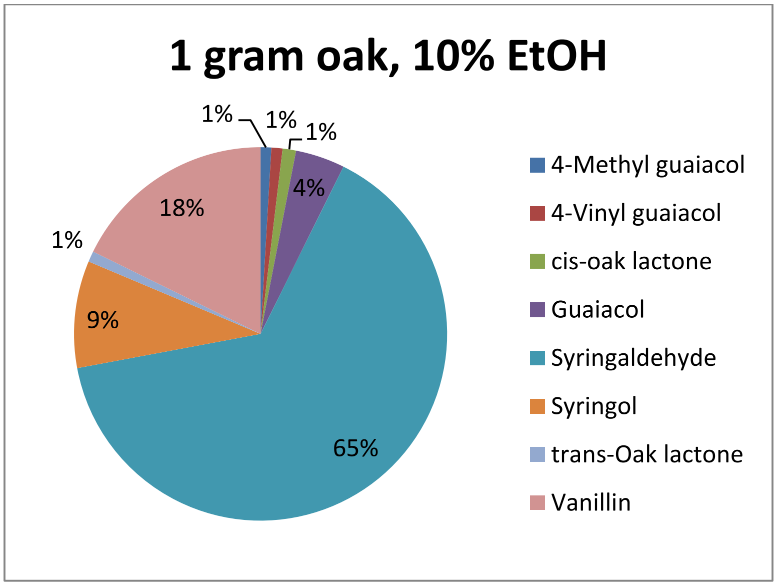
b. If not, restart the process with pure water instead. Allow this to run for some time to rinse out the system and then turn off the heat and the pump. Allow the column to cool down while the chiller still runs.
c. Once water at the base of the column ceases to boil and vapor is no longer seen at the top of the column, turn off the chiller.
d. Disassemble and clean glassware once cool

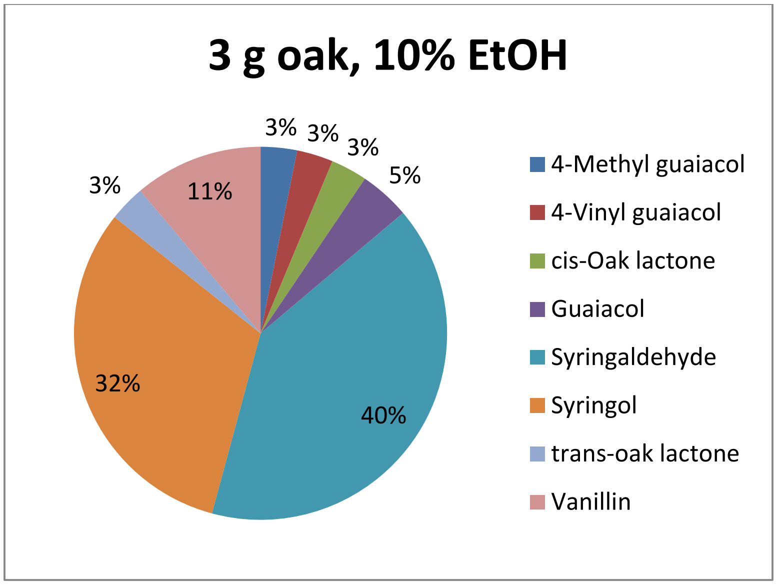
Appendix II: Important Data/Graphs/Pictures

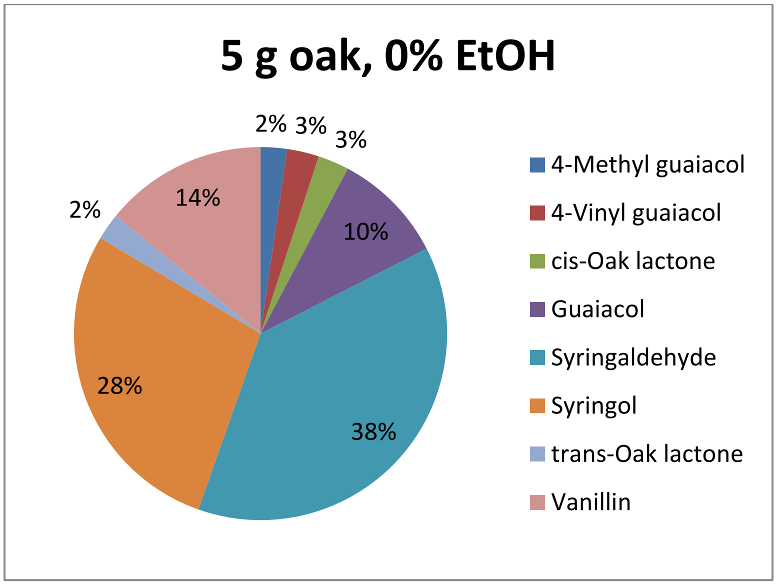
II.A Cooking

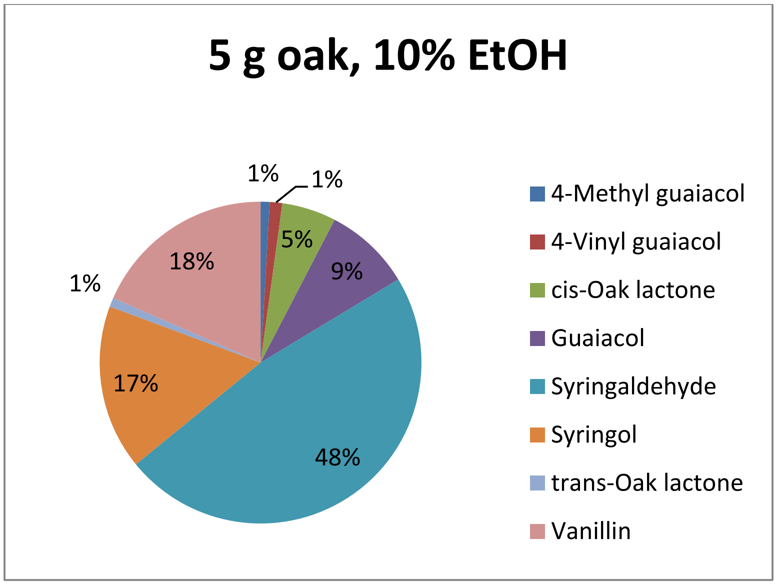
TSS & Percentage Alcohol

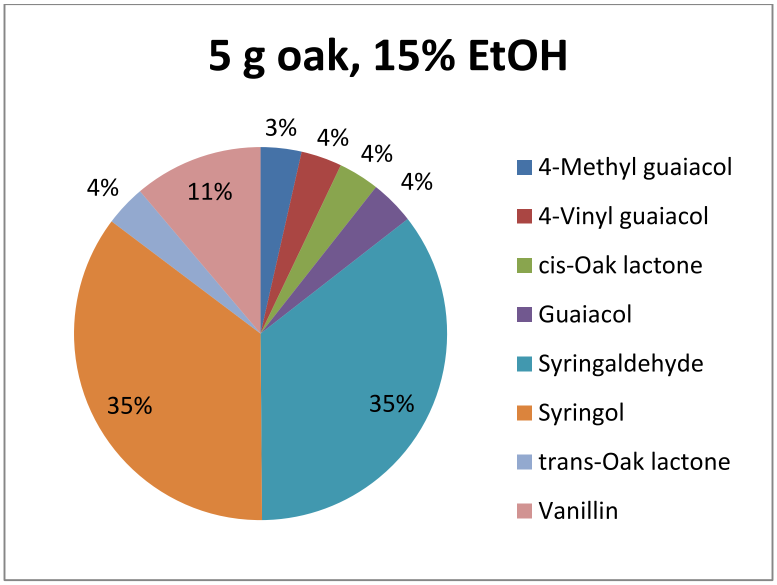


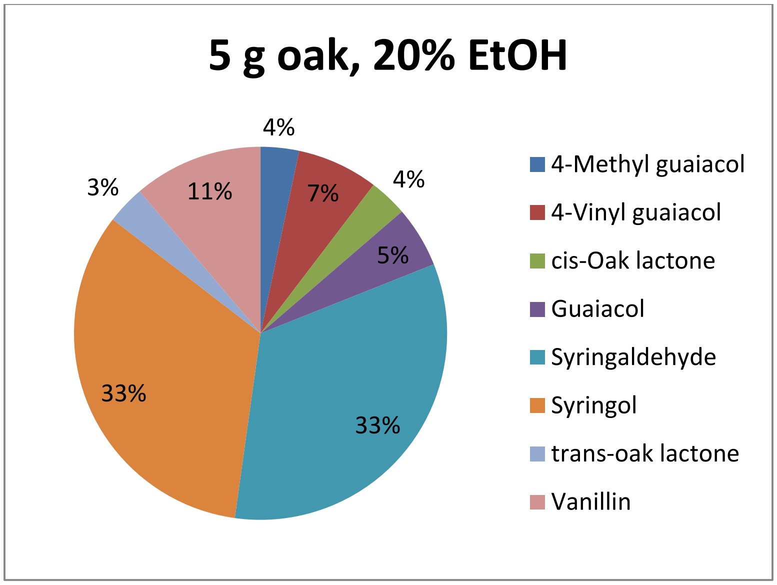


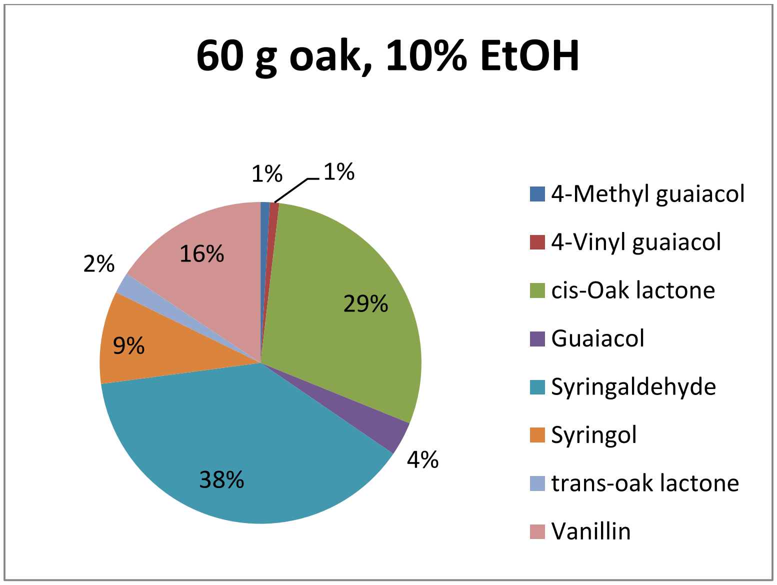

3 g oak, 10% EtOH
3%
3%
3%
5%
3%
11%
32%
40%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin

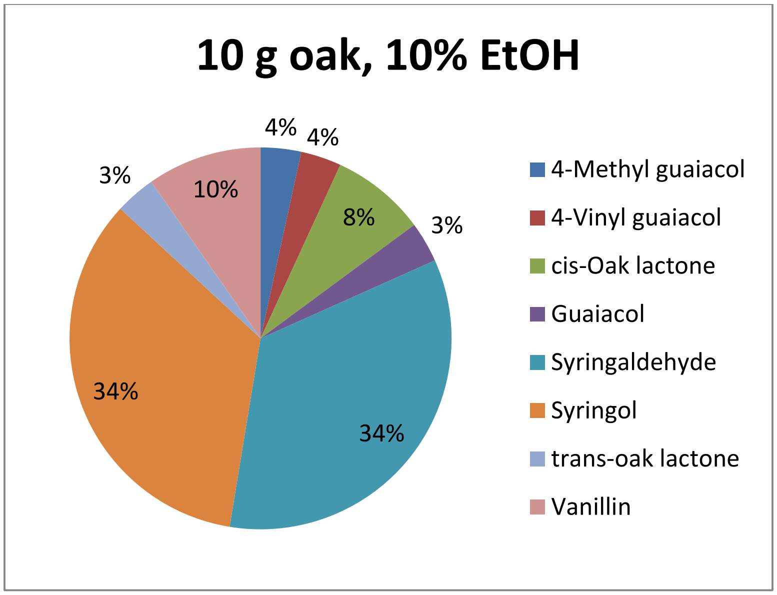

5 g oak, 0% EtOH
2%
3%
3%
2%
14%
10%
28%
38%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-Oak lactone
Vanillin


5 g oak, 10% EtOH
1%
1%
5%
9%
18%
1%
17%
48%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-Oak lactone
Vanillin

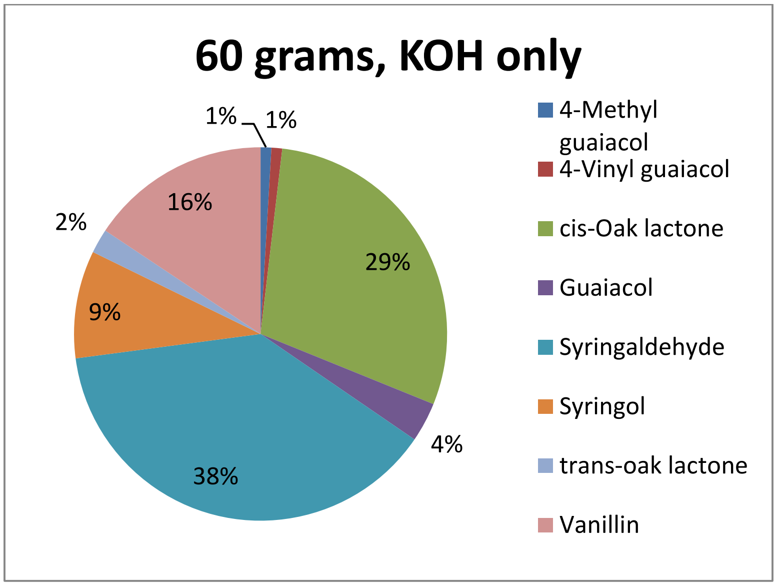

5 g oak, 15% EtOH
3%
4%
4%
4%
4%
11%
35%
35%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-Oak lactone
Vanillin

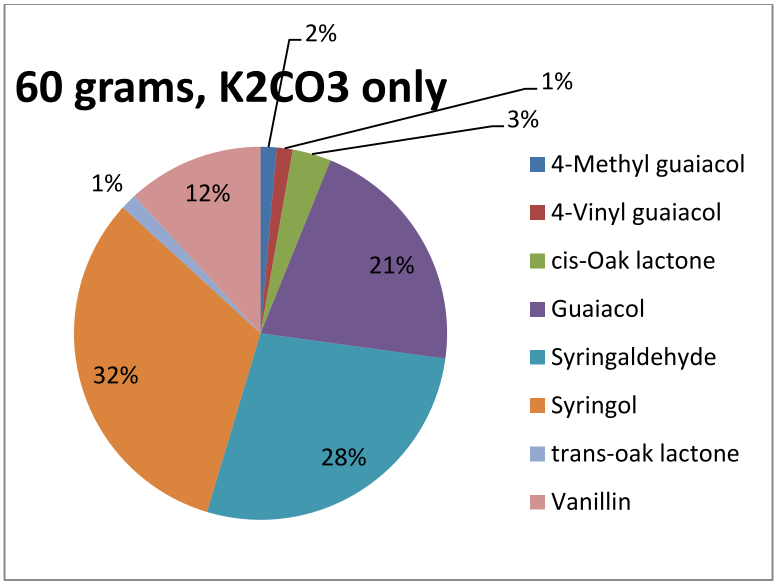

5 g oak, 20% EtOH
4%
7%
4%
5%
33%
33%
3%
11%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin

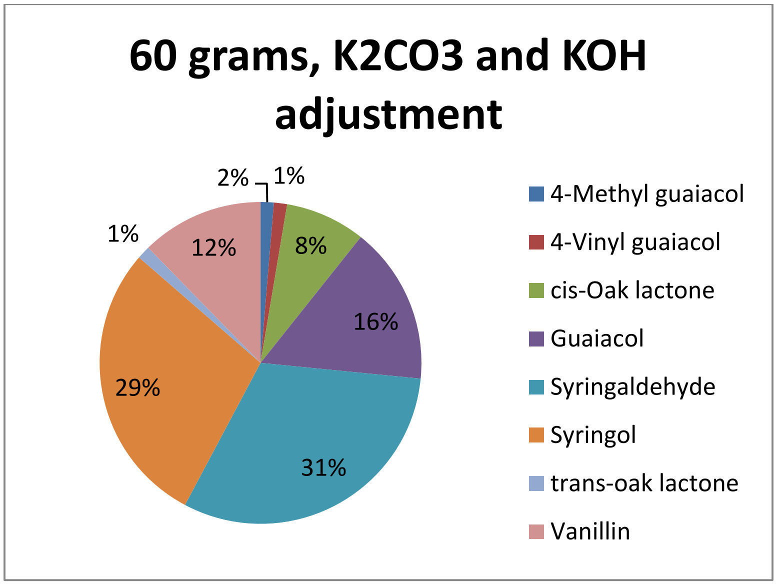

60 g oak, 10% EtOH
1%
1%
29%
4%
38%
9%
2%
16%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin



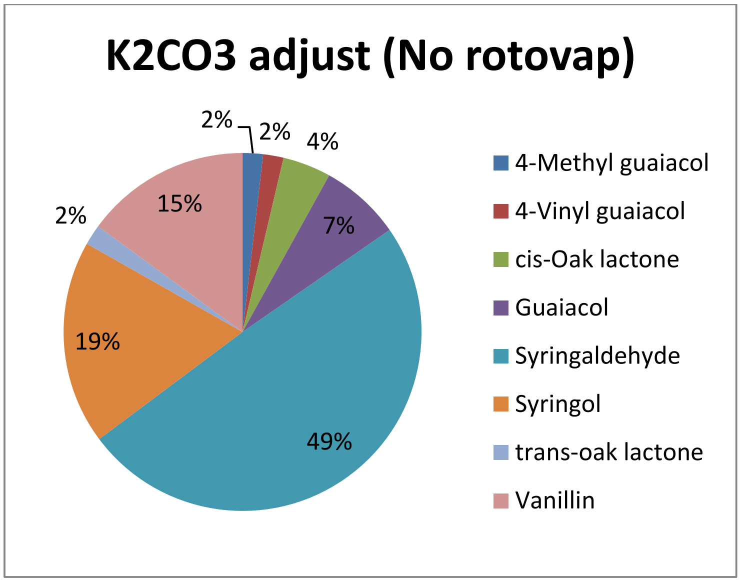
Effect of Base on Methoxyphenols

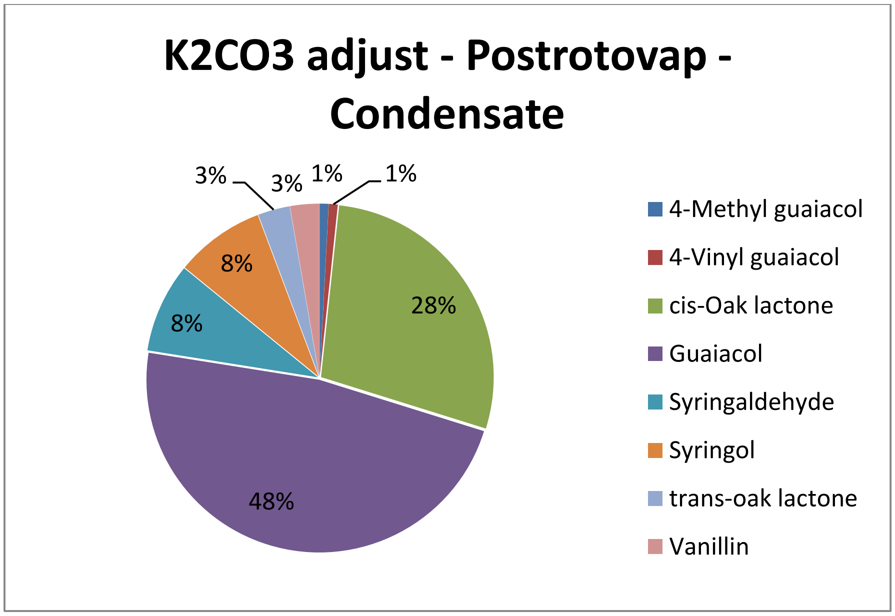


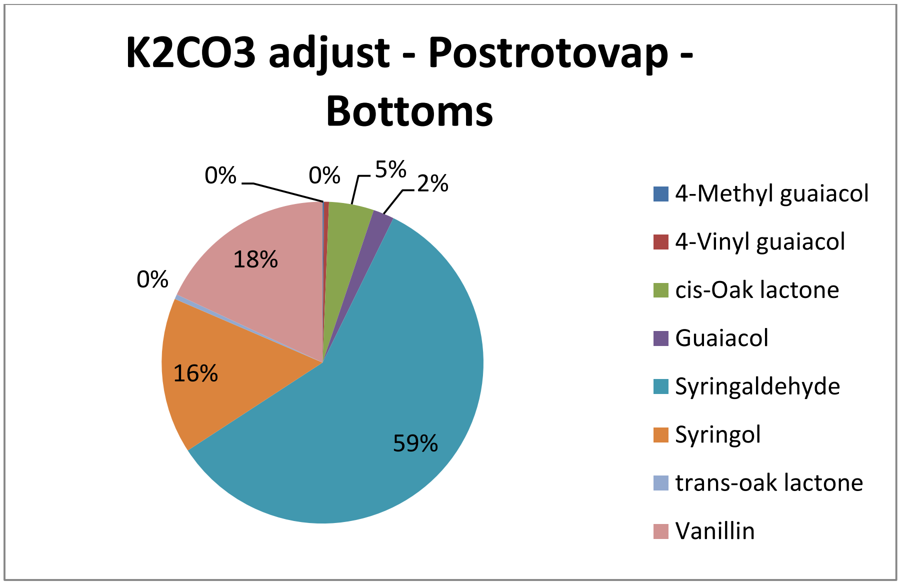

60 grams, K2CO3 only
2%
1%
3%
21%
28%
32%
1%
12%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin

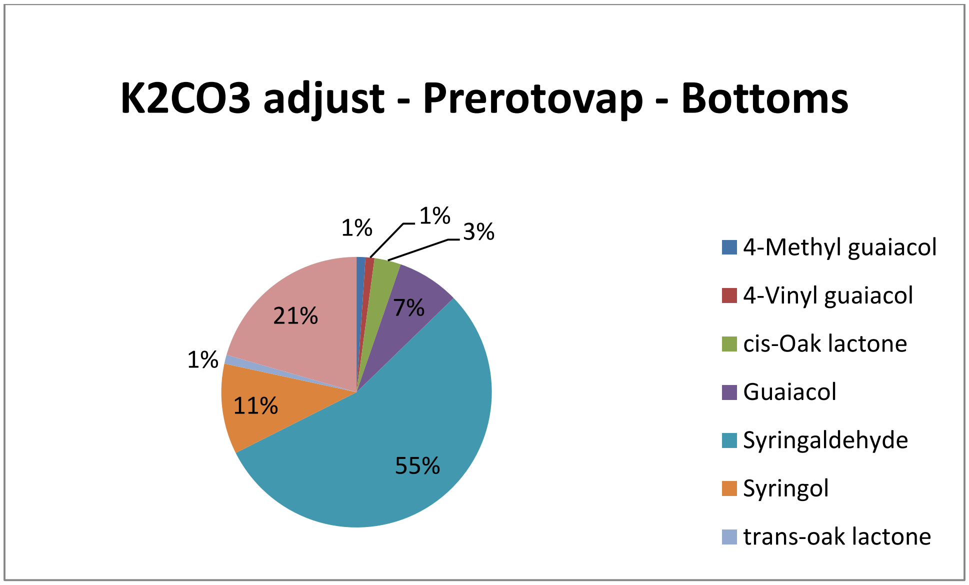

60 grams, K2CO3 and KOH adjustment
2%
1%
8%
16%
31%
29%
1%
12%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin

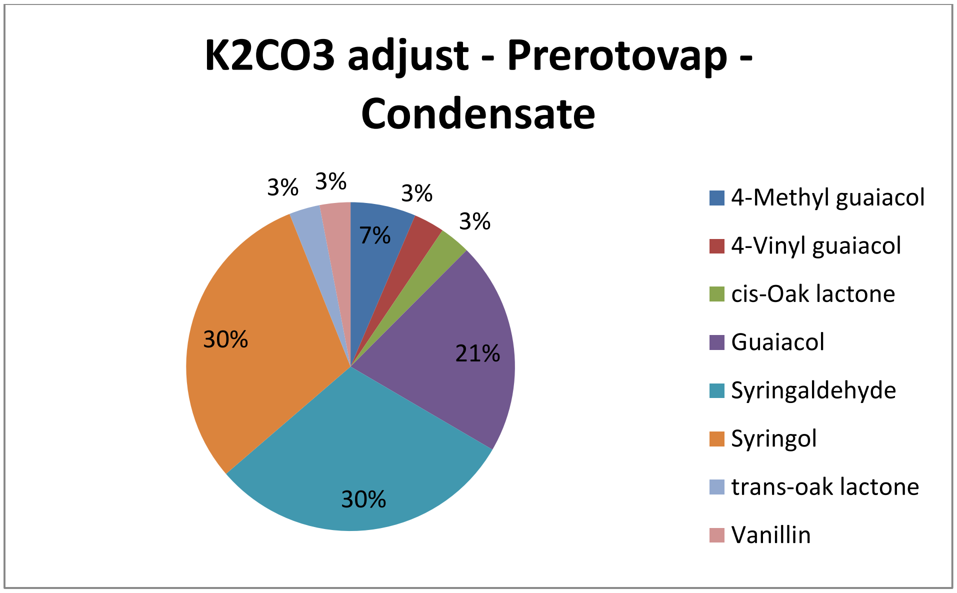
Evaporation



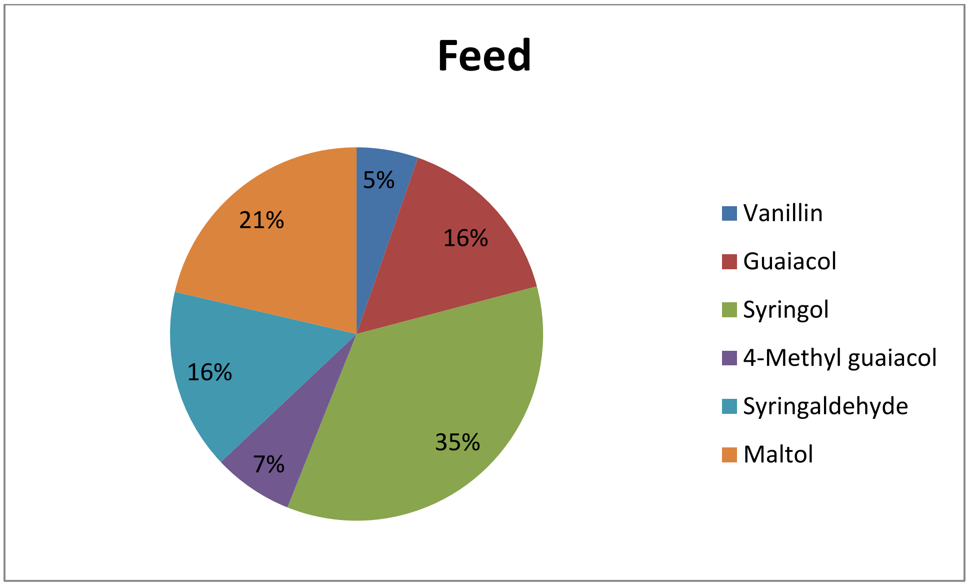


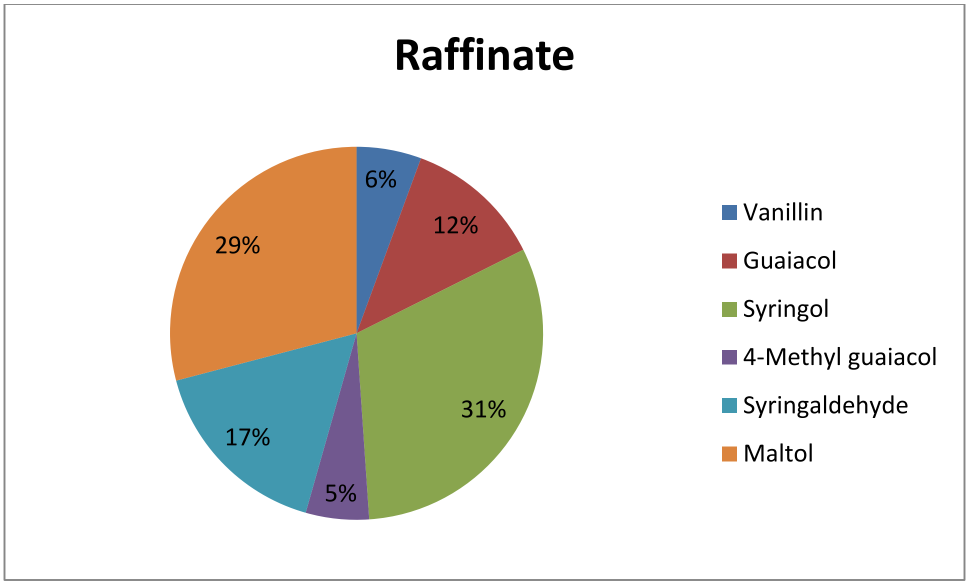

K2CO3 adjust - Postrotovap - Bottoms
0%
0%
5%
2%
0%
18%
16%
59%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone
Vanillin

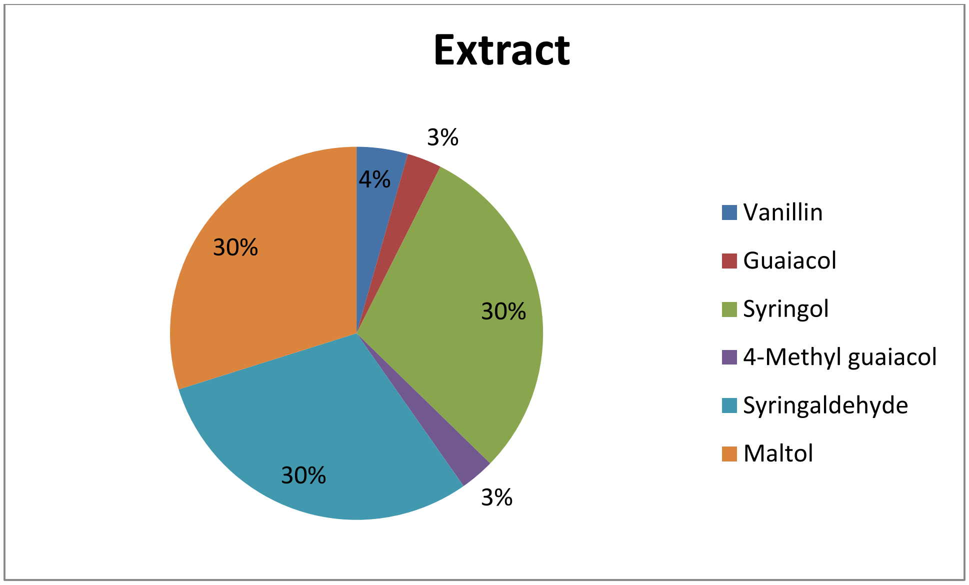

K2CO3 adjust - Prerotovap - Bottoms
1%
1%
3%
7%
21%
1%
11%
55%
4-Methyl guaiacol
4-Vinyl guaiacol
cis-Oak lactone
Guaiacol
Syringaldehyde
Syringol
trans-oak lactone

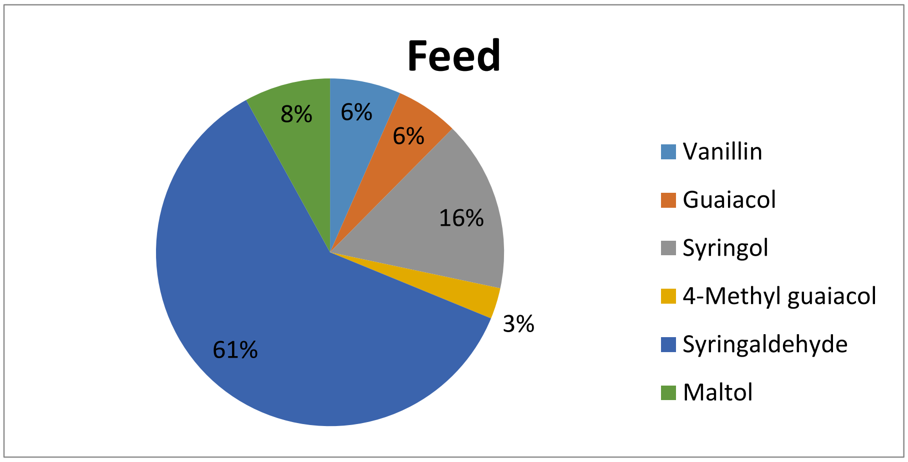


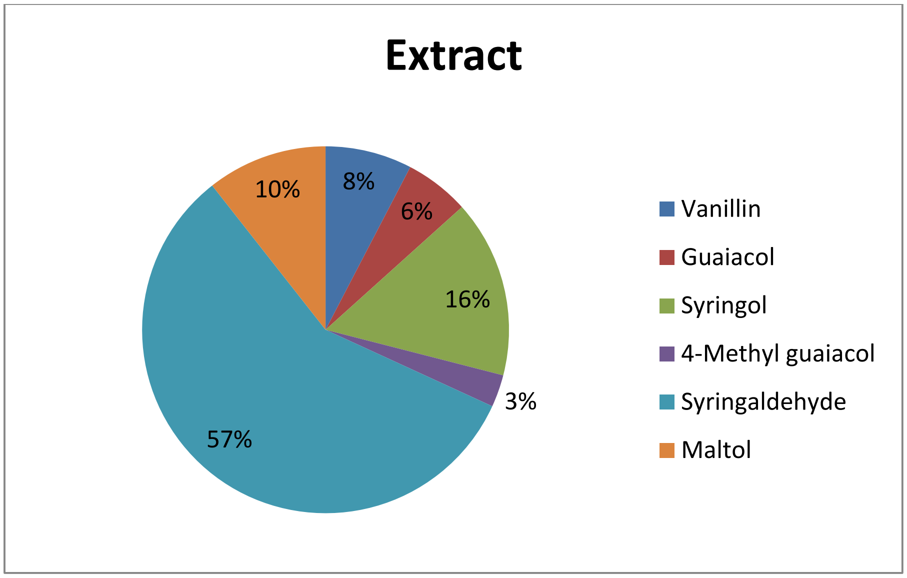
II.B Liquid-Liquid Extraction

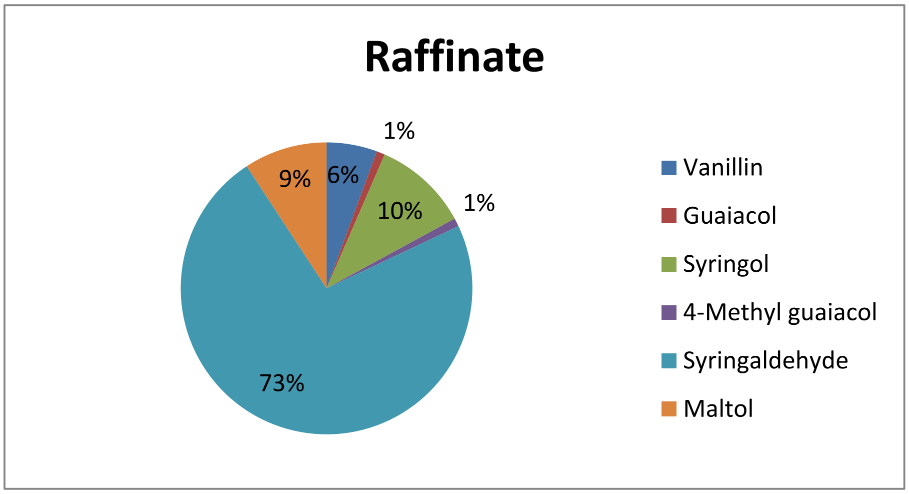



Raffinate
6%
12%
31%
5%
17%
29%
Vanillin
Guaiacol
Syringol
4-Methyl guaiacol
Syringaldehyde
Maltol

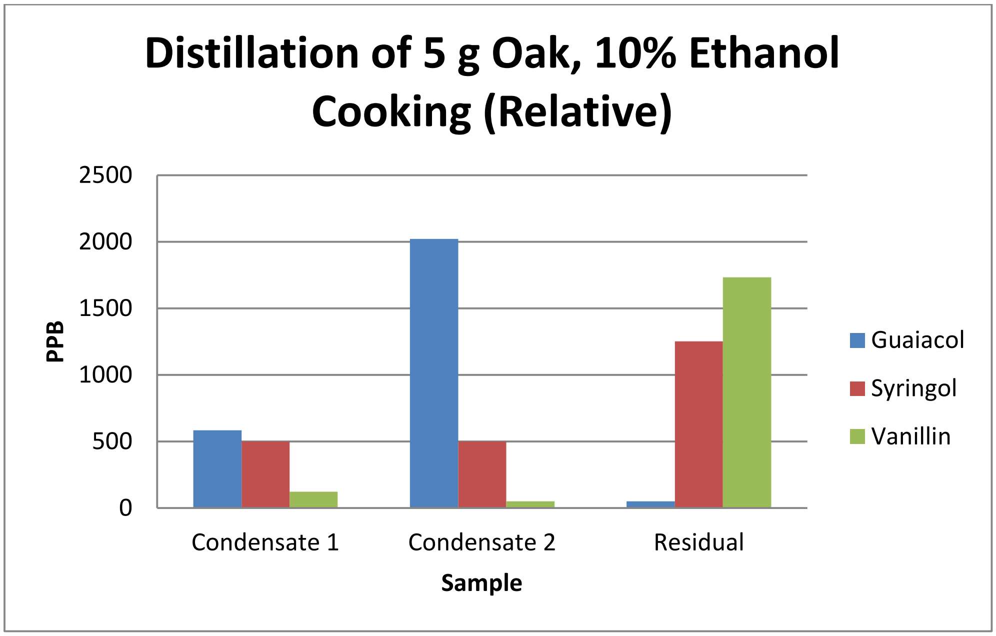

Extract
4%
3%
30%
3%
30%
30%
Vanillin
Guaiacol
Syringol
4-Methyl guaiacol
Syringaldehyde
Maltol

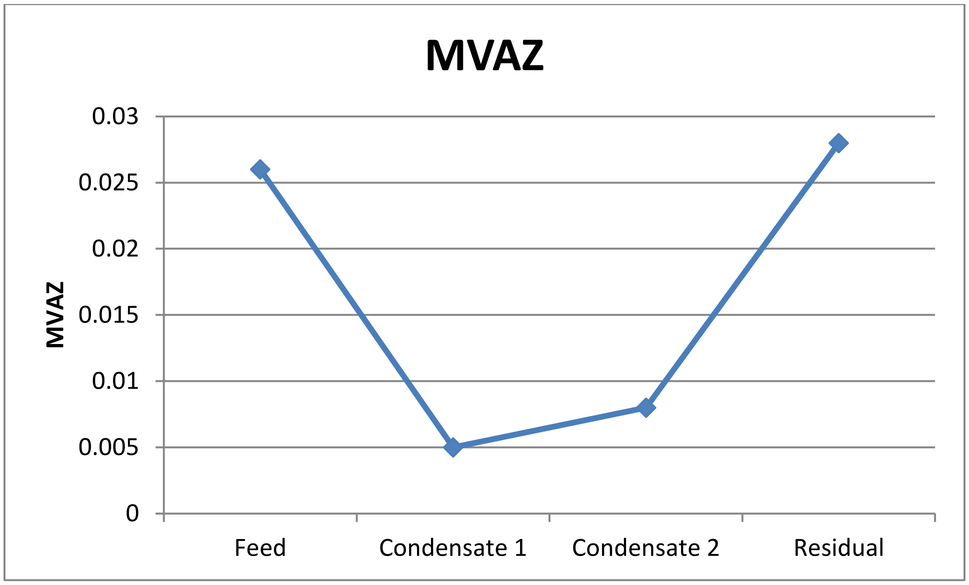

Feed
6%
6%
16%
3%
61%
8%
Vanillin
Guaiacol
Syringol
4-Methyl guaiacol
Syringaldehyde
Maltol

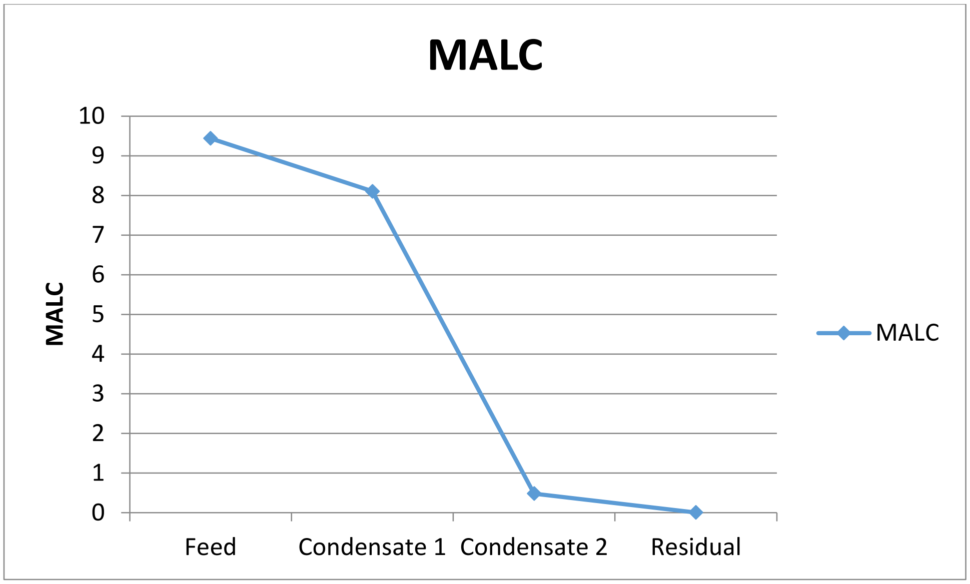

Extract
8%
6%
16%
3%
57%
10%
Vanillin
Guaiacol
Syringol
4-Methyl guaiacol
Syringaldehyde
Maltol

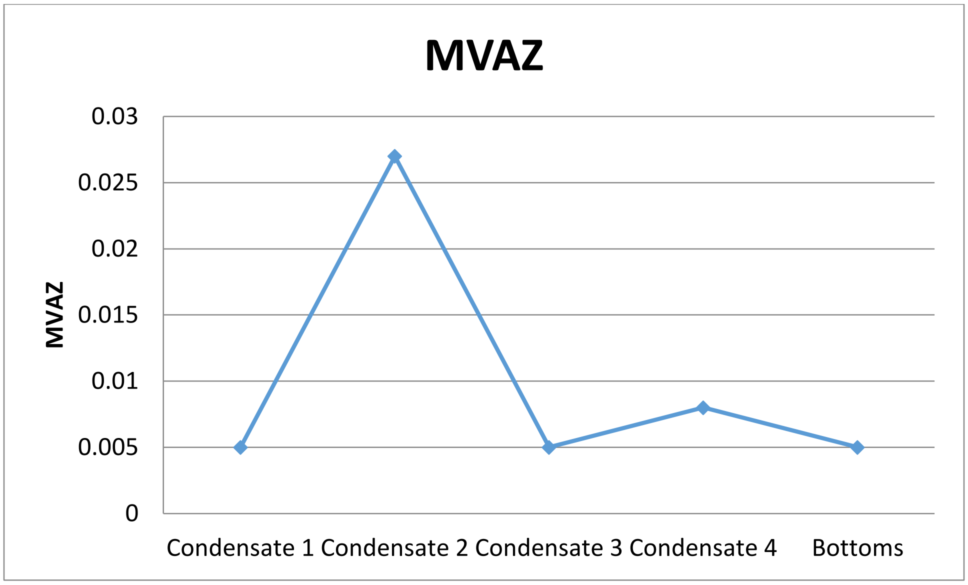

Raffinate
1%
6%
10%
1%
73%
9%
Vanillin
Guaiacol
Syringol
4-Methyl guaiacol
Syringaldehyde
Maltol

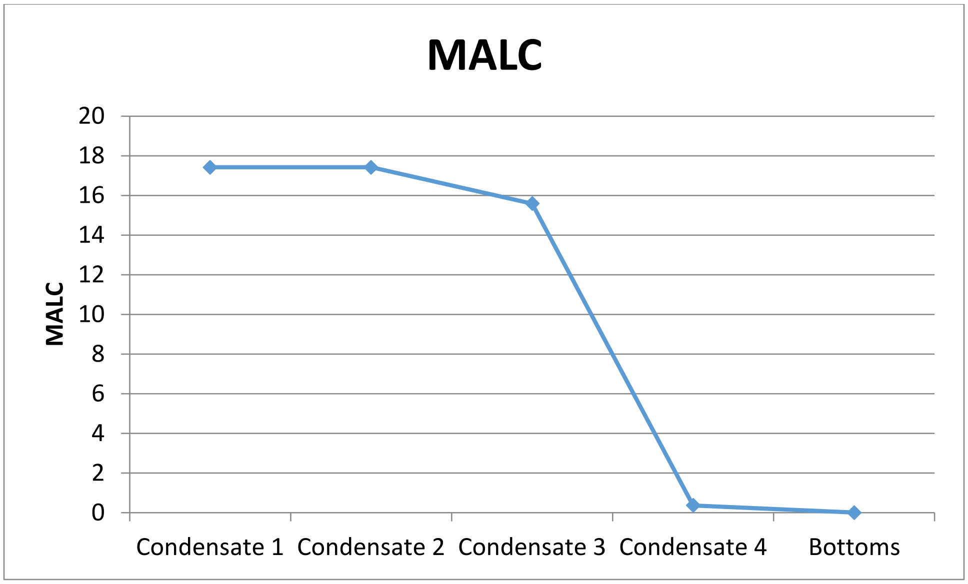
II.C Batch Distillation

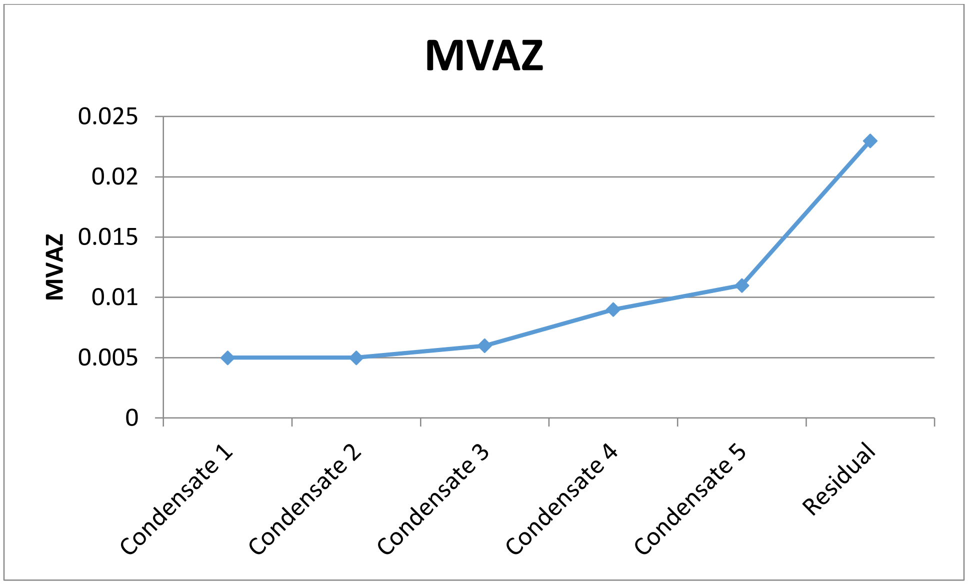





MALC
MALC
10
9
8
7
6
5
4
3
2
1
0
Feed
Condensate 1
Condensate 2
Residual
MALC


MVAZ
MVAZ
0.03
0.025
0.02
0.015
0.01
0.005
0
Condensate 1
Condensate 2
Condensate 3
Condensate 4
Bottoms


MALC
MALC
20
18
16
14
12
10
8
6
4
2
0
Condensate 1
Condensate 2
Condensate 3
Condensate 4
Bottoms


MVAZ
MVAZ
0.025
0.02
0.015
0.01
0.005
0
Condensate 1
Condensate 2
Condensate 3
Condensate 4
Condensate 5
Residual

pKa Results

Trial	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*
Description	**1g, 10%**	**3 g, 10%**	**5 g, 0%**	**5 g, 10%**	**5 g, 15%**	**5 g, 20%**	**10 g, 10%**	**60 g, 10%**
Pre-cook	11.98	12.01	12.03	11.98	12	12.07	12	11.96
Post-Cook	11.5	10.67	10.31	8.06	8.98	8.77	6.98	6.23

	Trial 1								
					CH3COOH +	H20	↔	$HCO3^{+}$ +	$CH3COO^{-}$
		x=acetic acid		I	1.04713E-12			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			3.16228E-12	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*3.16228E-12*			
					2.11515E-12	*M of acetic acid production*			

	Trial 2								
					CH3COOH +	H20	↔	$HCO3^{+}$ +	$CH3COO^{-}$
		x=acetic acid		I	9.77237E-13			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			2.13796E-11	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*2.13796E-11*			
					2.04024E-11	*M of acetic acid production*			

Trial 3									
					CH3COOH +	H20	↔	$HCO3^{+}$ +	$CH3COO^{-}$
		x=acetic acid		I	9.33254E-13			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			4.89779E-11	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*4.89779E-11*			
					4.80446E-11	*M of acetic acid production*			

Trial 4									
					CH3COOH +	H20	↔	$HCO3^{+}$ +	$CH3COO^{-}$
		x=acetic acid		I	1.04713E-12			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			8.70964E-09	

Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*8.70964E-09*			
					8.70859E-09	*M of acetic acid production*			

Trial 5									
					CH3COOH +	H20	↔	HCO3^{+} +	CH3COO^{-}
		x=acetic acid		I	1E-12			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			1.04713E-09	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*1.04713E-09*			
					1.04613E-09	*M of acetic acid production*			

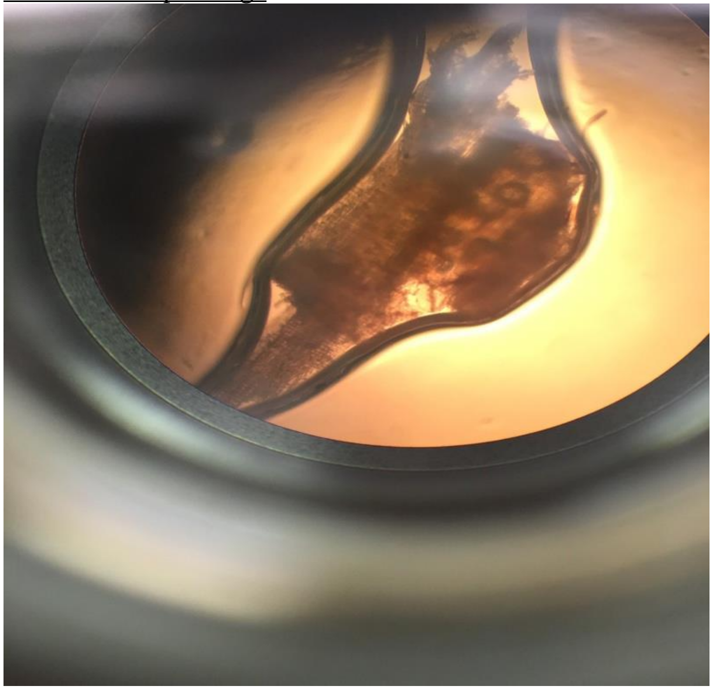
Trial 6									
					CH3COOH +	H20	↔	HCO3^{+} +	CH3COO^{-}
		x=acetic acid		I	8.51138E-13			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			1.69824E-09	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*1.69824E-09*			
					1.69739E-09	*M of acetic acid production*			
Trial 8									
					CH3COOH +	H20	↔	HCO3^{+} +	CH3COO^{-}

		x=acetic acid		I	1.09648E-12			0	0
		Ka = 1.75*10^-5		C	x+			x+	x+
		pKa=4.75		E	**???**			5.88844E-07	
Ka=([HCO3+][CH3COO-]) / [CH3COOH]									
					x=	*5.88844E-07*			
					5.88843E-07	*M of acetic acid production*			

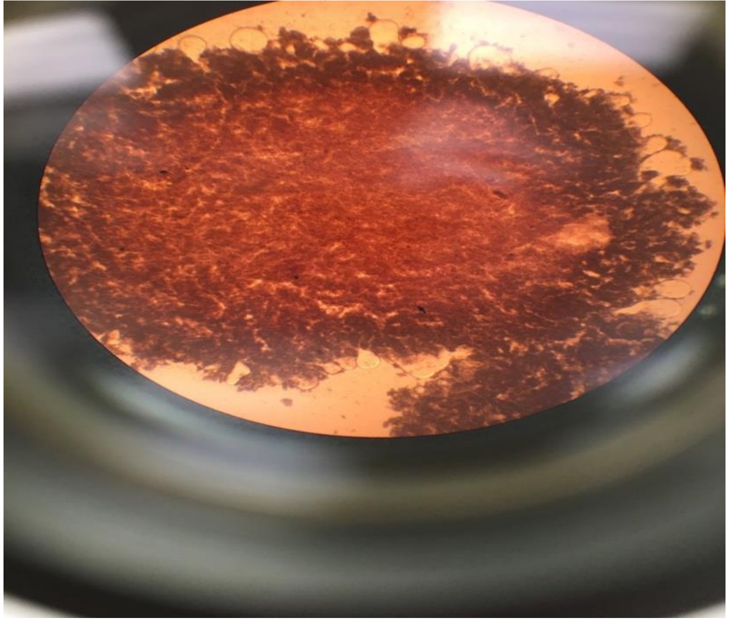
II.D Steam Stripping

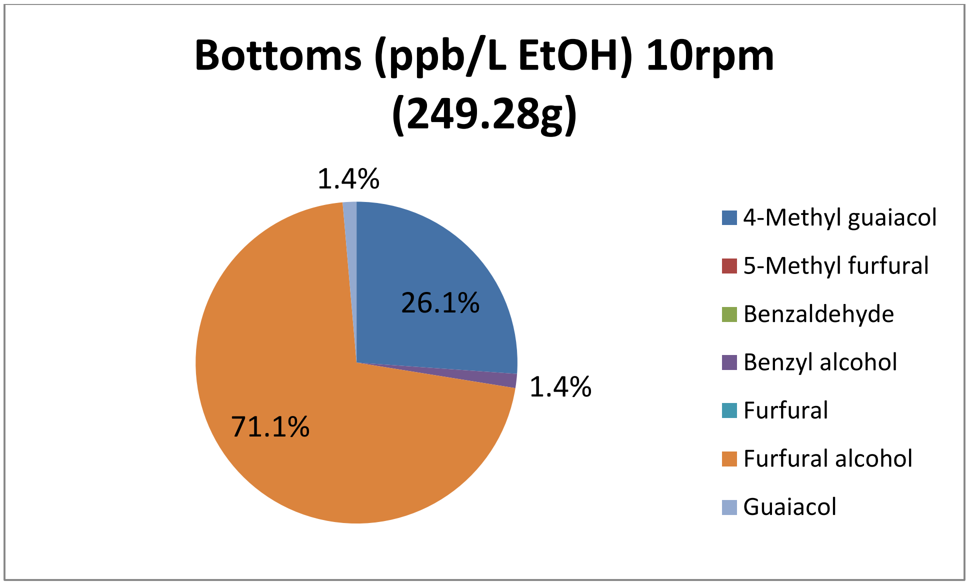
Microscope Images of Solid Oak and Solids Found in CLT04 Stream

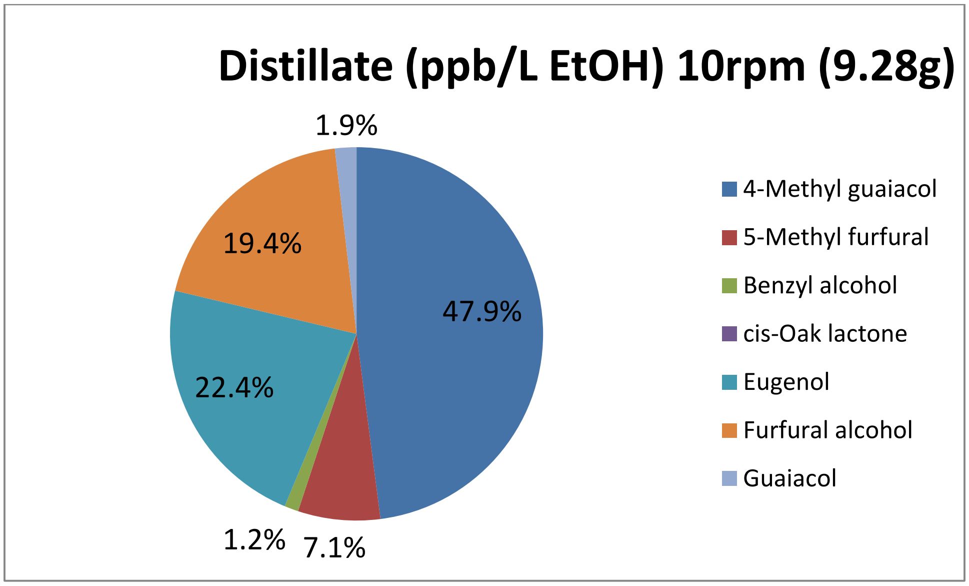
Oak Microscope Image

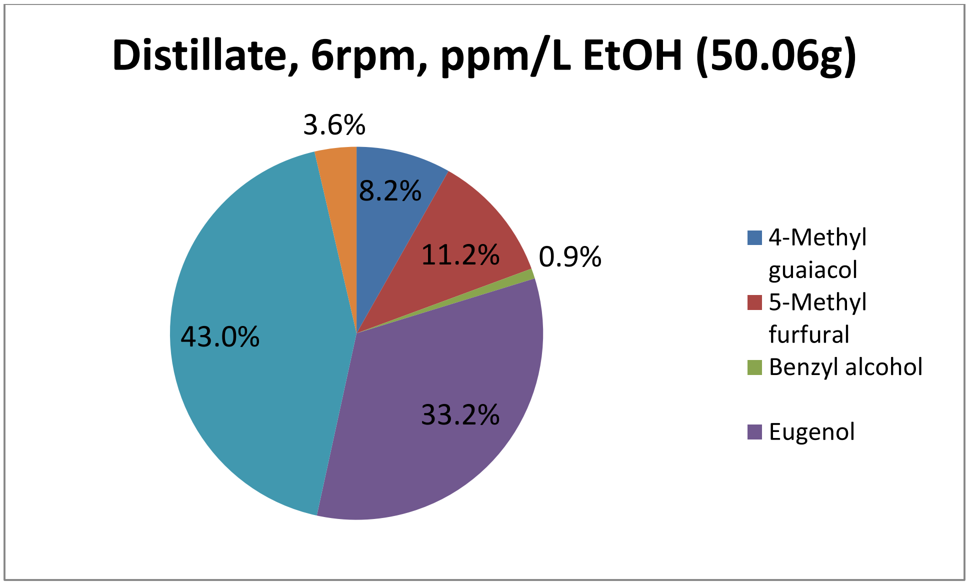


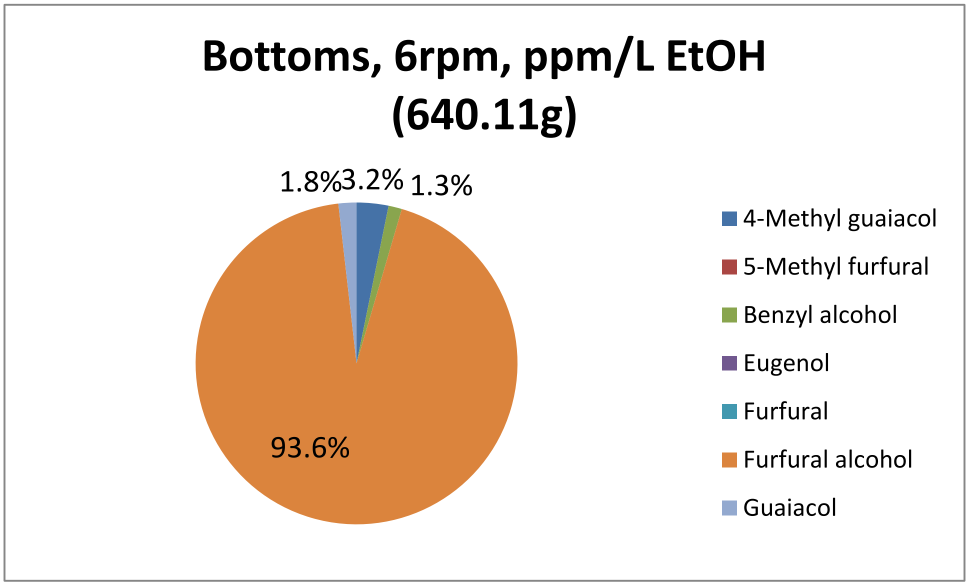
VDR Solid Microscope Image

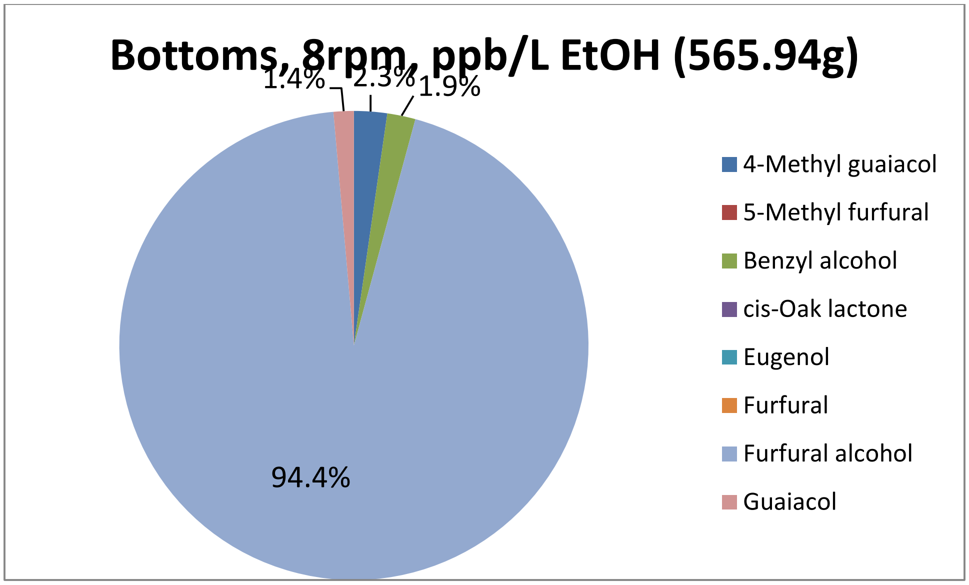



Bottoms (ppb/L EtOH) 10rpm (249.28g)
1.4%
26.1%
1.4%
71.1%
4-Methyl guaiacol
5-Methyl furfural
Benzaldehyde
Benzyl alcohol
Furfural
Furfural alcohol
Guaiacol

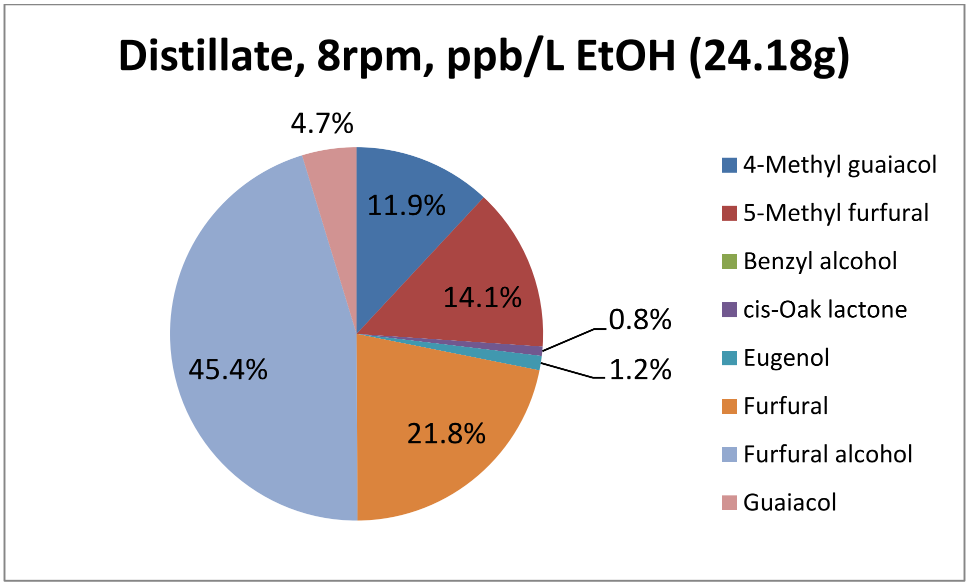

Distillate (ppb/L EtOH) 10rpm (9.28g)
1.9%
19.4%
47.9%
22.4%
1.2%
7.1%
4-Methyl guaiacol
5-Methyl furfural
Benzyl alcohol
cis-Oak lactone
Eugenol
Furfural alcohol
Guaiacol


Distillate, 6rpm, ppm/L EtOH (50.06g)
3.6%
8.2%
11.2%
0.9%
43.0%
33.2%
4-Methyl guaiacol
5-Methyl furfural
Benzyl alcohol
Eugenol

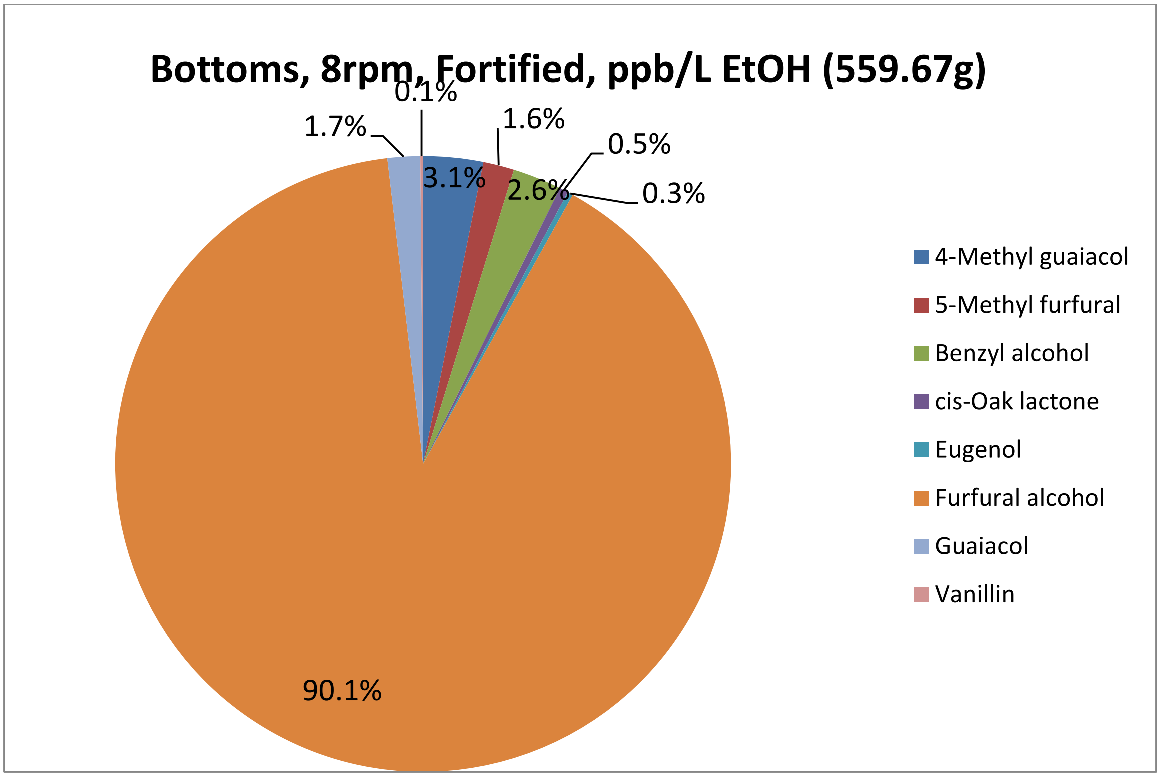

Bottoms, 6rpm, ppm/L EtOH (640.11g)
1.8%
3.2%
1.3%
93.6%
4-Methyl guaiacol
5-Methyl furfural
Benzyl alcohol
Eugenol
Furfural
Furfural alcohol
Guaiacol

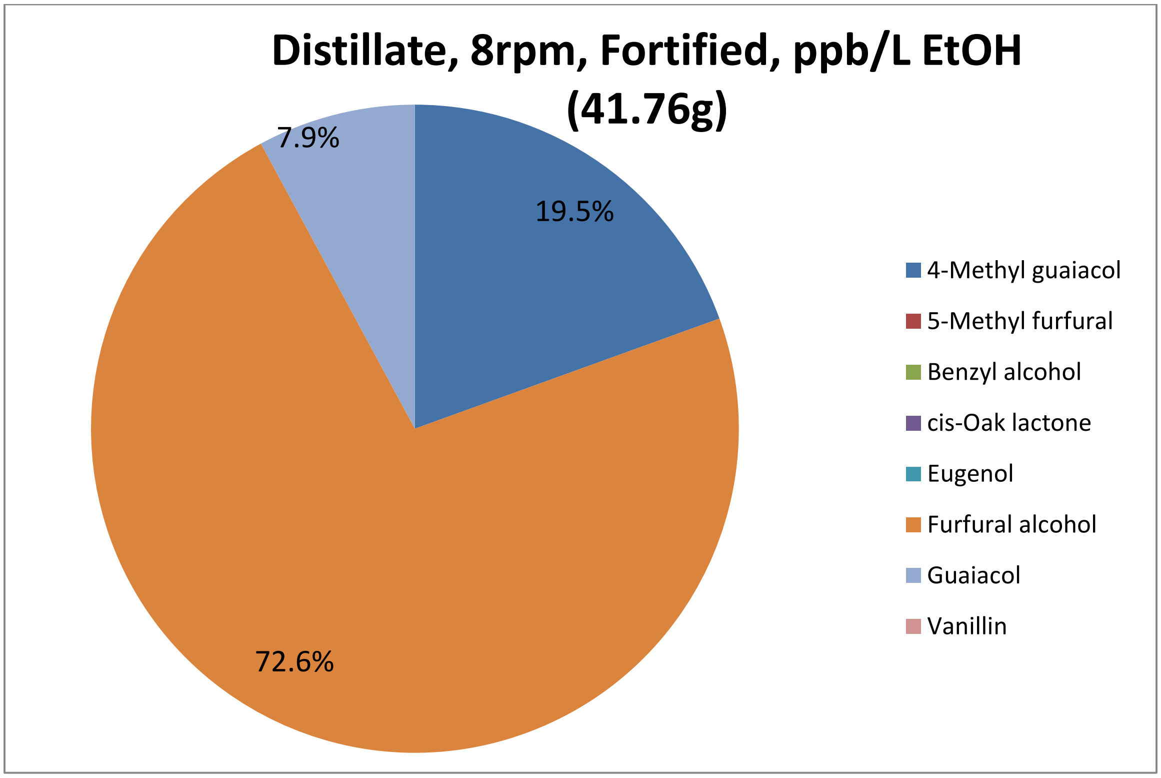


Bottoms, 8rpm, ppb/L EtOH (565.94g)



Distillate, 8rpm, ppb/L EtOH (24.18g)


Bottoms, 8rpm, Fortified, ppb/L EtOH (559.67g)
0.1%
1.7%
1.6%
0.5%
3.1%
2.6%
0.3%
90.1%
4-Methyl guaiacol
5-Methyl furfural
Benzyl alcohol
cis-Oak lactone
Eugenol
Furfural alcohol
Guaiacol
Vanillin


Distillate, 8rpm, Fortified, ppb/L EtOH (41.76g)
7.9%
19.5%
72.6%
4-Methyl guaiacol
5-Methyl furfural
Benzyl alcohol
cis-Oak lactone
Eugenol
Furfural alcohol
Guaiacol
Vanillin

II.E Wine Dosing

Bed Volume #6 (1st Pass Elution)		
Guaiacol	41034.4	PPB
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**
0.18	1.8	**73.86192**
0.09	0.9	**36.93096**

Bed Volume #5 (1st Pass Elution) Rotary Evaporation					
Guaiacol = 28812.1PPB			Syringol = 144965 PPB		
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**	Volume syringol added (mL) in 100 mL wine	Volume syringol in 1L	**PPB Syringol**
0.3	3	**86.4363**	1.3	13	**1884.545**
0.15	1.5	**43.21815**	0.65	6.5	**942.2725**

Bed Volume #4 (1st Pass Elution) Rotary Evaporation					
Guaiacol = 21226.1 PPB			Syringol = 94148 PPB		
Volume guaiacol added (mL) in 100 mL wine	Volume guaiacol in 1L	**PPB Guaiacol**	Volume syringol added (mL) in 100 mL wine	Volume syringol in 1L	**PPB Syringol**
0.5	5	**106.1305**	1.4	14	**1318.072**
0.25	2.5	**53.06525**	0.7	7	**659.036**

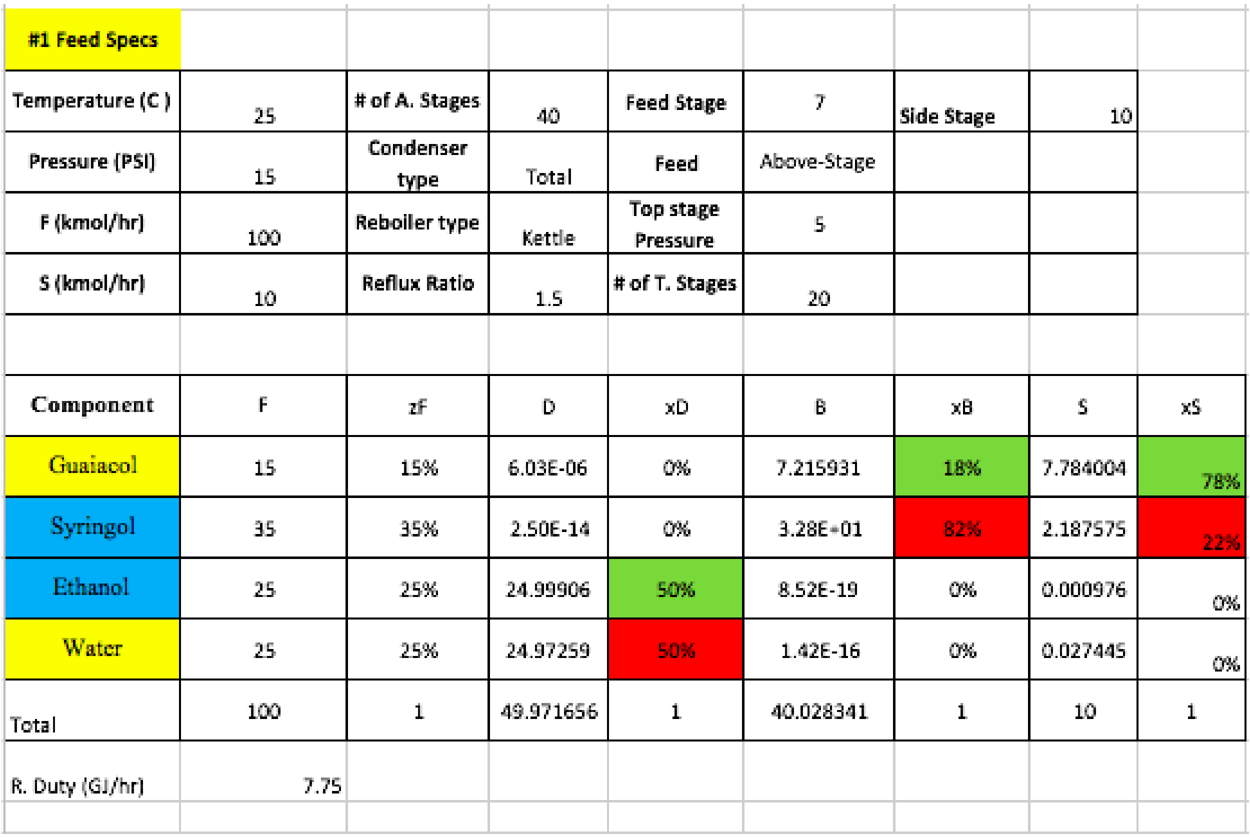
Appendix III: Aspen Results

III.A: Distillation Column

Properties

Units	*METBAR*
Method Filter	*Common*
Base Method	*UNIQAC*

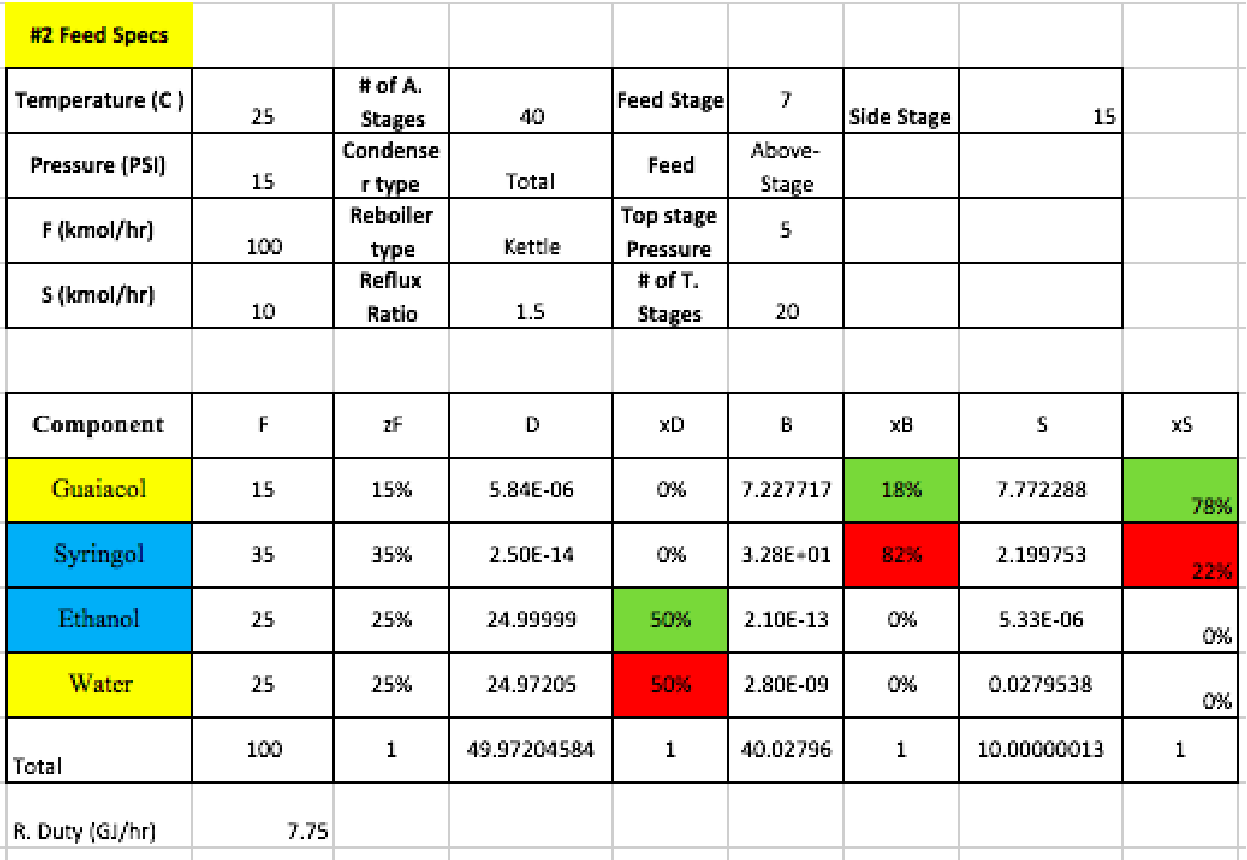
Side Stage Variation



#1 Feed Specs								
Temperature (C)	25	# of A. Stages	40	Feed Stage	7	Side Stage	10	
Pressure (PSI)	15	Condenser type	Total	Feed	Above-Stage			
F (kmol/hr)	100	Reboiler type	Kettle	Top stage Pressure	5			
S (kmol/hr)	10	Reflux Ratio	1.5	# of T. Stages	20			
Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	6.03E-06	0%	7.215931	18%	7.784004	78%
Syringol	35	35%	2.50E-14	0%	3.28E+01	82%	2.187575	22%
Ethanol	25	25%	24.99906	50%	8.52E-19	0%	0.000976	0%
Water	25	25%	24.97259	50%	1.42E-16	0%	0.027445	0%
Total	100	1	49.971656	1	40.028341	1	10	1
R. Duty (GJ/hr)	7.75							

Stages	*Stage Composition*			
	Guaiacol	*Syringol*	*Ethanol*	*Water*
1	*0%*	*0%*	*72%*	*28%*
2	*0%*	*0%*	*29%*	*71%*
3	*0%*	*0%*	*9%*	*91%*
4	*0%*	*0%*	*6%*	*94%*

5	*0%*	*0%*	*6%*	*94%*
6	*2%*	*0%*	*6%*	*92%*
7	*19%*	*53%*	*6%*	*22%*
8	*39%*	*45%*	*2%*	*14%*
9	*69%*	*30%*	*0%*	*1%*
10	*74%*	*26%*	*0%*	*0%*
11	*74%*	*26%*	*0%*	*0%*
12	*74%*	*26%*	*0%*	*0%*
13	*74%*	*26%*	*0%*	*0%*
14	*74%*	*26%*	*0%*	*0%*
15	*74%*	*26%*	*0%*	*0%*
16	*74%*	*26%*	*0%*	*0%*
17	*71%*	*29%*	*0%*	*0%*
18	*61%*	*39%*	*0%*	*0%*
19	*38%*	*62%*	*0%*	*0%*
20	*15%*	*85%*	*0%*	*0%*



#2 Feed Specs

Temperature (C)	25	# of A. Stages	40	Feed Stage	7	Side Stage	15
Pressure (PSI)	15	Condenser type	Total	Feed	Above-Stage		
F (kmol/hr)	100	Reboiler type	Kettle	Top stage Pressure	5		
S (kmol/hr)	10	Reflux Ratio	1.5	# of T. Stages	20		

Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	5.84E-06	0%	7.227717	18%	7.772288	78%
Syringol	35	35%	2.50E-14	0%	3.28E+01	82%	2.199753	22%
Ethanol	25	25%	24.99999	50%	2.10E-13	0%	5.33E-06	0%
Water	25	25%	24.97205	50%	2.80E-09	0%	0.0279538	0%
Total	100	1	49.97204584	1	40.02796	1	10.00000013	1

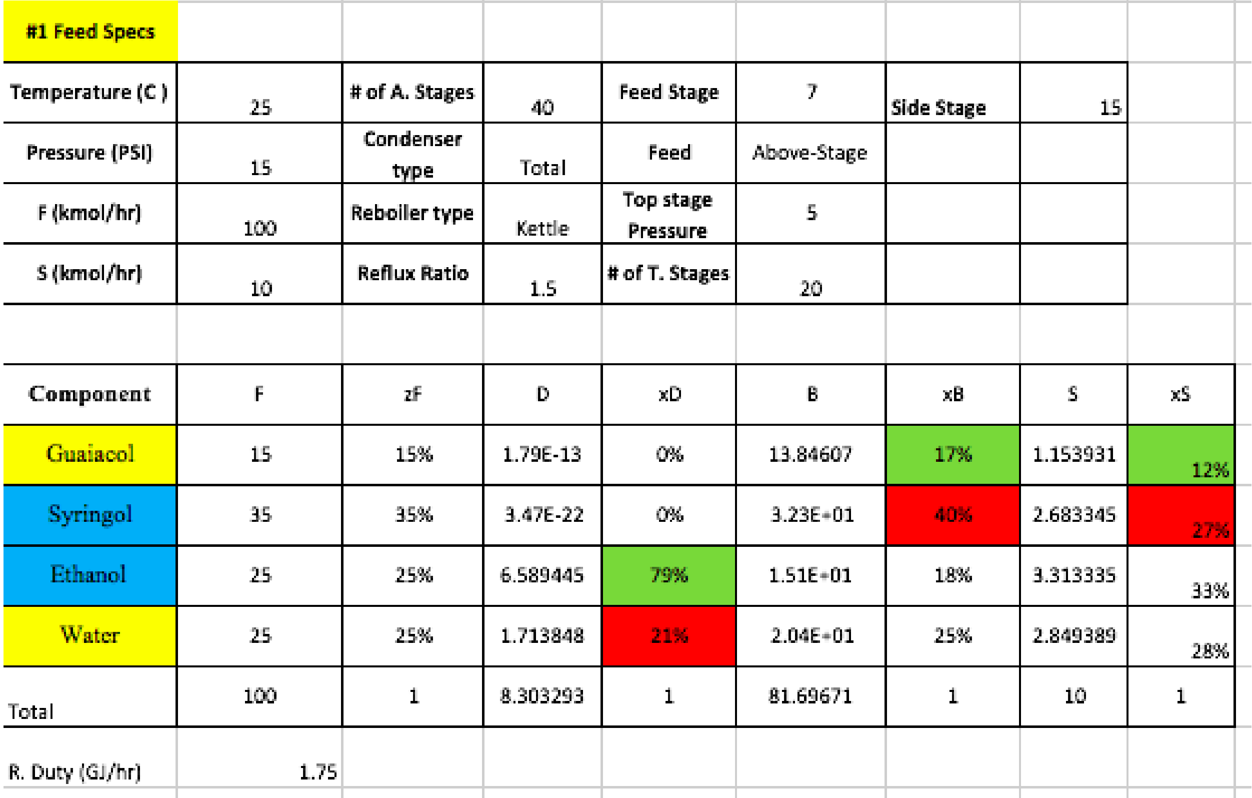
R. Duty (GJ/hr) 7.75

Stages	*Stage Composition*			
	Guaiacol	*Syringol*	*Ethanol*	*Water*
1	*4.52E-07*	*2.40E-15*	*72%*	*28%*
2	*2.36E-05*	*4.29E-12*	*29%*	*71%*
3	*0%*	*8.30E-10*	*9%*	*91%*

4	0%	1.10E-07	6%	94%
5	0%	1.42E-05	6%	94%
6	2%	0%	6%	92%
7	19%	53%	6%	22%
8	19%	54%	3%	25%
9	19%	54%	1%	26%
10	19%	55%	0%	26%
11	19%	55%	0%	26%
12	20%	54%	0%	26%
13	41%	45%	0%	14%
14	69%	30%	5.54E-06	1%
15	74%	26%	1.88E-07	0%
16	74%	26%	5.55E-09	1.35E-05
17	71%	29%	1.60E-10	4.63E-07
18	61%	39%	4.32E-12	1.50E-08
19	38%	62%	9.61E-14	4.07E-10

20	*15%*	*85%*	*1.62E-15*	*8.46E-12*

Reboiler Heat Duty Variation



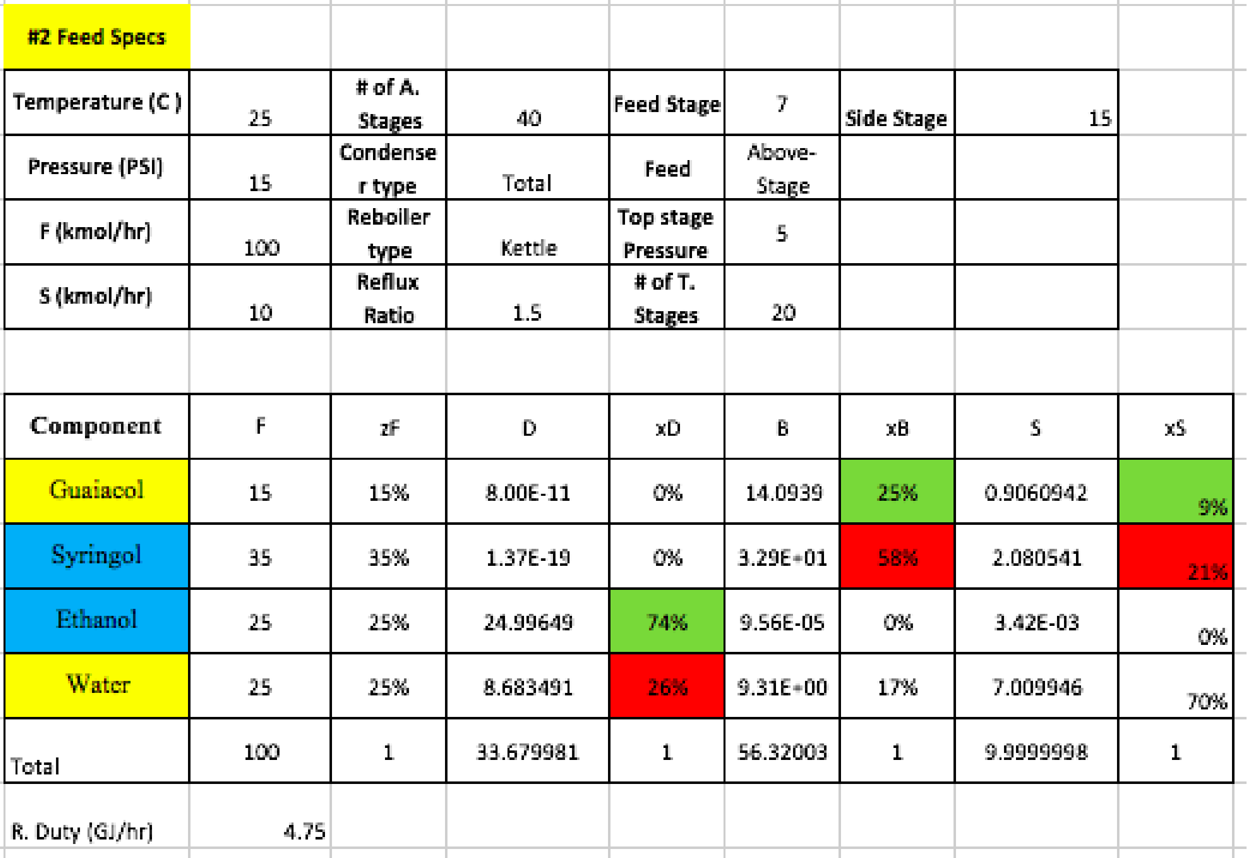
#1 Feed Specs							
Temperature (C)	25	**# of A. Stages**	40	**Feed Stage**	7	**Side Stage**	15
Pressure (PSI)	15	**Condenser type**	Total	**Feed**	Above-Stage		
F (kmol/hr)	100	**Reboiler type**	Kettle	**Top stage Pressure**	5		
S (kmol/hr)	10	**Reflux Ratio**	1.5	**# of T. Stages**	20		

Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	1.79E-13	0%	13.84607	17%	1.153931	12%
Syringol	35	35%	3.47E-22	0%	3.23E+01	40%	2.683345	27%
Ethanol	25	25%	6.589445	79%	1.51E+01	18%	3.313335	33%
Water	25	25%	1.713848	21%	2.04E+01	25%	2.849389	28%
Total	100	1	8.303293	1	81.69671	1	10	1
R. Duty (GJ/hr)	1.75							

Stages	*Stage Composition*			
	Guaiacol	*Syringol*	*Ethanol*	*Water*
1	*6.66E-14*	*1.60E-22*	*91%*	*9%*
2	*1.80E-11*	*1.46E-18*	*89%*	*11%*
3	*2.87E-09*	*8.03E-15*	*86%*	*14%*

4	4.44E-07	4.38E-11	84%	16%
5	6.54E-05	2.33E-07	80%	20%
6	1%	0%	74%	25%
7	19%	54%	20%	7%
8	19%	54%	20%	7%
9	19%	54%	20%	7%
10	19%	54%	20%	7%
11	19%	54%	20%	7%
12	19%	54%	20%	7%
13	19%	54%	20%	7%
14	19%	54%	20%	7%
15	19%	54%	20%	7%
16	19%	54%	20%	7%
17	19%	54%	20%	7%
18	19%	55%	19%	7%
19	19%	55%	18%	8%

20	*22%*	*64%*	*9%*	*5%*



#2 Feed Specs

Temperature (C)	25	**# of A. Stages**	40	**Feed Stage**	7	**Side Stage**	15
Pressure (PSI)	15	**Condenser type**	Total	**Feed**	Above-Stage		
F (kmol/hr)	100	**Reboiler type**	Kettle	**Top stage Pressure**	5		
S (kmol/hr)	10	**Reflux Ratio**	1.5	**# of T. Stages**	20		

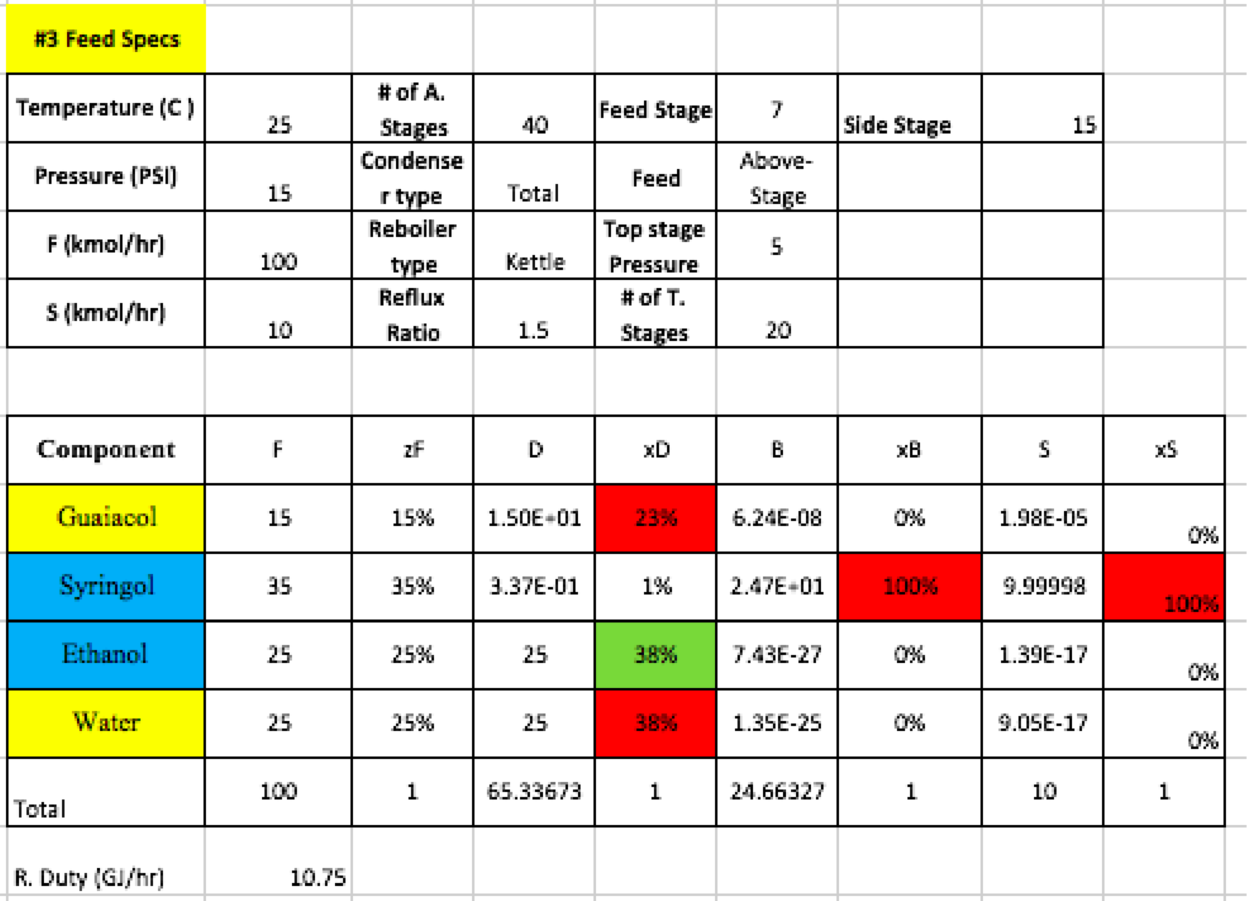
Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	8.00E-11	0%	14.0939	25%	0.9060942	9%
Syringol	35	35%	1.37E-19	0%	3.29E+01	58%	2.080541	21%
Ethanol	25	25%	24.99649	74%	9.56E-05	0%	3.42E-03	0%
Water	25	25%	8.683491	26%	9.31E+00	17%	7.009946	70%
Total	100	1	33.679981	1	56.32003	1	9.9999998	1

R. Duty (GJ/hr) 4.75

Stages	*Stage Composition*			
	Guaiacol	*Syringol*	*Ethanol*	*Water*
1	*7.59E-12*	*1.61E-20*	*88%*	*12%*
2	*2.00E-09*	*1.51E-16*	*83%*	*17%*
3	*2.85E-07*	*7.89E-13*	*76%*	*24%*

4	2.93E-05	3.08E-09	62%	38%
5	0%	4.13E-06	36%	64%
6	1%	0%	18%	81%
7	19%	54%	11%	17%
8	19%	55%	7%	19%
9	20%	56%	3%	21%
10	20%	57%	2%	22%
11	20%	57%	1%	22%
12	20%	57%	0%	22%
13	20%	57%	0%	22%
14	20%	57%	6.56E-04	23%
15	20%	57%	2.81E-04	23%
16	20%	57%	1.16E-04	2.26E-01
17	20%	57%	4.79E-05	2.26E-01
18	20%	57%	1.96E-05	2.25E-01
19	24%	56%	7.55E-06	2.01E-01

20	*25%*	*73%*	*6.30E-07*	*2.40E-02*



#3 Feed Specs

Temperature (C)	25	# of A. Stages	40	Feed Stage	7	Side Stage	15
Pressure (PSI)	15	Condenser type	Total	Feed	Above-Stage		
F (kmol/hr)	100	Reboiller type	Kettle	Top stage Pressure	5		
S (kmol/hr)	10	Reflux Ratio	1.5	# of T. Stages	20		

Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	1.50E+01	23%	6.24E-08	0%	1.98E-05	0%
Syringol	35	35%	3.37E-01	1%	2.47E+01	100%	9.99998	100%
Ethanol	25	25%	25	38%	7.43E-27	0%	1.39E-17	0%
Water	25	25%	25	38%	1.35E-25	0%	9.05E-17	0%
Total	100	1	65.33673	1	24.66327	1	10	1

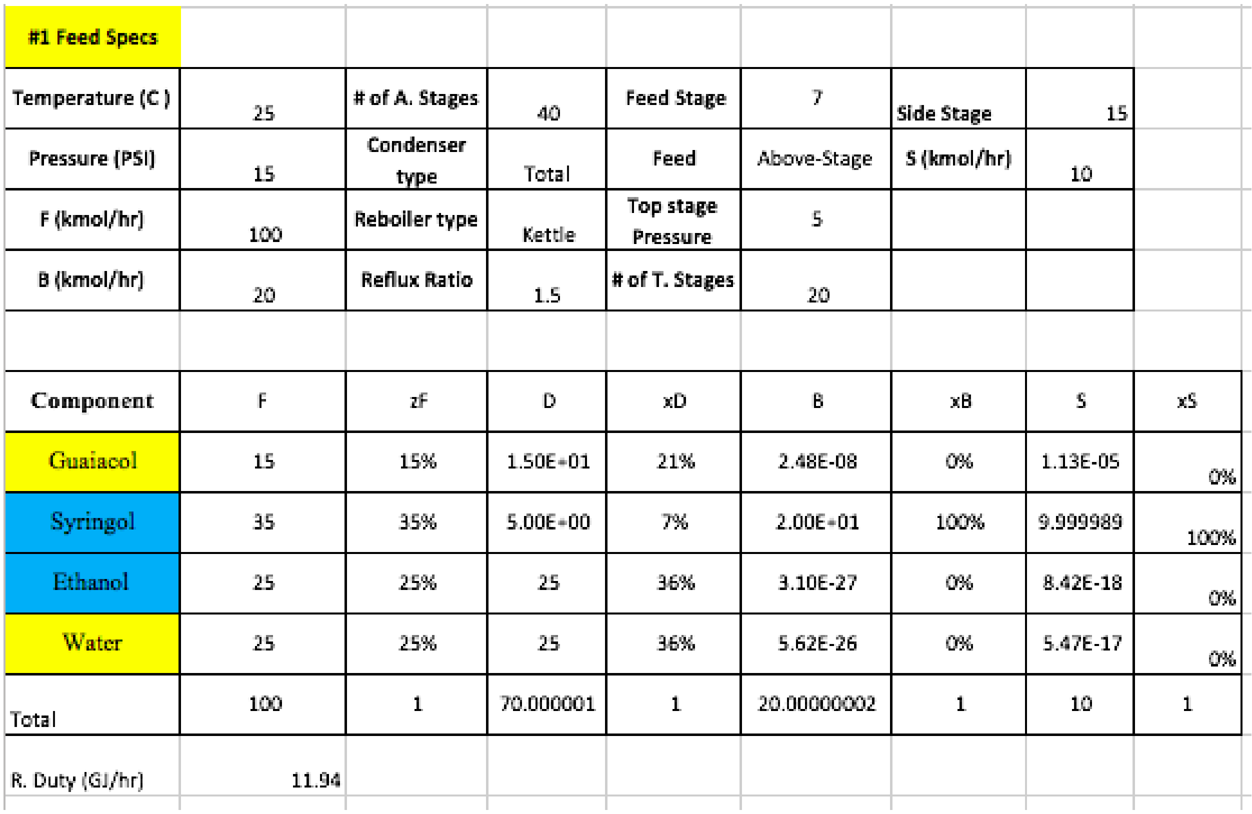
R. Duty (GJ/hr) 10.75

Stages	*Stage Composition*			
	Guaiacol	*Syringol*	*Ethanol*	*Water*
1	*5.30E-01*	*1.48E-02*	*33%*	*13%*
2	*7.82E-01*	*2.05E-01*	*1%*	*0%*

3	*4.18E-01*	*5.80E-01*	*0%*	*0%*
4	*1.56E-01*	*8.43E-01*	*0%*	*0%*
5	*7%*	*9.29E-01*	*0%*	*0%*
6	*5%*	*95%*	*0%*	*0%*
7	*4%*	*96%*	*0%*	*0%*
8	*1%*	*99%*	*9.93E-06*	*6.01E-06*
9	*0%*	*100%*	*1.22E-07*	*9.11E-08*
10	*0%*	*100%*	*1.50E-09*	*1.37E-09*
11	*0%*	*100%*	*1.84E-11*	*2.07E-11*
12	*7.49E-05*	*100%*	*2.26E-13*	*3.11E-13*
13	*2.09E-05*	*100%*	*2.76E-15*	*4.68E-15*
14	*5.80E-06*	*100%*	*3.39E-17*	*7.04E-17*
15	*1.60E-06*	*100%*	*4.15E-19*	*1.06E-18*
16	*4.25E-07*	*100%*	*4.84E-21*	*1.52E-20*
17	*1.13E-07*	*100%*	*5.66E-23*	*2.18E-22*
18	*3.00E-08*	*100%*	*6.61E-25*	*3.12E-24*

19	*7.90E-09*	*100%*	*7.72E-27*	*4.48E-26*
20	*2.04E-09*	*100%*	*9.00E-29*	*6.41E-28*

Bottoms Flow Variation

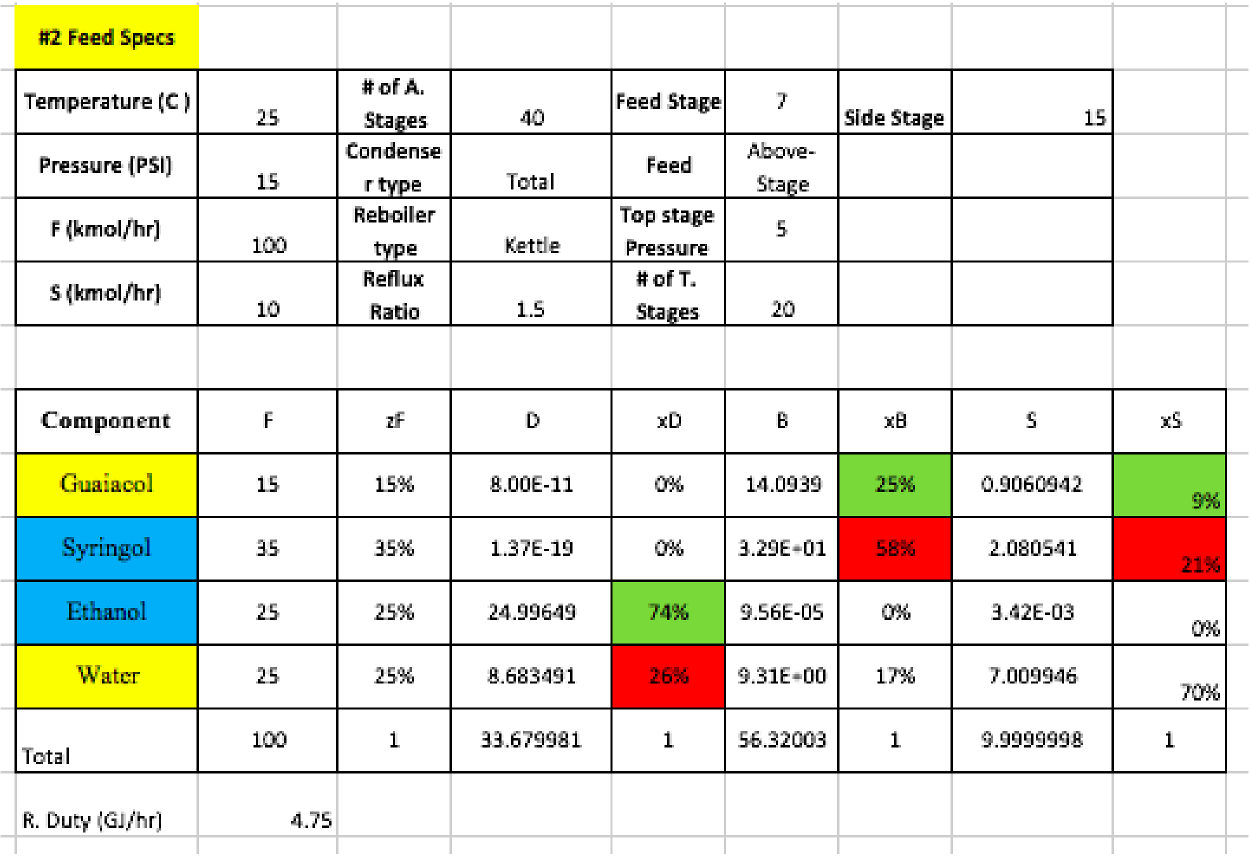


#1 Feed Specs								
Temperature (C)	25	**# of A. Stages**	40	**Feed Stage**	7	**Side Stage**	15	
Pressure (PSI)	15	**Condenser type**	Total	**Feed**	Above-Stage	**S (kmol/hr)**	10	
F (kmol/hr)	100	**Reboiler type**	Kettle	**Top stage Pressure**	5			
B (kmol/hr)	20	**Reflux Ratio**	1.5	**# of T. Stages**	20			
Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	1.50E+01	21%	2.48E-08	0%	1.13E-05	0%
Syringol	35	35%	5.00E+00	7%	2.00E+01	100%	9.999989	100%
Ethanol	25	25%	25	36%	3.10E-27	0%	8.42E-18	0%
Water	25	25%	25	36%	5.62E-26	0%	5.47E-17	0%
Total	100	1	70.000001	1	20.00000002	1	10	1
R. Duty (GJ/hr)	11.94							

Stages	Stage Composition			
	Guaiacol	Syringol	Ethanol	Water
1	4.40E-01	1.82E-01	27%	11%
2	2.48E-01	7.46E-01	0%	0%

3	8.87E-02	9.10E-01	0%	0%
4	4.54E-02	9.54E-01	0%	0%
5	3.52E-02	9.64E-01	0%	0%
6	3%	97%	0%	0%
7	3%	97%	0%	0%
8	1%	99%	7.79E-06	4.70E-06
9	0%	100%	9.24E-08	6.85E-08
10		100%	1.09E-09	9.94E-10
11	0%	100%	1.29E-11	1.44E-11
12	4.76E-05	100%	1.53E-13	2.09E-13
13	1.28E-05	100%	1.80E-15	3.04E-15
14	3.43E-06	100%	2.13E-17	4.41E-17
15	9.12E-07	100%	2.51E-19	6.39E-19
16	2.35E-07	100%	2.84E-21	8.87E-21
17	6.03E-08	100%	3.21E-23	1.23E-22
18	1.55E-08	100%	3.63E-25	1.71E-24

19	3.97E-09	100%	4.10E-27	2.37E-26
20	9.97E-10	100%	4.63E-29	3.28E-28



#2 Feed Specs

Temperature (C)	25	# of A. Stages	40	Feed Stage	7	Side Stage	15
Pressure (PSI)	15	Condenser type	Total	Feed	Above-Stage		
F (kmol/hr)	100	Reboiler type	Kettle	Top stage Pressure	5		
S (kmol/hr)	10	Reflux Ratio	1.5	# of T. Stages	20		

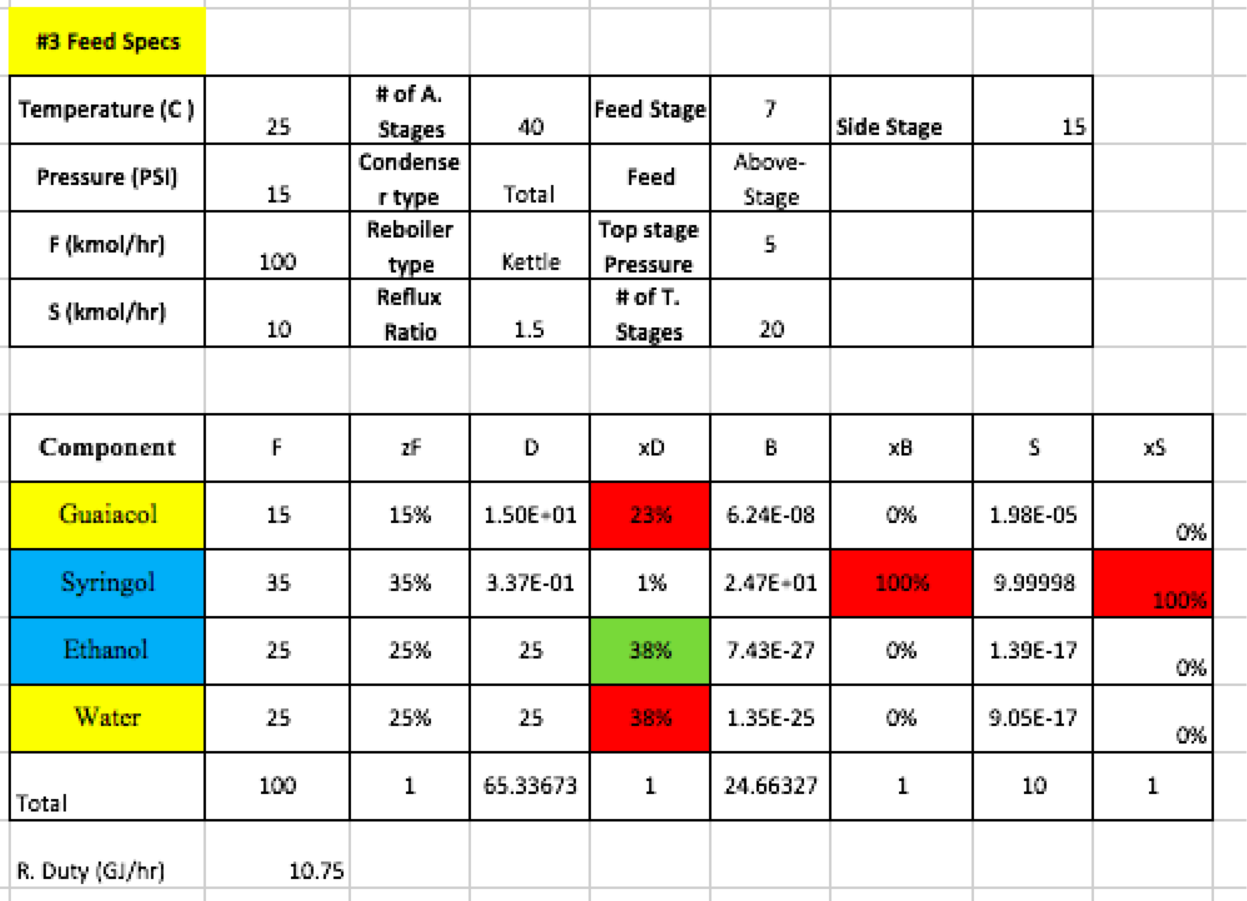
Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	8.00E-11	0%	14.0939	25%	0.9060942	9%
Syringol	35	35%	1.37E-19	0%	3.29E+01	58%	2.080541	21%
Ethanol	25	25%	24.99649	74%	9.56E-05	0%	3.42E-03	0%
Water	25	25%	8.683491	26%	9.31E+00	17%	7.009946	70%
Total	100	1	33.679981	1	56.32003	1	9.9999998	1

R. Duty (GJ/hr) 4.75

Stages	Stage Composition			
	Guaiacol	Syringol	Ethanol	Water
1	7.59E-12	1.61E-20	88%	12%
2	2.00E-09	1.51E-16	83%	17%

3	2.85E-07	7.89E-13	76%	24%
4	2.93E-05	3.08E-09	62%	38%
5	0%	4.13E-06	36%	64%
6	1%	0%	18%	81%
7	19%	54%	11%	17%
8	19%	55%	7%	19%
9	20%	56%	3%	21%
10	20%	57%	2%	22%
11	20%	57%	1%	22%
12	20%	57%	0%	22%
13	20%	57%	0%	22%
14	20%	57%	6.56E-04	23%
15	20%	57%	2.81E-04	23%
16	20%	57%	1.16E-04	2.26E-01
17	20%	57%	4.79E-05	2.26E-01
18	20%	57%	1.96E-05	2.25E-01

19	24%	56%	7.55E-06	2.01E-01
20	25%	73%	6.30E-07	2.40E-02



#3 Feed Specs

Temperature (C)	25	# of A. Stages	40	Feed Stage	7	Side Stage	15
Pressure (PSI)	15	Condenser type	Total	Feed	Above-Stage		
F (kmol/hr)	100	Reboiler type	Kettle	Top stage Pressure	5		
S (kmol/hr)	10	Reflux Ratio	1.5	# of T. Stages	20		

Component	F	zF	D	xD	B	xB	S	xS
Guaiacol	15	15%	1.50E+01	23%	6.24E-08	0%	1.98E-05	0%
Syringol	35	35%	3.37E-01	1%	2.47E+01	100%	9.99998	100%
Ethanol	25	25%	25	38%	7.43E-27	0%	1.39E-17	0%
Water	25	25%	25	38%	1.35E-25	0%	9.05E-17	0%
Total	100	1	65.33673	1	24.66327	1	10	1

R. Duty (GJ/hr) 10.75

Stages	Stage Composition			
	Guaiacol	Syringol	Ethanol	Water
1	5.30E-01	1.48E-02	33%	13%

2	7.82E-01	2.05E-01	1%	0%
3	4.18E-01	5.80E-01	0%	0%
4	1.56E-01	8.43E-01	0%	0%
5	7%	9.29E-01	0%	0%
6	5%	95%	0%	0%
7	4%	96%	0%	0%
8	1%	99%	9.93E-06	6.01E-06
9	0%	100%	1.22E-07	9.11E-08
10	0%	100%	1.50E-09	1.37E-09
11	0%	100%	1.84E-11	2.07E-11
12	7.49E-05	100%	2.26E-13	3.11E-13
13	2.09E-05	100%	2.76E-15	4.68E-15
14	5.80E-06	100%	3.39E-17	7.04E-17
15	1.60E-06	100%	4.15E-19	1.06E-18
16	4.25E-07	100%	4.84E-21	1.52E-20
17	1.13E-07	100%	5.66E-23	2.18E-22

18	3.00E-08	100%	6.61E-25	3.12E-24
19	7.90E-09	100%	7.72E-27	4.48E-26
20	2.04E-09	100%	9.00E-29	6.41E-28

III.B: Liquid-Liquid Extraction

n=40
100 C

	ELT-03	EXTRACT	RAFF	SOLVENT
	EXTCOL			EXTCOL
		EXTCOL	EXTCOL	
	MIXED	LIQUID	LIQUID	LIQUID
Substream: MIXED				
Mole Flow kmol/hr				
WATER	70	65.92259	4.077409	0
ETHAN-01	20	18.83503	1.164974	0
GUAIA-01	10	9.417513	0.582487	0
N-BUT-01	0	3.83E-20	30	30
Total Flow kmol/hr	100	94.17513	35.82487	30

Total Flow kg/hr	3423.844	3224.409	2423.118	2223.684
Total Flow l/min	38998.21	63.35919	55.93381	50.68645
Temperature C	100	151.5665	122.5104	100
Pressure bar	1.01325	1.01325	1.01325	1.378951
Vapor Frac	0.7703777	0	0	0
Liquid Frac	0.2296223	1	1	1
Solid Frac	0	0	0	0
Enthalpy cal/mol	-59367.46	-65394.93	-71955.12	-74399.18
Enthalpy cal/gm	-1733.942	-1909.986	-1063.829	-1003.729
Enthalpy cal/sec	-1.65E+06	-1.71E+06	-7.16E+05	-6.20E+05
Entropy cal/mol-K	-30.70769	-47.52202	-105.7445	-120.9597
Entropy cal/gm-K	-0.8968778	-1.387973	-1.563392	-1.631882
Density amol/cc	4.27E-05	0.0247728	0.0106747	9.86E-03
Density gm/cc	1.46E-03	0.8481825	0.7220196	0.7311895
Average MW	34.23844	34.23844	67.63788	74.1228
Liq Vol 60F l/min	58.69607	55.2771	49.06972	45.65075

10 stages at 50 degrees celsius

n=10
50 C

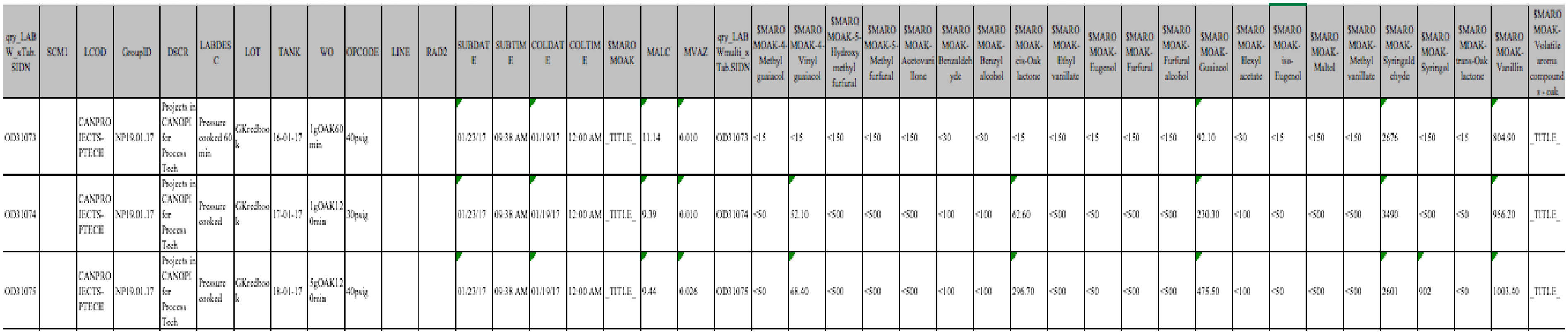
	ELT-03	EXTRACT	RAFF	SOLVENT

	EXTCOL			EXTCOL
		EXTCOL	EXTCOL	
	LIQUID	LIQUID	LIQUID	LIQUID
Substream: MIXED				
Mole Flow kmol/hr				
WATER	70	66.50127	3.498728	0
ETHAN-01	20	19.00039	0.999614	0
GUAIA-01	10	9.500153	0.4998473	0
N-BUT-01	0	9.29E-03	49.99071	50
Total Flow kmol/hr	100	95.0111	54.9889	50
Total Flow kg/hr	3423.844	3253.4	3876.584	3706.14
Total Flow l/min	56.35166	53.54873	78.60838	76.06382
Temperature C	20	20.00064	20.41434	20
Pressure bar	1.01325	1.01325	1.01325	1.378951
Vapor Frac	0	0	0	0
Liquid Frac	1	1	1	1
Solid Frac	0	0	0	0
Enthalpy cal/mol	-69057.25	-69058.19	-77860.57	-78740.72
Enthalpy cal/gm	-2016.951	-2016.75	-1104.443	-1062.301
Enthalpy cal/sec	-1.92E+06	-1.82E+06	-1.19E+06	-1.09E+06

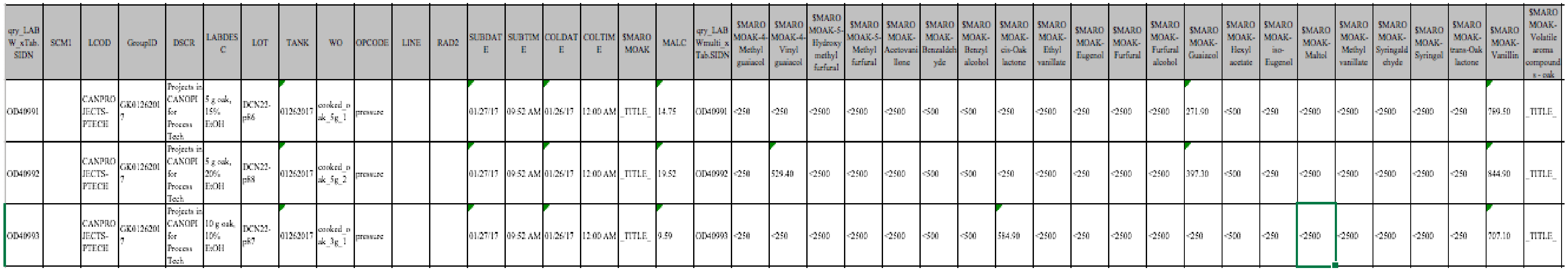
Entropy cal/mol-K	-57.70512	-57.71072	-126.5617	-134.0085
Entropy cal/gm-K	-1.68539	-1.685363	-1.795263	-1.807926
Density mol/cc	0.0295761	0.0295715	0.0116588	0.0109557
Density gm/cc	1.012642	1.012598	0.8219191	0.8120681
Average MW	34.23844	34.24231	70.49757	74.1228
Liq Vol 60F l/min	58.69607	55.77644	79.00421	76.08458

Appendix IV: Raw Data

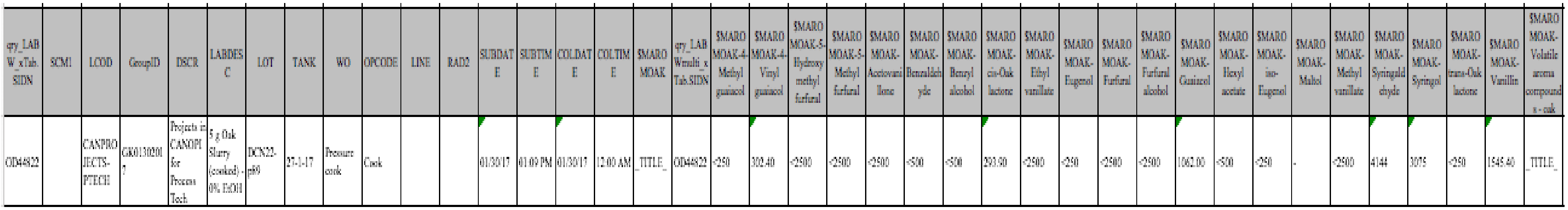
IV.A Cooking



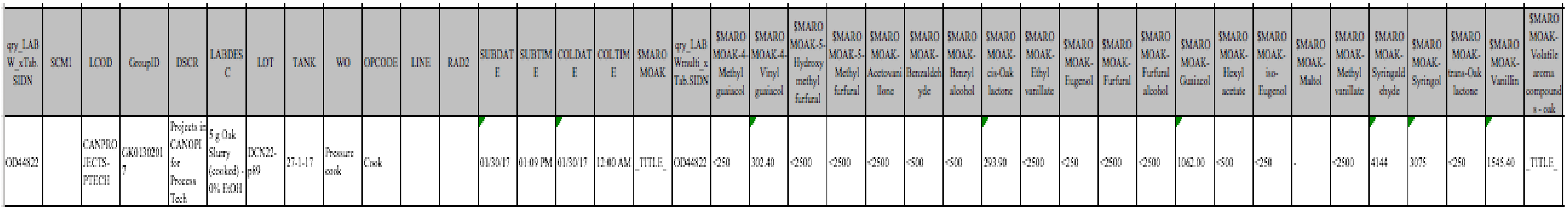
qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD31073		CANPROJECTS-PTECH	NP19.01.17	Projects in CANOPI for Process Tech	Pressure cooked 60 min	GKredhook	16-01-17	1gOAK60min	40psig			01/23/17	09:38 AM	01/19/17	12:00 AM	_TITLE_	11.14	0.010	OD31073	<15	<15	<150	<150	<150	<30	<30	<15	<150	<15	<150	<150	92.10	<30	<15	<150	<150	2676	<150	<15	804.90	_TITLE_
OD31074		CANPROJECTS-PTECH	NP19.01.17	Projects in CANOPI for Process Tech	Pressure cooked	GKredhook	17-01-17	1gOAK120min	30psig			01/23/17	09:38 AM	01/19/17	12:00 AM	_TITLE_	9.39	0.010	OD31074	<50	52.10	<500	<500	<500	<100	<100	62.60	<500	<50	<500	<500	230.30	<100	<50	<500	<500	3490	<500	<50	956.20	_TITLE_
OD31075		CANPROJECTS-PTECH	NP19.01.17	Projects in CANOPI for Process Tech	Pressure cooked	GKredhook	18-01-17	5gOAK120min	40psig			01/23/17	09:38 AM	01/19/17	12:00 AM	_TITLE_	9.44	0.026	OD31075	<50	68.40	<500	<500	<500	<100	<100	296.70	<500	<50	<500	<500	475.50	<100	<50	<500	<500	2601	902	<50	1003.40	_TITLE_



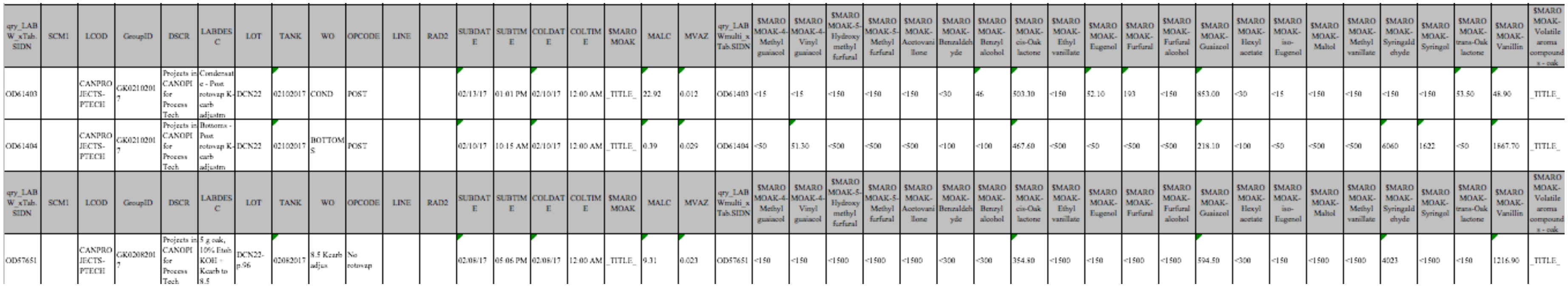
qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	MALC	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD40991		CANPROJECTS-PTECH	GK01262017	Projects in CANOPI for Process Tech	5 g oak, 15% EtOH	DCN22-p86	01262017	cooked_oak_5g_1	pressure			01/27/17	09:52 AM	01/26/17	12:00 AM	_TITLE_	14.75	OD40991	<250	<250	<2500	<2500	<2500	<500	<500	<250	<2500	<250	<2500	<2500	271.90	<500	<250	<2500	<2500	<2500	<2500	<250	769.50	_TITLE_
OD40992		CANPROJECTS-PTECH	GK01262017	Projects in CANOPI for Process Tech	5 g oak, 20% EtOH	DCN22-p88	01262017	cooked_oak_5g_2	pressure			01/27/17	09:52 AM	01/26/17	12:00 AM	_TITLE_	19.52	OD40992	<250	529.40	<2500	<2500	<2500	<500	<500	<250	<2500	<250	<2500	<2500	397.30	<500	<250	<2500	<2500	<2500	<2500	<250	844.90	_TITLE_
OD40993		CANPROJECTS-PTECH	GK01262017	Projects in CANOPI for Process Tech	10 g oak, 10% EtOH	DCN22-p87	01262017	cooked_oak_3g_1	pressure			01/27/17	09:52 AM	01/26/17	12:00 AM	_TITLE_	9.59	OD40993	<250	<250	<2500	<2500	<2500	<500	<500	584.90	<2500	<250	<2500	<2500	<250	<500	<250	<2500	<2500	<2500	<2500	<250	707.10	_TITLE_



qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD44822		CANPROJECTS-PTECH	GK01302017	Projects in CANOPI for Process Tech	5 g Oak Slurry (cooked) - 0% EtOH	DCN22-p89	27-1-17	Pressure cook	Cook			01/30/17	01:09 PM	01/30/17	12:00 AM	_TITLE_	OD44822	<250	302.40	<2500	<2500	<2500	<500	<500	293.90	<2500	<250	<2500	<2500	1062.00	<500	<250	-	<2500	4144	3075	<250	1545.40	_TITLE_

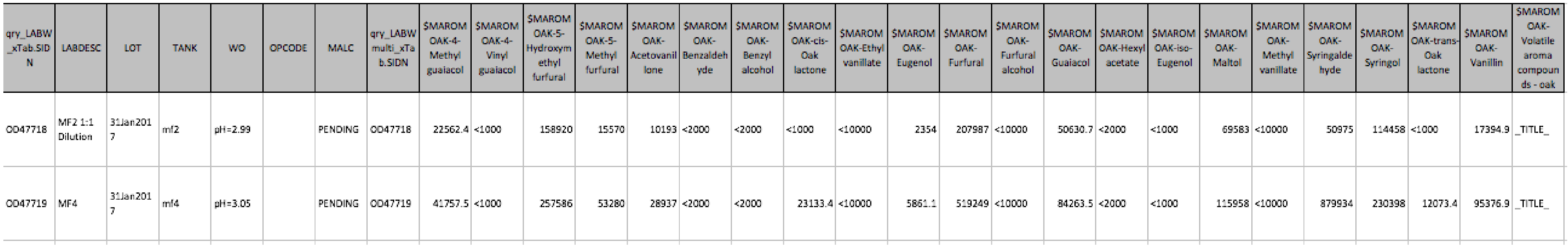


qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD44822		CANPROJECTS-PTECH	GK01302017	Projects in CANOPI for Process Tech	5 g Oak Slurry (cooked) - 0% EtOH	DCN22-p89	27-1-17	Pressure cook	Cook			01/30/17	01:09 PM	01/30/17	12:00 AM	_TITLE_	OD44822	<250	302.40	<2500	<2500	<2500	<500	<500	293.90	<2500	<250	<2500	<2500	1062.00	<500	<250	-	<2500	4144	3075	<250	1545.40	_TITLE_

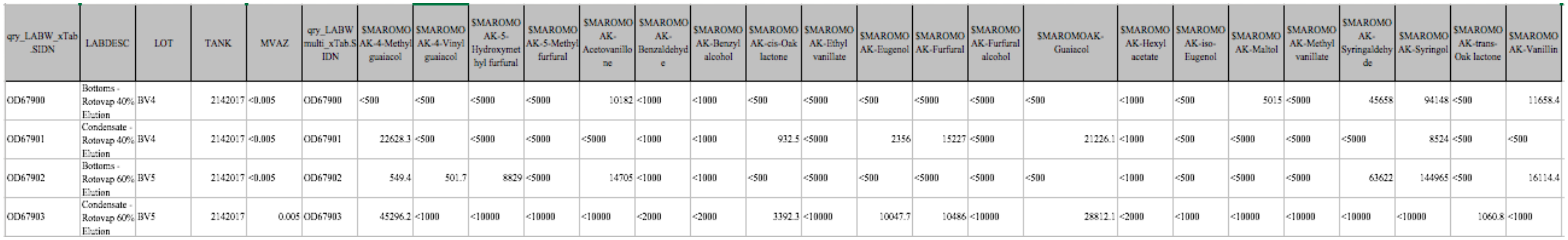


qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD61403		CANPROJECTS-PTECH	GK02102017	Projects in CANOPI for Process Tech	Condensate - Post rotovap K-carb adjustm	DCN22	02102017	COND	POST			02/13/17	01:01 PM	02/10/17	12:00 AM	_TITLE_	22.92	0.012	OD61403	<15	<15	<150	<150	<150	<30	46	503.30	<150	52.10	193	<150	853.00	<30	<15	<150	<150	<150	<150	53.50	48.90	_TITLE_
OD61404		CANPROJECTS-PTECH	GK02102017	Projects in CANOPI for Process Tech	Bottoms - Post rotovap K-carb adjustm	DCN22	02102017	BOTTOMS	POST			02/10/17	10:15 AM	02/10/17	12:00 AM	_TITLE_	0.39	0.029	OD61404	<50	51.30	<500	<500	<500	<100	<100	467.60	<500	<50	<500	<500	218.10	<100	<50	<500	<500	6060	1622	<50	1867.70	_TITLE_
qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	LINE	RAD2	SUBDATE	SUBTIME	COLDATE	COLTIME	$MAROMOAK	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxy methyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD57651		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	5 g oak, 10% Etoh KOH + Kcarb to 8.5	DCN22-p.96	02082017	8.5 Kcarb adjus	No rotovap			02/08/17	05:06 PM	02/08/17	12:00 AM	_TITLE_	9.31	0.023	OD57651	<150	<150	<1500	<1500	<1500	<300	<300	354.80	<1500	<150	<1500	<1500	594.50	<300	<150	<1500	<1500	4023	<1500	<150	1216.90	_TITLE_

IV.B Liquid-Liquid Extraction

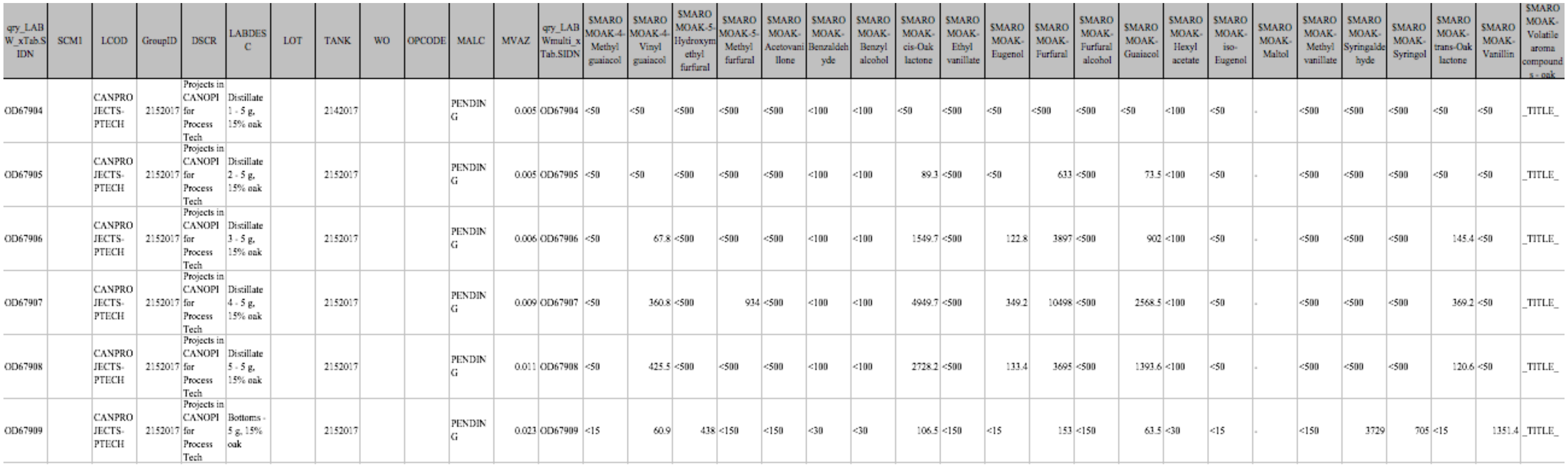


qry_LABW_xTab.SIDN	LABDESC	LOT	TANK	WO	OPCODE	MALC	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD47718	MF2 1:1 Dilution	31Jan2017	mf2	pH=2.99		PENDING	OD47718	22562.4	<1000	158920	15570	10193	<2000	<2000	<1000	<10000	2354	207987	<10000	50630.7	<2000	<1000	69583	<10000	50975	114458	<1000	17394.9	_TITLE_
OD47719	MF4	31Jan2017	mf4	pH=3.05		PENDING	OD47719	41757.5	<1000	257586	53280	28937	<2000	<2000	23133.4	<10000	5861.1	519249	<10000	84263.5	<2000	<1000	115958	<10000	879934	230398	12073.4	95376.9	_TITLE_



qry_LABW_xTab.SIDN	LABDESC	LOT	TANK	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin
OD67900	Bottoms - Rotovap 40% Elution	BV4	2142017	<0.005	OD67900	<500	<500	<5000	<5000	10182	<1000	<1000	<500	<5000	<500	<5000	<5000	<500	<1000	<500	5015	<5000	45658	94148	<500	11658.4
OD67901	Condensate - Rotovap 40% Elution	BV4	2142017	<0.005	OD67901	22628.3	<500	<5000	<5000	<5000	<1000	<1000	932.5	<5000	2356	15227	<5000	21226.1	<1000	<500	<5000	<5000	<5000	8524	<500	<500
OD67902	Bottoms - Rotovap 60% Elution	BV5	2142017	<0.005	OD67902	549.4	501.7	8829	<5000	14705	<1000	<1000	<500	<5000	<500	<5000	<5000	<500	<1000	<500	<5000	<5000	63622	144965	<500	16114.4
OD67903	Condensate - Rotovap 60% Elution	BV5	2142017	0.005	OD67903	45296.2	<1000	<10000	<10000	<10000	<2000	<2000	3392.3	<10000	10047.7	10486	<10000	28812.1	<2000	<1000	<10000	<10000	<10000	<10000	1060.8	<1000

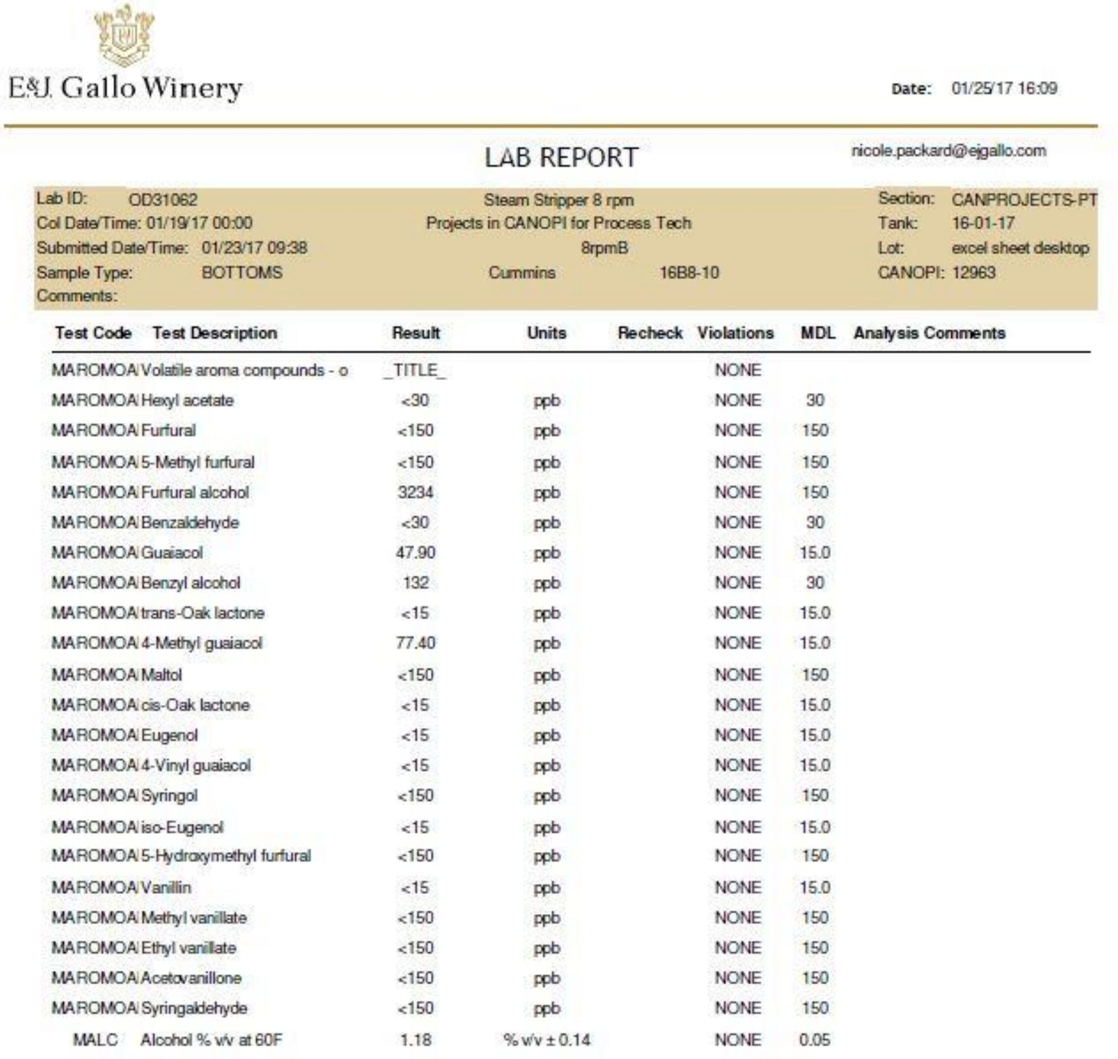
IV.C Batch Distillation



qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD67904		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Distillate 1 - 5 g, 15% oak		2142017			PENDING	0.005	OD67904	<50	<50	<500	<500	<500	<100	<100	<50	<500	<50	<500	<500	<50	<100	<50	-	<500	<500	<500	<50	<50	_TITLE_
OD67905		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Distillate 2 - 5 g, 15% oak		2152017			PENDING	0.005	OD67905	<50	<50	<500	<500	<500	<100	<100	89.3	<500	<50	633	<500	73.5	<100	<50	-	<500	<500	<500	<50	<50	_TITLE_
OD67906		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Distillate 3 - 5 g, 15% oak		2152017			PENDING	0.006	OD67906	<50	67.8	<500	<500	<500	<100	<100	1549.7	<500	122.8	3897	<500	902	<100	<50	-	<500	<500	<500	145.4	<50	_TITLE_
OD67907		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Distillate 4 - 5 g, 15% oak		2152017			PENDING	0.009	OD67907	<50	360.8	<500	934	<500	<100	<100	4949.7	<500	349.2	10498	<500	2568.5	<100	<50	-	<500	<500	<500	369.2	<50	_TITLE_
OD67908		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Distillate 5 - 5 g, 15% oak		2152017			PENDING	0.011	OD67908	<50	425.5	<500	<500	<500	<100	<100	2728.2	<500	133.4	3695	<500	1393.6	<100	<50	-	<500	<500	<500	120.6	<50	_TITLE_
OD67909		CANPROJECTS-PTECH	2152017	Projects in CANOPI for Process Tech	Bottoms - 5 g, 15% oak		2152017			PENDING	0.023	OD67909	<15	60.9	438	<150	<150	<30	<30	106.5	<150	<15	153	<150	63.5	<30	<15	-	<150	3729	705	<15	1351.4	_TITLE_

qry_LABW_xTab.SIDN	SCM1	LCOD	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin	$MAROMOAK-Volatile aroma compounds - oak
OD57655		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Condensate 1 - 5 g, 10%	DCN22	2082017			85.86	<0.005	OD57655	<50	<50	<500	<500	<500	110	122	926.7	<500	84.2	2379	<500	584.3	<100	<50	<500	<500	<500	<500	106.2	122.1	_TITLE_
OD57656		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Condensate 2 - 5 g, 10%	DCN22	2082017			5.13	0.008	OD57656	<50	945.3	<500	973	<500	<100	293	2925.7	<500	149.8	6000	<500	2020.8	<100	<50	<500	<500	<500	<500	130.8	<50	_TITLE_
OD57657		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Bottoms - 5 g, 10%	DCN22	2082017			0.08	0.028	OD57657	<50	63.4	<500	<500	<500	<100	<100	<50	<500	<50	<500	<500	<50	<100	<50	<500	<500	4131	1252	<50	1733	_TITLE_
OD57658		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Condensate 1 - 5 g, 20%	DCN22	2082017			89.25	<0.005	OD57658	<50	<50	<500	<500	<500	<100	<100	<50	<500	<50	<500	<500	<50	<100	<50	<500	<500	<500	<500	<50	89.9	_TITLE_
OD57659		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Condensate 2 - 5 g, 20%	DCN22	2082017			89.24	<0.005	OD57659	<50	<50	<500	<500	<500	<100	<100	<50	<500	<50	<500	<500	<50	<100	<50	<500	<500	<500	<500	<50	97.4	_TITLE_
OD57660		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	Bottoms - 5 g, 20%	DCN22	2082017			<0.05	0.027	OD57660	<50	159	<500	<500	<500	<100	<100	<50	<500	<50	<500	<500	<50	<100	<50	<500	<500	4116	1168	<50	1564.5	_TITLE_
OD58350		CANPROJECTS-PTECH	GK02082017	Projects in CANOPI for Process Tech	CONDENSATE 4 - 5 g oak, 20% EtOH	DCN22	2082017		pH=5.73	1.91	0.008	OD58350	<25	2944.8	351	893	<250	<50	64	3285	<250	184.8	4641	<250	2024.1	<50	87.3	<250	<250	<250	<250	138.9	28.9	_TITLE_

IV.D Steam Stripping



E&J. Gallo Winery

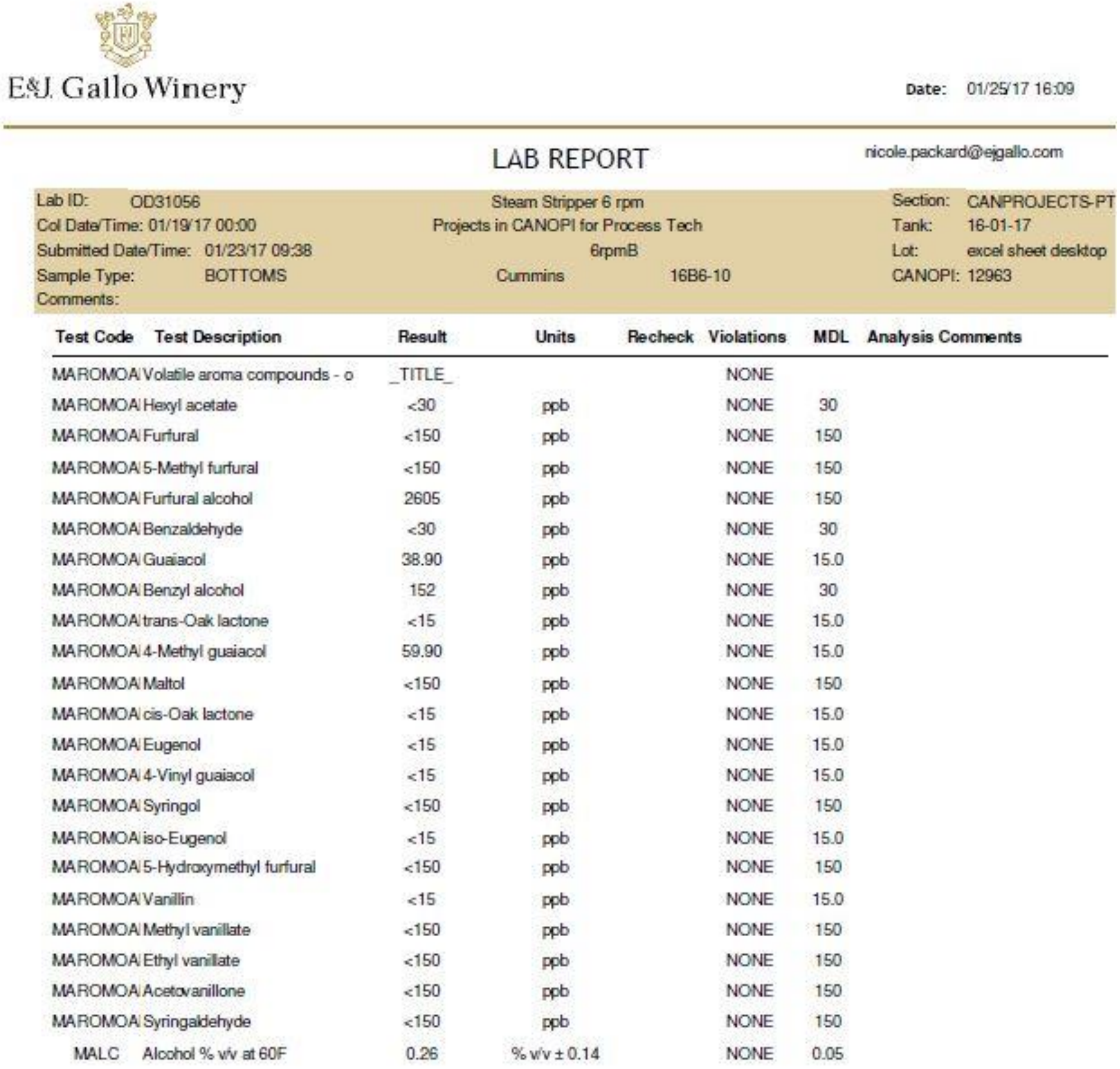
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31062	Steam Stripper 8 rpm		Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech		Tank: 16-01-17
Submitted Date/Time: 01/23/17 09:38	8rpmB		Lot: excel sheet desktop
Sample Type: BOTTOMS	Cummins	16B8-10	CANOPI: 12963
Comments:			

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	3234	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	47.90	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	132	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	77.40	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	1.18	% v/v ± 0.14		NONE	0.05	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

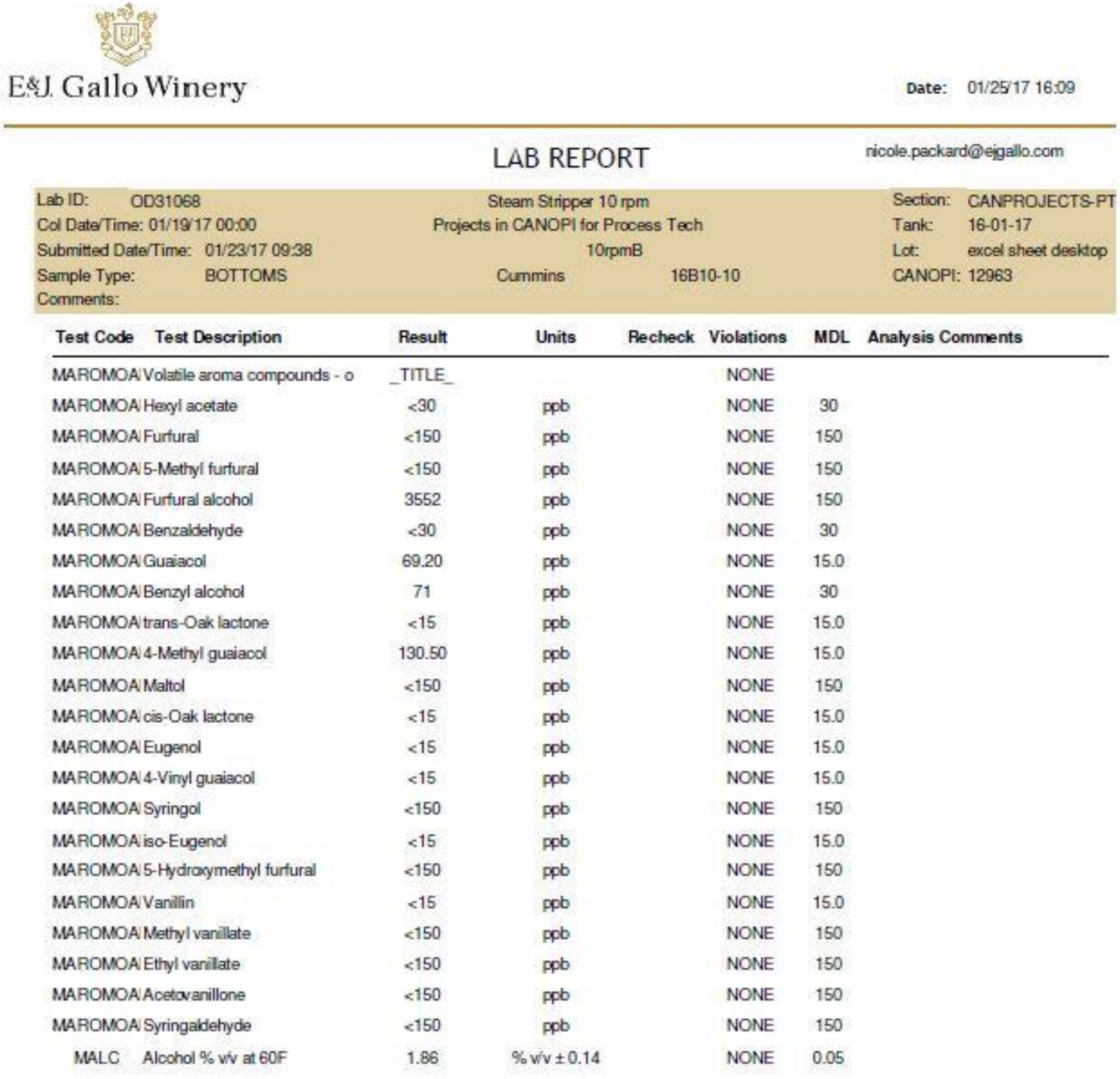
nicole.packard@ejgallo.com

Lab ID: OD31056
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: BOTTOMS
Comments:

Steam Stripper 6 rpm
Projects in CANOPI for Process Tech
6rpmB
Cummins 16B6-10

Section: CANPROJECTS-PT
Tank: 16-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	2605	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	38.90	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	152	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	59.90	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	0.26	% v/v ± 0.14		NONE	0.05	



E&J Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

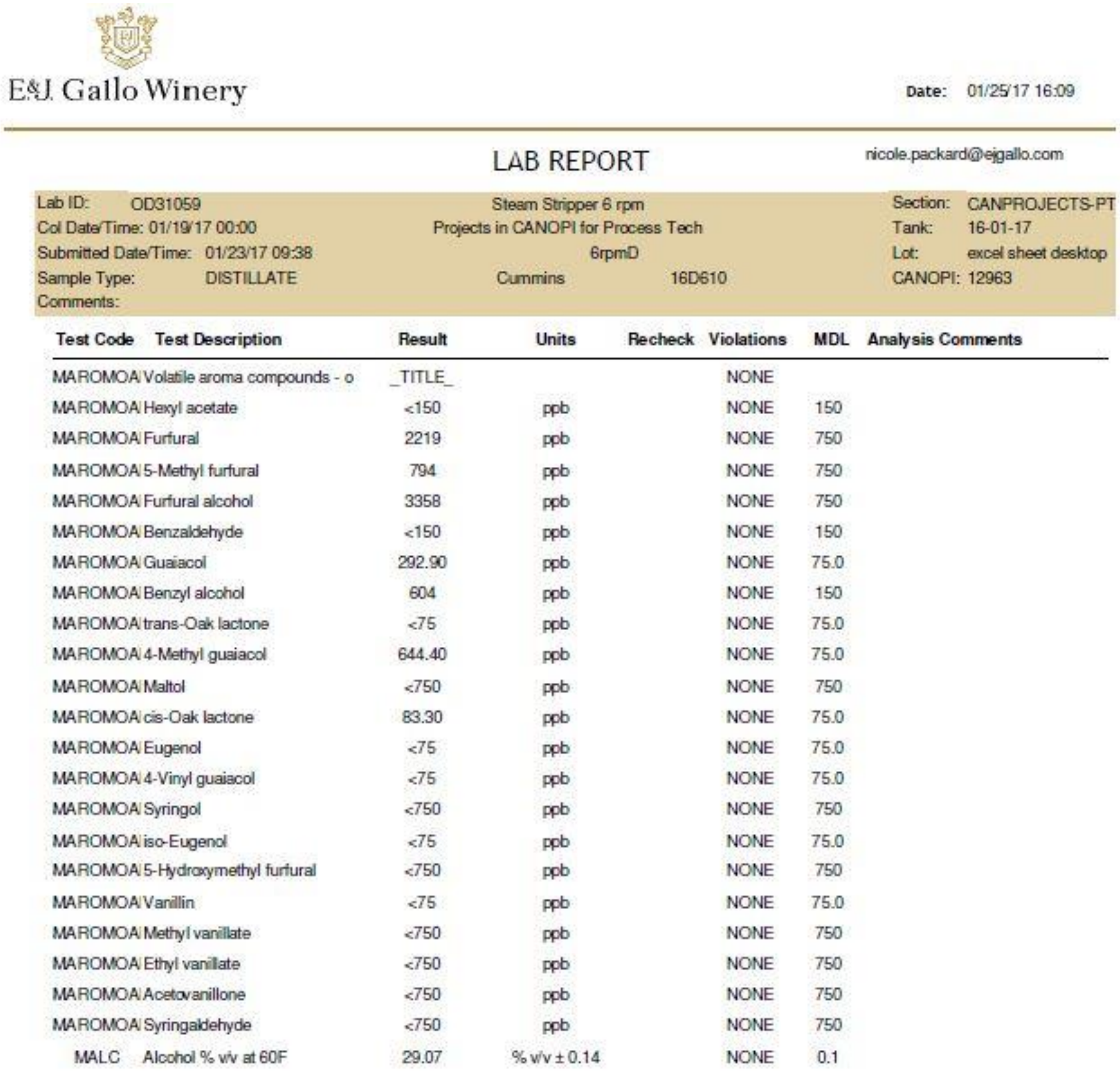
nicole.packard@ejgallo.com

Lab ID: OD31068
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: BOTTOMS
Comments:

Steam Stripper 10 rpm
Projects in CANOPI for Process Tech
10rpmB
Cummins 16B10-10

Section: CANPROJECTS-PT
Tank: 16-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	3552	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	69.20	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	71	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	130.50	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	1.86	% v/v ± 0.14		NONE	0.05	



E&J Gallo Winery

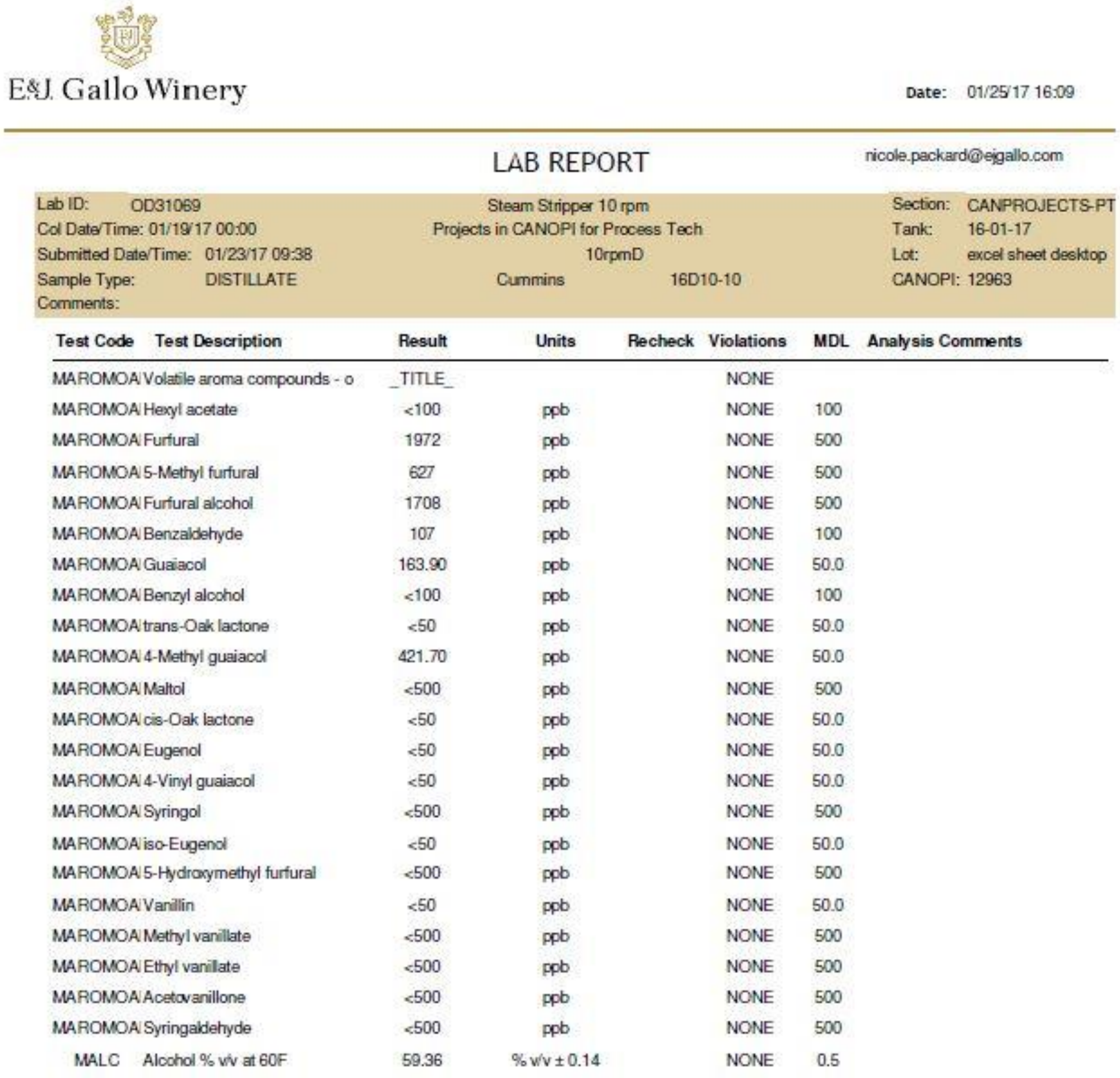
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31059	Steam Stripper 6 rpm	Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech	Tank: 16-01-17
Submitted Date/Time: 01/23/17 09:38	6rpmD	Lot: excel sheet desktop
Sample Type: DISTILLATE	Cummins 16D610	CANOPI: 12963
Comments:		

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<150	ppb		NONE	150	
MAROMOA	Furfural	2219	ppb		NONE	750	
MAROMOA	5-Methyl furfural	794	ppb		NONE	750	
MAROMOA	Furfural alcohol	3358	ppb		NONE	750	
MAROMOA	Benzaldehyde	<150	ppb		NONE	150	
MAROMOA	Guaiacol	292.90	ppb		NONE	75.0	
MAROMOA	Benzyl alcohol	604	ppb		NONE	150	
MAROMOA	trans-Oak lactone	<75	ppb		NONE	75.0	
MAROMOA	4-Methyl guaiacol	644.40	ppb		NONE	75.0	
MAROMOA	Maltol	<750	ppb		NONE	750	
MAROMOA	cis-Oak lactone	83.30	ppb		NONE	75.0	
MAROMOA	Eugenol	<75	ppb		NONE	75.0	
MAROMOA	4-Vinyl guaiacol	<75	ppb		NONE	75.0	
MAROMOA	Syringol	<750	ppb		NONE	750	
MAROMOA	iso-Eugenol	<75	ppb		NONE	75.0	
MAROMOA	5-Hydroxymethyl furfural	<750	ppb		NONE	750	
MAROMOA	Vanillin	<75	ppb		NONE	75.0	
MAROMOA	Methyl vanillate	<750	ppb		NONE	750	
MAROMOA	Ethyl vanillate	<750	ppb		NONE	750	
MAROMOA	Acetovanillone	<750	ppb		NONE	750	
MAROMOA	Syringaldehyde	<750	ppb		NONE	750	
MALC	Alcohol % v/v at 60F	29.07	% v/v ± 0.14		NONE	0.1	



E&J. Gallo Winery

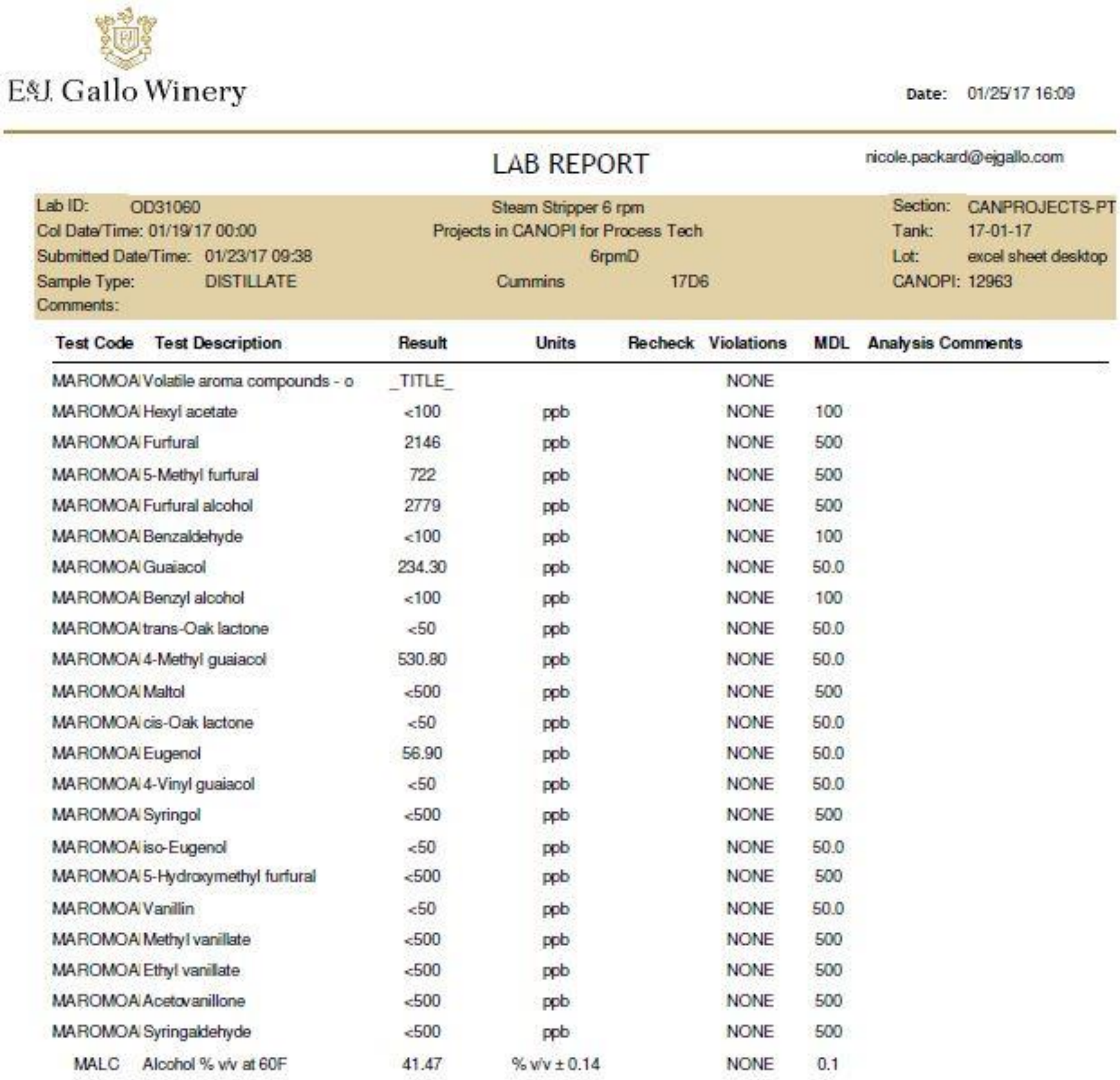
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID:	OD31069	Steam Stripper 10 rpm	Section:	CANPROJECTS-PT
Col Date/Time:	01/19/17 00:00	Projects in CANOPI for Process Tech	Tank:	16-01-17
Submitted Date/Time:	01/23/17 09:38	10rpmD	Lot:	excel sheet desktop
Sample Type:	DISTILLATE	Cummins 16D10-10	CANOPI:	12963
Comments:				

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<100	ppb		NONE	100	
MAROMOA	Furfural	1972	ppb		NONE	500	
MAROMOA	5-Methyl furfural	627	ppb		NONE	500	
MAROMOA	Furfural alcohol	1708	ppb		NONE	500	
MAROMOA	Benzaldehyde	107	ppb		NONE	100	
MAROMOA	Guaiacol	163.90	ppb		NONE	50.0	
MAROMOA	Benzyl alcohol	<100	ppb		NONE	100	
MAROMOA	trans-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	4-Methyl guaiacol	421.70	ppb		NONE	50.0	
MAROMOA	Maltol	<500	ppb		NONE	500	
MAROMOA	cis-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	Eugenol	<50	ppb		NONE	50.0	
MAROMOA	4-Vinyl guaiacol	<50	ppb		NONE	50.0	
MAROMOA	Syringol	<500	ppb		NONE	500	
MAROMOA	iso-Eugenol	<50	ppb		NONE	50.0	
MAROMOA	5-Hydroxymethyl furfural	<500	ppb		NONE	500	
MAROMOA	Vanillin	<50	ppb		NONE	50.0	
MAROMOA	Methyl vanillate	<500	ppb		NONE	500	
MAROMOA	Ethyl vanillate	<500	ppb		NONE	500	
MAROMOA	Acetovanillone	<500	ppb		NONE	500	
MAROMOA	Syringaldehyde	<500	ppb		NONE	500	
MALC	Alcohol % v/v at 60F	59.36	% v/v ± 0.14		NONE	0.5	



E&J. Gallo Winery

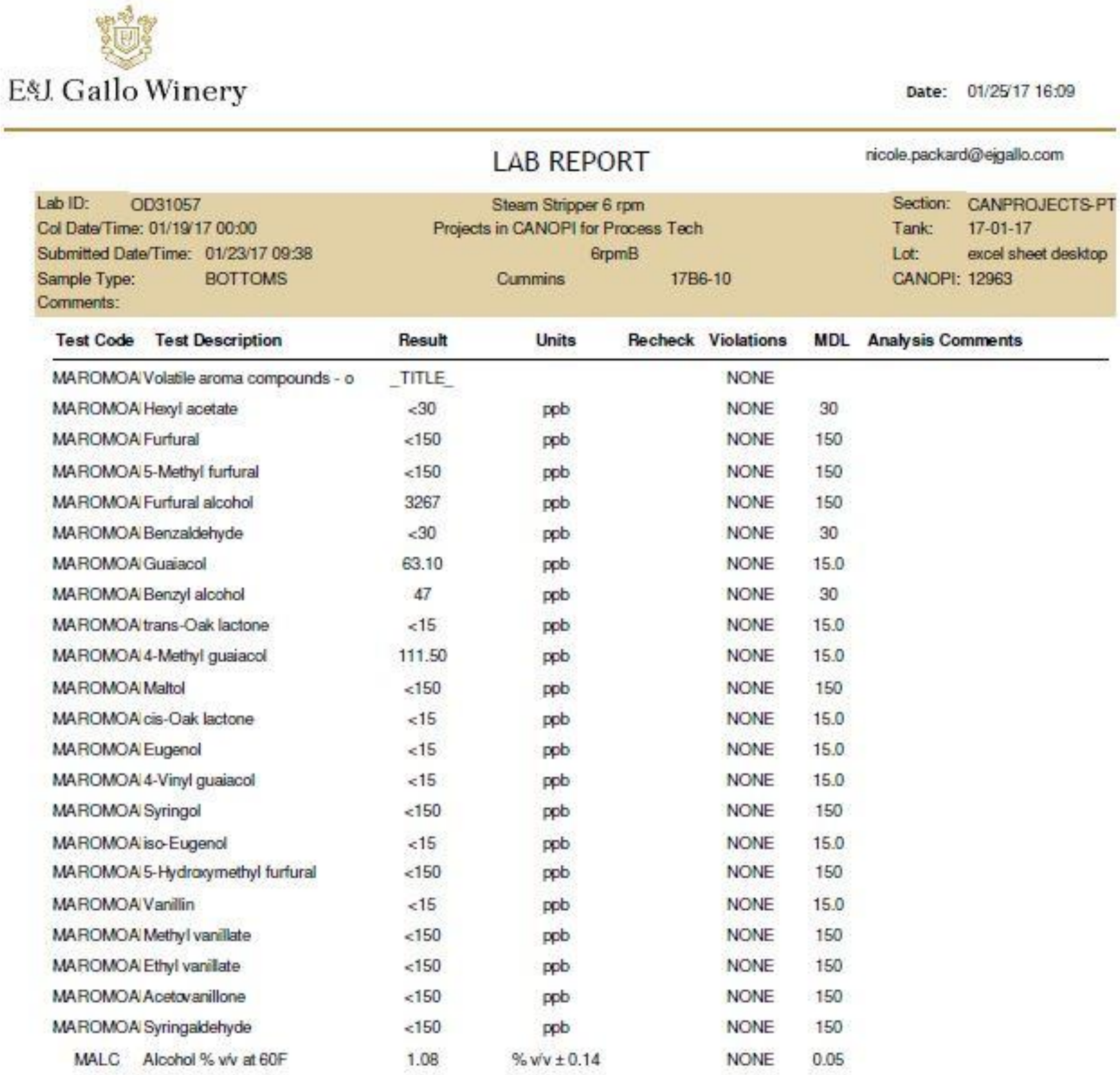
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31060	Steam Stripper 6 rpm		Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech		Tank: 17-01-17
Submitted Date/Time: 01/23/17 09:38	6rpmD		Lot: excel sheet desktop
Sample Type: DISTILLATE	Cummins	17D6	CANOPI: 12963
Comments:			

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<100	ppb		NONE	100	
MAROMOA	Furfural	2146	ppb		NONE	500	
MAROMOA	5-Methyl furfural	722	ppb		NONE	500	
MAROMOA	Furfural alcohol	2779	ppb		NONE	500	
MAROMOA	Benzaldehyde	<100	ppb		NONE	100	
MAROMOA	Guaiacol	234.30	ppb		NONE	50.0	
MAROMOA	Benzyl alcohol	<100	ppb		NONE	100	
MAROMOA	trans-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	4-Methyl guaiacol	530.80	ppb		NONE	50.0	
MAROMOA	Maltol	<500	ppb		NONE	500	
MAROMOA	cis-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	Eugenol	56.90	ppb		NONE	50.0	
MAROMOA	4-Vinyl guaiacol	<50	ppb		NONE	50.0	
MAROMOA	Syringol	<500	ppb		NONE	500	
MAROMOA	iso-Eugenol	<50	ppb		NONE	50.0	
MAROMOA	5-Hydroxymethyl furfural	<500	ppb		NONE	500	
MAROMOA	Vanillin	<50	ppb		NONE	50.0	
MAROMOA	Methyl vanillate	<500	ppb		NONE	500	
MAROMOA	Ethyl vanillate	<500	ppb		NONE	500	
MAROMOA	Acetovanillone	<500	ppb		NONE	500	
MAROMOA	Syringaldehyde	<500	ppb		NONE	500	
MALC	Alcohol % v/v at 60F	41.47	% v/v ± 0.14		NONE	0.1	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

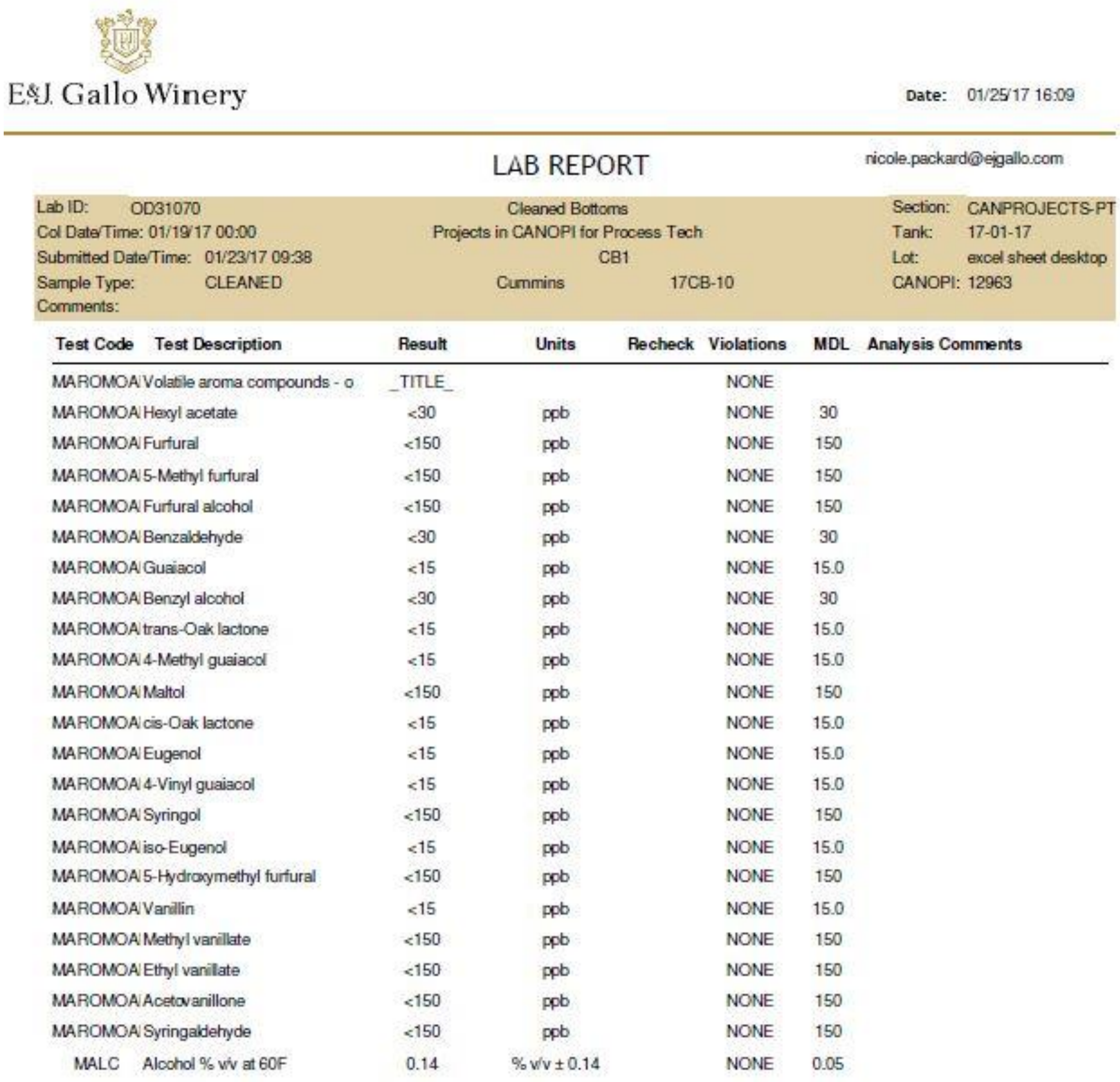
nicole.packard@ejgallo.com

Lab ID: OD31057
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: BOTTOMS
Comments:

Steam Stripper 6 rpm
Projects in CANOPI for Process Tech
6rpmB
Cummins 17B6-10

Section: CANPROJECTS-PT
Tank: 17-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	3267	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	63.10	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	47	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	111.50	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	1.08	% v/v ± 0.14		NONE	0.05	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

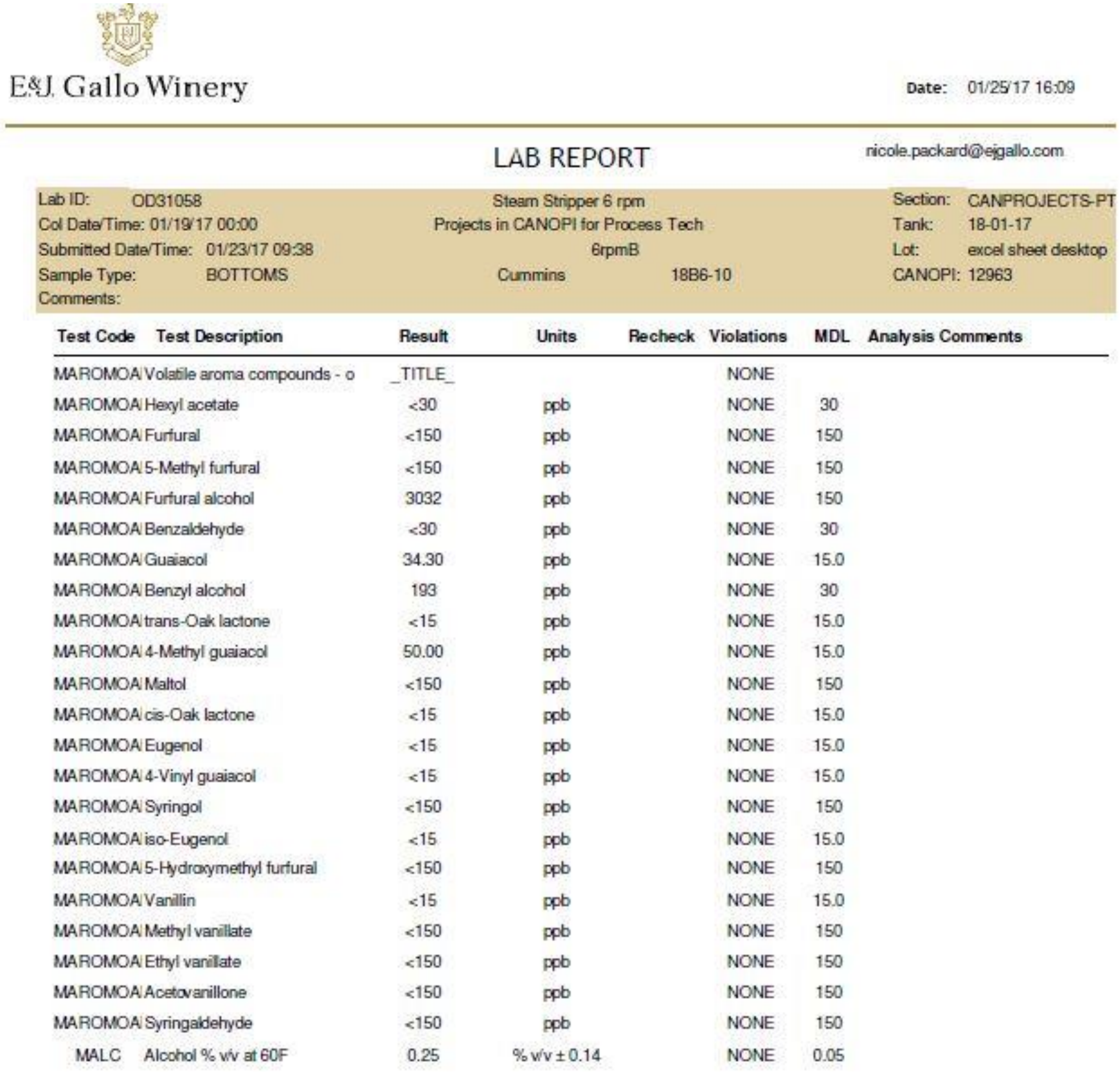
nicole.packard@ejgallo.com

Lab ID: OD31070
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: CLEANED
Comments:

Cleaned Bottoms
Projects in CANOPI for Process Tech
CB1
Cummins 17CB-10

Section: CANPROJECTS-PT
Tank: 17-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	<150	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	<30	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	0.14	% v/v ± 0.14		NONE	0.05	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

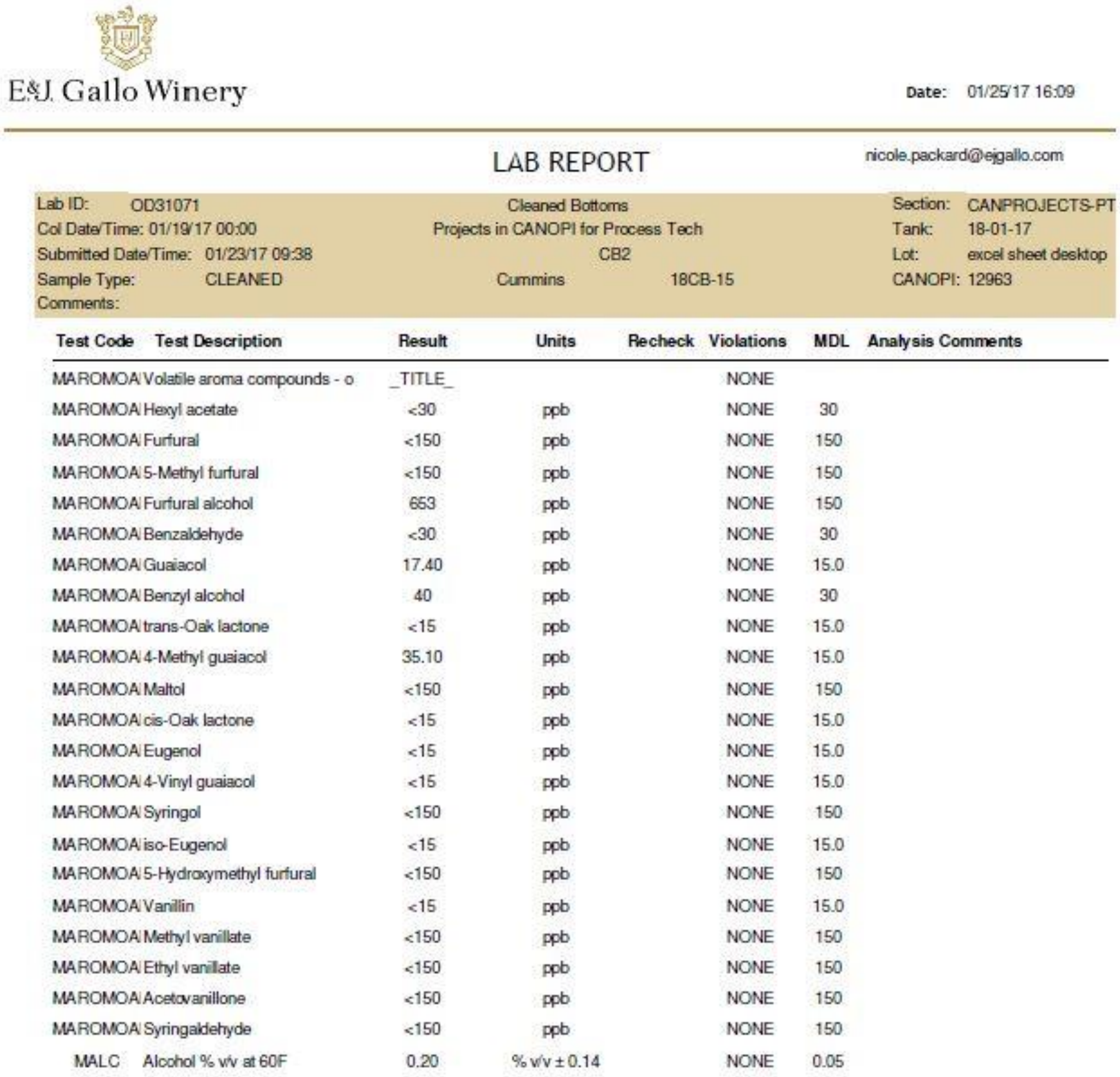
nicole.packard@ejgallo.com

Lab ID: OD31058
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: BOTTOMS
Comments:

Steam Stripper 6 rpm
Projects in CANOPI for Process Tech
6rpmB
Cummins 18B6-10

Section: CANPROJECTS-PT
Tank: 18-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	3032	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	34.30	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	193	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	50.00	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	0.25	% v/v ± 0.14		NONE	0.05	



E&J Gallo Winery

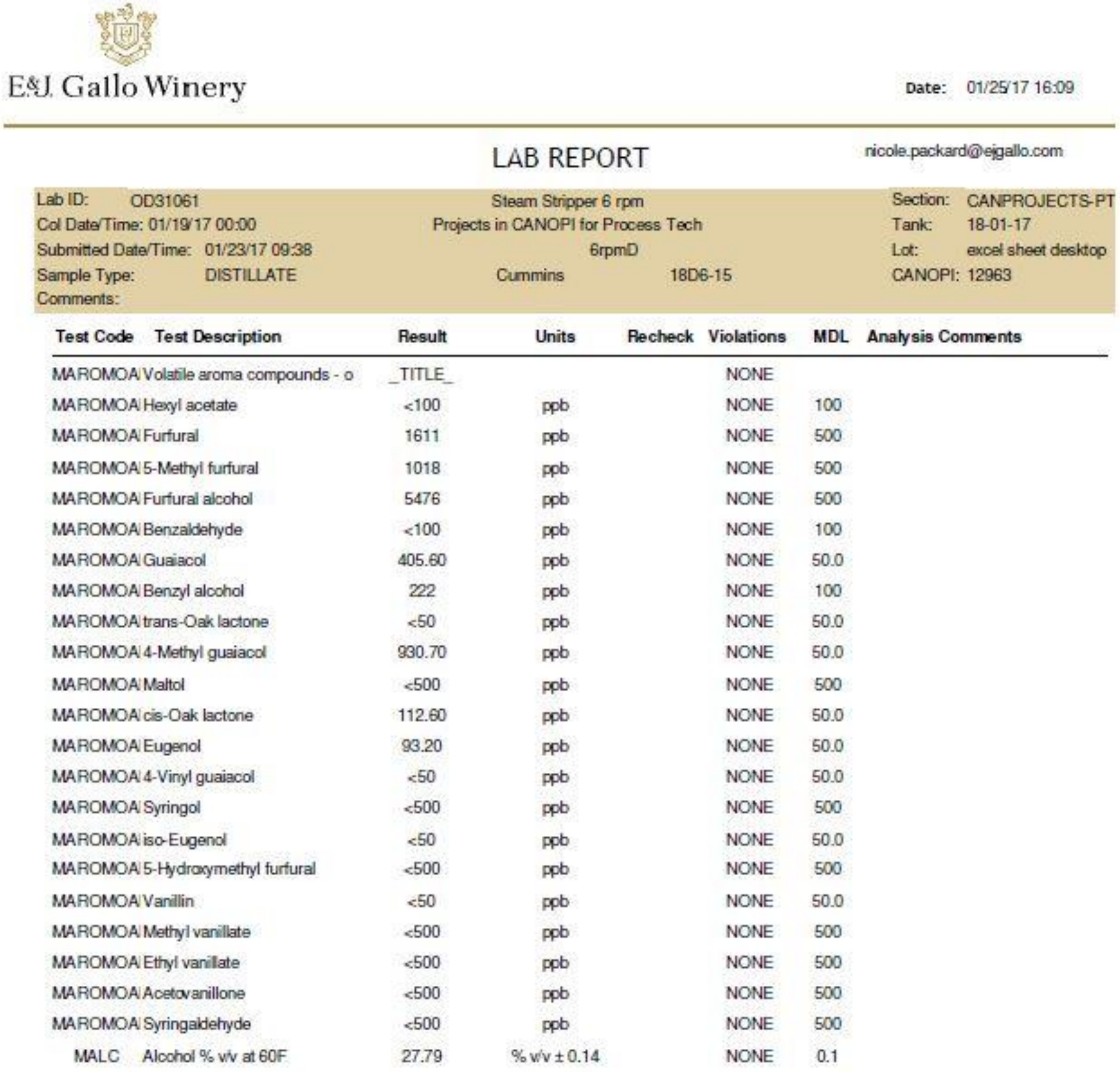
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31071	Cleaned Bottoms		Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech		Tank: 18-01-17
Submitted Date/Time: 01/23/17 09:38	CB2		Lot: excel sheet desktop
Sample Type: CLEANED	Cummins	18CB-15	CANOPI: 12963
Comments:			

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<30	ppb		NONE	30	
MAROMOA	Furfural	<150	ppb		NONE	150	
MAROMOA	5-Methyl furfural	<150	ppb		NONE	150	
MAROMOA	Furfural alcohol	653	ppb		NONE	150	
MAROMOA	Benzaldehyde	<30	ppb		NONE	30	
MAROMOA	Guaiacol	17.40	ppb		NONE	15.0	
MAROMOA	Benzyl alcohol	40	ppb		NONE	30	
MAROMOA	trans-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	4-Methyl guaiacol	35.10	ppb		NONE	15.0	
MAROMOA	Maltol	<150	ppb		NONE	150	
MAROMOA	cis-Oak lactone	<15	ppb		NONE	15.0	
MAROMOA	Eugenol	<15	ppb		NONE	15.0	
MAROMOA	4-Vinyl guaiacol	<15	ppb		NONE	15.0	
MAROMOA	Syringol	<150	ppb		NONE	150	
MAROMOA	iso-Eugenol	<15	ppb		NONE	15.0	
MAROMOA	5-Hydroxymethyl furfural	<150	ppb		NONE	150	
MAROMOA	Vanillin	<15	ppb		NONE	15.0	
MAROMOA	Methyl vanillate	<150	ppb		NONE	150	
MAROMOA	Ethyl vanillate	<150	ppb		NONE	150	
MAROMOA	Acetovanillone	<150	ppb		NONE	150	
MAROMOA	Syringaldehyde	<150	ppb		NONE	150	
MALC	Alcohol % v/v at 60F	0.20	% v/v ± 0.14		NONE	0.05	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

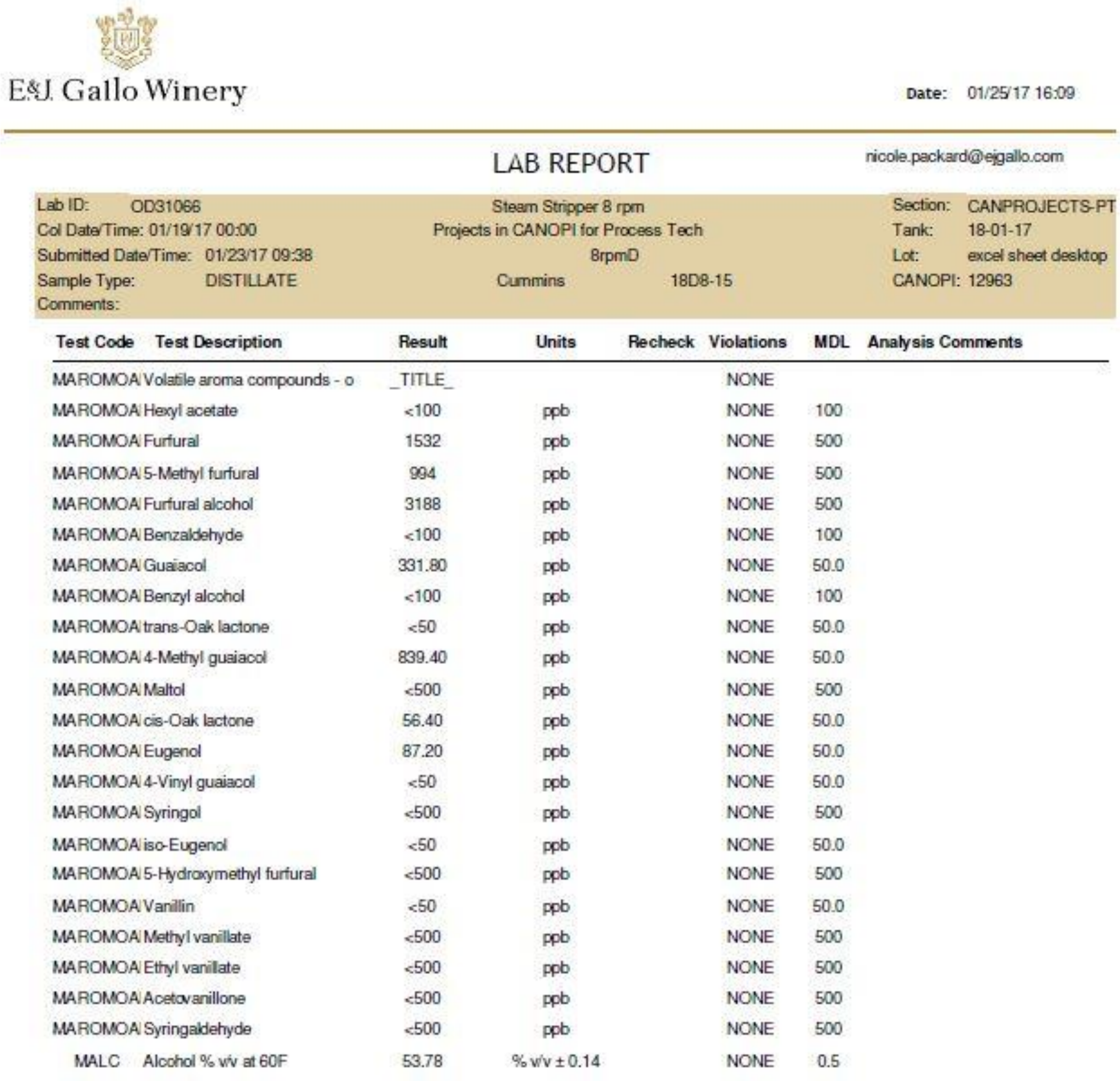
nicole.packard@ejgallo.com

Lab ID: OD31061
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: DISTILLATE
Comments:

Steam Stripper 6 rpm
Projects in CANOPI for Process Tech
6rpmD
Cummins 18D6-15

Section: CANPROJECTS-PT
Tank: 18-01-17
Lot: excel sheet desktop
CANOPI: 12963

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<100	ppb		NONE	100	
MAROMOA	Furfural	1611	ppb		NONE	500	
MAROMOA	5-Methyl furfural	1018	ppb		NONE	500	
MAROMOA	Furfural alcohol	5476	ppb		NONE	500	
MAROMOA	Benzaldehyde	<100	ppb		NONE	100	
MAROMOA	Guaiacol	405.60	ppb		NONE	50.0	
MAROMOA	Benzyl alcohol	222	ppb		NONE	100	
MAROMOA	trans-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	4-Methyl guaiacol	930.70	ppb		NONE	50.0	
MAROMOA	Maltol	<500	ppb		NONE	500	
MAROMOA	cis-Oak lactone	112.60	ppb		NONE	50.0	
MAROMOA	Eugenol	93.20	ppb		NONE	50.0	
MAROMOA	4-Vinyl guaiacol	<50	ppb		NONE	50.0	
MAROMOA	Syringol	<500	ppb		NONE	500	
MAROMOA	iso-Eugenol	<50	ppb		NONE	50.0	
MAROMOA	5-Hydroxymethyl furfural	<500	ppb		NONE	500	
MAROMOA	Vanillin	<50	ppb		NONE	50.0	
MAROMOA	Methyl vanillate	<500	ppb		NONE	500	
MAROMOA	Ethyl vanillate	<500	ppb		NONE	500	
MAROMOA	Acetovanillone	<500	ppb		NONE	500	
MAROMOA	Syringaldehyde	<500	ppb		NONE	500	
MALC	Alcohol % v/v at 60F	27.79	% v/v ± 0.14		NONE	0.1	



E&J Gallo Winery

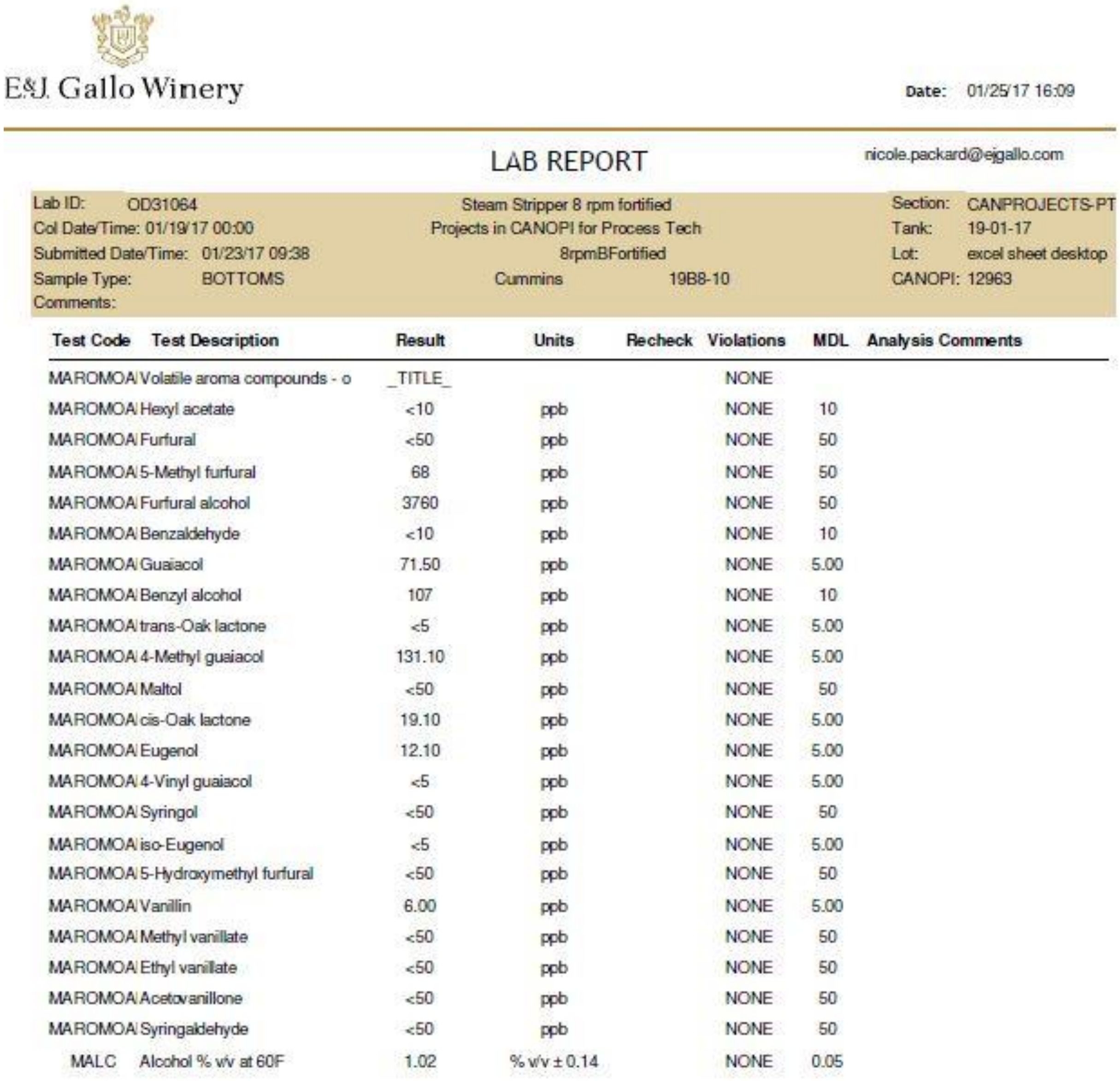
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31066	Steam Stripper 8 rpm		Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech		Tank: 18-01-17
Submitted Date/Time: 01/23/17 09:38	8rpmD		Lot: excel sheet desktop
Sample Type: DISTILLATE	Cummins	18D8-15	CANOPI: 12963
Comments:			

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<100	ppb		NONE	100	
MAROMOA	Furfural	1532	ppb		NONE	500	
MAROMOA	5-Methyl furfural	994	ppb		NONE	500	
MAROMOA	Furfural alcohol	3188	ppb		NONE	500	
MAROMOA	Benzaldehyde	<100	ppb		NONE	100	
MAROMOA	Guaiacol	331.80	ppb		NONE	50.0	
MAROMOA	Benzyl alcohol	<100	ppb		NONE	100	
MAROMOA	trans-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	4-Methyl guaiacol	839.40	ppb		NONE	50.0	
MAROMOA	Maltol	<500	ppb		NONE	500	
MAROMOA	cis-Oak lactone	56.40	ppb		NONE	50.0	
MAROMOA	Eugenol	87.20	ppb		NONE	50.0	
MAROMOA	4-Vinyl guaiacol	<50	ppb		NONE	50.0	
MAROMOA	Syringol	<500	ppb		NONE	500	
MAROMOA	iso-Eugenol	<50	ppb		NONE	50.0	
MAROMOA	5-Hydroxymethyl furfural	<500	ppb		NONE	500	
MAROMOA	Vanillin	<50	ppb		NONE	50.0	
MAROMOA	Methyl vanillate	<500	ppb		NONE	500	
MAROMOA	Ethyl vanillate	<500	ppb		NONE	500	
MAROMOA	Acetovanillone	<500	ppb		NONE	500	
MAROMOA	Syringaldehyde	<500	ppb		NONE	500	
MALC	Alcohol % v/v at 60F	53.78	% v/v ± 0.14		NONE	0.5	



E&J. Gallo Winery

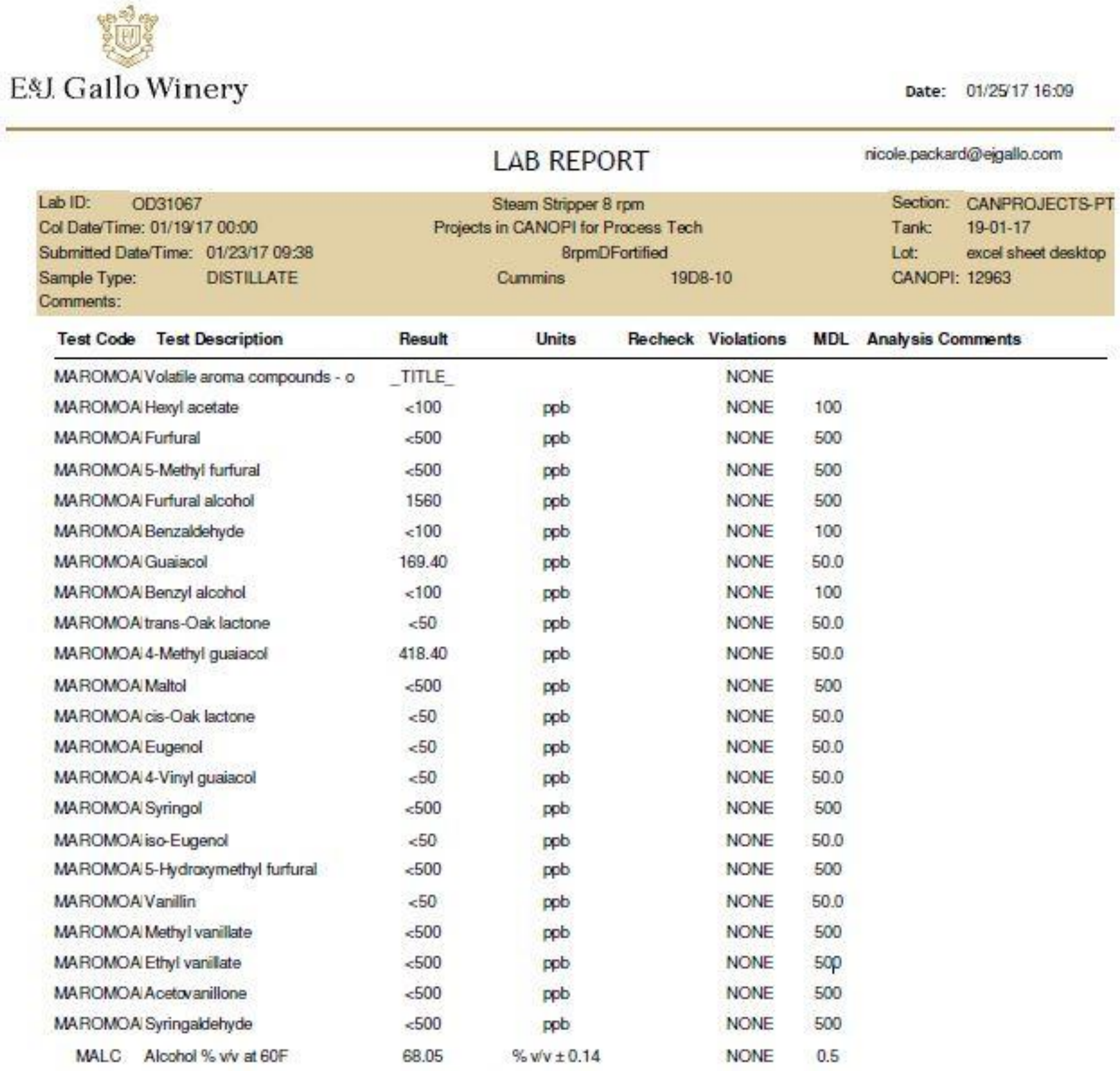
Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

Lab ID: OD31064	Steam Stripper 8 rpm fortified		Section: CANPROJECTS-PT
Col Date/Time: 01/19/17 00:00	Projects in CANOPI for Process Tech		Tank: 19-01-17
Submitted Date/Time: 01/23/17 09:38	8rpmBFortified		Lot: excel sheet desktop
Sample Type: BOTTOMS	Cummins	19B8-10	CANOPI: 12963
Comments:			

Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<10	ppb		NONE	10	
MAROMOA	Furfural	<50	ppb		NONE	50	
MAROMOA	5-Methyl furfural	68	ppb		NONE	50	
MAROMOA	Furfural alcohol	3760	ppb		NONE	50	
MAROMOA	Benzaldehyde	<10	ppb		NONE	10	
MAROMOA	Guaiacol	71.50	ppb		NONE	5.00	
MAROMOA	Benzyl alcohol	107	ppb		NONE	10	
MAROMOA	trans-Oak lactone	<5	ppb		NONE	5.00	
MAROMOA	4-Methyl guaiacol	131.10	ppb		NONE	5.00	
MAROMOA	Maltol	<50	ppb		NONE	50	
MAROMOA	cis-Oak lactone	19.10	ppb		NONE	5.00	
MAROMOA	Eugenol	12.10	ppb		NONE	5.00	
MAROMOA	4-Vinyl guaiacol	<5	ppb		NONE	5.00	
MAROMOA	Syringol	<50	ppb		NONE	50	
MAROMOA	iso-Eugenol	<5	ppb		NONE	5.00	
MAROMOA	5-Hydroxymethyl furfural	<50	ppb		NONE	50	
MAROMOA	Vanillin	6.00	ppb		NONE	5.00	
MAROMOA	Methyl vanillate	<50	ppb		NONE	50	
MAROMOA	Ethyl vanillate	<50	ppb		NONE	50	
MAROMOA	Acetovanillone	<50	ppb		NONE	50	
MAROMOA	Syringaldehyde	<50	ppb		NONE	50	
MALC	Alcohol % v/v at 60F	1.02	% v/v ± 0.14		NONE	0.05	



E&J. Gallo Winery

Date: 01/25/17 16:09

LAB REPORT

nicole.packard@ejgallo.com

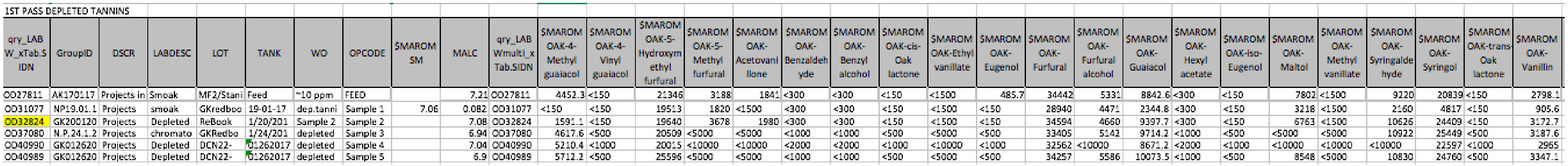
Lab ID: OD31067
Col Date/Time: 01/19/17 00:00
Submitted Date/Time: 01/23/17 09:38
Sample Type: DISTILLATE
Comments:

Steam Stripper 8 rpm
Projects in CANOPI for Process Tech
8rpmDFortified
Cummins 19D8-10

Section: CANPROJECTS-PT
Tank: 19-01-17
Lot: excel sheet desktop
CANOPI: 12963

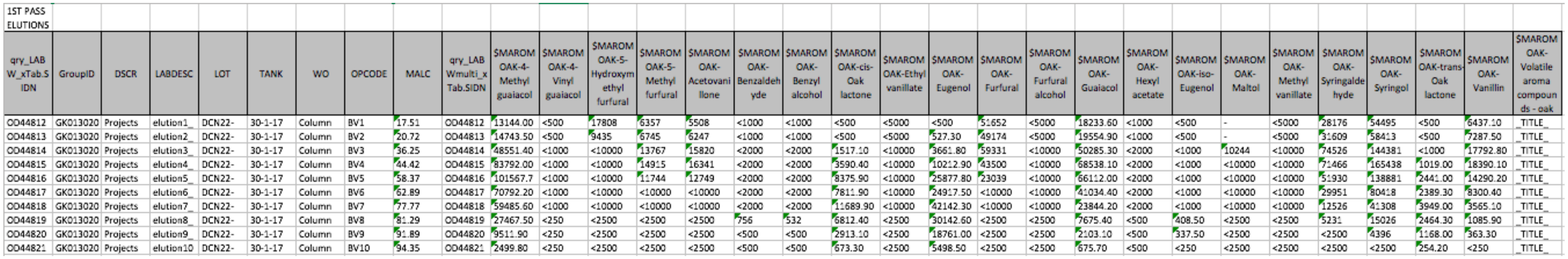
Test Code	Test Description	Result	Units	Recheck	Violations	MDL	Analysis Comments
MAROMOA	Volatile aroma compounds - o	_TITLE_			NONE		
MAROMOA	Hexyl acetate	<100	ppb		NONE	100	
MAROMOA	Furfural	<500	ppb		NONE	500	
MAROMOA	5-Methyl furfural	<500	ppb		NONE	500	
MAROMOA	Furfural alcohol	1560	ppb		NONE	500	
MAROMOA	Benzaldehyde	<100	ppb		NONE	100	
MAROMOA	Guaiacol	169.40	ppb		NONE	50.0	
MAROMOA	Benzyl alcohol	<100	ppb		NONE	100	
MAROMOA	trans-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	4-Methyl guaiacol	418.40	ppb		NONE	50.0	
MAROMOA	Maltol	<500	ppb		NONE	500	
MAROMOA	cis-Oak lactone	<50	ppb		NONE	50.0	
MAROMOA	Eugenol	<50	ppb		NONE	50.0	
MAROMOA	4-Vinyl guaiacol	<50	ppb		NONE	50.0	
MAROMOA	Syringol	<500	ppb		NONE	500	
MAROMOA	iso-Eugenol	<50	ppb		NONE	50.0	
MAROMOA	5-Hydroxymethyl furfural	<500	ppb		NONE	500	
MAROMOA	Vanillin	<50	ppb		NONE	50.0	
MAROMOA	Methyl vanillate	<500	ppb		NONE	500	
MAROMOA	Ethyl vanillate	<500	ppb		NONE	500	
MAROMOA	Acetovanillone	<500	ppb		NONE	500	
MAROMOA	Syringaldehyde	<500	ppb		NONE	500	
MALC	Alcohol % v/v at 60F	68.05	% v/v ± 0.14		NONE	0.5	

IV.E Column Chromatography



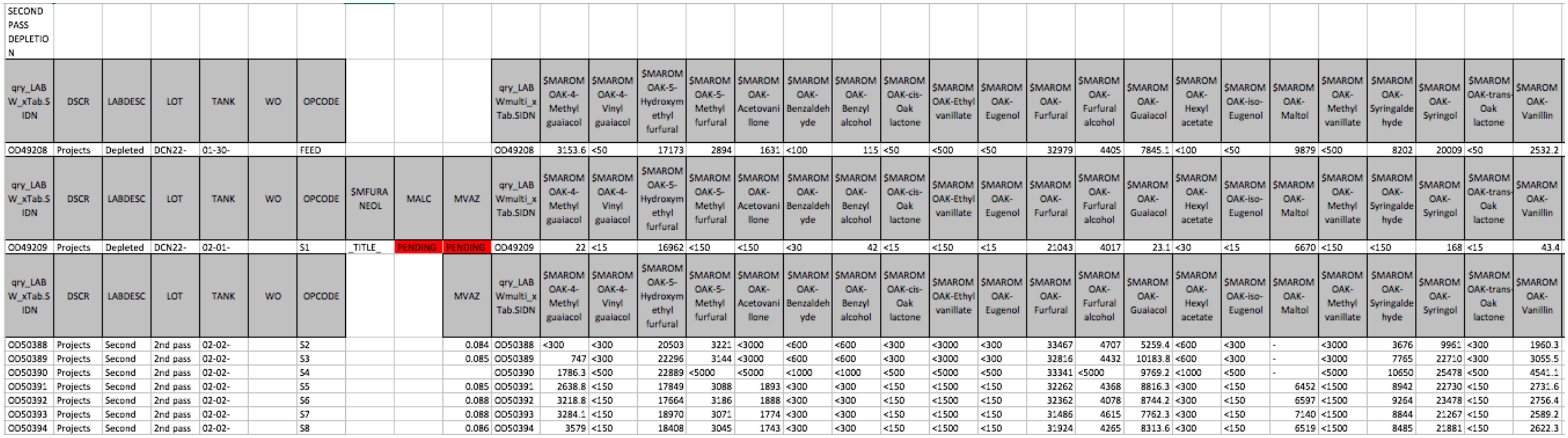
1ST PASS DEPLETED TANNINS

qry_LAB W_xTab.SIDN	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	$MAROM SM	MALC	qry_LAB Wmulti_x Tab.SIDN	$MAROM OAK-4-Methyl guaiacol	$MAROM OAK-4-Vinyl guaiacol	$MAROM OAK-5-Hydroxymethyl furfural	$MAROM OAK-5-Methyl furfural	$MAROM OAK-Acetovanillone	$MAROM OAK-Benzaldehyde	$MAROM OAK-Benzyl alcohol	$MAROM OAK-cis-Oak lactone	$MAROM OAK-Ethyl vanillate	$MAROM OAK-Eugenol	$MAROM OAK-Furfural	$MAROM OAK-Furfural alcohol	$MAROM OAK-Guaiacol	$MAROM OAK-Hexyl acetate	$MAROM OAK-iso-Eugenol	$MAROM OAK-Maltol	$MAROM OAK-Methyl vanillate	$MAROM OAK-Syringaldehyde	$MAROM OAK-Syringol	$MAROM OAK-trans-Oak lactone	$MAROM OAK-Vanillin
OD27811	AK170117	Projects in	Smoak	MF2/Stani	Feed	~10 ppm	FEED		7.21	OD27811	4452.3	<150	21346	3188	1841	<300	<300	<150	<1500	485.7	34442	5331	8842.6	<300	<150	7802	<1500	9220	20839	<150	2798.1
OD31077	NP19.01.1	Projects	smoak	GKredboo	19-01-17	dep.tanni	Sample 1	7.06	0.082	OD31077	<150	<150	19513	1820	<1500	<300	<300	<150	<1500	<150	28940	4471	2344.8	<300	<150	3218	<1500	2160	4817	<150	905.6
OD32824	GK200120	Projects	Depleted	ReBook	1/20/201	Sample 2	Sample 2		7.08	OD32824	1591.1	<150	19640	3678	1980	<300	<300	<150	<1500	<150	34594	4660	9397.7	<300	<150	6763	<1500	10626	24409	<150	3172.7
OD37080	N.P.24.1.2	Projects	chromato	GKRedbo	1/24/201	depleted	Sample 3		6.94	OD37080	4617.6	<500	20509	<5000	<5000	<1000	<1000	<500	<5000	<500	33405	5142	9714.2	<1000	<500	<5000	<5000	10922	25449	<500	3187.6
OD40990	GK012620	Projects	Depleted	DCN22-	01262017	depleted	Sample 4		7.04	OD40990	5210.4	<1000	20015	<10000	<10000	<2000	<2000	<1000	<10000	<1000	32562	<10000	8671.2	<2000	<1000	<10000	<10000	<10000	22597	<1000	2965
OD40989	GK012620	Projects	Depleted	DCN22-	01262017	depleted	Sample 5		6.9	OD40989	5712.2	<500	25596	<5000	<5000	<1000	<1000	<500	<5000	<500	34257	5586	10073.5	<1000	<500	8548	<5000	10830	24760	<500	3347.1

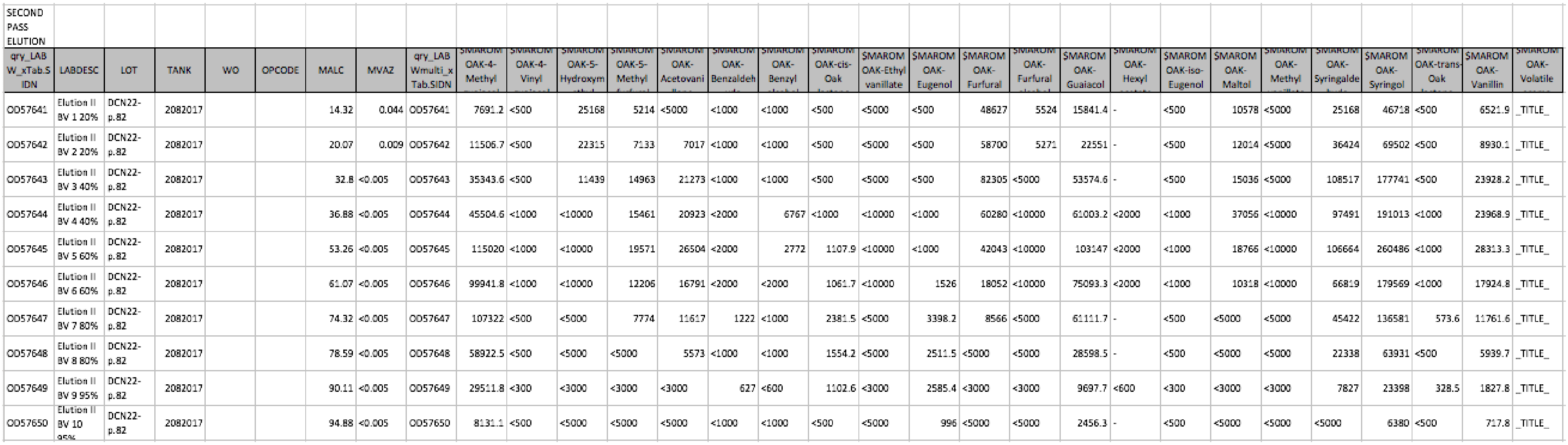


1ST PASS ELUTIONS

qry_LAB W_xTab.SIDN	GroupID	DSCR	LABDESC	LOT	TANK	WO	OPCODE	MALC	qry_LAB Wmulti_x Tab.SIDN	$MAROM OAK-4-Methyl guaiacol	$MAROM OAK-4-Vinyl guaiacol	$MAROM OAK-5-Hydroxymethyl furfural	$MAROM OAK-5-Methyl furfural	$MAROM OAK-Acetovanillone	$MAROM OAK-Benzaldehyde	$MAROM OAK-Benzyl alcohol	$MAROM OAK-cis-Oak lactone	$MAROM OAK-Ethyl vanillate	$MAROM OAK-Eugenol	$MAROM OAK-Furfural	$MAROM OAK-Furfural alcohol	$MAROM OAK-Guaiacol	$MAROM OAK-Hexyl acetate	$MAROM OAK-iso-Eugenol	$MAROM OAK-Maltol	$MAROM OAK-Methyl vanillate	$MAROM OAK-Syringaldehyde	$MAROM OAK-Syringol	$MAROM OAK-trans-Oak lactone	$MAROM OAK-Vanillin	$MAROM OAK-Volatile aroma compounds - oak
OD44812	GK013020	Projects	elution1_	DCN22-	30-1-17	Column	BV1	17.51	OD44812	13144.00	<500	17808	6357	5508	<1000	<1000	<500	<5000	<500	51652	<5000	18233.60	<1000	<500	-	<5000	28176	54495	<500	6437.10	_TITLE_
OD44813	GK013020	Projects	elution2_	DCN22-	30-1-17	Column	BV2	20.72	OD44813	14743.50	<500	9435	6745	6247	<1000	<1000	<500	<5000	527.30	49174	<5000	19554.90	<1000	<500	-	<5000	31609	58413	<500	7287.50	_TITLE_
OD44814	GK013020	Projects	elution3_	DCN22-	30-1-17	Column	BV3	36.25	OD44814	48551.40	<1000	<10000	13767	15820	<2000	<2000	1517.10	<10000	3661.80	59331	<10000	50285.30	<2000	<1000	10244	<10000	74526	144381	<1000	17792.80	_TITLE_
OD44815	GK013020	Projects	elution4_	DCN22-	30-1-17	Column	BV4	44.42	OD44815	83792.00	<1000	<10000	14915	16341	<2000	<2000	3590.40	<10000	10212.90	43500	<10000	68538.10	<2000	<1000	<10000	<10000	71466	165438	1019.00	18390.10	_TITLE_
OD44816	GK013020	Projects	elution5_	DCN22-	30-1-17	Column	BV5	58.37	OD44816	101567.7	<1000	<10000	11744	12749	<2000	<2000	8375.90	<10000	25877.80	23039	<10000	66112.00	<2000	<1000	<10000	<10000	51930	138881	2441.00	14290.20	_TITLE_
OD44817	GK013020	Projects	elution6_	DCN22-	30-1-17	Column	BV6	62.89	OD44817	70792.20	<1000	<10000	<10000	<10000	<2000	<2000	7811.90	<10000	24917.50	<10000	<10000	41034.40	<2000	<1000	<10000	<10000	29951	80418	2389.30	8300.40	_TITLE_
OD44818	GK013020	Projects	elution7_	DCN22-	30-1-17	Column	BV7	77.77	OD44818	59485.60	<1000	<10000	<10000	<10000	<2000	<2000	11689.90	<10000	42142.30	<10000	<10000	23844.20	<2000	<1000	<10000	<10000	12526	41308	3949.00	3565.10	_TITLE_
OD44819	GK013020	Projects	elution8_	DCN22-	30-1-17	Column	BV8	81.29	OD44819	27467.50	<250	<2500	<2500	<2500	756	532	6812.40	<2500	30142.60	<2500	<2500	7675.40	<500	408.50	<2500	<2500	5231	15026	2464.30	1085.90	_TITLE_
OD44820	GK013020	Projects	elution9_	DCN22-	30-1-17	Column	BV9	91.89	OD44820	9511.90	<250	<2500	<2500	<2500	<500	<500	2913.10	<2500	18761.00	<2500	<2500	2103.10	<500	337.50	<2500	<2500	<2500	4396	1168.00	363.30	_TITLE_
OD44821	GK013020	Projects	elution10	DCN22-	30-1-17	Column	BV10	94.35	OD44821	2499.80	<250	<2500	<2500	<2500	<500	<500	673.30	<2500	5498.50	<2500	<2500	675.70	<500	<250	<2500	<2500	<2500	<2500	254.20	<250	_TITLE_



SECOND PASS DEPLETION																															
qry_LABW_xTab.SIDN	DSCR	LABDESC	LOT	TANK	WO	OPCODE				qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin
OD49208	Projects	Depleted	DCN22-	01-30-		FEED				OD49208	3153.6	<50	17173	2894	1631	<100	115	<50	<500	<50	32979	4405	7845.1	<100	<50	9879	<500	8202	20009	<50	2532.2
qry_LABW_xTab.SIDN	DSCR	LABDESC	LOT	TANK	WO	OPCODE	$MFURANEOL	MALC	MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin
OD49209	Projects	Depleted	DCN22-	02-01-		S1	_TITLE_	PENDING	PENDING	OD49209	22	<15	16962	<150	<150	<30	42	<15	<150	<15	21043	4017	23.1	<30	<15	6670	<150	<150	168	<15	43.4
qry_LABW_xTab.SIDN	DSCR	LABDESC	LOT	TANK	WO	OPCODE			MVAZ	qry_LABWmulti_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin
OD50388	Projects	Second	2nd pass	02-02-		S2			0.084	OD50388	<300	<300	20503	3221	<3000	<600	<600	<300	<3000	<300	33467	4707	5259.4	<600	<300	-	<3000	3676	9961	<300	1960.3
OD50389	Projects	Second	2nd pass	02-02-		S3			0.085	OD50389	747	<300	22296	3144	<3000	<600	<600	<300	<3000	<300	32816	4432	10183.8	<600	<300	-	<3000	7765	22710	<300	3055.5
OD50390	Projects	Second	2nd pass	02-02-		S4				OD50390	1786.3	<500	22889	<5000	<5000	<1000	<1000	<500	<5000	<500	33341	<5000	9769.2	<1000	<500	-	<5000	10650	25478	<500	4541.1
OD50391	Projects	Second	2nd pass	02-02-		S5			0.085	OD50391	2638.8	<150	17849	3088	1893	<300	<300	<150	<1500	<150	32262	4368	8816.3	<300	<150	6452	<1500	8942	22730	<150	2731.6
OD50392	Projects	Second	2nd pass	02-02-		S6			0.088	OD50392	3218.8	<150	17664	3186	1888	<300	<300	<150	<1500	<150	32362	4078	8744.2	<300	<150	6597	<1500	9264	23478	<150	2756.4
OD50393	Projects	Second	2nd pass	02-02-		S7			0.088	OD50393	3284.1	<150	18970	3071	1774	<300	<300	<150	<1500	<150	31486	4615	7762.3	<300	<150	7140	<1500	8844	21267	<150	2589.2
OD50394	Projects	Second	2nd pass	02-02-		S8			0.086	OD50394	3579	<150	18408	3045	1743	<300	<300	<150	<1500	<150	31924	4265	8313.6	<300	<150	6519	<1500	8485	21881	<150	2622.3



SECOND PASS ELUTION																														
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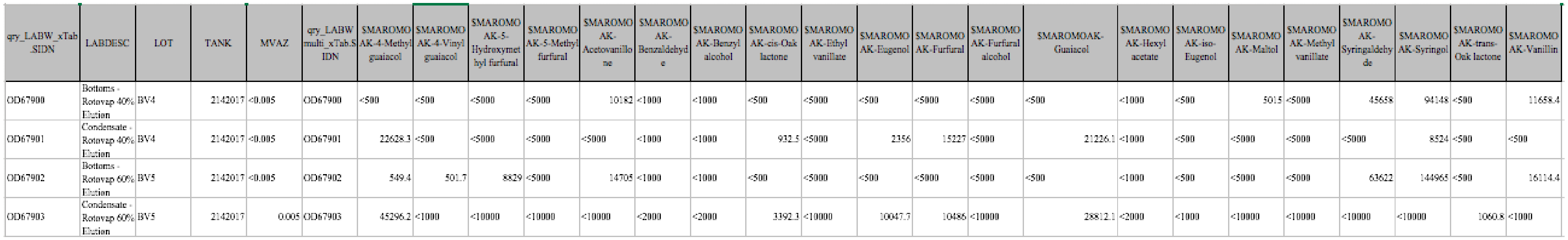
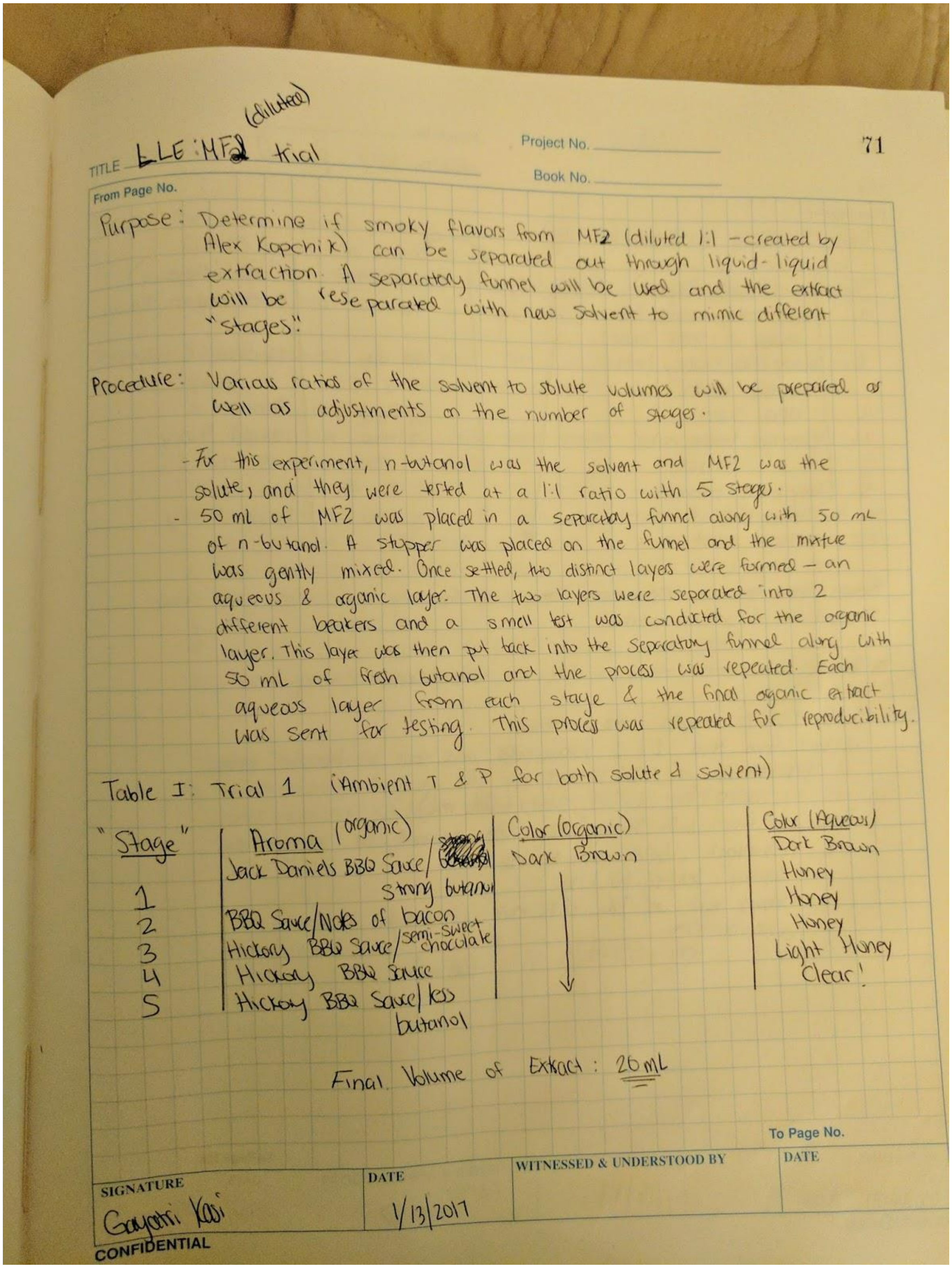
OD57641	Elution II BV 1 20%	DCN22-p.82	2082017			14.32	0.044	OD57641	7691.2	<500	25168	5214	<5000	<1000	<1000	<500	<5000	<500	48627	5524	15841.4	-	<500	10578	<5000	25168	46718	<500	6521.9	_TITLE_
OD57642	Elution II BV 2 20%	DCN22-p.82	2082017			20.07	0.009	OD57642	11506.7	<500	22315	7133	7017	<1000	<1000	<500	<5000	<500	58700	5271	22551	-	<500	12014	<5000	36424	69502	<500	8930.1	_TITLE_
OD57643	Elution II BV 3 40%	DCN22-p.82	2082017			32.8	<0.005	OD57643	35343.6	<500	11439	14963	21273	<1000	<1000	<500	<5000	<500	82305	<5000	53574.6	-	<500	15036	<5000	108517	177741	<500	23928.2	_TITLE_
OD57644	Elution II BV 4 40%	DCN22-p.82	2082017			36.88	<0.005	OD57644	45504.6	<1000	<10000	15461	20923	<2000	6767	<1000	<10000	<1000	60280	<10000	61003.2	<2000	<1000	37056	<10000	97491	191013	<1000	23968.9	_TITLE_
OD57645	Elution II BV 5 60%	DCN22-p.82	2082017			53.26	<0.005	OD57645	115020	<1000	<10000	19571	26504	<2000	2772	1107.9	<10000	<1000	42043	<10000	103147	<2000	<1000	18766	<10000	106664	260486	<1000	28313.3	_TITLE_
OD57646	Elution II BV 6 60%	DCN22-p.82	2082017			61.07	<0.005	OD57646	99941.8	<1000	<10000	12206	16791	<2000	<2000	1061.7	<10000	1526	18052	<10000	75093.3	<2000	<1000	10318	<10000	66819	179569	<1000	17924.8	_TITLE_
OD57647	Elution II BV 7 80%	DCN22-p.82	2082017			74.32	<0.005	OD57647	107322	<500	<5000	7774	11617	1222	<1000	2381.5	<5000	3398.2	8566	<5000	61111.7	-	<500	<5000	<5000	45422	136581	573.6	11761.6	_TITLE_
OD57648	Elution II BV 8 80%	DCN22-p.82	2082017			78.59	<0.005	OD57648	58922.5	<500	<5000	<5000	5573	<1000	<1000	1554.2	<5000	2511.5	<5000	<5000	28598.5	-	<500	<5000	<5000	22338	63931	<500	5939.7	_TITLE_
OD57649	Elution II BV 9 95%	DCN22-p.82	2082017			90.11	<0.005	OD57649	29511.8	<300	<3000	<3000	<3000	627	<600	1102.6	<3000	2585.4	<3000	<3000	9697.7	<600	<300	<3000	<3000	7827	23398	328.5	1827.8	_TITLE_
OD57650	Elution II BV 10 95%	DCN22-p.82	2082017			94.88	<0.005	OD57650	8131.1	<500	<5000	<5000	<5000	<1000	<1000	<500	<5000	996	<5000	<5000	2456.3	-	<500	<5000	<5000	<5000	6380	<500	717.8	_TITLE_

IV.F Dosing



qry_LABW_xTab.SIDN	LABDESC	LOT	TANK	MVAZ	qry_LABW multi_xTab.SIDN	$MAROMOAK-4-Methyl guaiacol	$MAROMOAK-4-Vinyl guaiacol	$MAROMOAK-5-Hydroxymethyl furfural	$MAROMOAK-5-Methyl furfural	$MAROMOAK-Acetovanillone	$MAROMOAK-Benzaldehyde	$MAROMOAK-Benzyl alcohol	$MAROMOAK-cis-Oak lactone	$MAROMOAK-Ethyl vanillate	$MAROMOAK-Eugenol	$MAROMOAK-Furfural	$MAROMOAK-Furfural alcohol	$MAROMOAK-Guaiacol	$MAROMOAK-Hexyl acetate	$MAROMOAK-iso-Eugenol	$MAROMOAK-Maltol	$MAROMOAK-Methyl vanillate	$MAROMOAK-Syringaldehyde	$MAROMOAK-Syringol	$MAROMOAK-trans-Oak lactone	$MAROMOAK-Vanillin
OD67900	Bottoms - Rotovap 40% Elution	BV4	2142017	<0.005	OD67900	<500	<500	<5000	<5000	10182	<1000	<1000	<500	<5000	<500	<5000	<5000	<500	<1000	<500	5015	<5000	45658	94148	<500	11658.4
OD67901	Condensate - Rotovap 40% Elution	BV4	2142017	<0.005	OD67901	22628.3	<500	<5000	<5000	<5000	<1000	<1000	932.5	<5000	2356	15227	<5000	21226.1	<1000	<500	<5000	<5000	<5000	8524	<500	<500
OD67902	Bottoms - Rotovap 60% Elution	BV5	2142017	<0.005	OD67902	549.4	501.7	8829	<5000	14705	<1000	<1000	<500	<5000	<500	<5000	<5000	<500	<1000	<500	<5000	<5000	63622	144965	<500	16114.4
OD67903	Condensate - Rotovap 60% Elution	BV5	2142017	0.005	OD67903	45296.2	<1000	<10000	<10000	<10000	<2000	<2000	3392.3	<10000	10047.7	10486	<10000	28812.1	<2000	<1000	<10000	<10000	<10000	<10000	1060.8	<1000

Appendix V: Lab Notebook



TITLE LLE: MF2 (diluted) trial

Project No.

Book No.

71

From Page No.

Purpose: Determine if smoky flavors from MF2 (diluted 1:1 - created by Alex Kopchik) can be separated out through liquid-liquid extraction. A separatory funnel will be used and the extract will be reseparated with new solvent to mimic diffelent "stages".

Procedure: Various ratios of the solvent to solute volumes will be prepared as well as adjustments on the number of stages.

- For this experiment, n-butanol was the solvent and MF2 was the solute, and they were tested at a 1:1 ratio with 5 stages.
- 50 mL of MF2 was placed in a separatory funnel along with 50 mL of n-butanol. A stopper was placed on the funnel and the mixture was gently mixed. Once settled, two distinct layers were formed - an aqueous & organic layer. The two layers were separated into 2 different beakers and a smell test was conducted for the organic layer. This layer was then put back into the separatory funnel along with 50 mL of fresh butanol and the process was repeated. Each aqueous layer from each stage & the final organic extract was sent for testing. This process was repeated for reproducibility.

Table I: Trial 1 (Ambient T & P for both solute & solvent)

"Stage"	Aroma (organic)	Color (organic)	Color (Aqueous)
1	Jack Daniels BBQ Sauce/ [illegible] strong butanol	Dark Brown ↓	Dark Brown
2	BBQ Sauce/Notes of bacon		Honey
3	Hickory BBQ Sauce/semi-sweet chocolate		Honey
4	Hickory BBQ Sauce		Honey
5	Hickory BBQ Sauce/less butanol		Light Honey
			Clear!

Final Volume of Extract: 20 mL

To Page No.

SIGNATURE Gayatri Kasi

DATE 1/13/2017

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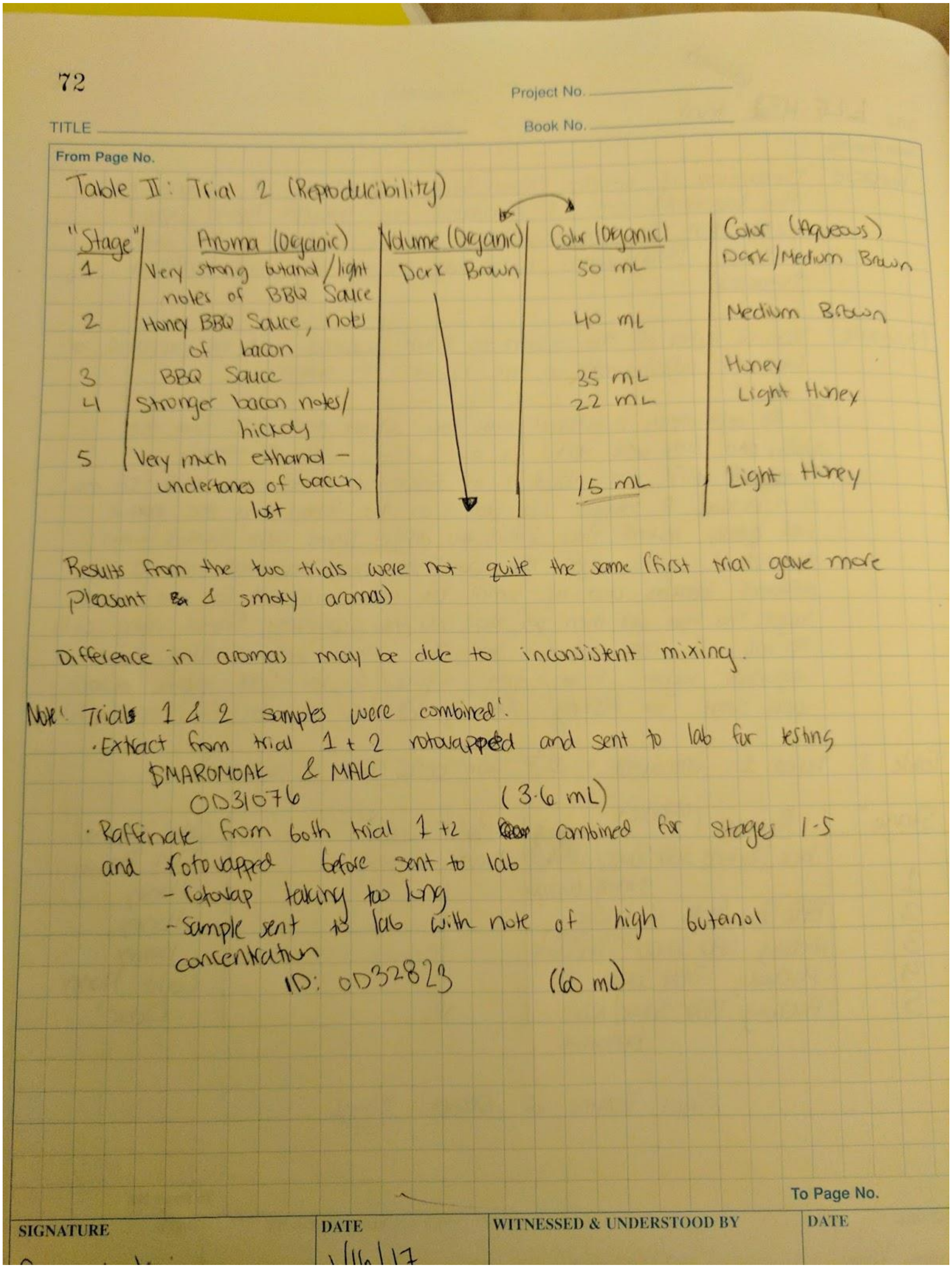

72

Project No.

TITLE

Book No.

From Page No.

Table II: Trial 2 (Reproducibility)

"Stage"	Aroma (Organic)	Volume (Organic)	Color (Organic)	Color (Aqueous)
1	Very strong ethanol/light notes of BBQ Sauce	Dark Brown	50 mL	Dark/Medium Brown
2	Honey BBQ Sauce, notes of bacon		40 mL	Medium Brown
3	BBQ Sauce		35 mL	Honey
4	Stronger bacon notes/ hickory		22 mL	Light Honey
5	Very much ethanol – undertones of bacon lost		15 mL	Light Honey

Results from the two trials were not quite the same (first trial gave more pleasant & smoky aromas)

Difference in aromas may be due to inconsistent mixing.

Note! Trials 1 & 2 samples were combined!

- Extract from trial 1 + 2 rotavapped and sent to lab for testing
  $MAROMOAK & MALC
  OD31076 (3.6 mL)
- Raffinate from both trial 1 + 2 combined for stages 1-5 and rotovapped before sent to lab
  - rotovap taking too long
  - Sample sent to lab with note of high butanol concentration
    ID: OD32823 (60 mL)

To Page No.

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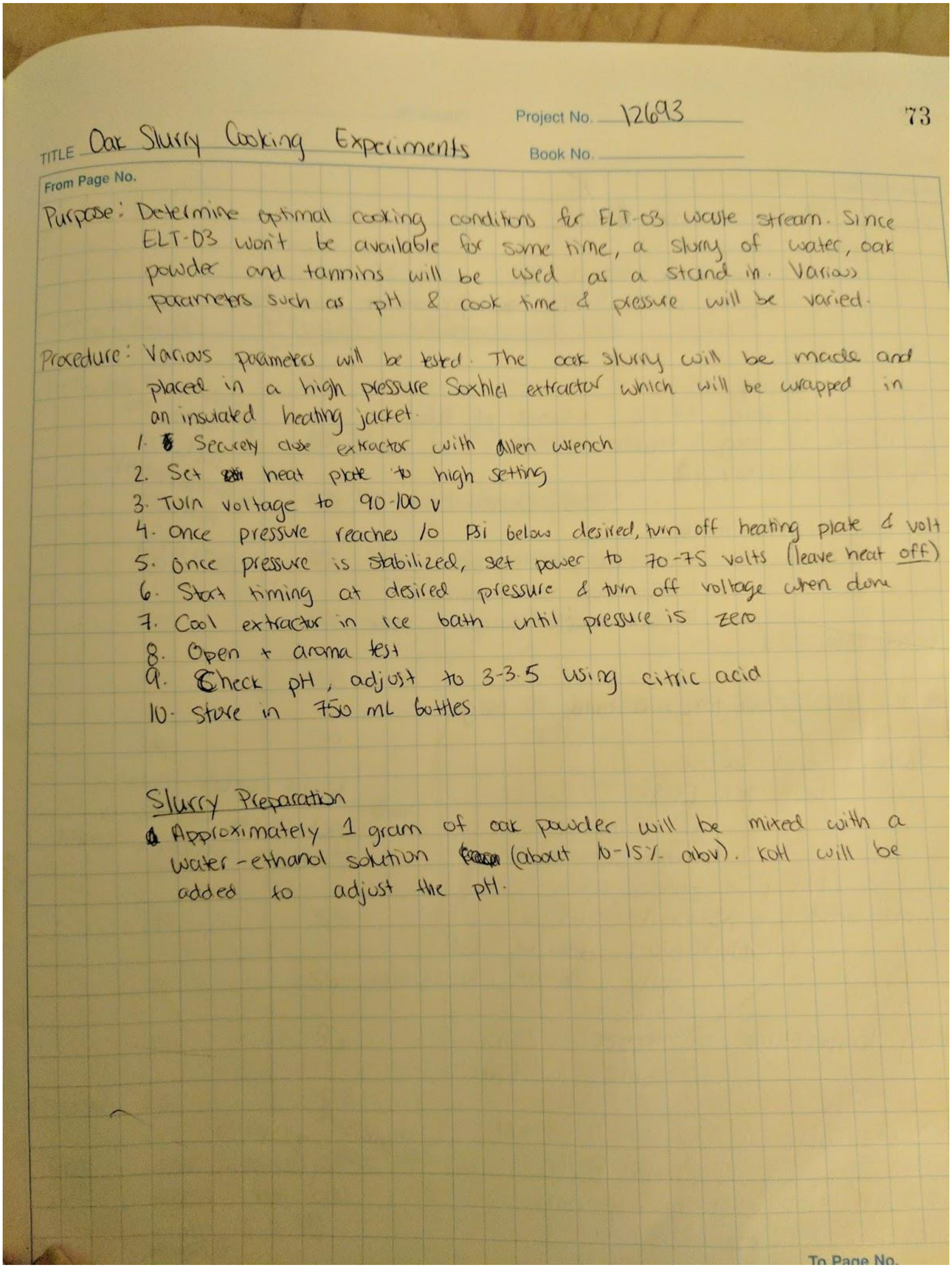
	1/16/17		



Project No. 12693

73

TITLE Oak Slurry Cooking Experiments

Book No.

From Page No.

Purpose: Determine optimal cooking conditions for ELT-03 waste stream. Since ELT-03 won't be available for some time, a slurry of water, oak powder and tannins will be used as a stand in. Various parameters such as pH & cook time & pressure will be varied.

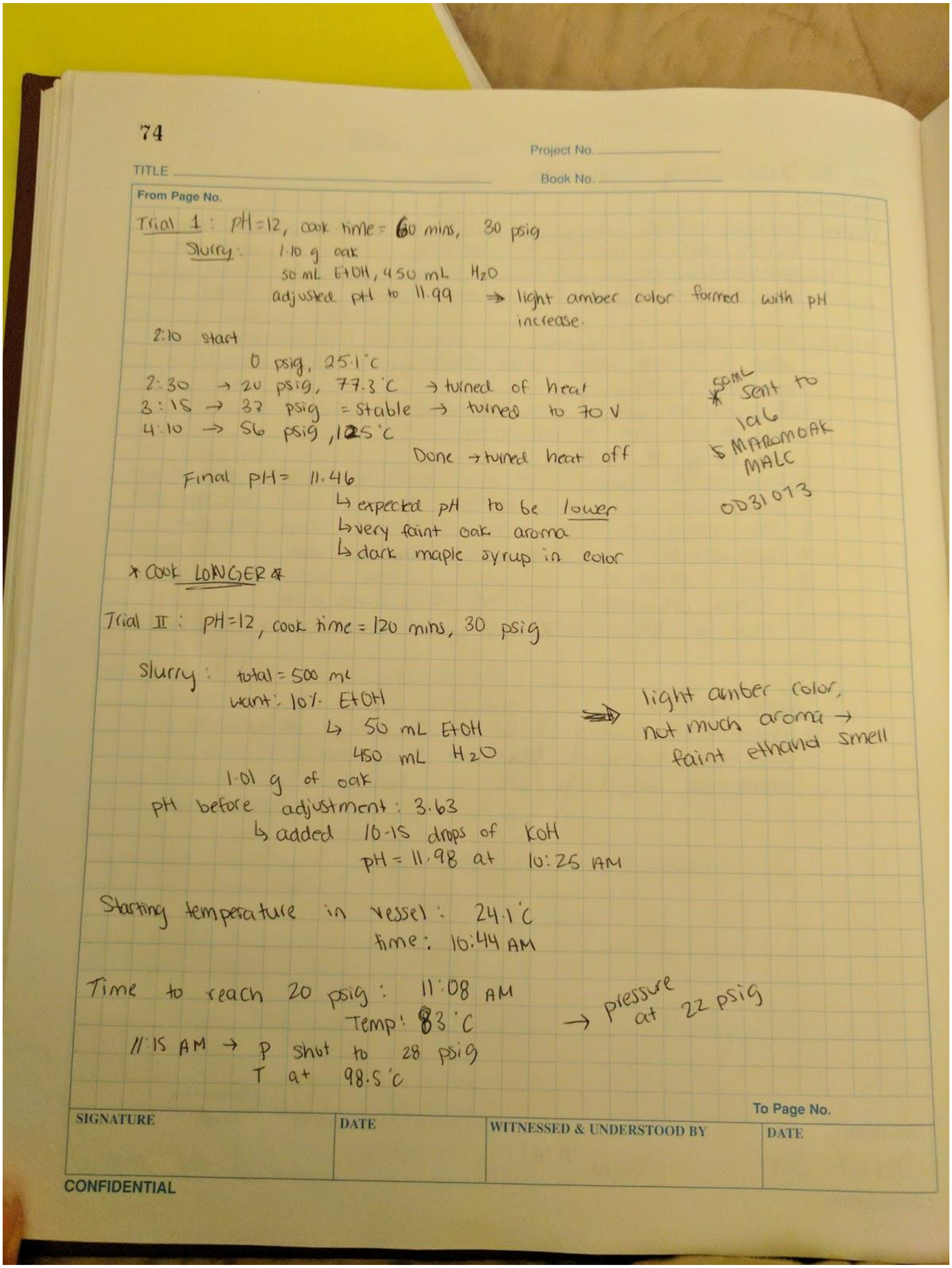
Procedure: Various parameters will be tested. The oak slurry will be made and placed in a high pressure Soxhlet extractor which will be wrapped in an insulated heating jacket.

1. Securely close extractor with allen wrench
2. Set heat plate to high setting
3. Turn voltage to 90-100 v
4. Once pressure reaches 10 psi below desired, turn off heating plate & volt
5. Once pressure is stabilized, set power to 70-75 volts (leave heat off)
6. Start timing at desired pressure & turn off voltage when done
7. Cool extractor in ice bath until pressure is zero
8. Open + aroma test
9. Check pH, adjust to 3-3.5 using citric acid
10. Store in 750 mL bottles

Slurry Preparation

Approximately 1 gram of oak powder will be mixed with a water-ethanol solution (about 10-15% abv). KOH will be added to adjust the pH.

To Page No.



74

TITLE

Project No.

Book No.

From Page No.

Trial 1 : pH = 12, cook time = 60 mins, 30 psig

Slurry: 1.10 g oak
50 mL EtOH, 450 mL H_2O
adjusted pH to 11.99 ⇒ light amber color formed with pH increase.

2:10 start
0 psig, 25.1 °C
2:30 → 20 psig, 77.3 °C → turned of heat
3:15 → 33 psig = stable → turned to 70 V
4:10 → 56 psig, 125 °C
Done → turned heat off

Final pH = 11.46
↳ expected pH to be lower
↳ very faint oak aroma
↳ dark maple syrup in color

* 50mL sent to lab
↳ MAROMOAK MALC
OD31073

* Cook LONGER *

Trial II : pH = 12, cook time = 120 mins, 30 psig

Slurry: total = 500 mL
want: 10% EtOH
↳ 50 mL EtOH
450 mL H_2O
1.01 g of oak

⇒ light amber color, not much aroma → faint ethanol smell

pH before adjustment: 3.63
↳ added 10-15 drops of KOH
pH = 11.98 at 10:25 AM

Starting temperature in vessel: 24.1 °C
time: 10:44 AM

Time to reach 20 psig: 11:08 AM
Temp: 83 °C → pressure at 22 psig
11:15 AM → P shot to 28 psig
T at 98.5 °C

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To Page No.

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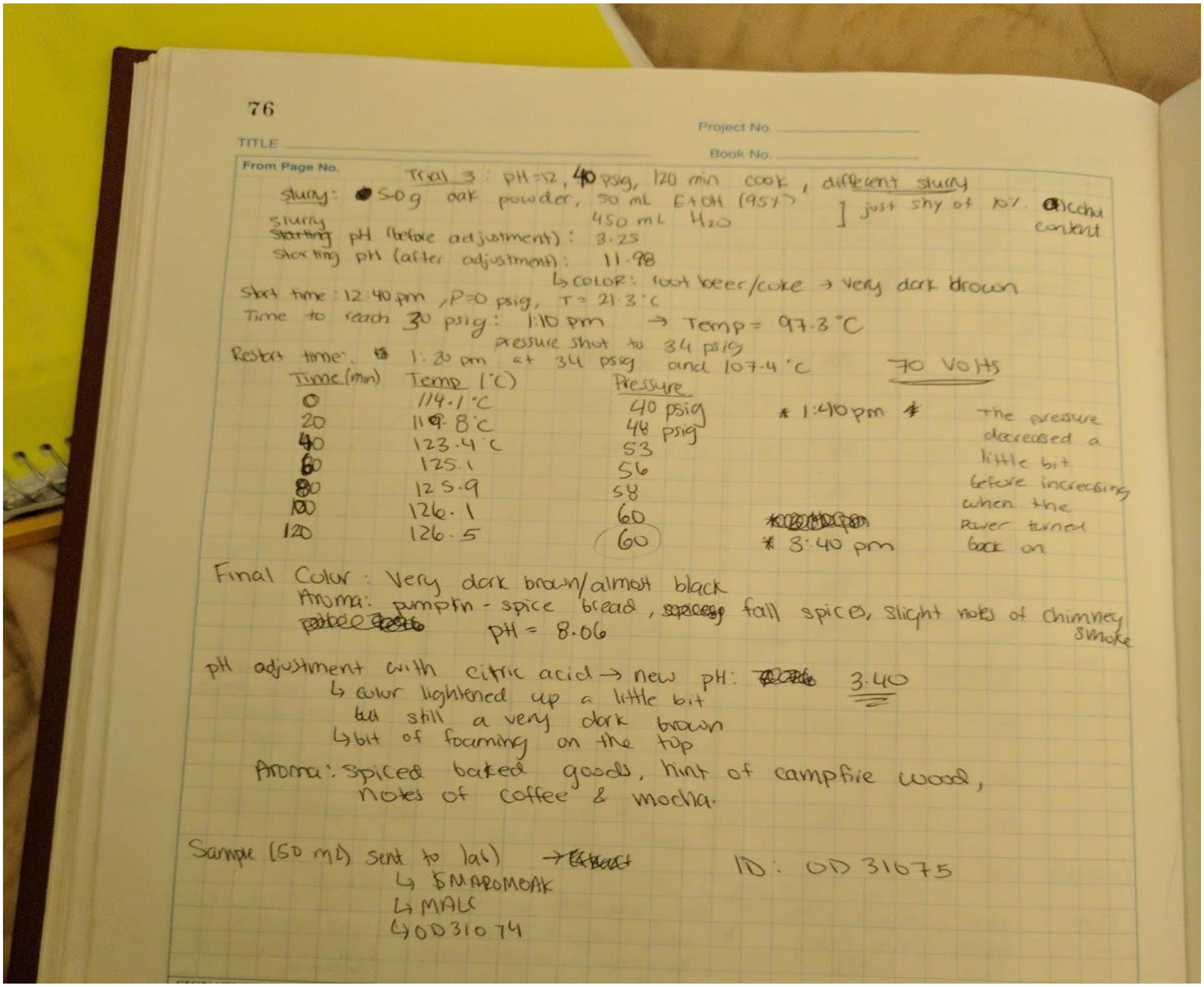


76

TITLE ______ Project No. ______

Book No. ______

From Page No.

Trial 3 : pH=12, 40 psig, 120 min cook, different slurry

slurry: 50g oak powder, 50 mL EtOH (95%)] just shy of 10% alcohol content
450 mL H_2O

~~Starting~~ slurry pH (before adjustment): 3.25
Starting pH (after adjustment): 11.98
↳ COLOR: root beer/coke → very dark brown

Start time: 12:40 pm, P=0 psig, T= 21.3°C
Time to reach 30 psig: 1:10 pm → Temp = 97.3°C
pressure shot to 34 psig
Restart time: 1:20 pm at 34 psig and 107.4°C

70 Volts

Time (min)	Temp (°C)	Pressure
0	114.1°C	40 psig
20	119.8°C	48 psig
40	123.4°C	53
60	125.1	56
80	125.9	58
100	126.1	60
120	126.5	60

* 1:40 pm *
[illegible]
* 3:40 pm

the pressure decreased a little bit before increasing when the power turned back on

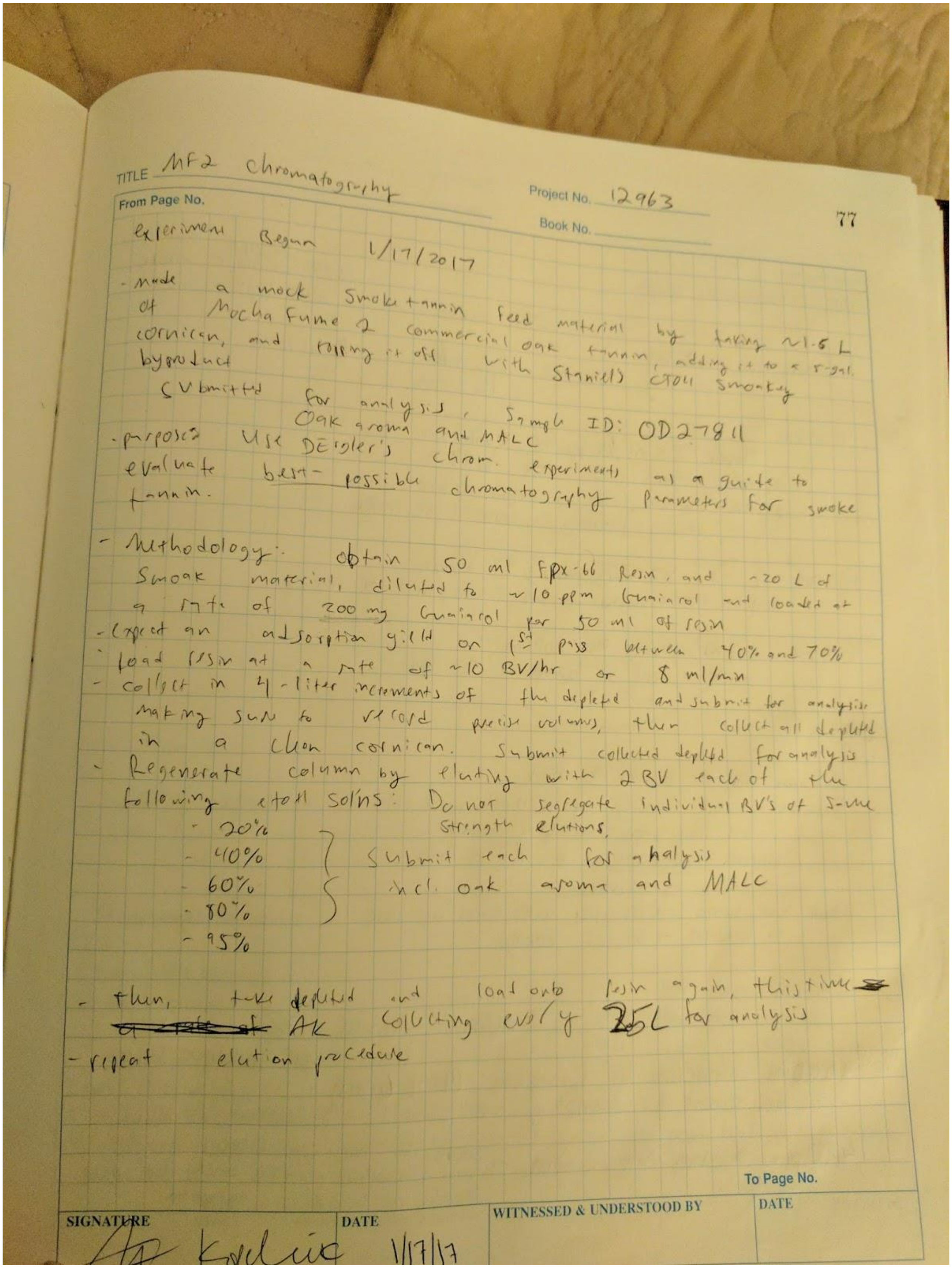
Final Color: Very dark brown/almost black
Aroma: pumpkin-spice bread, [illegible] fall spices, slight notes of chimney smoke [illegible]
pH = 8.06

pH adjustment with citric acid → new pH: [illegible] 3.40
↳ color lightened up a little bit but still a very dark brown
↳ bit of foaming on the top

Aroma: spiced baked goods, hint of campfire wood, notes of coffee & mocha.

Sample (50 mL) sent to lab → [illegible]
↳ SMAROMOAK
↳ MALC
↳ OD31074

ID: OD 31675



TITLE MF2 chromatography

Project No. 12963

From Page No.

Book No.

77

Experiment Begun 1/17/2017

- made a mock smoke tannin feed material by taking ~1.5 L of Mocha Fume 2 commercial oak tannin, adding it to a 5-gal. cornican, and topping it off with Staniel's CTOU smokey byproduct
  Submitted for analysis, Sample ID: OD27811
  Oak aroma and MALC
- purpose: Use Degler's chrom. experiments as a guide to evaluate best-possible chromatography parameters for smoke tannin.

- Methodology: obtain 50 ml FPX-66 Resin, and ~20 L of Smoak material, diluted to ~10 ppm Guaiacol and loaded at a rate of 200 mg Guaiacol per 50 ml of resin
- Expect an adsorption yield on 1st pass between 40% and 70%
- load resin at a rate of ~10 BV/hr or 8 ml/min
- collect in 4-liter increments of the depleted and submit for analysis making sure to record precise volumes, then collect all depleted in a clean cornican. Submit collected depleted for analysis
- Regenerate column by eluting with 2 BV each of the following EtOH soln's: Do not segregate individual BV's of same strength elutions.
  - 20%
  - 40%
  - 60%
  - 80%
  - 95%

  } Submit each for analysis incl. oak aroma and MALC

- then, take depleted and load onto resin again, this time AK collecting every 25L for analysis
- repeat elution procedure

To Page No.

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DATE 1/17/17

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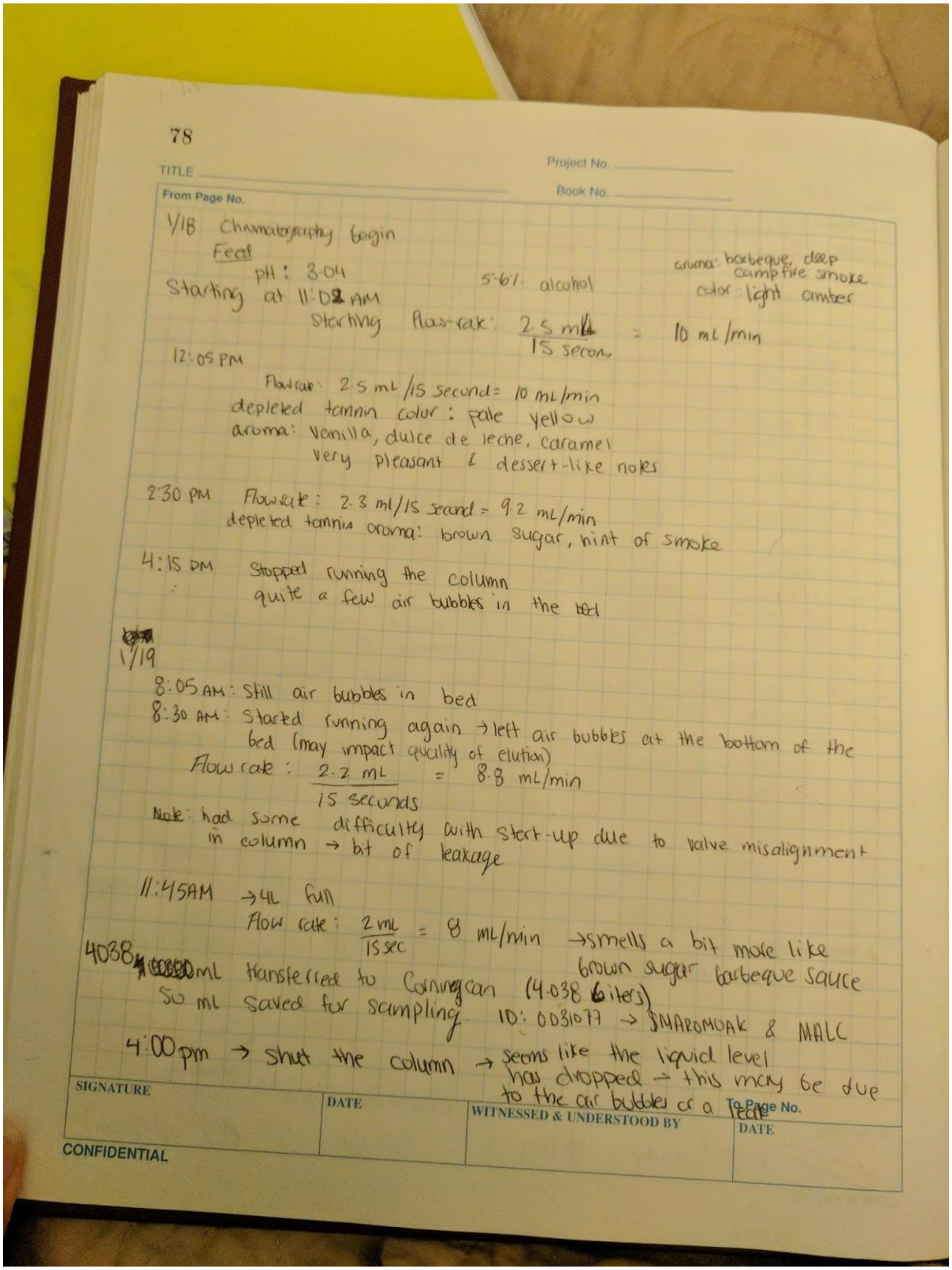

78

TITLE

Project No.

From Page No.

Book No.

1/18 Chromatography begin

Feed

pH : 3.04

5.6% alcohol

aroma: barbeque, deep campfire smoke

color: light amber

Starting at 11:02 AM

Starting flow-rate: $\frac{2.5\ mL}{15\ secon} = 10\ mL/min$

12:05 PM

Flowrate: 2.5 mL /15 second = 10 mL/min

depleted tannin color : pale yellow

aroma: vanilla, dulce de leche, caramel

very pleasant & dessert-like notes

2:30 PM Flowrate: 2.3 ml/15 second = 9.2 mL/min

depleted tannin aroma: brown sugar, hint of smoke

4:15 PM Stopped running the column

quite a few air bubbles in the bed

1/19

8:05 AM: Still air bubbles in bed

8:30 AM: Started running again → left air bubbles at the bottom of the bed (may impact quality of elution)

Flow rate : $\frac{2.2\ mL}{15\ seconds} = 8.8\ mL/min$

Note: had some difficulty with start-up due to valve misalignment in column → bit of leakage

11:45AM → 4L full

Flow rate: $\frac{2\ mL}{15\ sec} = 8\ mL/min$ → smells a bit more like brown sugar barbeque sauce

4038 mL transferred to Corning can (4.038 liters)

50 mL saved for sampling ID: 0031077 → SMAROMOAK & MALC

4:00 pm → shut the column → seems like the liquid level has dropped → this may be due to the air bubbles or a leak

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To Page No.

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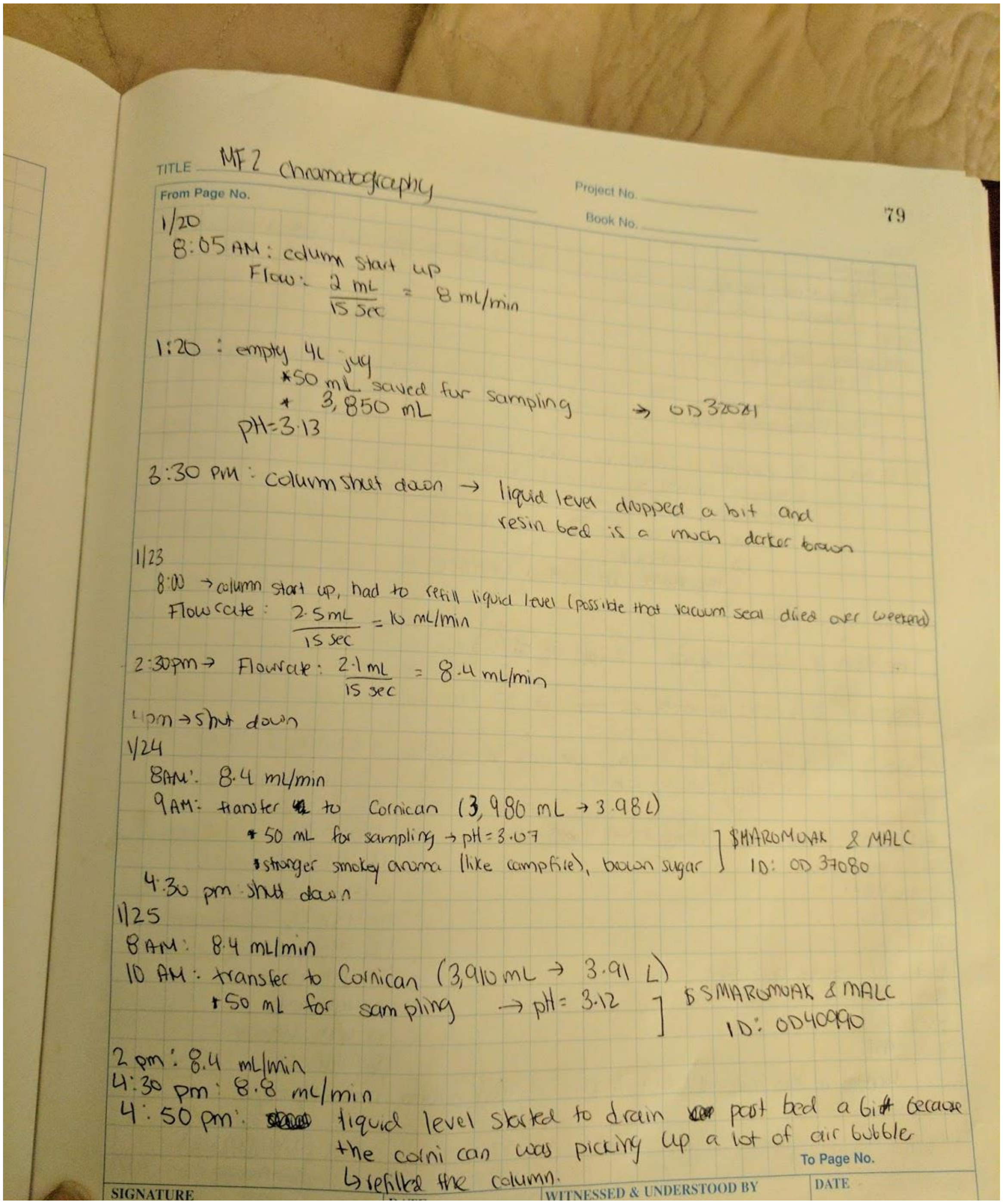

TITLE MF2 Chromatography
From Page No.
Project No.
Book No.
79

1/20
8:05 AM: column start up
Flow: $\frac{2\ mL}{15\ sec} = 8\ mL/min$

1:20 : empty 4L jug
*50 mL saved for sampling → OD32081
* 3,850 mL
pH=3.13

3:30 PM: column shut down → liquid level dropped a bit and resin bed is a much darker brown

1/23
8:00 → column start up, had to refill liquid level (possible that vacuum seal dried over weekend)
Flow rate: $\frac{2.5\ mL}{15\ sec} = 10\ mL/min$

2:30pm → Flowrate: $\frac{2.1\ mL}{15\ sec} = 8.4\ mL/min$

4pm → shut down

1/24
8AM: 8.4 mL/min
9AM: transfer to Cornican (3,980 mL → 3.98L)
* 50 mL for sampling → pH = 3.07
*stronger smokey aroma (like campfire), brown sugar] $HMAROMVAK & MALC ID: OD 37080
4:30 pm shut down

1/25
8 AM: 8.4 mL/min
10 AM: transfer to Cornican (3,910 mL → 3.91 L)
*50 mL for sampling → pH = 3.12] $ SMAROMVAK & MALC ID: OD40990

2 pm: 8.4 mL/min
4:30 pm: 8.8 mL/min
4:50 pm: liquid level started to drain past bed a bit because the corni can was picking up a lot of air bubble
↳ refilled the column.

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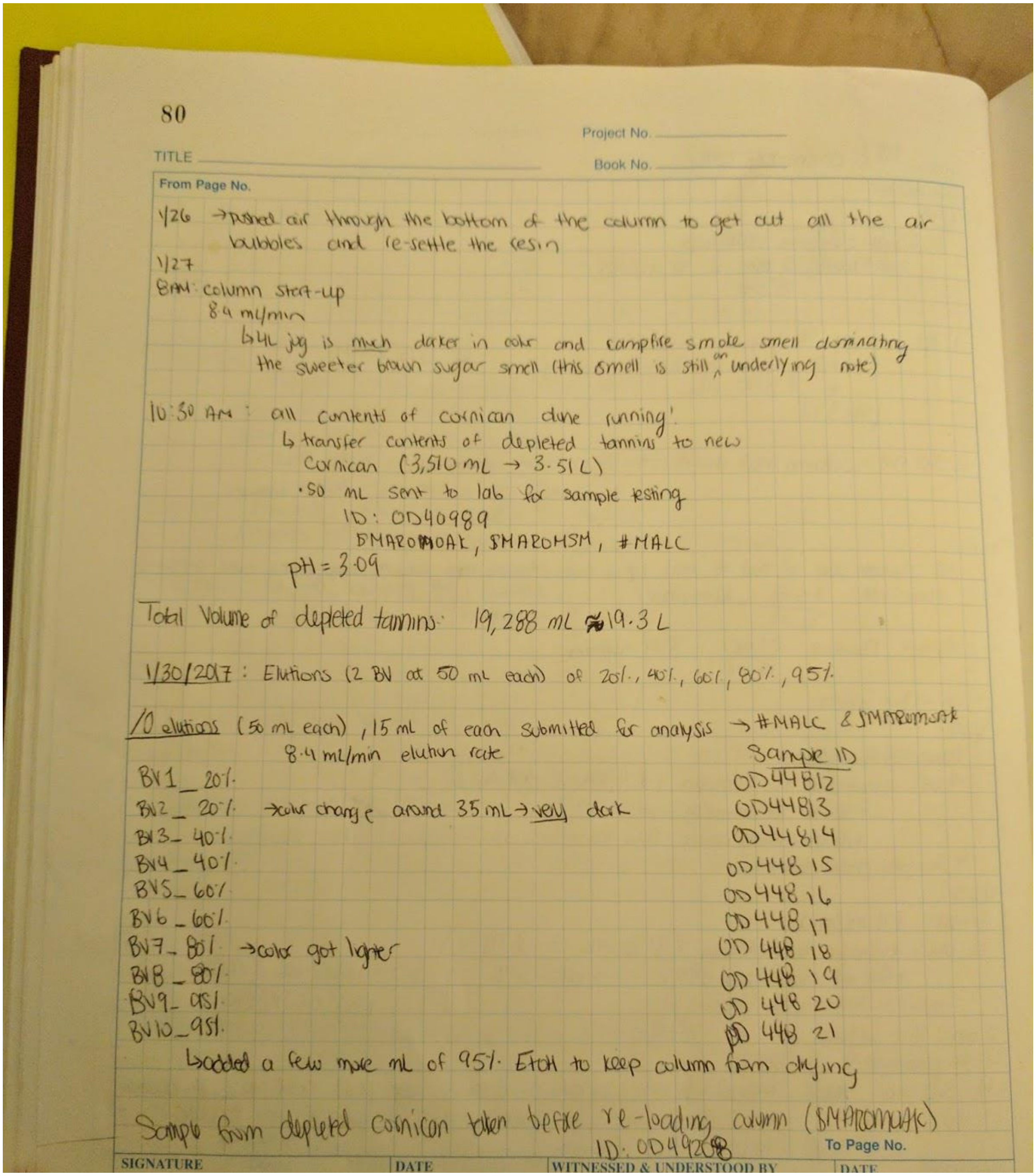

80

TITLE

Project No.

Book No.

From Page No.

1/26 → pushed air through the bottom of the column to get out all the air bubbles and re-settle the resin

1/27

8AM: column start-up

8.4 ml/min

↳ 4L jug is much darker in color and campfire smoke smell dominating the sweeter brown sugar smell (this smell is still an underlying note)

10:30 AM: all contents of cornican done running!

↳ transfer contents of depleted tannins to new cornican (3,510 mL → 3.51 L)

• 50 mL sent to lab for sample testing

ID: OD40989

$MAROMOAK, $MAROHSM, #MALC

pH = 3.09

Total Volume of depleted tannins: 19,288 mL ≈ 19.3 L

1/30/2017: Elutions (2 BV at 50 mL each) of 20%, 40%, 60%, 80%, 95%

10 elutions (50 mL each), 15 mL of each submitted for analysis → #MALC & $MAROMOAK

8.4 mL/min elution rate

		Sample ID
BV1_20%		OD44812
BV2_20%	→ color change around 35 mL → very dark	OD44813
BV3_40%		OD44814
BV4_40%		OD44815
BV5_60%		OD44816
BV6_60%		OD44817
BV7_80%	→ color got lighter	OD44818
BV8_80%		OD44819
BV9_95%		OD44820
BV10_95%		OD44821

↳ added a few more mL of 95% EtOH to keep column from drying

Sample from depleted cornican taken before re-loading column ($MAROMOAK)

ID: OD49208

To Page No.

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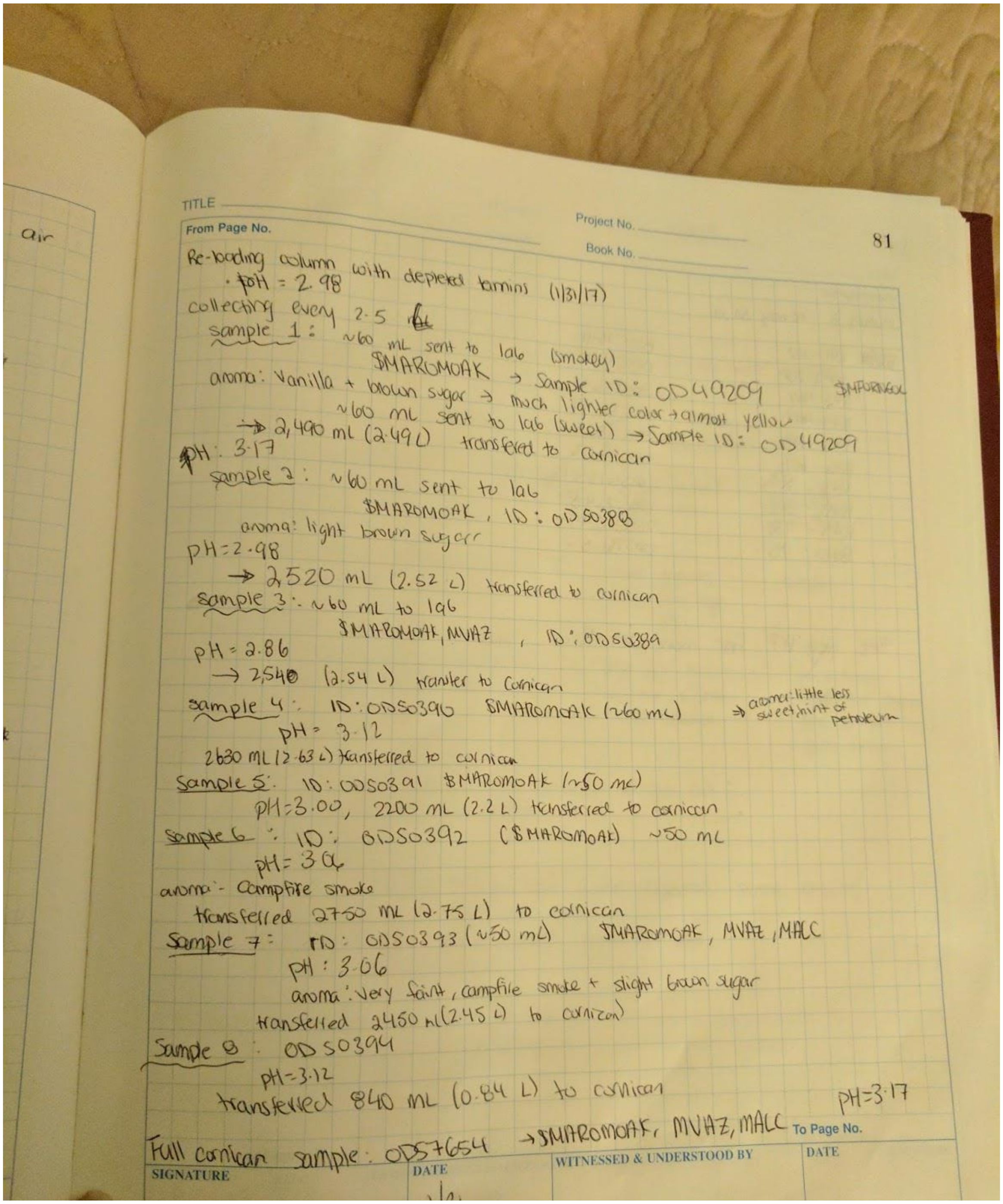

air

TITLE

From Page No.

Project No.

Book No.

81

Re-loading column with depleted tannins (1/31/17)
· ~~pH~~ pH = 2.98
collecting every 2.5 L
sample 1: ~60 mL sent to lab (smokey)
$MAROMOAK → Sample ID: OD49209 $MFORNGOL
aroma: Vanilla + brown sugar → much lighter color → almost yellow
~60 mL sent to lab (sweet) → Sample ID: OD49209
→ 2,490 mL (2.49 L) transfered to cornican
pH: 3.17
sample 2: ~60 mL sent to lab
$MAROMOAK, ID: OD50388
aroma: light brown sugar
pH = 2.98
→ 2,520 mL (2.52 L) transferred to cornican
sample 3: ~60 mL to lab
$MAROMOAK, MVAZ, ID: ODS0389
pH = 2.86
→ 2,540 (2.54 L) transfer to Cornican
sample 4: ID: ODS0390 $MAROMOAK (~60 mL) ⇒ aroma: little less sweet, hint of petroleum
pH = 3.12
2630 mL (2.63 L) transferred to cornican
Sample 5: ID: ODS0391 $MAROMOAK (~50 mL)
pH = 3.00, 2200 mL (2.2 L) transferred to cornican
Sample 6: ID: ODS0392 ($MAROMOAK) ~50 mL
pH = 3.06
aroma - Campfire smoke
transferred 2750 mL (2.75 L) to cornican
Sample 7: ID: ODS0393 (~50 mL) $MAROMOAK, MVAZ, MALC
pH: 3.06
aroma: very faint, campfire smoke + slight brown sugar
transferred 2450 mL (2.45 L) to cornican
Sample 8: OD S0394
pH = 3.12
transferred 840 mL (0.84 L) to cornican
pH = 3.17
Full cornican sample: ODS7654 → $MAROMOAK, MVAZ, MALC

To Page No.

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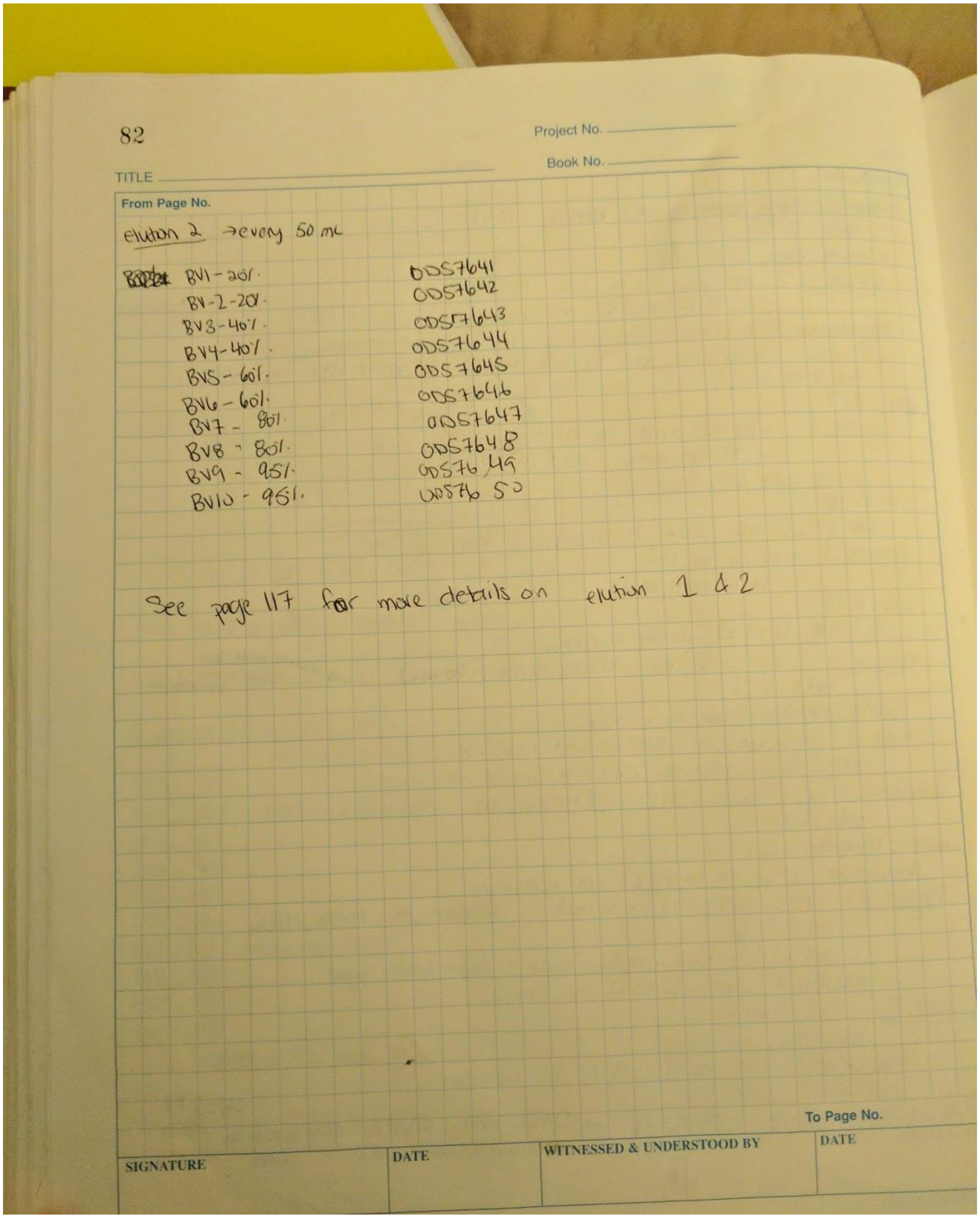


82

Project No.

Book No.

TITLE

From Page No.

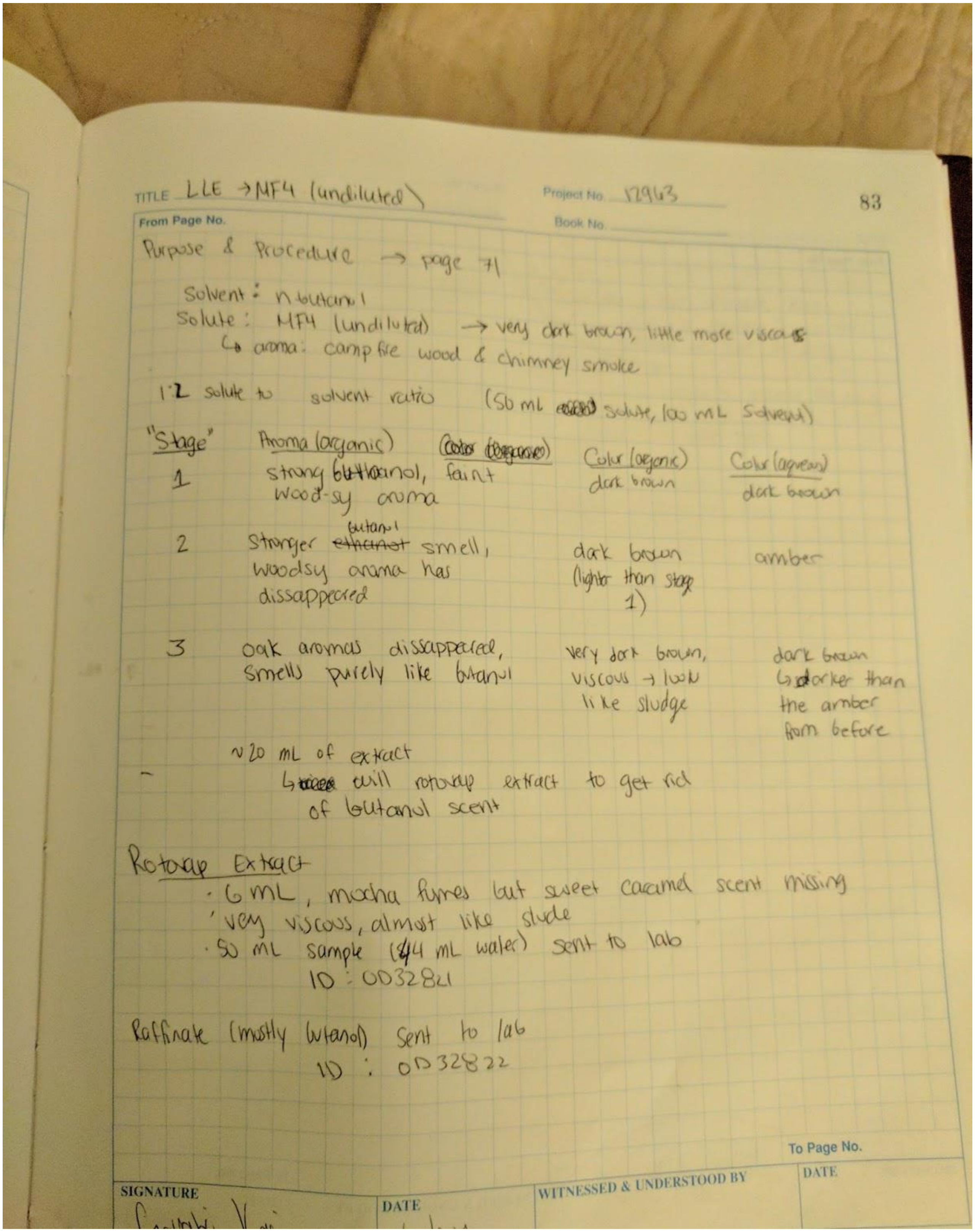
elution 2 →every 50 mL

BV1 - 20%	ODS7641
BV-2-20%	ODS7642
BV3-40%	ODS7643
BV4-40%	ODS7644
BV5 - 60%	ODS7645
BV6 - 60%	ODS7646
BV7 - 80%	ODS7647
BV8 - 80%	ODS7648
BV9 - 95%	ODS7649
BV10 - 95%	ODS7650

See page 117 for more details on elution 1 & 2

To Page No.

SIGNATURE	DATE	WITNESSED & UNDERSTOOD BY	DATE



TITLE LLE → MF4 (undiluted)

Project No. 12963

83

From Page No.

Book No.

Purpose & Procedure → page 71

Solvent: n butanol
Solute: MF4 (undiluted) → very dark brown, little more viscous
↳ aroma: camp fire wood & chimney smoke

1:2 solute to solvent ratio (50 mL solute, 100 mL solvent)

"Stage"	Aroma (organic)	Color (organic)	Color (aqueous)
1	strong butanol, faint wood-sy aroma	dark brown	dark brown
2	Stronger ~~ethanol~~ butanol smell, woodsy aroma has dissappeared	dark brown (lighter than stage 1)	amber
3	oak aromas dissappeared, smells purely like butanol	very dark brown, viscous → look like sludge	dark brown ↳ darker than the amber from before

~20 mL of extract
↳ will rotovap extract to get rid of butanol scent

Rotovap Extract
- 6 mL, mocha fumes but sweet caramel scent missing
- very viscous, almost like slude
- 50 mL sample (44 mL water) sent to lab
  ID: 0D32821

Raffinate (mostly butanol) sent to lab
ID: 0D32822

To Page No.

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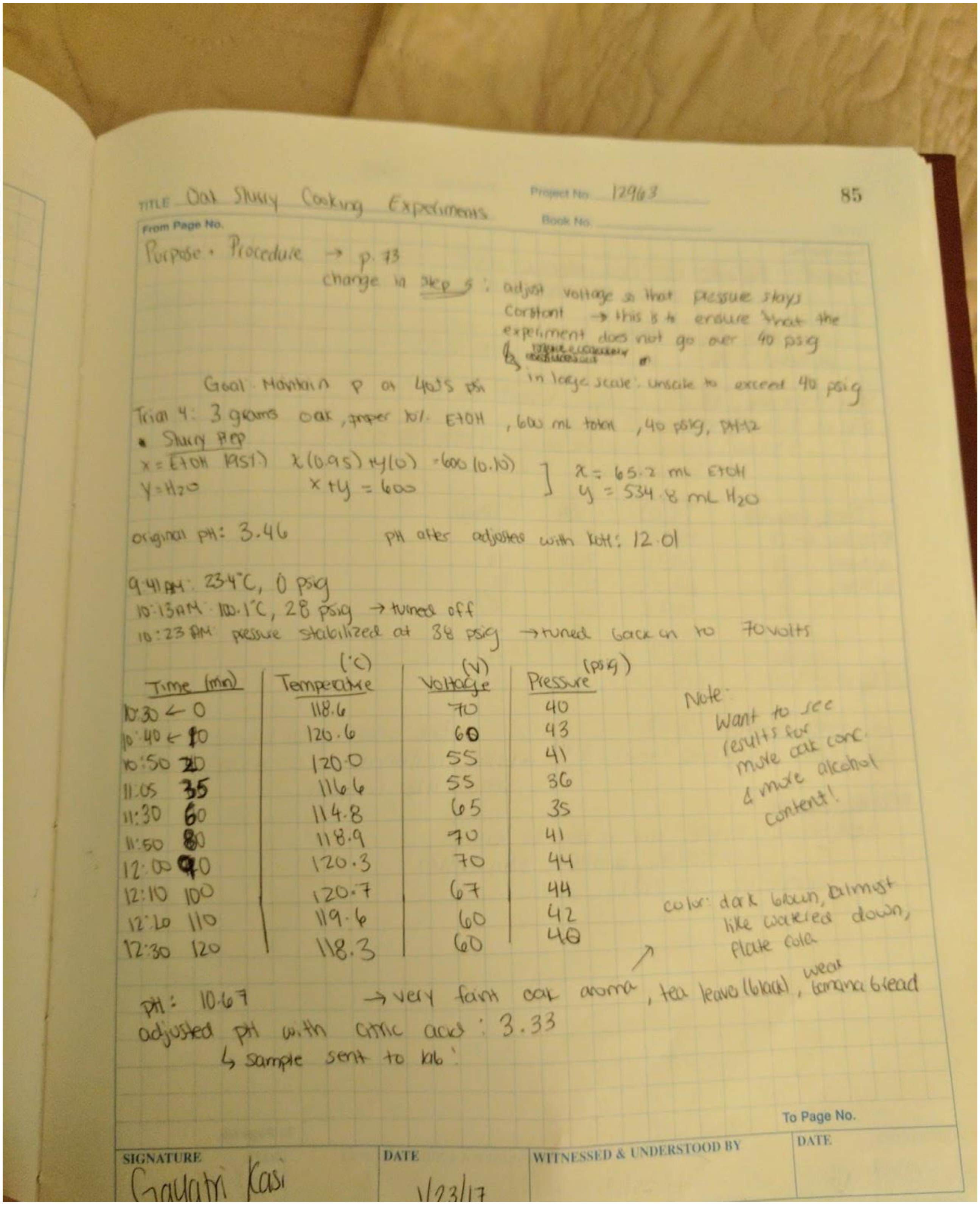

TITLE Oak Slurry Cooking Experiments

Project No. 12963

Book No.

85

From Page No.

Purpose + Procedure → p. 73

change in step 5: adjust voltage so that pressure stays constant → this is to ensure that the experiment does not go over 40 psig

↳ in large scale: unsafe to exceed 40 psig

Goal: Maintain P at 40±5 psi

Trial 4: 3 grams oak, proper 10% EtOH, 600 mL total, 40 psig, pH 12

- Slurry Prep

x = EtOH (95%) x(0.95) + y(0) = 600 (0.10)
y = H₂O x + y = 600

] x = 65.2 mL EtOH
y = 534.8 mL H₂O

original pH: 3.46

pH after adjusted with KOH: 12.01

9:41 AM: 23.4°C, 0 psig
10:13 AM: 100.1°C, 28 psig → turned off
10:23 AM: pressure stabilized at 38 psig → turned back on to 70 volts

Time (min)	Temperature (°C)	Voltage (V)	Pressure (psig)
10:30 ← 0	118.6	70	40
10:40 ← 10	120.6	60	43
10:50 20	120.0	55	41
11:05 35	116.6	55	36
11:30 60	114.8	65	35
11:50 80	118.9	70	41
12:00 90	120.3	70	44
12:10 100	120.7	67	44
12:20 110	119.6	60	42
12:30 120	118.3	60	40

Note:
Want to see results for more oak conc. & more alcohol content!

color: dark brown, almost like watered down, flat cola

pH: 10.67 → very faint oak aroma, tea leaves (black), weak banana bread

adjusted pH with citric acid: 3.33
↳ sample sent to lab.

To Page No.

SIGNATURE Gayatri Kasi

DATE 1/23/17

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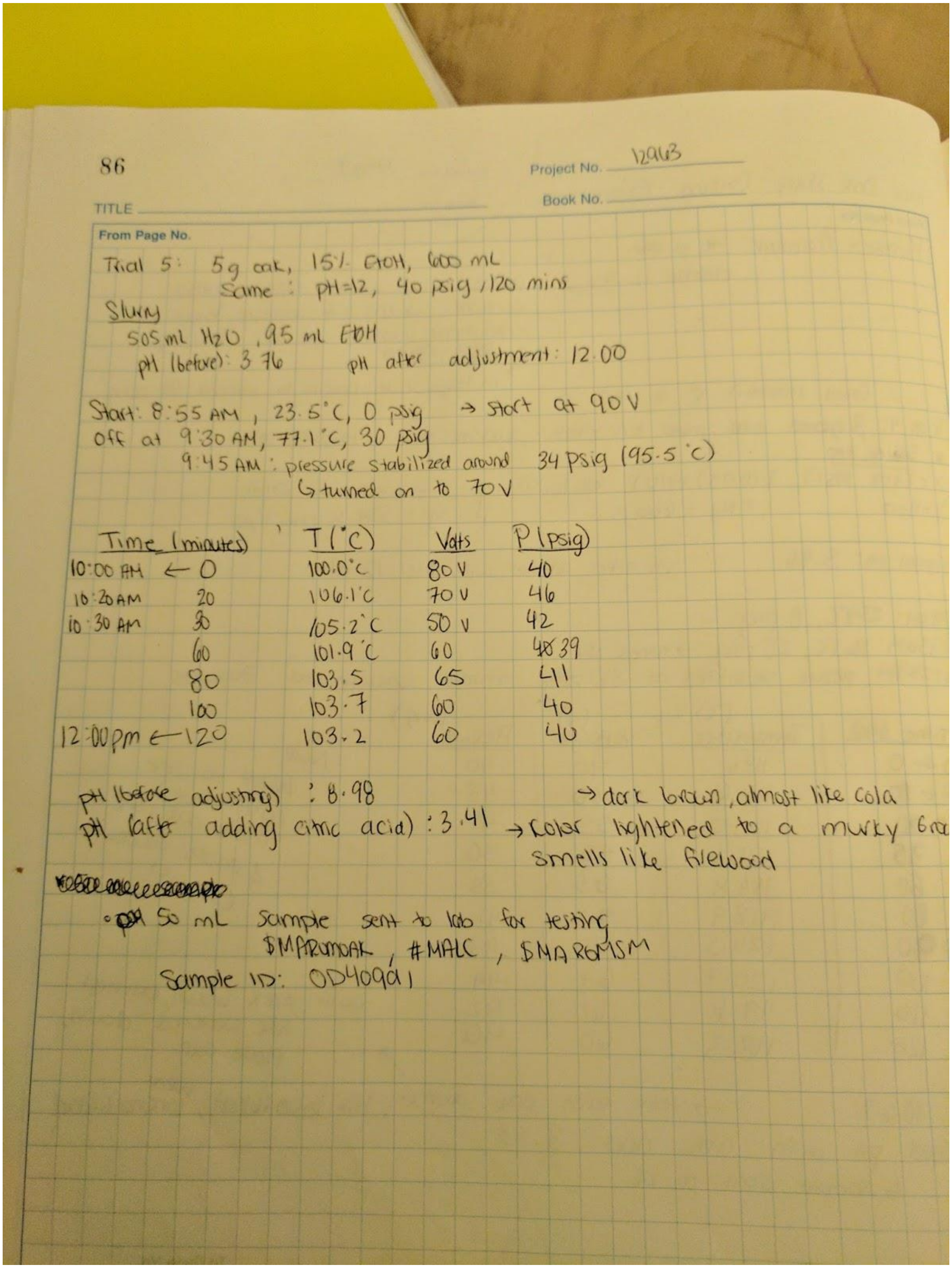


86

Project No. 12963

TITLE

Book No.

From Page No.

Trial 5: 5g oak, 15% EtOH, 600 mL
Same: pH=12, 40 psig /120 mins

Slurry

505 mL H_2O, 95 mL EtOH
pH (before): 3.76 pH after adjustment: 12.00

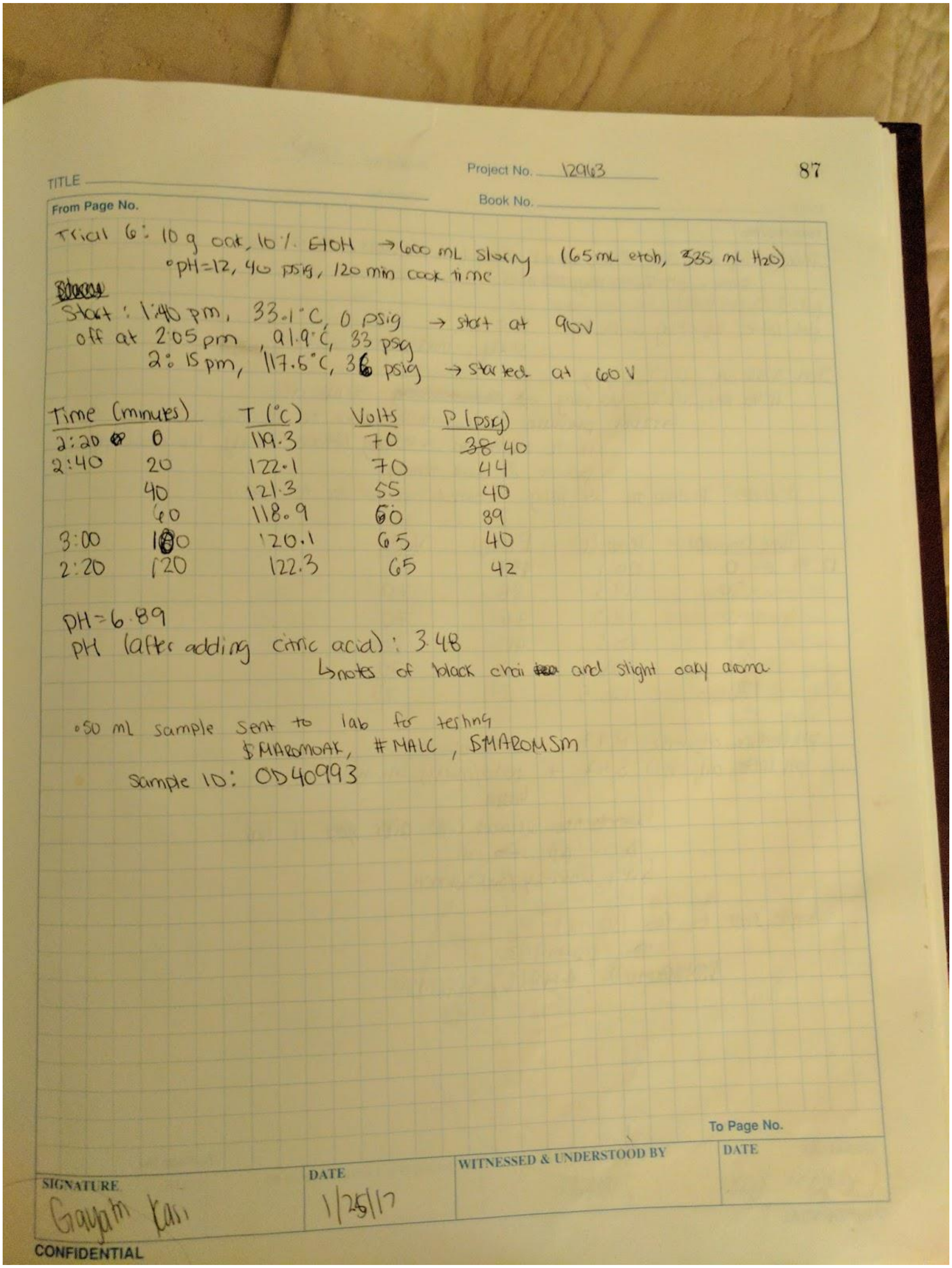
Start: 8:55 AM, 23.5°C, 0 psig → start at 90 V
off at 9:30 AM, 77.1°C, 30 psig
9:45 AM: pressure stabilized around 34 psig (95.5°C)
↳ turned on to 70V

	Time (minutes)	T (°C)	Volts	P (psig)
10:00 AM ←	0	100.0°C	80 V	40
10:20 AM	20	106.1°C	70 V	46
10:30 AM	30	105.2°C	50 V	42
	60	101.9°C	60	~~40~~ 39
	80	103.5	65	41
	100	103.7	60	40
12:00pm ←	120	103.2	60	40

pH (before adjusting): 8.98 → dark brown, almost like cola
pH (after adding citric acid): 3.41 → color lightened to a murky Gra
smells like firewood

- 50 mL sample sent to lab for testing
  $MAROMOAK, #MALC, $MAROMSM

Sample ID: OD409a1



TITLE

Project No. 12963

87

From Page No.

Book No.

Trial 6: 10 g oak, 10% EtOH → 600 mL slurry (65 mL etoh, 335 mL H_2O)
∘ pH=12, 40 psig, 120 min cook time

Start: 1:40 pm, 33.1°C, 0 psig → start at 90V
off at 2:05 pm, 91.9°C, 33 psig
2:15 pm, 117.5°C, 36 psig → started at 60 V

Time	(minutes)	T (°C)	Volts	P (psig)
2:20	0	119.3	70	~~38~~ 40
2:40	20	122.1	70	44
	40	121.3	55	40
	60	118.9	60	39
3:00	100	120.1	65	40
2:20	120	122.3	65	42

pH=6.89
pH (after adding citric acid): 3.48
↳ notes of black chai and slight oaky aroma

∘ 50 mL sample sent to lab for testing
$MAROMOAK, #MALC, $MAROMSM
Sample ID: OD40993

To Page No.

SIGNATURE	DATE	WITNESSED & UNDERSTOOD BY	DATE
Gayatri Kari	1/25/17		

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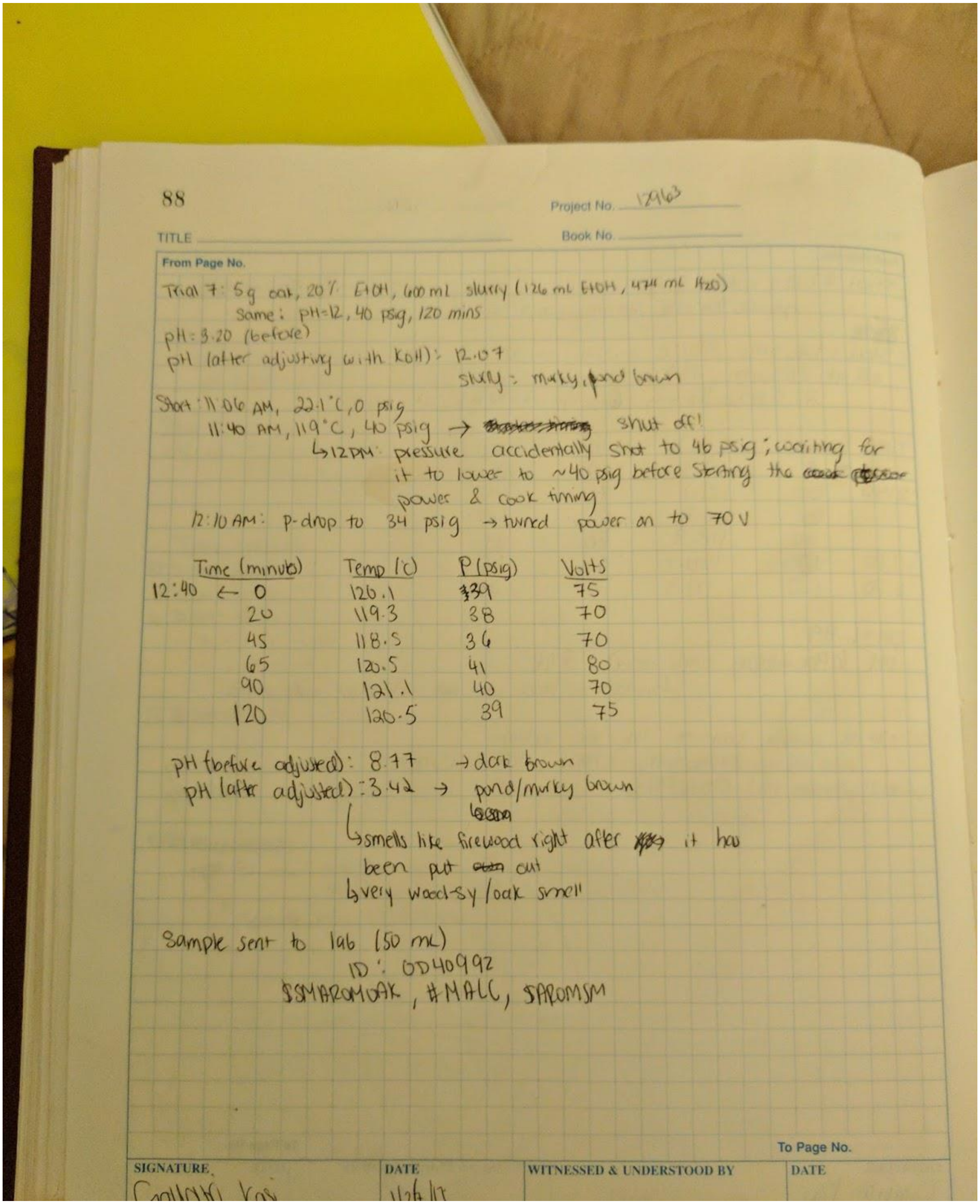

88

Project No. 12963

TITLE

Book No.

From Page No.

Trial 7: 5g oak, 20% EtOH, 600 mL slurry (126 mL EtOH, 474 mL H2O)
Same: pH=12, 40 psig, 120 mins
pH: 3.20 (before)
pH (after adjusting with KOH): 12.07
slurry: murky, pond brown

Start: 11:06 AM, 22.1°C, 0 psig
11:40 AM, 119°C, 40 psig → shut off!
↳ 12PM: pressure accidentally shot to 46 psig; waiting for it to lower to ~40 psig before starting the power & cook timing
12:10 AM: p-drop to 34 psig → turned power on to 70 V

	Time (minutes)	Temp (°C)	P (psig)	Volts
12:40 ←	0	120.1	39	75
	20	119.3	38	70
	45	118.5	36	70
	65	120.5	41	80
	90	121.1	40	70
	120	120.5	39	75

pH (before adjusted): 8.77 → dark brown
pH (after adjusted): 3.42 → pond/murky brown
↳ smells like firewood right after it has been put out
↳ very wood-sy / oak smell

Sample sent to lab (50 mL)
ID: OD40992
$SMAROMOAK, #MALC, $AROMSM

To Page No.

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DATE 1/26/17

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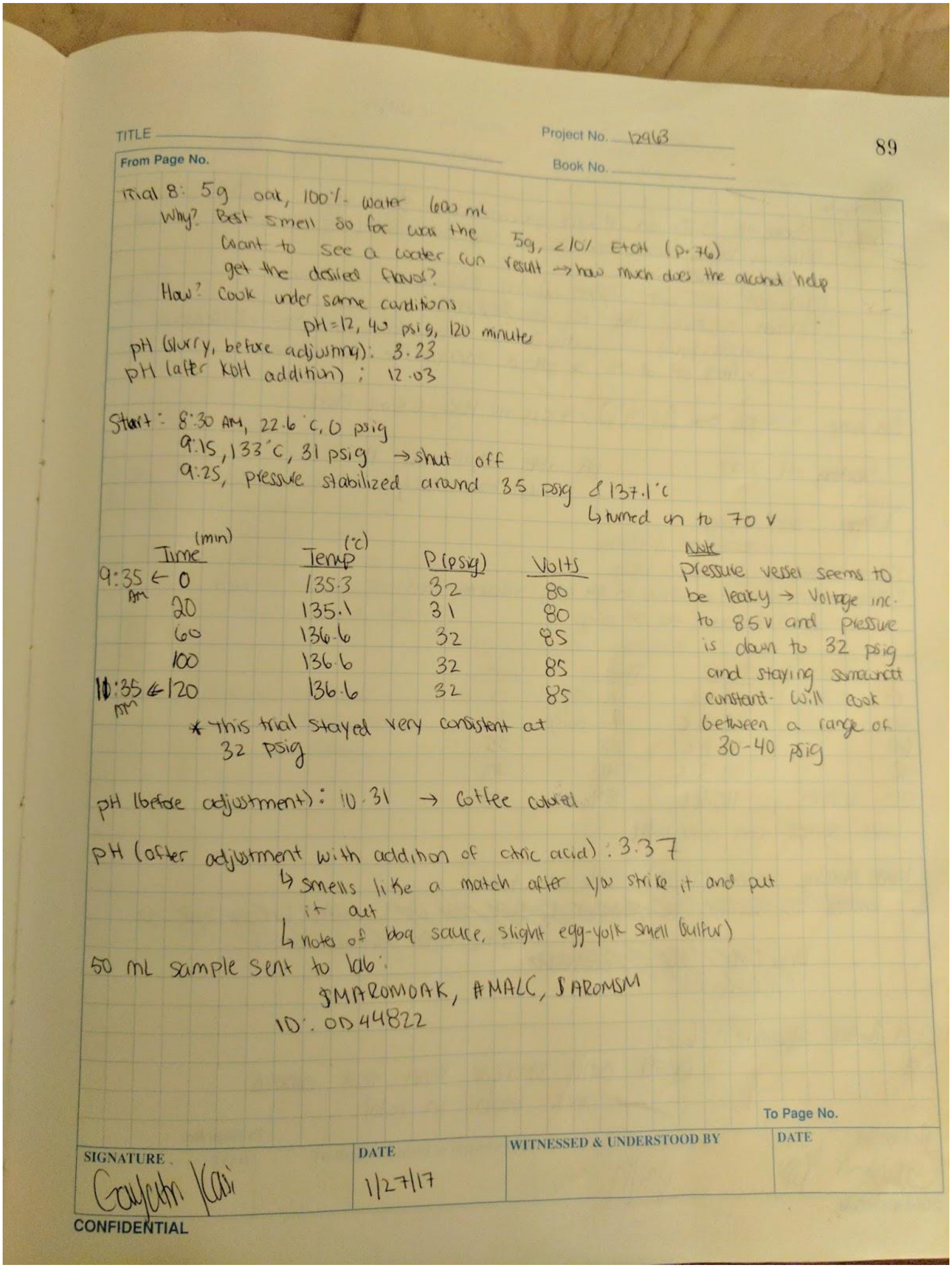

TITLE

Project No. 12963

89

From Page No.

Book No.

Trial 8: 5g oak, 100% water 600 mL
Why? Best smell so far was the 5g, <10% EtOH (p. 76)
Want to see a water run result → how much does the alcohol help get the desired flavor?
How? Cook under same conditions
pH=12, 40 psig, 120 minute
pH (slurry, before adjusting): 3.23
pH (after KOH addition) ; 12.03

Start: 8:30 AM, 22.6 °C, 0 psig
9:15, 133 °C, 31 psig → shut off
9:25, pressure stabilized around 35 psig & 137.1 °C
↳ turned on to 70 V

	Time (min)	Temp (°C)	P (psig)	Volts
9:35 AM ←	0	135.3	32	80
	20	135.1	31	80
	60	136.6	32	85
	100	136.6	32	85
10:35 AM ←	120	136.6	32	85

* this trial stayed very consistent at 32 psig

Note
Pressure vessel seems to be leaky → Voltage inc. to 85 V and pressure is down to 32 psig and staying somewhat constant. Will cook between a range of 30-40 psig

pH (before adjustment): 10.31 → coffee colored

pH (after adjustment with addition of citric acid): 3.37
↳ smells like a match after you strike it and put it out
↳ notes of bbq sauce, slight egg-yolk smell (sulfur)

50 mL sample sent to lab:
$MAROMOAK, #MALC, $AROMSM
ID: OD44822

To Page No.

SIGNATURE	DATE	WITNESSED & UNDERSTOOD BY	DATE
Gaylethn Kai	1/27/17		

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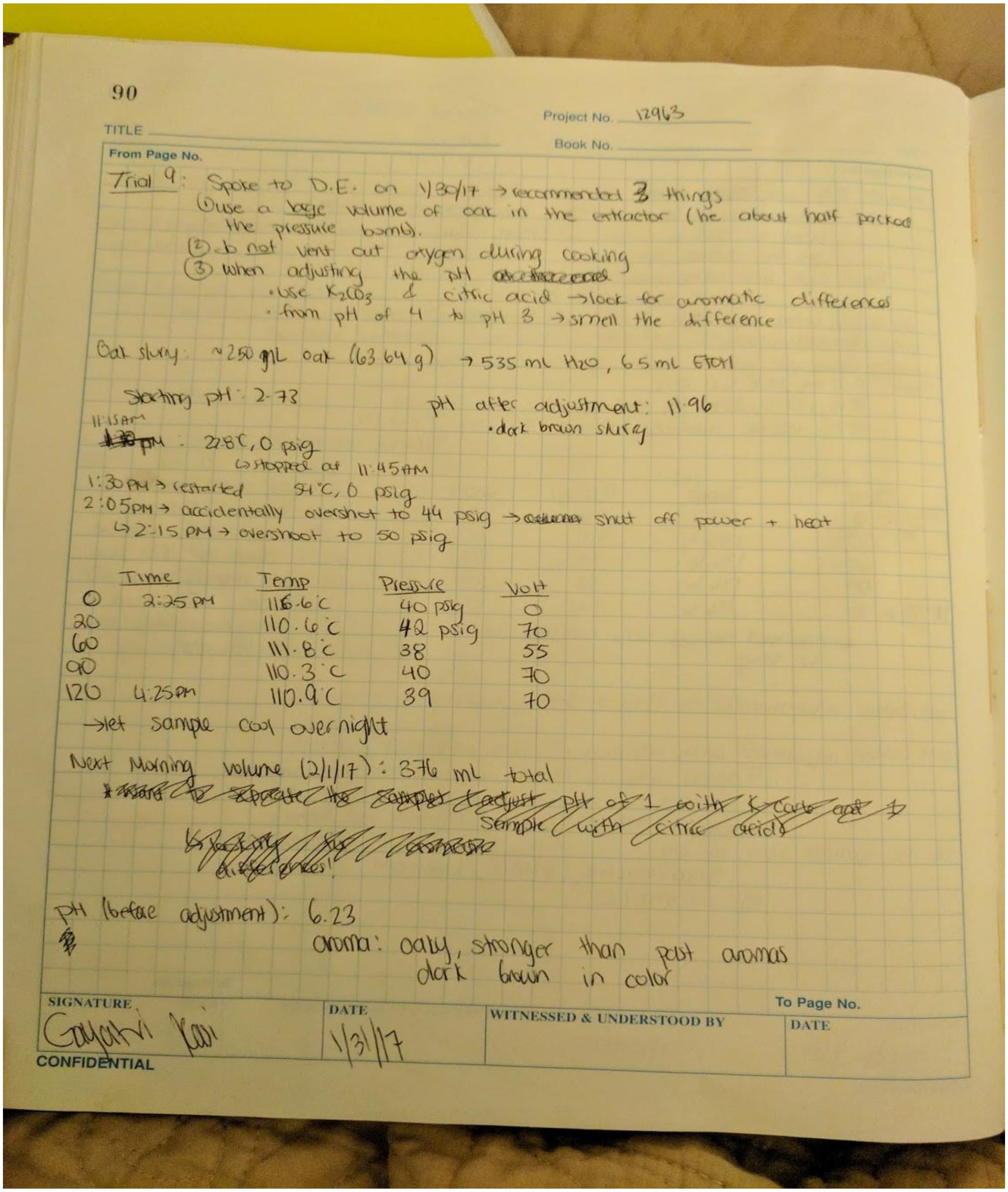

90

TITLE

Project No. 12963

Book No.

From Page No.

Trial 9: Spoke to D.E. on 1/30/17 → recommended 3 things
① use a large volume of oak in the extractor (he about half packed the pressure bomb).
② do not vent out oxygen during cooking
③ when adjusting the pH ~~[illegible]~~
- use K_2CO_3 & citric acid → look for aromatic differences
- from pH of 4 to pH 3 → smell the difference

Oak slurry: ~250 mL oak (63.64 g) → 535 mL H_2O, 65 mL EtOH

Starting pH: 2.73

pH after adjustment: 11.96
- dark brown slurry

11:15AM ~~1:30~~ PM: 22.8°C, 0 psig
↳ stopped at 11:45AM
1:30 PM → restarted 54°C, 0 psig
2:05 PM → accidentally overshot to 44 psig → ~~[illegible]~~ shut off power + heat
↳ 2:15 PM → overshoot to 50 psig

	Time	Temp	Pressure	Volt
0	2:25 PM	116.6°C	40 psig	0
20		110.6°C	42 psig	70
60		111.8°C	38	55
90		110.3°C	40	70
120	4:25 PM	110.9°C	39	70

→ let sample cool overnight

Next Morning volume (2/1/17): 376 mL total
~~* want to separate the samples & adjust pH of 1 with K_2CO_3 and 1 sample with citric acid~~
~~& [illegible] the [illegible] differences!~~

pH (before adjustment): 6.23
aroma: oaky, stronger than past aromas
dark brown in color

SIGNATURE Gayatri Rai

DATE 1/31/17

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To Page No.

DATE

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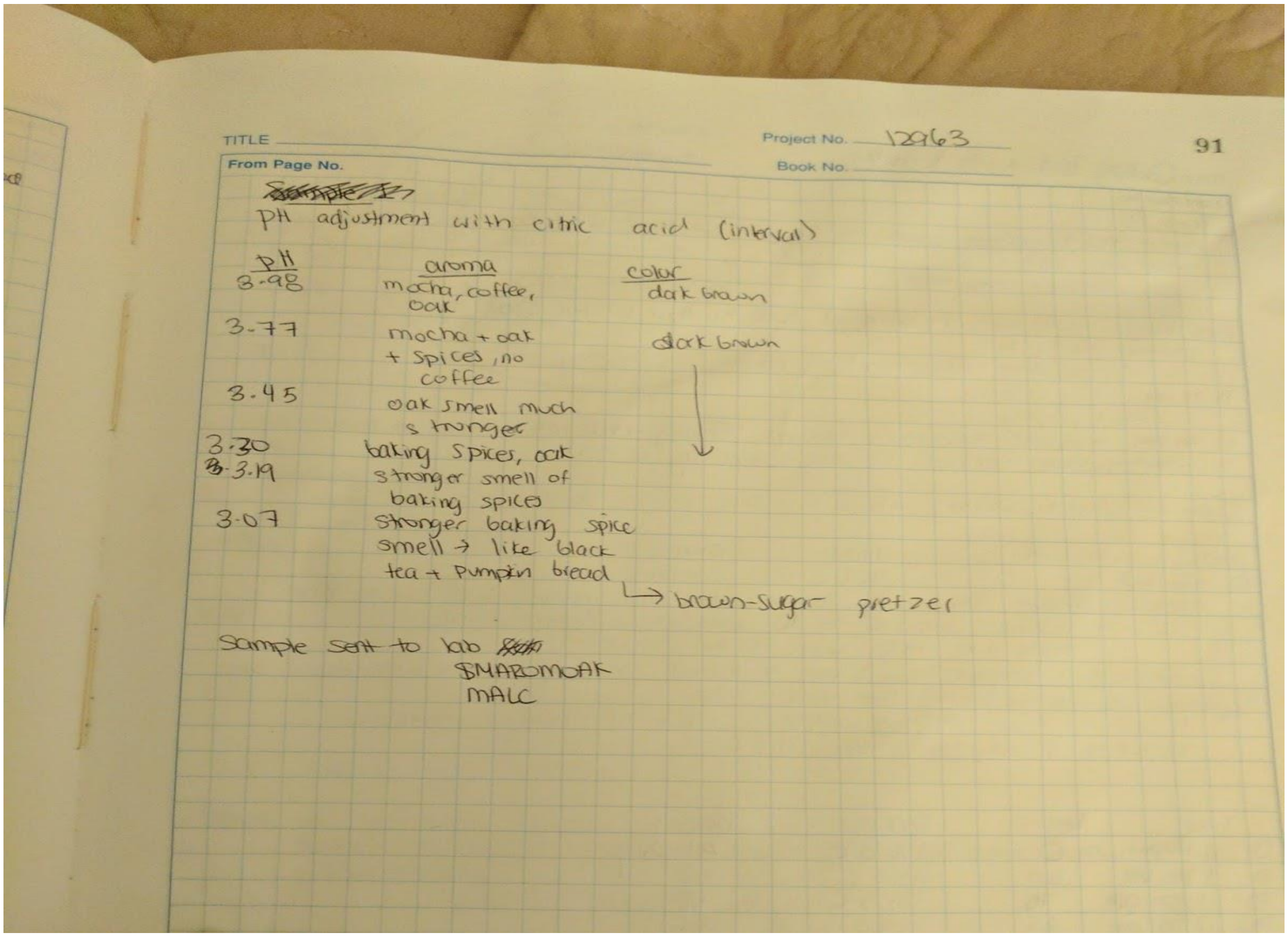


TITLE

From Page No.

Project No. 12963

Book No.

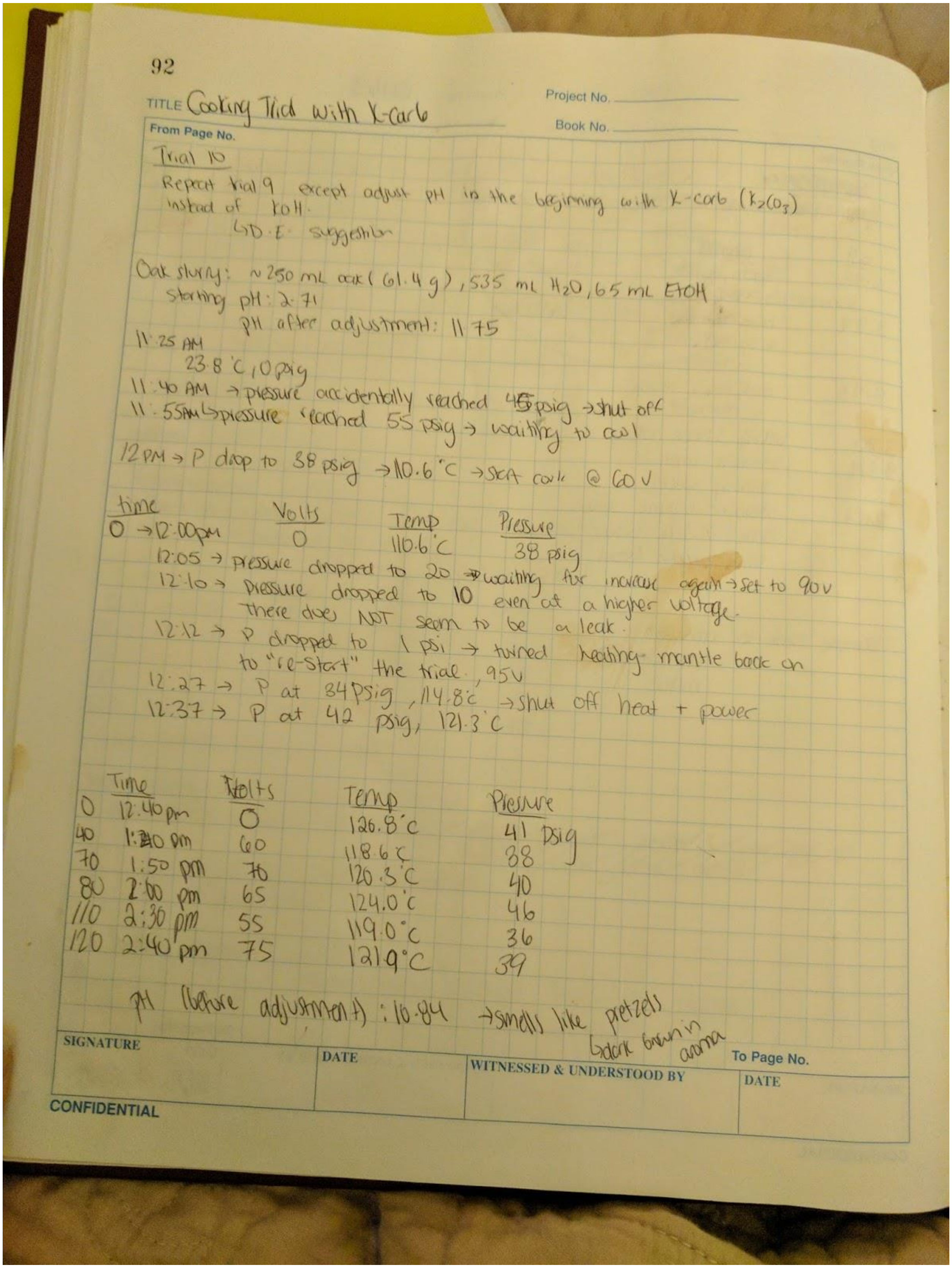
91

~~Sample 1~~

pH adjustment with citric acid (interval)

pH	aroma	color
3.98	mocha, coffee, oak	dak brown
3.77	mocha + oak + spices, no coffee	dark brown
3.45	oak smell much stronger	↓
3.30	baking spices, oak	
3.19	stronger smell of baking spices	
3.07	stronger baking spice smell → like black tea + pumpkin bread ↳ brown-sugar pretzel	

Sample sent to lab ~~[illegible]~~
SMAROMOAK
MALC



92

TITLE Cooking Trial with K-carb

Project No.

Book No.

From Page No.

Trial 10

Repeat trial 9 except adjust pH in the beginning with K-carb (K_2CO_3) instead of KOH.
↳D.E. suggestion

Oak slurry: ~250 mL oak (61.4 g), 535 mL H_2O, 65 mL EtOH
starting pH: 2.71
pH after adjustment: 11.75

11:25 AM
23.8 °C, 0 psig
11:40 AM → pressure accidentally reached 45 psig → shut off
11:55 AM ↳ pressure reached 55 psig → waiting to cool

12 PM → P drop to 38 psig → 110.6 °C → Set coils @ 60 V

time	Volts	Temp	Pressure
0 → 12:00pm	0	110.6 °C	38 psig

12:05 → pressure dropped to 20 → waiting for increase again → set to 90 V
12:10 → Pressure dropped to 10 even at a higher voltage.
There does NOT seem to be a leak.
12:12 → P dropped to 1 psi → turned heating mantle back on to "re-start" the trial, 95 V
12:27 → P at 34 psig, 114.8 °C → shut off heat + power
12:37 → P at 42 psig, 121.3 °C

	Time	Volts	Temp	Pressure
0	12:40 pm	0	126.8 °C	41 psig
40	1:20 pm	60	118.6 °C	38
70	1:50 pm	70	120.3 °C	40
80	2:00 pm	65	124.0 °C	46
110	2:30 pm	55	119.0 °C	36
120	2:40 pm	75	121.9 °C	39

pH (before adjustment): 10.84 → smells like pretzels
↳dark brown in aroma

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To Page No.

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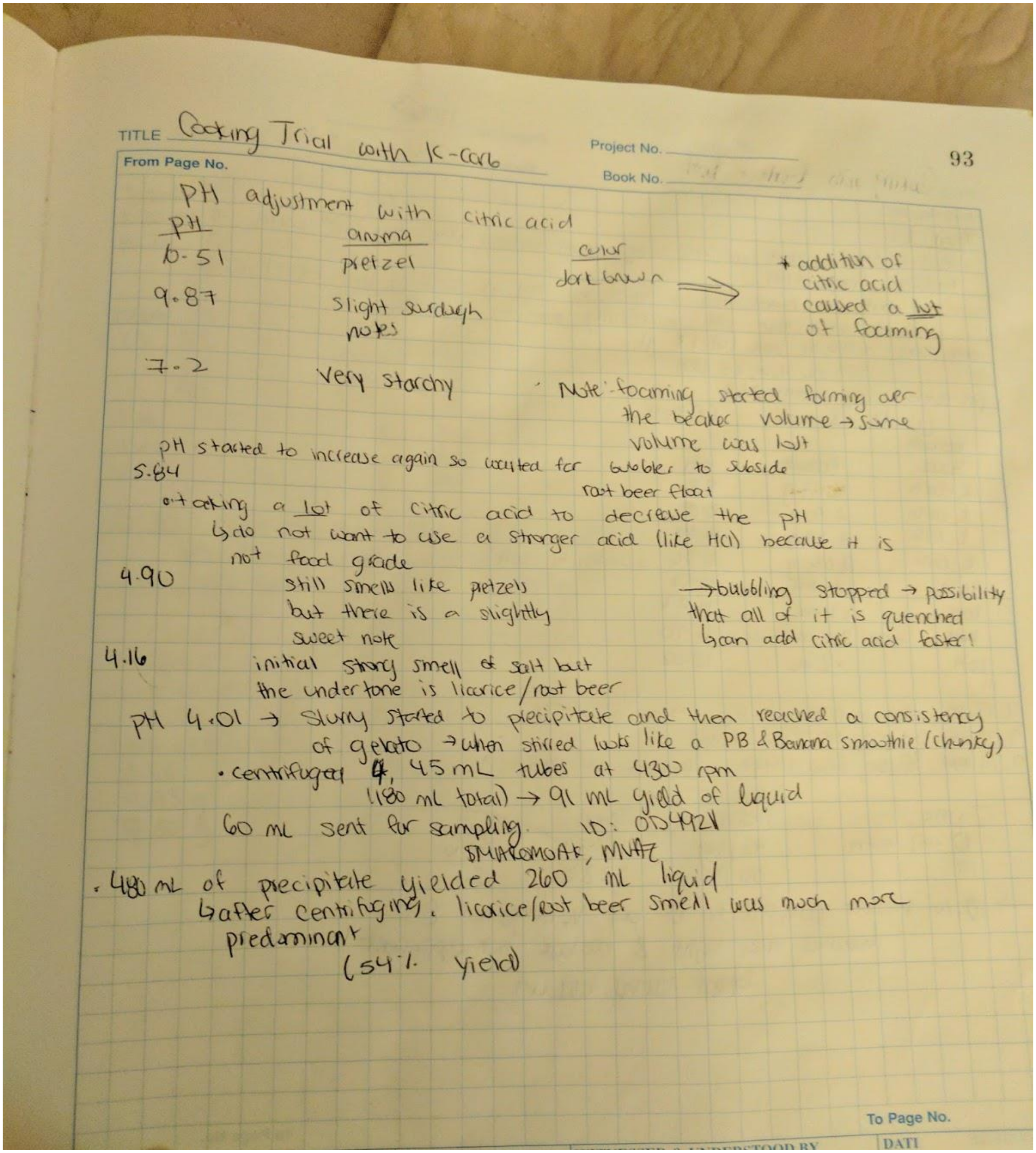


TITLE Cooking Trial with K-carb

From Page No.

Project No.

Book No.

93

PH adjustment with citric acid

pH	aroma	color
10.51	pretzel	dark brown ⟹
9.87	slight sourdough notes	
7.2	very starchy	

* addition of citric acid caused a lot of foaming

Note: foaming started forming over the beaker volume → some volume was lost

pH started to increase again so waited for bubbles to subside

5.84 root beer float

- taking a lot of citric acid to decrease the pH
  - ↳ do not want to use a stronger acid (like HCl) because it is not food grade

4.90 still smells like pretzels but there is a slightly sweet note

→ bubbling stopped → possibility that all of it is quenched
↳ can add citric acid faster!

4.16 initial strong smell of salt but the undertone is licorice/root beer

pH 4.01 → slurry started to precipitate and then reached a consistency of gelato → when stirred looks like a PB & Banana smoothie (chunky)

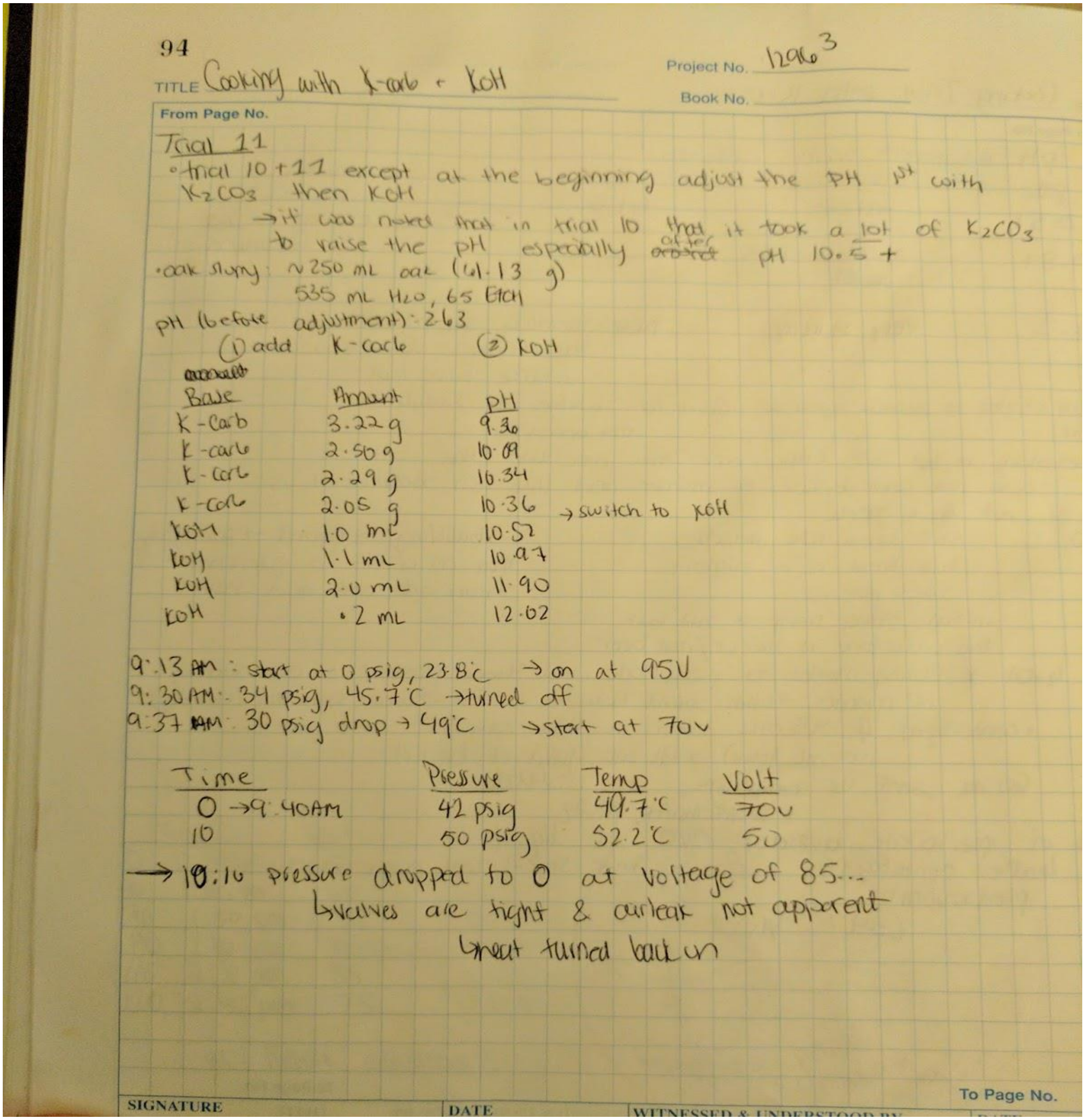
- centrifuged 4, 45 mL tubes at 4300 rpm (180 mL total) → 91 mL yield of liquid

60 mL sent for sampling. ID: OD4921
SMIAROMOAK, MVAE

- 480 mL of precipitate yielded 260 mL liquid
  - ↳ after centrifuging. licorice/root beer smell was much more predominant

(54% yield)

To Page No.



94

TITLE Cooking with K-carb + KOH

Project No. 1296^{3}

Book No.

From Page No.

Trial 11

- trial 10 + 11 except at the beginning adjust the PH 1st with K_2CO_3 then KOH
  → it was noted that in trial 10 that it took a lot of K_2CO_3 to raise the pH especially ~~around~~ after pH 10.5 +
- oak slurry: ~250 mL oak (61.13 g)
  535 mL H_2O, 65 EtOH

pH (before adjustment): 2.63

① add K-carb ② KOH

Base	Amount	pH
K-Carb	3.22 g	9.36
K-carb	2.50 g	10.09
K-carb	2.29 g	10.34
K-carb	2.05 g	10.36 → switch to KOH
KOH	1.0 mL	10.52
KOH	1.1 mL	10.97
KOH	2.0 mL	11.90
KOH	.2 mL	12.02

9:13 AM: start at 0 psig, 23.8°C → on at 95V
9:30 AM: 34 psig, 45.7°C → turned off
9:37 AM: 30 psig drop → 49°C → start at 70v

Time	Pressure	Temp	Volt
0 → 9:40AM	42 psig	49.7°C	70v
10	50 psig	52.2°C	50

→ 10:10 pressure dropped to 0 at voltage of 85...
↳ valves are tight & airleak not apparent
↳ heat turned back on

To Page No.

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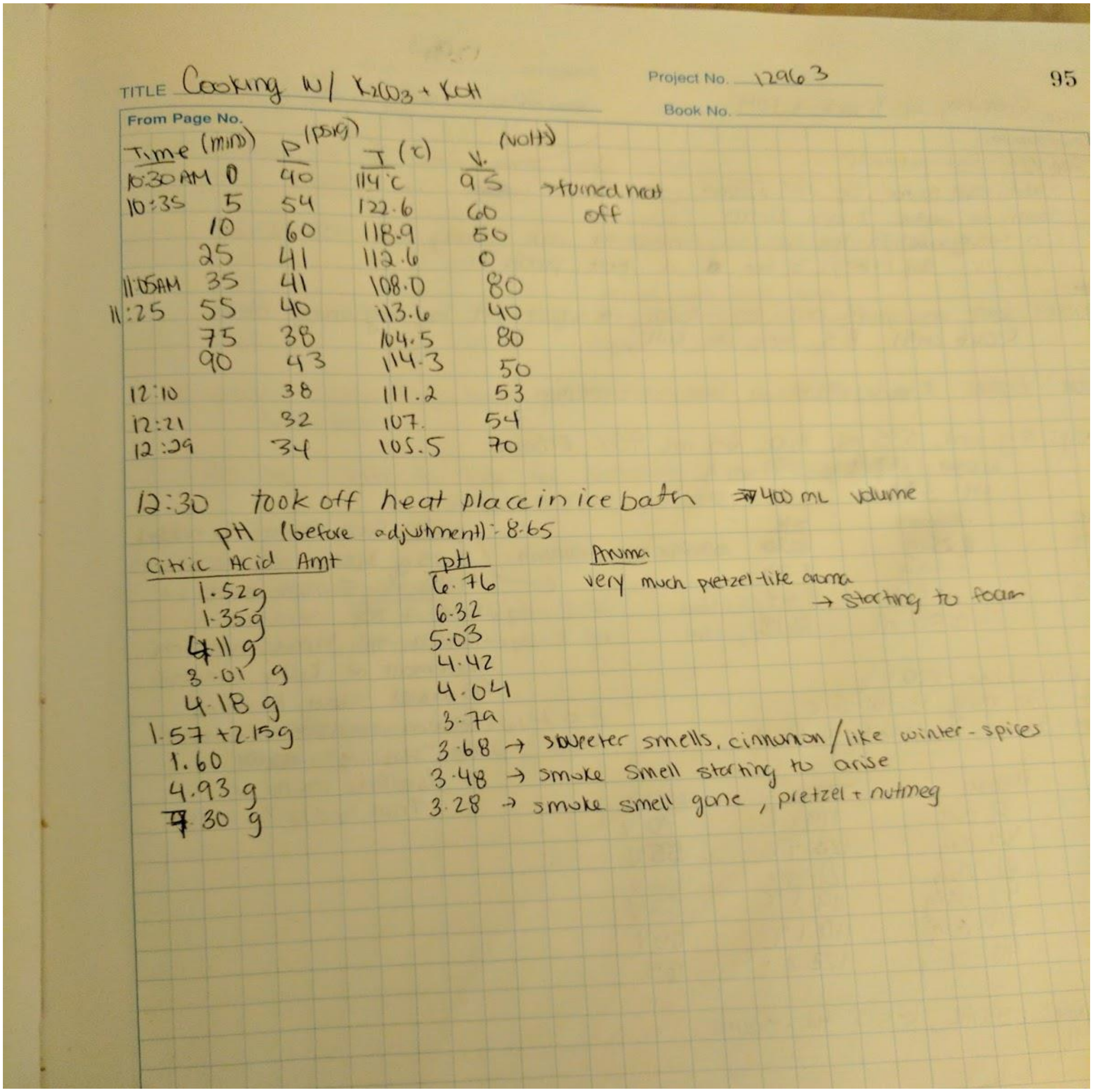


TITLE Cooking w/ K_2CO_3 + KOH

Project No. 12963

Book No.

95

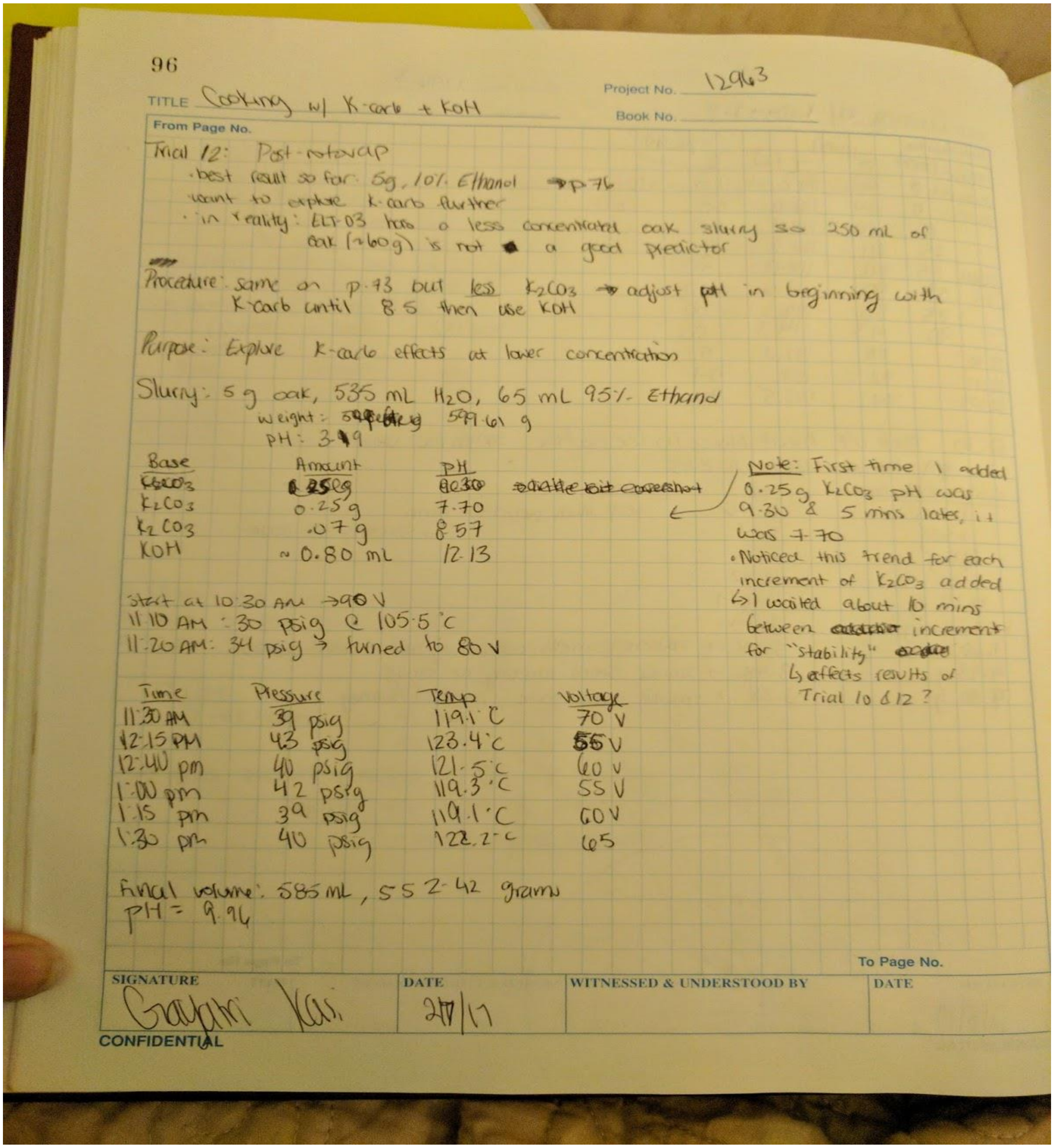
From Page No.

Time	(min)	P (psig)	T (°C)	V. (volts)	
10:30 AM	0	40	114 °C	95	→ turned heat off
10:35	5	54	122.6	60	
	10	60	118.9	50	
	25	41	112.6	0	
11:05AM	35	41	108.0	80	
11:25	55	40	113.6	40	
	75	38	104.5	80	
	90	43	114.3	50	
12:10		38	111.2	53	
12:21		32	107.	54	
12:29		34	105.5	70	

12:30 took off heat place in ice bath ⇒ 400 mL volume

pH (before adjustment) - 8.65

Citric Acid Amt	pH	Aroma
1.52g	6.76	very much pretzel-like aroma → starting to foam
1.35g	6.32	
4.11 g	5.03	
3.01 g	4.42	
4.18 g	4.04	
1.57 + 2.15g	3.79	
1.60	3.68	→ sweeter smells, cinnamon/like winter-spices
4.93 g	3.48	→ smoke smell starting to arise
7.30 g	3.28	→ smoke smell gone, pretzel + nutmeg



96

TITLE Cooking w/ K-carb + KOH

Project No. 12963

Book No.

From Page No.

Trial 12: Post-rotovap
- best result so far: 5g, 10% Ethanol → p.76
- want to explore K-carb further
- in reality: ELT-03 has a less concentrated oak slurry so 250 mL of oak (~60g) is not a good predictor

Procedure: same on p.43 but <u>less</u> K_2CO_3 → adjust pH in beginning with K-carb until 8.5 then use KOH

Purpose: Explore K-carb effects at lower concentration

Slurry: 5 g oak, 535 mL H_2O, 65 mL 95% Ethanol
weight: ~~[illegible]~~ 599.61 g
pH: 3.[illegible]9

Base	Amount	pH	
~~K_2CO_3~~	~~0.25g~~	~~9.30~~	~~[illegible]~~
K_2CO_3	0.25 g	7.70	
K_2CO_3	.07 g	8.57	
KOH	~ 0.80 mL	12.13	

<u>Note:</u> First time I added 0.25g K_2CO_3 pH was 9.30 & 5 mins later, it was 7.70
- Noticed this trend for each increment of K_2CO_3 added
↳ I waited about 10 mins between ~~[illegible]~~ increments for "stability" ~~[illegible]~~
↳ affects results of Trial 10 & 12?

start at 10:30 AM → 90 V
11:10 AM: 30 psig @ 105.5 °C
11:20 AM: 34 psig → turned to 80 V

Time	Pressure	Temp	Voltage
11:30 AM	39 psig	119.1 °C	70 V
12:15 PM	43 psig	123.4 °C	55 V
12:40 pm	40 psig	121.5 °C	60 V
1:00 pm	42 psig	119.3 °C	55 V
1:15 pm	39 psig	119.1 °C	60 V
1:30 pm	40 psig	122.2 °C	65

final volume: 585 mL, 552.42 grams
pH = 9.96

To Page No.

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Gayatri Vasi	2/7/17		

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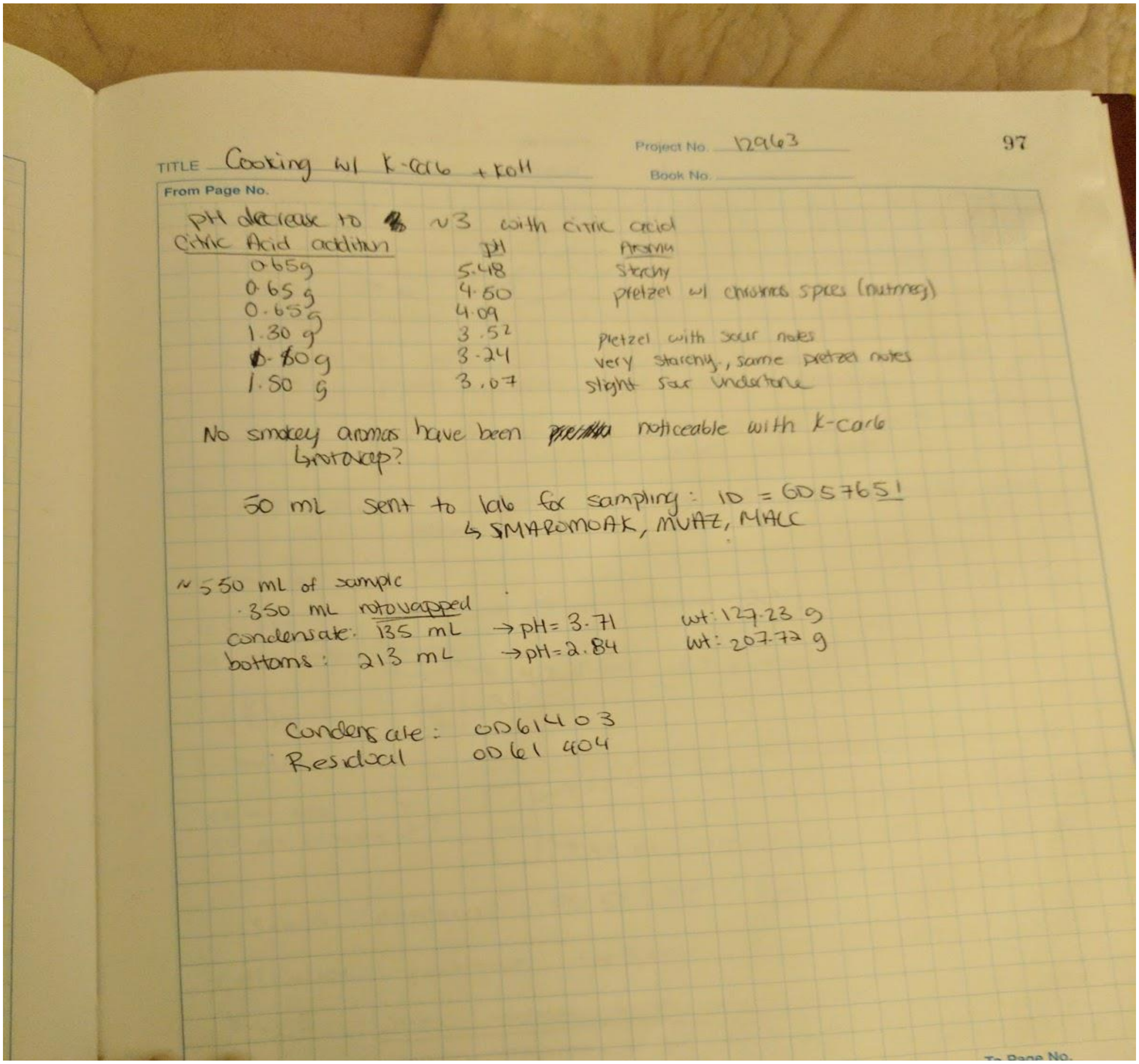


TITLE Cooking w/ K-carb + KOH

Project No. 12963

Book No.

97

From Page No.

pH decrease to ~3 with citric acid

Citric Acid addition	pH	Aroma
0.65g	5.48	Starchy
0.65 g	4.60	pretzel w/ christmas spices (nutmeg)
0.65 g	4.09	
1.30 g	3.52	pretzel with sour notes
0.80 g	3.24	very starchy, same pretzel notes
1.50 g	3.07	slight sour undertone

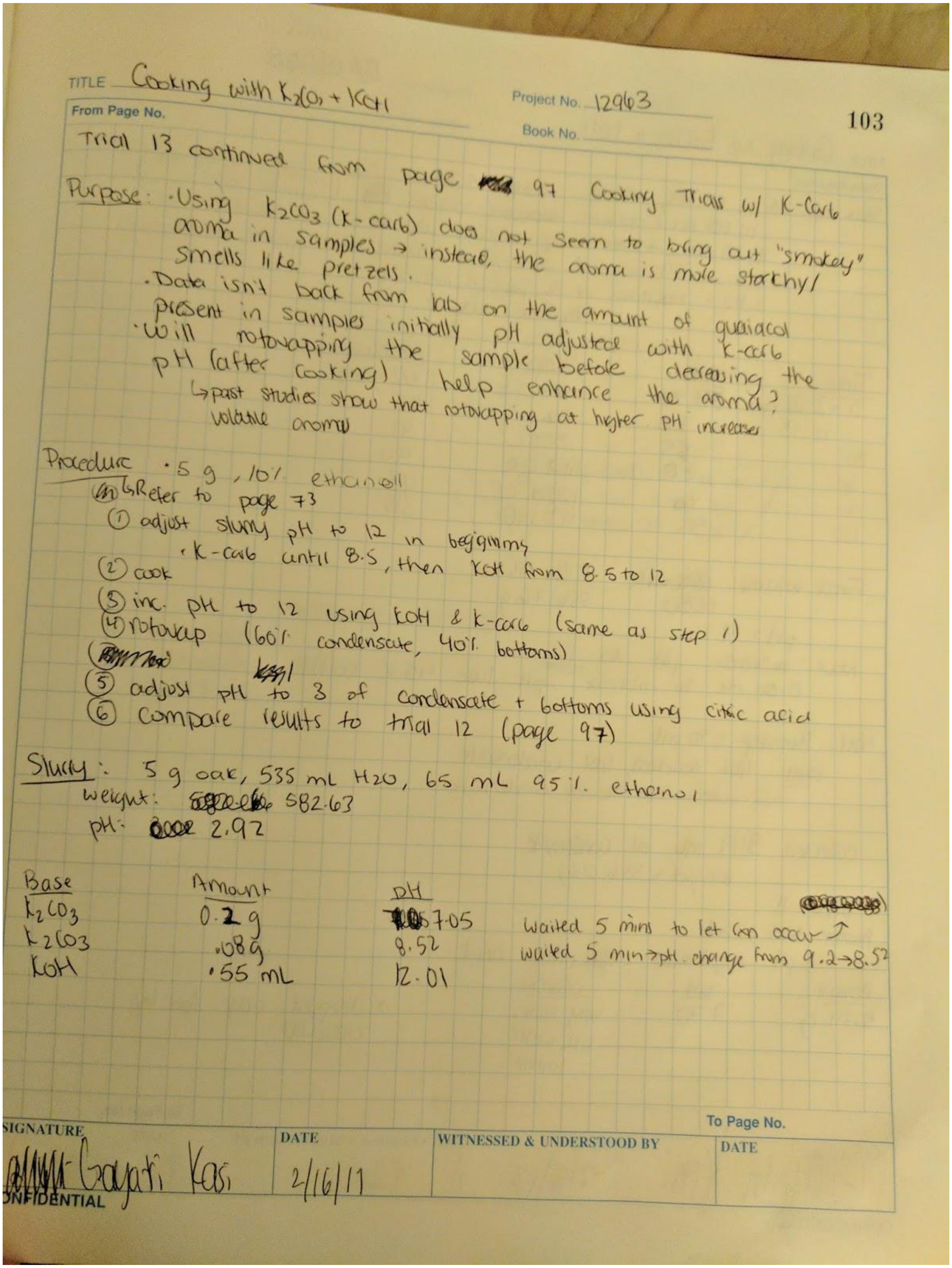
No smokey aromas have been noticeable with K-carb
↳ rotovap?

50 mL sent to lab for sampling: ID = GD57651
↳ SMAROMOAK, MVAZ, MACC

~550 mL of sample
· 350 mL rotovapped
condensate: 135 mL → pH = 3.71 wt: 127.23 g
bottoms: 213 mL → pH = 2.84 wt: 207.72 g

Condensate: OD61403
Residual OD61404

To Page No.



TITLE Cooking with K_2CO_3 + KOH

Project No. 12963

From Page No.

Book No.

103

Trial 13 continued from page 97 Cooking Trials w/ K-Carb

Purpose:
- Using K_2CO_3 (K-carb) does not seem to bring out "smokey" aroma in samples → instead, the aroma is more starchy/ smells like pretzels.
- Data isn't back from lab on the amount of guaiacol present in samples initially pH adjusted with K-carb
- Will rotovapping the sample before decreasing the pH (after cooking) help enhance the aroma?
  - ↳ past studies show that rotovapping at higher pH increases volatile aroma

Procedure
- 5 g, 10% ethanol
  - (in) ↳ Refer to page 73

① adjust slurry pH to 12 in beginning
- K-carb until 8.5, then KOH from 8.5 to 12

② cook

③ inc. pH to 12 using KOH & K-carb (same as step 1)

④ rotovap (60% condensate, 40% bottoms)

⑤ adjust pH to 3 of condensate + bottoms using citric acid

⑥ compare results to trial 12 (page 97)

Slurry: 5 g oak, 535 mL H_2O, 65 mL 95% ethanol

weight: 582.63

pH: 2.92

Base	Amount	pH	
K_2CO_3	0.2 g	7.05	waited 5 mins to let rxn occur ↗
K_2CO_3	.08 g	8.52	waited 5 min → pH change from 9.2 → 8.52
KOH	.55 mL	12.01	

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Gayatri Kasi	2/16/11		

To Page No.

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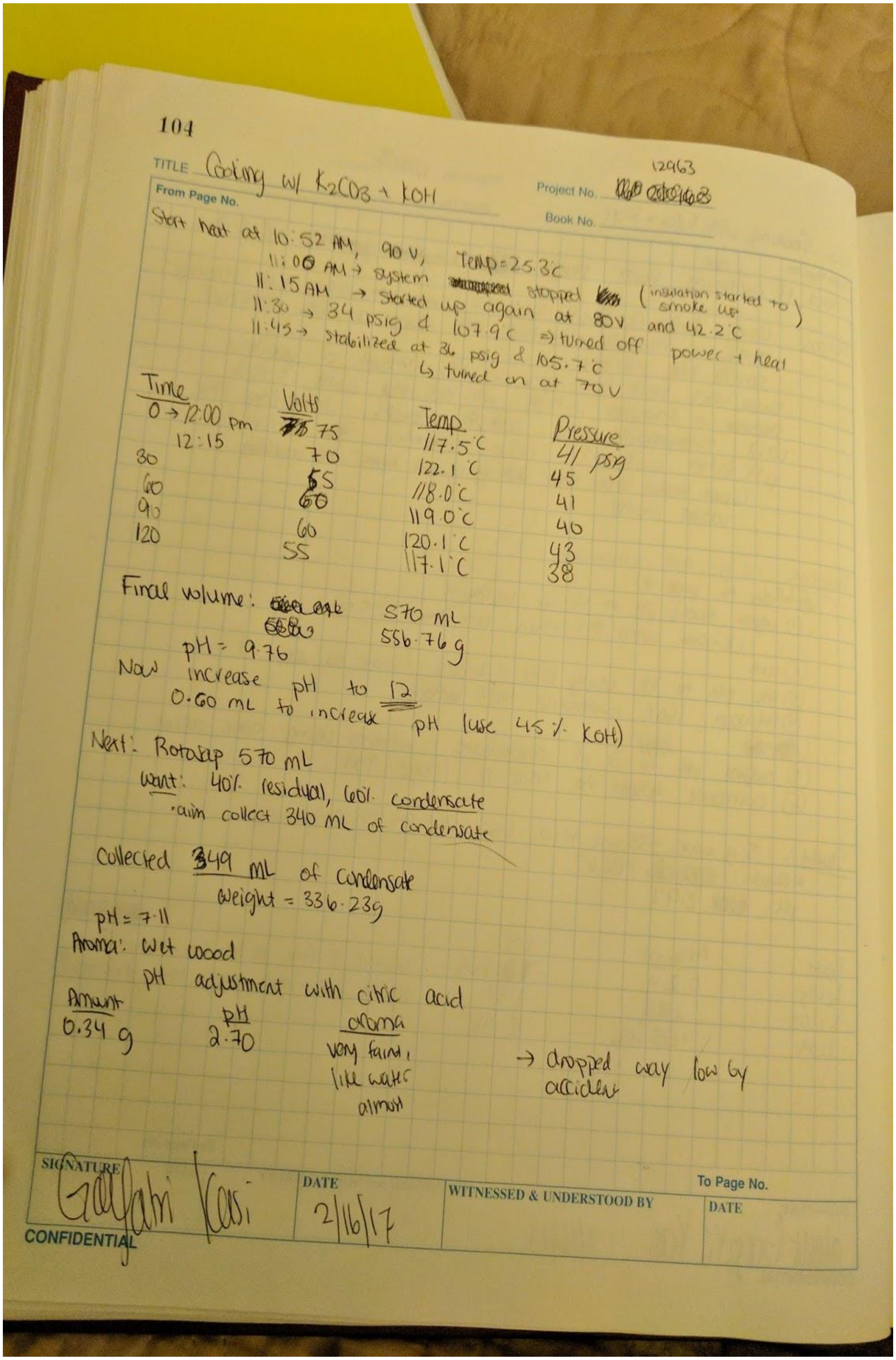

104

TITLE Cooking w/ K_2CO_3 + KOH

Project No. ~~[illegible]~~ 12963

From Page No.

Book No.

Start heat at 10:52 AM, 90 V, Temp = 25.3°C
11:00 AM → system ~~[illegible]~~ stopped ~~[illegible]~~ (insulation started to smoke up)
11:15 AM → Started up again at 80V and 42.2°C
11:30 → 34 psig & 107.9°C ⇒ turned off power + heat
11:45 → stabilized at 36 psig & 105.7°C
↳ turned on at 70V

Time	Volts	Temp	Pressure
0 → 12:00 pm	~~75~~ 75	117.5°C	41 psig
12:15	70	122.1°C	45
30	55	118.0°C	41
60	60	119.0°C	40
90	60	120.1°C	43
120	55	117.1°C	38

Final volume: ~~[illegible]~~ 570 mL
~~[illegible]~~ 556.76 g

pH = 9.76

Now increase pH to 12
0.60 mL to increase pH (use 45% KOH)

Next: Rotavap 570 mL
Want: 40% residual, 60% condensate
• aim collect 340 mL of condensate

Collected 349 mL of condensate
weight = 336.23g

pH = 7.11
Aroma: Wet wood

pH adjustment with citric acid

Amount	pH	aroma	
0.34 g	2.70	very faint, like water almost	→ dropped way low by accident

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To Page No.

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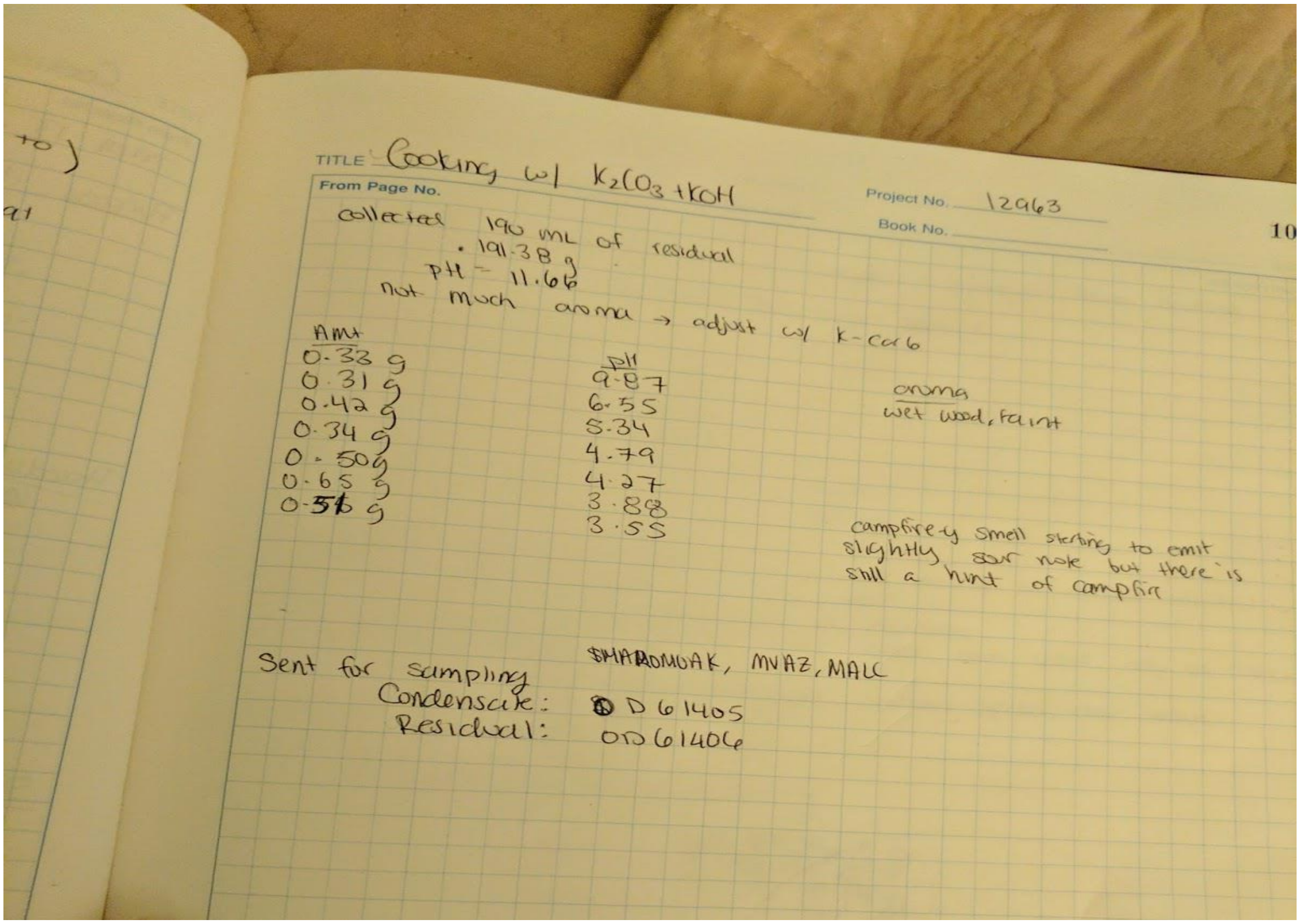


TITLE Cooking w/ K_2CO_3 + KOH

From Page No.

Project No. 12963

Book No.

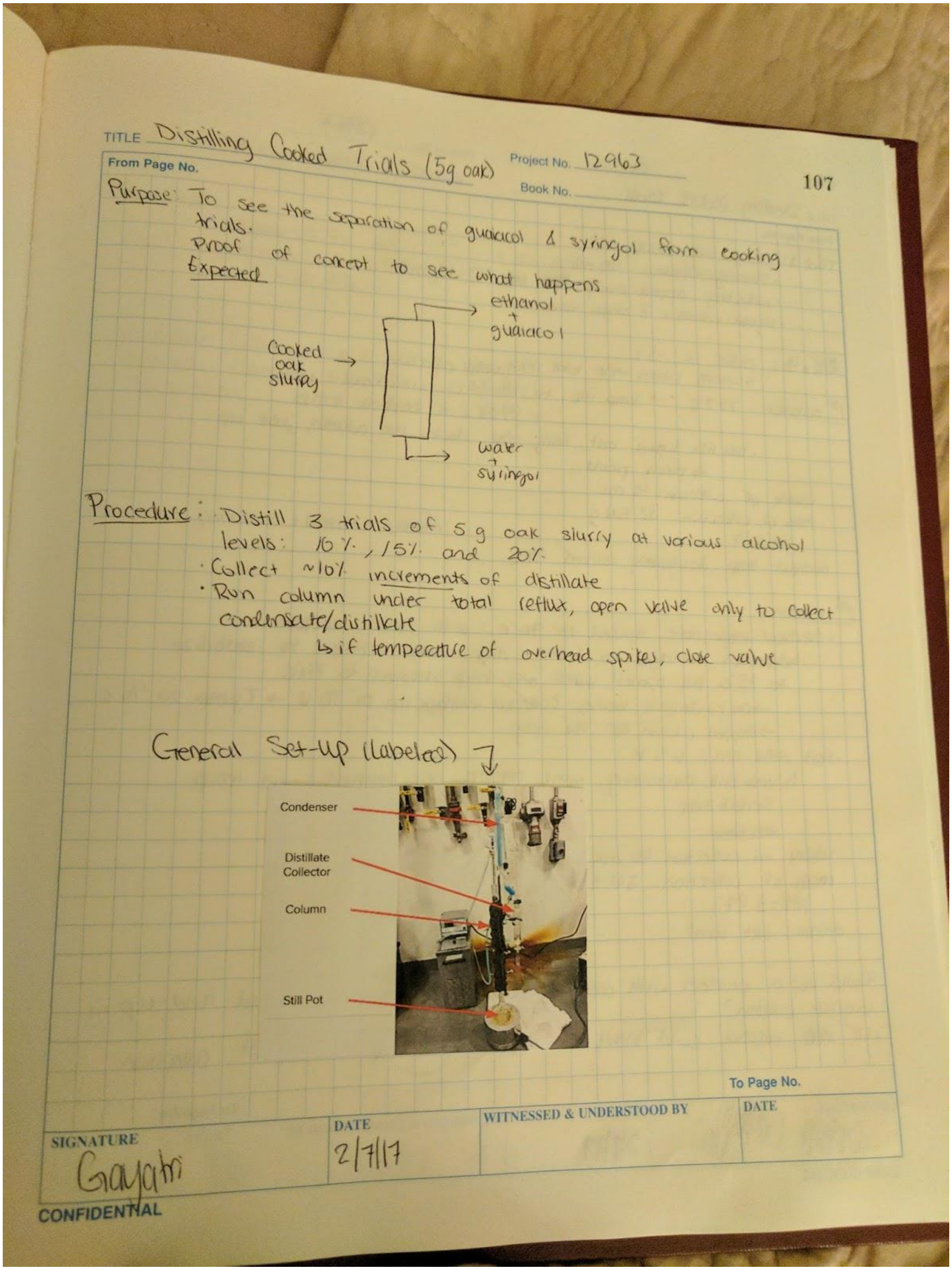
collected 190 mL of residual
• 191.38 g
pH = 11.66
not much aroma → adjust w/ K-Carb

Amt	pH
0.33 g	9.87
0.31 g	6.55
0.42 g	5.34
0.34 g	4.79
0.50 g	4.27
0.65 g	3.88
0.56 g	3.55

aroma
wet wood, faint

campfire-y smell starting to emit slightly sour note but there is still a hint of campfire

Sent for sampling SHARDMOAK, MVAZ, MALC
Condensate: 0D61405
Residual: OD61406



TITLE Distilling Cooked Trials (5g oak)

Project No. 12963

From Page No.

Book No.

107

Purpose: To see the separation of guaiacol & syringol from cooking trials.

Proof of concept to see what happens

Expected

Procedure: Distill 3 trials of 5 g oak slurry at various alcohol levels: 10 %, 15% and 20%.

- Collect ~10% increments of distillate
- Run column under total reflux, open valve only to collect condensate/distillate
  - ↳ if temperature of overhead spikes, close valve

General Set-Up (labeled) ↓

To Page No.

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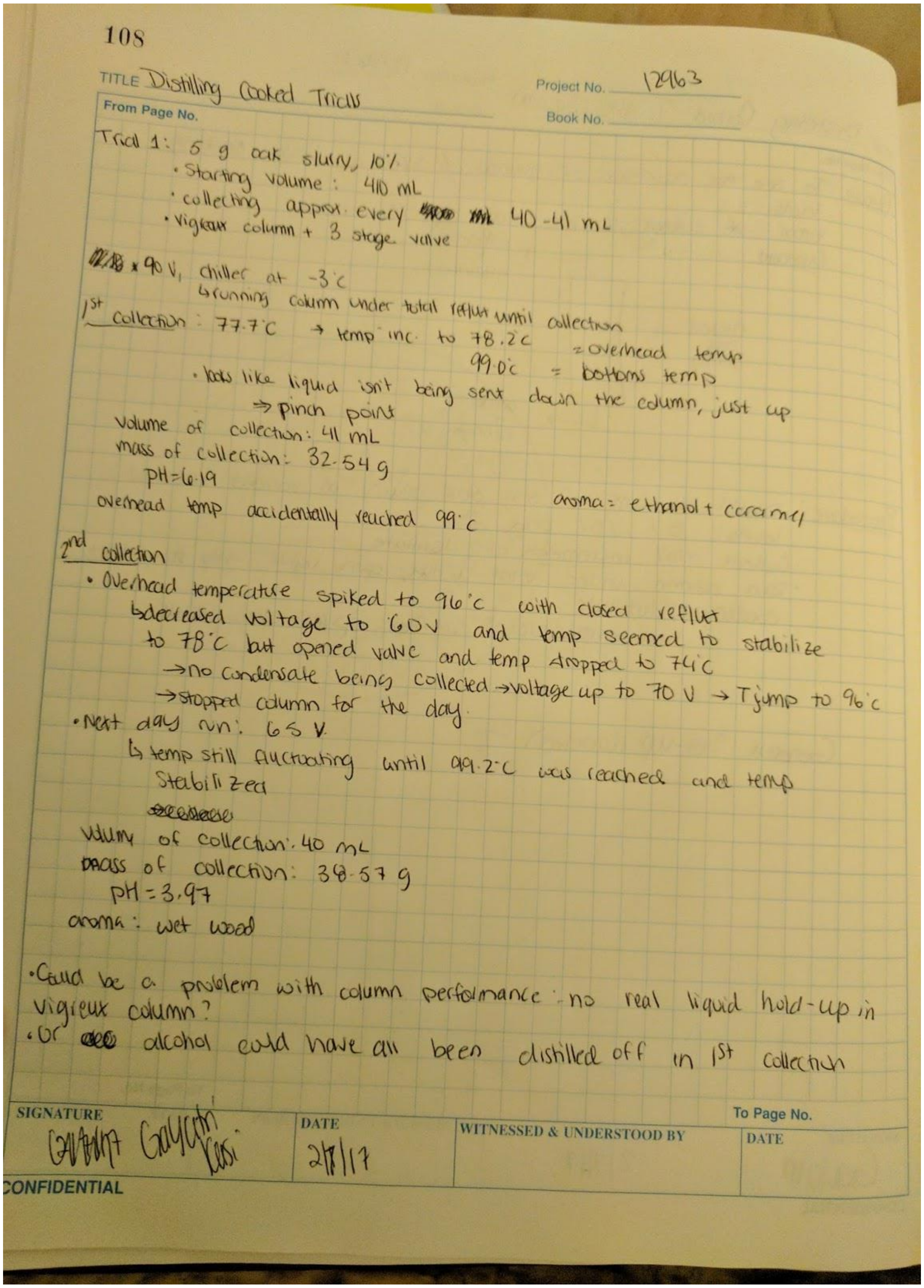

108

TITLE Distilling Cooked Trials

Project No. 12963

From Page No.

Book No.

Trial 1: 5 g oak slurry, 10%
- Starting volume: 410 mL
- collecting approx. every 40-41 mL
- Vigreux column + 3 stage valve

× 90 V, chiller at -3°C
↳ running column under total reflux until collection

1st collection: 77.7°C → temp inc. to 78.2°C = overhead temp
99.0°C = bottoms temp
- looks like liquid isn't being sent down the column, just up
⇒ pinch point

volume of collection: 41 mL
mass of collection: 32.54 g
pH = 6.19
aroma = ethanol + caramel

overhead temp accidentally reached 99°C

2nd collection
- Overhead temperature spiked to 96°C with closed reflux
↳ decreased voltage to 60 V and temp seemed to stabilize to 78°C but opened valve and temp dropped to 74°C
→ no condensate being collected → voltage up to 70 V → T jump to 96°C
→ stopped column for the day.
- Next day run: 65 V
↳ temp still fluctuating until 99.2°C was reached and temp stabilized

volume of collection: 40 mL
mass of collection: 38.57 g
pH = 3.97
aroma: wet wood

- Could be a problem with column performance: no real liquid hold-up in vigreux column?
- Or alcohol could have all been distilled off in 1st collection

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To Page No.

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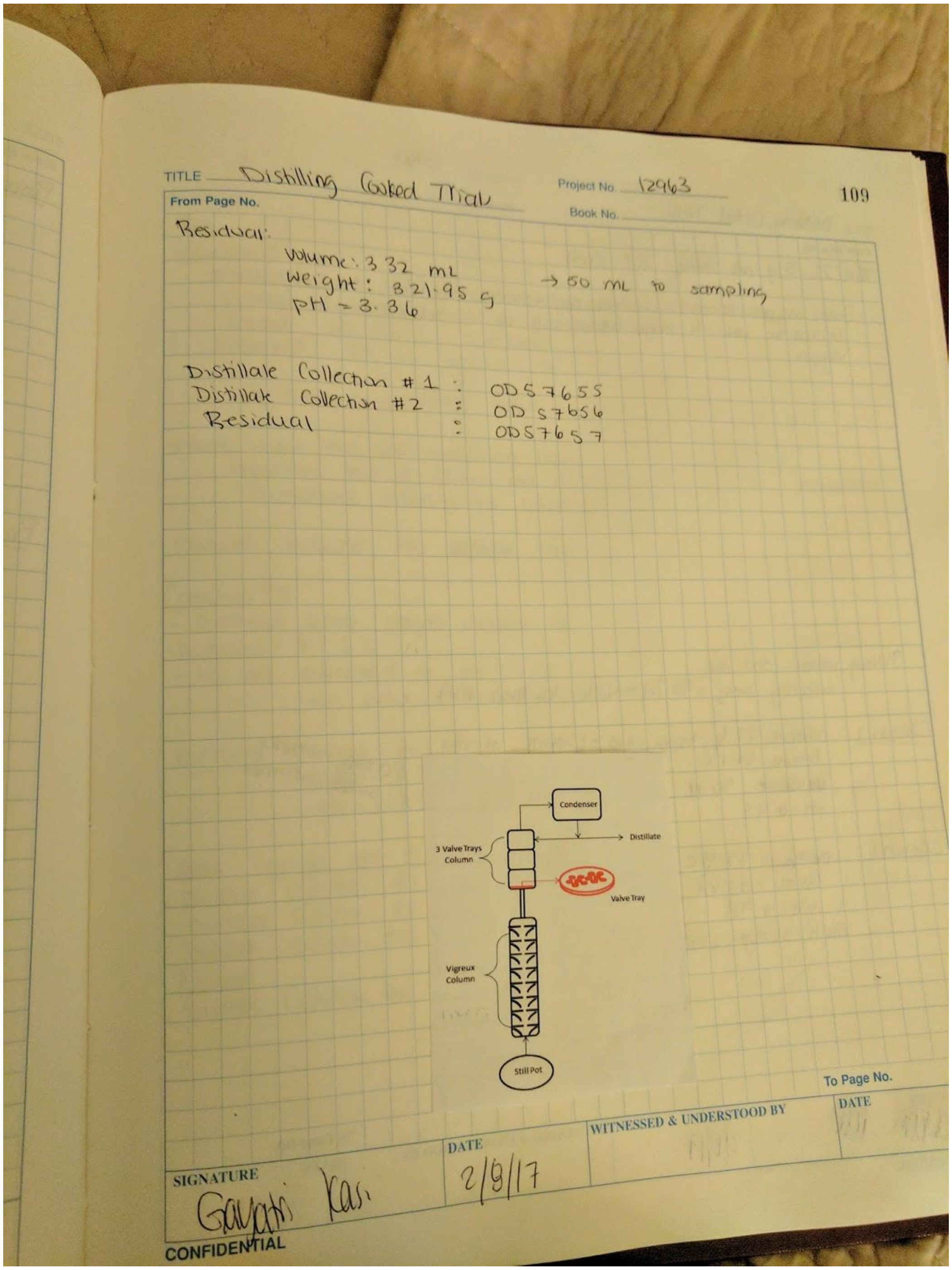

TITLE Distilling Cooked Trial
Project No. 12963
Book No.
109
From Page No.

Residual:
Volume: 332 mL
Weight: 321.95 g → 50 mL to sampling
pH = 3.36

Distillate Collection #1 : OD57655
Distillate Collection #2 : OD57656
Residual : OD57657

To Page No.

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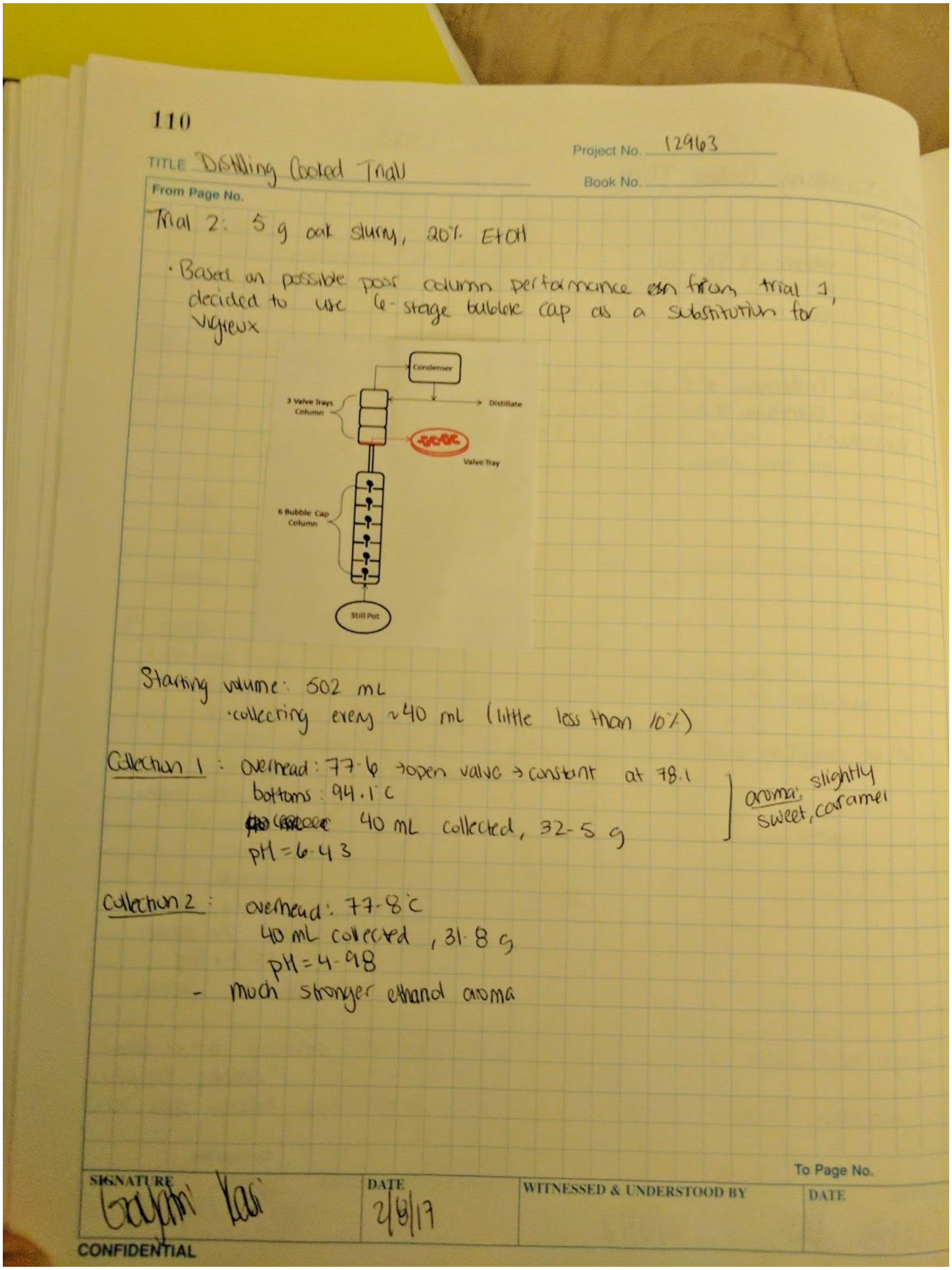

110

TITLE Distilling Cooked Trial

Project No. 12963

Book No.

From Page No.

Trial 2: 5 g oak slurry, 20% EtOH

- Based on possible poor column performance from trial 1, decided to use 6-stage bubble cap as a substitution for vigreux

Starting volume: 502 mL
·collecting every ~40 mL (little less than 10%)

Collection 1: overhead: 77.6 → open valve → constant at 78.1
bottoms: 94.1°C
40 mL collected, 32.5 g
pH = 6.43

aroma: slightly sweet, caramel

Collection 2: overhead: 77.8°C
40 mL collected, 31.8 g
pH = 4.98
- much stronger ethanol aroma

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Gayathri Vasi	2/8/17		

To Page No.

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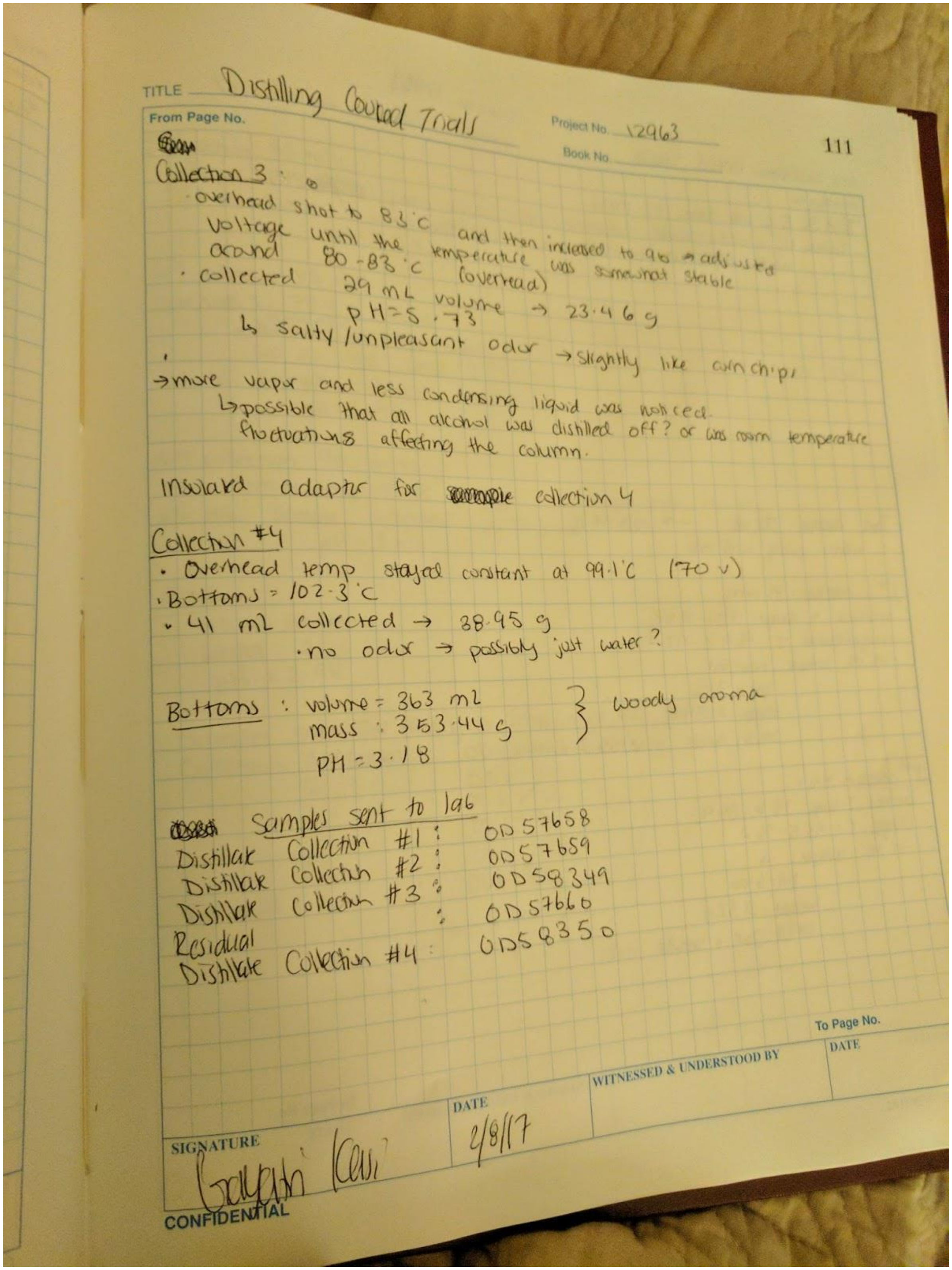


TITLE Distilling Couked Trials

From Page No.

Project No. 12963

Book No.

111

Collection 3 :

- overhead shot to 83 °C and then increased to 96 → adjusted voltage until the temperature was somewhat stable around 80 - 83 °C (overhead)
- collected 29 mL volume → 23.46 g
  pH = 5.73
  ↳ salty / unpleasant odor → slightly like corn chips

→ more vapor and less condensing liquid was noticed.
↳ possible that all alcohol was distilled off? or was room temperature fluctuations affecting the column.

Insulated adapter for ~~sample~~ collection 4

Collection #4

- Overhead temp stayed constant at 99.1 °C (170 v)
- Bottoms = 102.3 °C
- 41 mL collected → 38.95 g
  - no odor → possibly just water?

Bottoms : volume = 363 mL
mass : 353.44 g
pH = 3.18
} woody aroma

Samples sent to lab
Distillate Collection #1 : OD57658
Distillate Collection #2 : OD57659
Distillate Collection #3 : OD58349
Residual : OD57660
Distillate Collection #4 : OD58350

To Page No.

SIGNATURE

DATE 2/8/17

WITNESSED & UNDERSTOOD BY

DATE

CONFIDENTIAL

Project No.

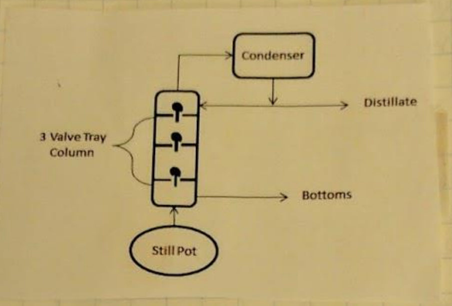
TITLE Distillation Cooked Trials

Book No.

From Page No.

Trial 3: 5 g oak slurry, 15% EtOH

Purpose: try a smaller/shorter column and collect every [illegible] 5% in volume ~~separation~~ to map out separation



Feed volume: 510 mL → 500.07 g

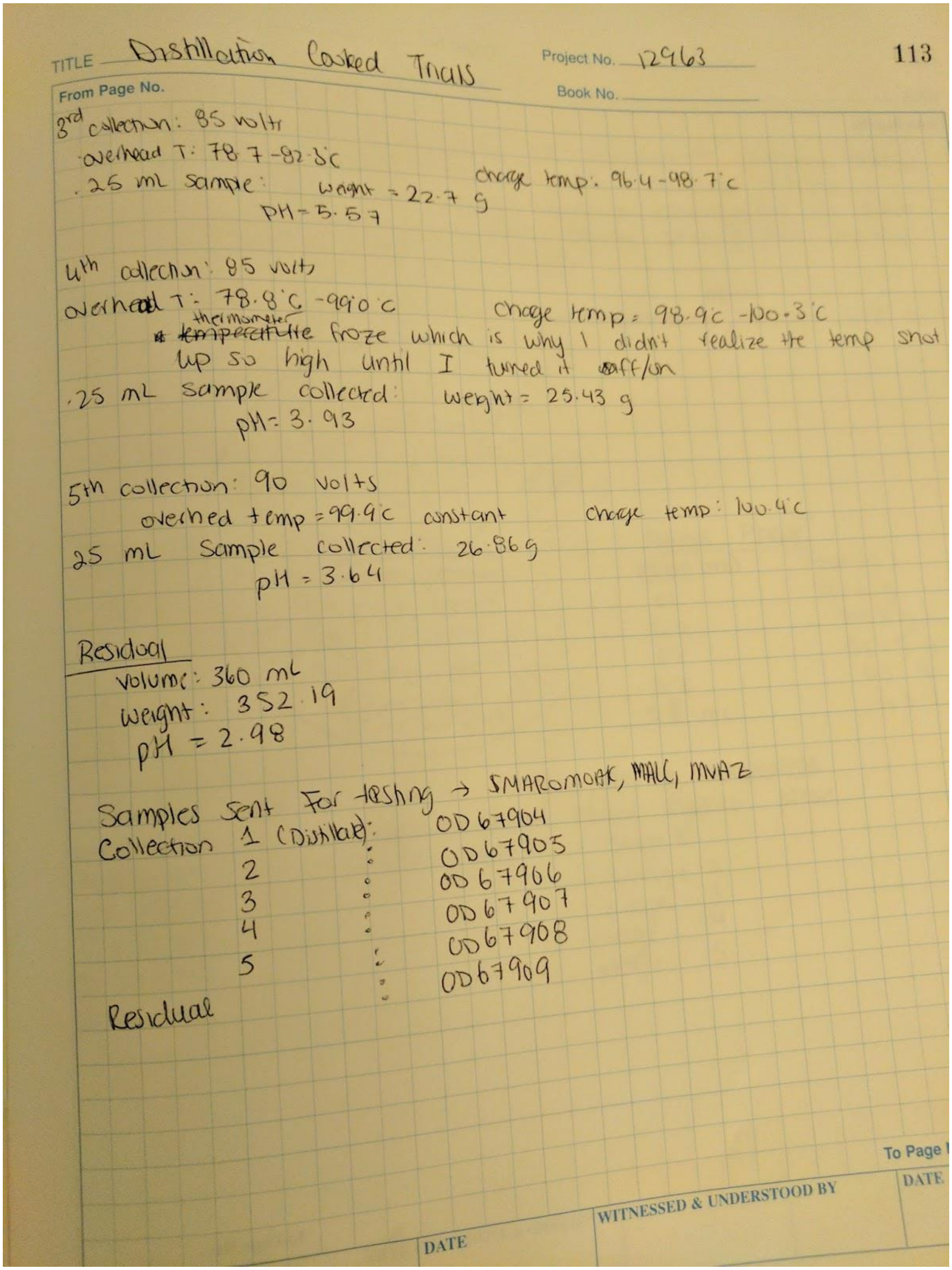
·collect every ~25 mL

1st collection: 85 volts
·overhead T range: 78.1 - 81.4°C
·charge/residual T: 91.9°C - 92.3
·25 mL sample collected:
pH = 7.86
weight: 21.34 g
smell: sweet potatoes & vanilla

2nd collection: 85 V
·overhead T-range: 78.6 - 84.4
charge T: 93.5 - 96.2
25 mL sample: →
pH = 6.00
weight = 21.32 g
smell: sour + vanilla notes

SIGNATURE	DATE	WITNESSED & UNDERSTOOD BY	DATE

To Page No.



TITLE Distillation Cooked Trials

Project No. 12963

113

Book No.

From Page No.

3rd collection: 85 volts
overhead T: 78.7-82.3°c
.25 mL sample: weight = 22.7 g charge temp: 96.4-98.7°c
pH = 5.57

4th collection: 85 volts
overhead T: 78.8°C -99.0°c charge temp = 98.9°c -100.3°c
* ~~temperature~~ thermometer froze which is why I didn't realize the temp shot up so high until I turned it off/on
.25 mL sample collected: weight = 25.43 g
pH = 3.93

5th collection: 90 volts
overhed temp = 99.9°c constant charge temp: 100.4°c
25 mL Sample collected: 26.86 g
pH = 3.64

Residual
volume: 360 mL
weight: 352.19
pH = 2.98

Samples sent for testing → SMAROMOAK, MALC, MVAZ
Collection 1 (Distillate): OD67904
2 : OD67905
3 : OD67906
4 : OD67907
5 : OD67908
: OD67909
Residual

To Page
DATE
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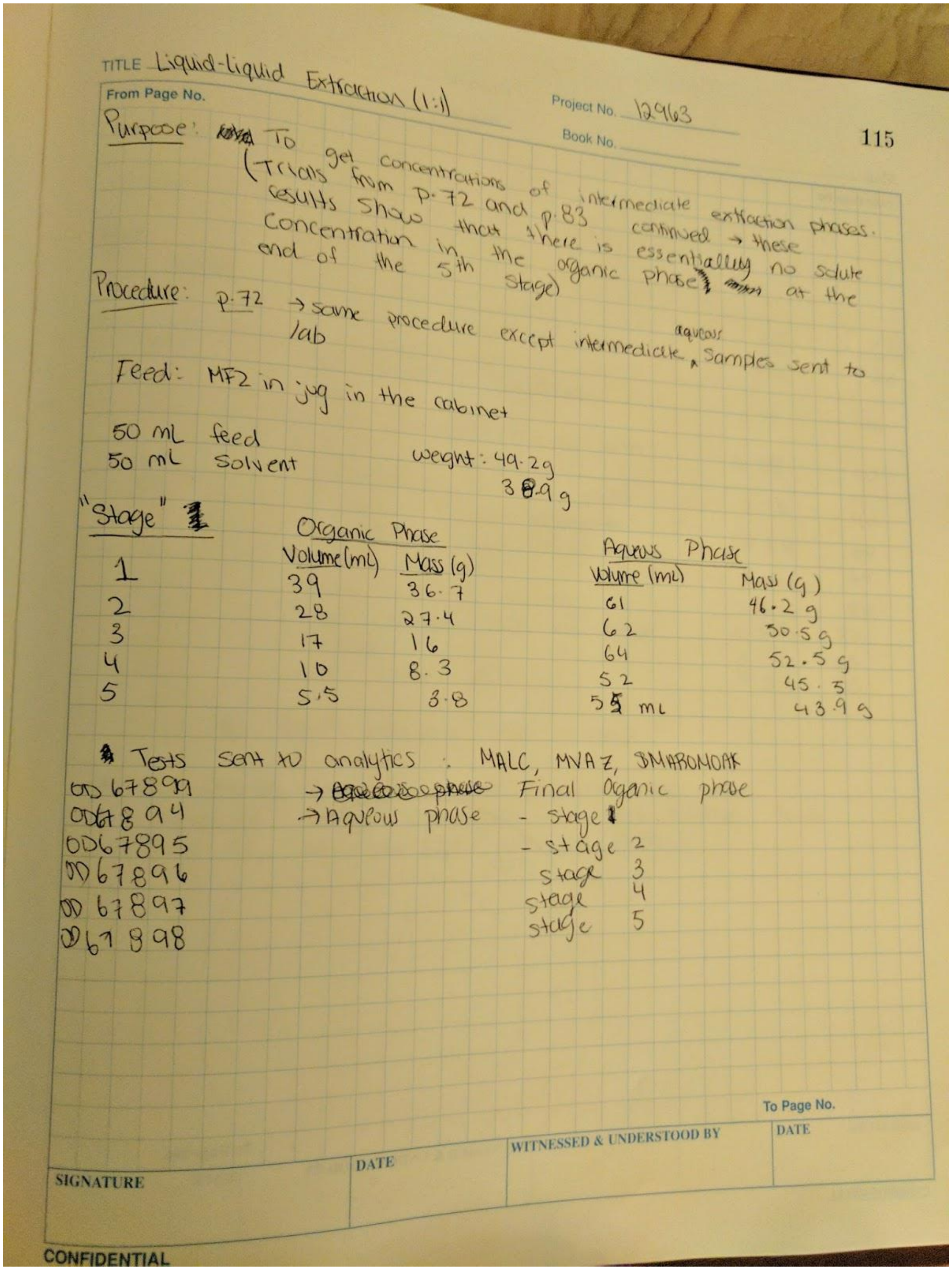
DATE



TITLE Liquid-liquid Extraction (1:1)

From Page No.

Project No. 12963

Book No.

115

Purpose: To get concentrations of intermediate extraction phases. (Trials from p.72 and p.83 continued → these results show that there is essentially no solute concentration in the organic phase at the end of the 5th stage)

Procedure: p.72 → same procedure except intermediate aqueous samples sent to lab

Feed: MF2 in jug in the cabinet

50 mL feed
50 mL solvent

weight: 49.2 g
38.9 g

"Stage"	Organic Phase		Aqueous Phase	
	Volume (mL)	Mass (g)	Volume (mL)	Mass (g)
1	39	36.7	61	46.2 g
2	28	27.4	62	50.5 g
3	17	16	64	52.5 g
4	10	8.3	52	45.5
5	5.5	3.8	55 mL	43.9 g

Tests sent to analytics: MALC, MVAZ, SMAROMOAK

OD67899 → Final Organic phase
OD67894 → Aqueous phase - stage 1
OD67895 - stage 2
OD67896 stage 3
OD67897 stage 4
OD67898 stage 5

To Page No.

SIGNATURE	DATE	WITNESSED & UNDERSTOOD BY	DATE

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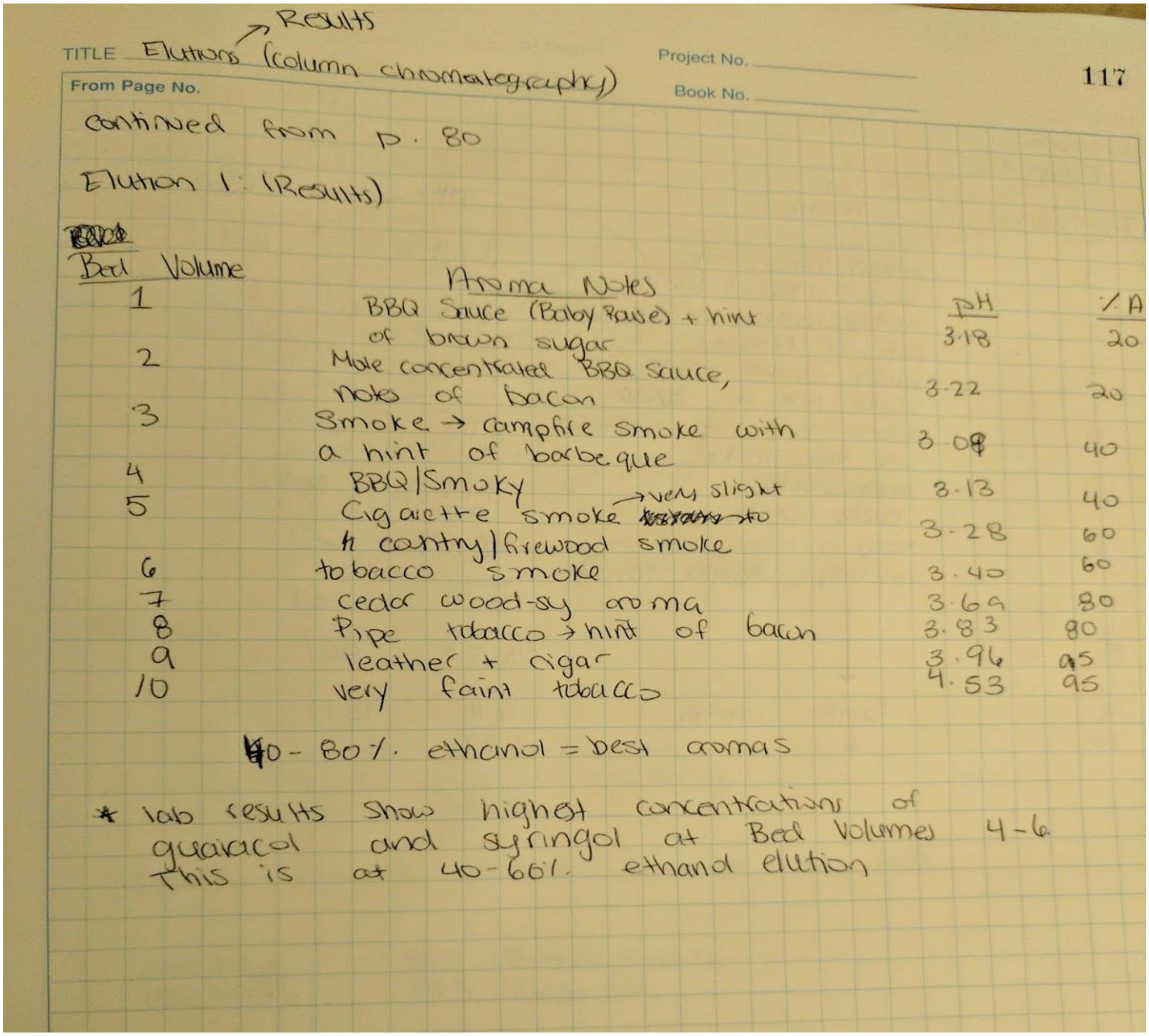


TITLE Elutions → Results (column chromatography)

Project No.

Book No.

117

From Page No.

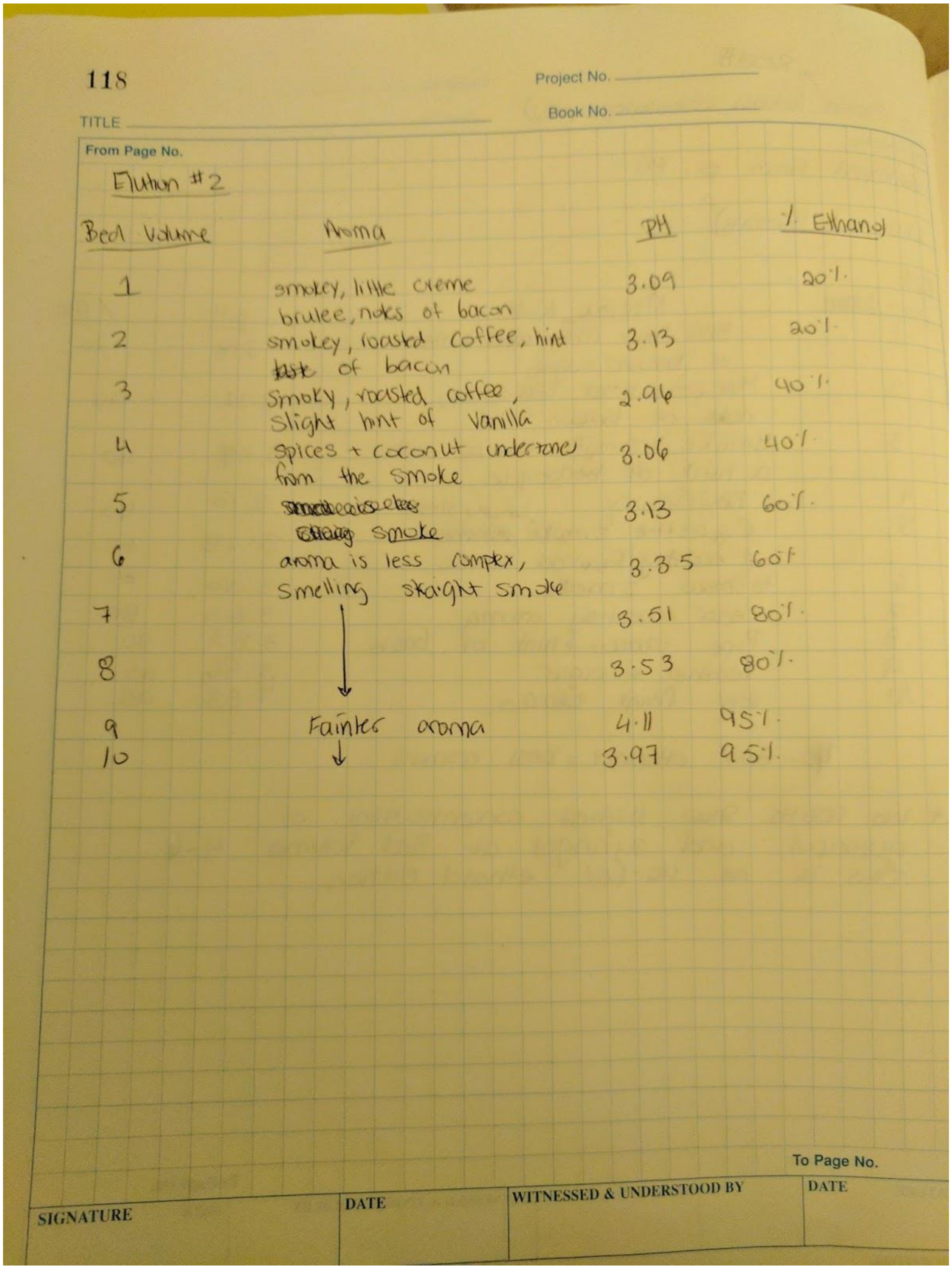
continued from p. 80

Elution 1: (Results)

Bed Volume	Aroma Notes	pH	% A
1	BBQ Sauce (Baby Rays) + hint of brown sugar	3.18	20
2	More concentrated BBQ sauce, notes of bacon	3.22	20
3	Smoke → campfire smoke with a hint of barbeque	3.08	40
4	BBQ/Smoky	3.13	40
5	Cigarette smoke → very slight to h country/firewood smoke	3.28	60
6	tobacco smoke	3.40	60
7	cedar wood-sy aroma	3.69	80
8	Pipe tobacco → hint of bacon	3.83	80
9	leather + cigar	3.96	95
10	very faint tobacco	4.53	95

40 - 80% ethanol = best aromas

* lab results show highest concentrations of guaiacol and syringol at Bed Volumes 4-6 This is at 40-60% ethanol elution



118

Project No.

TITLE

Book No.

From Page No.

Elution #2

Bed Volume	Aroma	PH	% Ethanol
1	smokey, little creme brulee, notes of bacon	3.09	20%
2	smokey, toasted coffee, hint ~~taste~~ of bacon	3.13	20%
3	Smoky, roasted coffee, slight hint of Vanilla	2.96	40%
4	spices + coconut undertone from the smoke	3.06	40%
5	[illegible] [illegible] smoke	3.13	60%
6	aroma is less complex, smelling straight smoke	3.35	60%
7	↓	3.51	80%
8	↓	3.53	80%
9	Fainter aroma	4.11	95%
10	↓	3.97	95%

To Page No.

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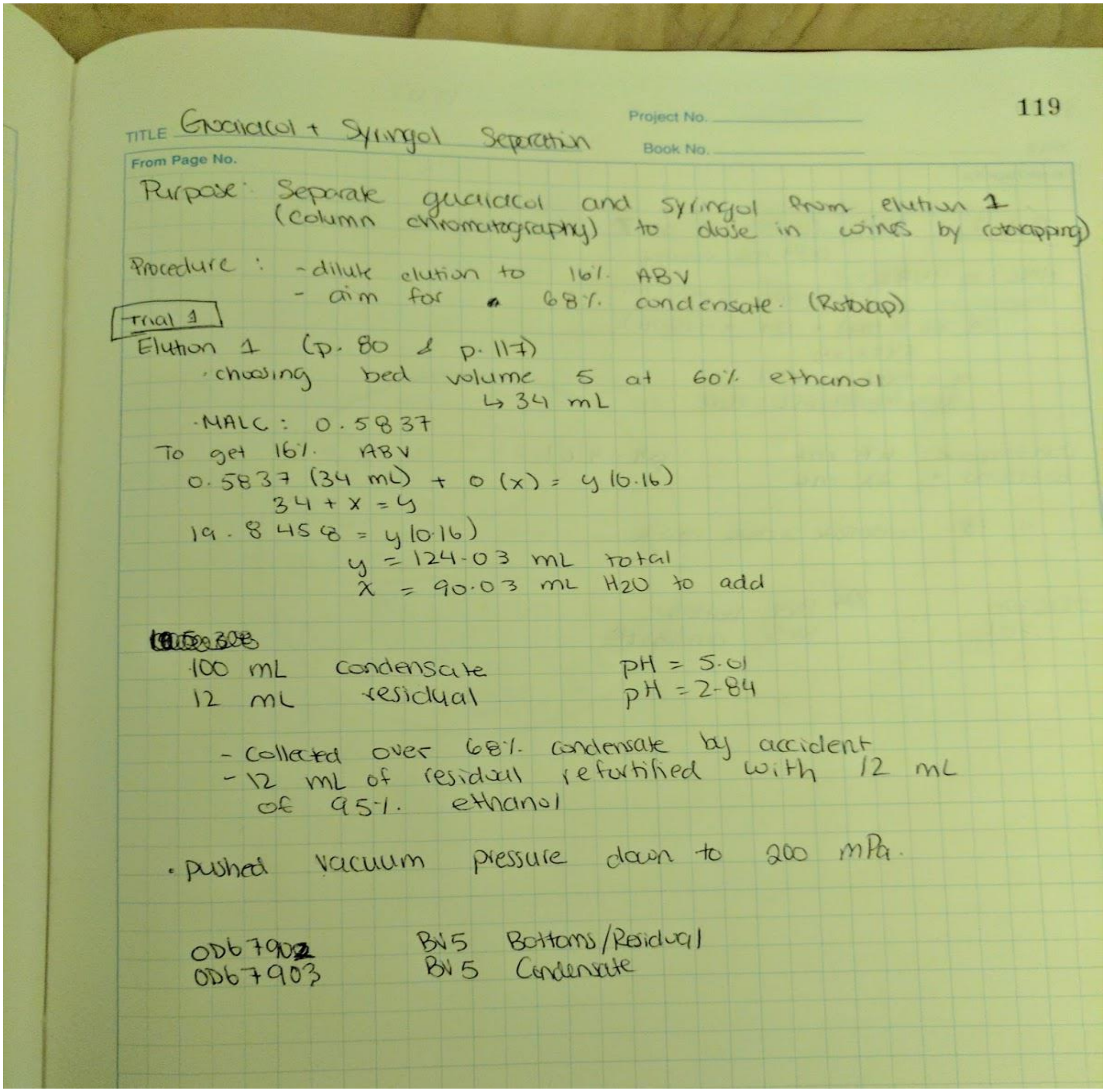


119

TITLE Guaiacol + Syringol Seperation
Project No.
Book No.
From Page No.

Purpose: Separate guaiacol and syringol from elution 1 (column chromatography) to dose in wines by (rotovapping)

Procedure: - dilute elution to 16% ABV
- aim for 68% condensate. (Rotovap)

Trial 1

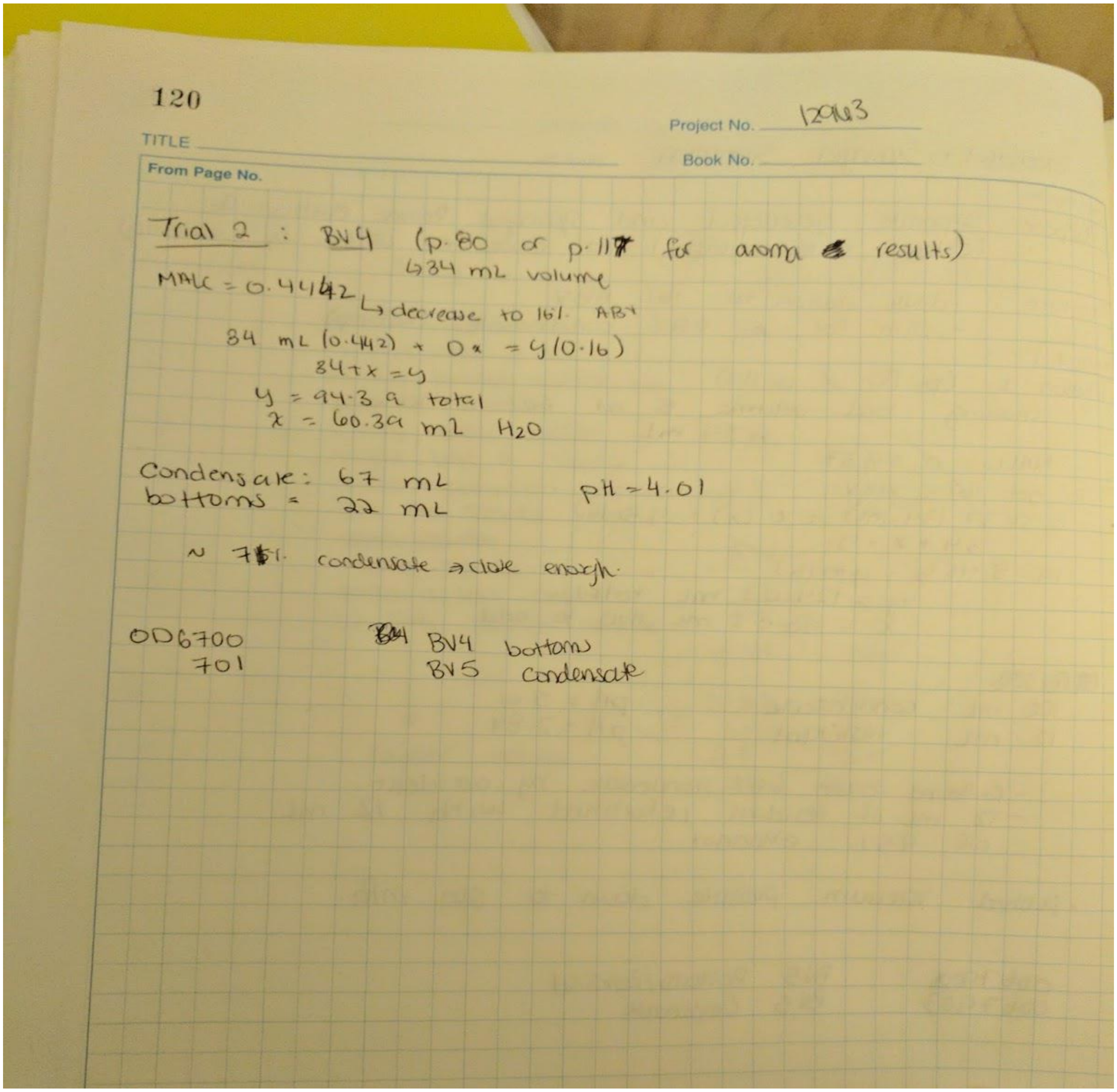
Elution 1 (p. 80 & p. 117)
· choosing bed volume 5 at 60% ethanol
↳ 34 mL
· MALC: 0.5837
To get 16% ABV
0.5837 (34 mL) + 0 (x) = y (0.16)
34 + x = y
19.8458 = y (0.16)
y = 124.03 mL total
x = 90.03 mL H_2O to add

[redacted]
100 mL condensate pH = 5.01
12 mL residual pH = 2.84

- Collected over 68% condensate by accident
- 12 mL of residual refortified with 12 mL of 95% ethanol

· pushed vacuum pressure down to 200 mPa.

OD67902 BV5 Bottoms/Residual
OD67903 BV5 Condensate



120

Project No. 12943

TITLE

Book No.

From Page No.

Trial 2 : BV4 (p. 80 or p. 11[illegible] for aroma [illegible] results)

↳ 34 mL volume

MALC = 0.4442

↳ decrease to 16% ABV

84 mL (0.442) + 0x = y(0.16)

84 + x = y

y = 94.3 g total

x = 60.39 mL H_2O

Condensate: 67 mL

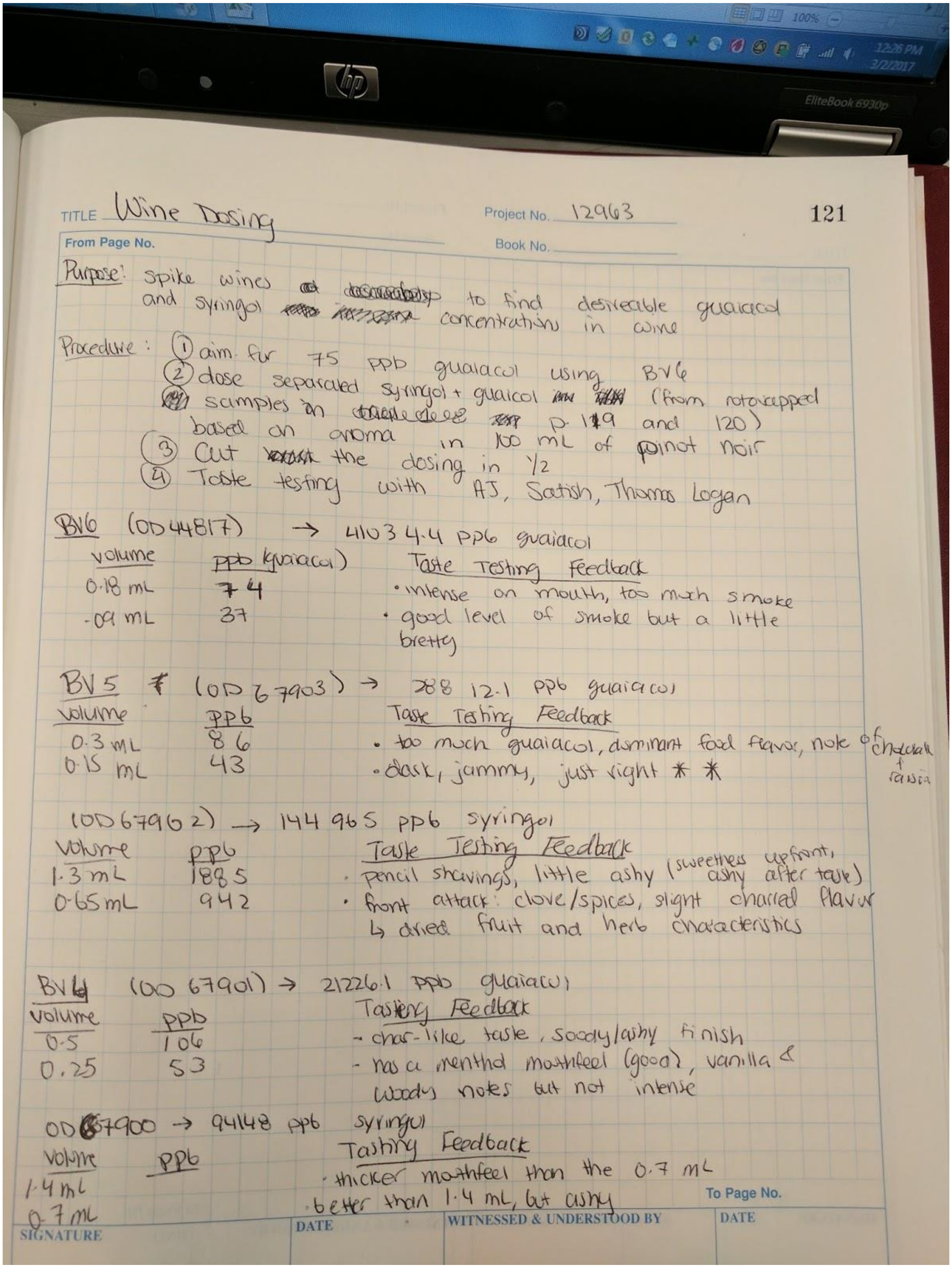
bottoms = 22 mL

pH = 4.01

~ 7[illegible]% condensate ⇒ close enough.

OD6700 ~~B4~~ BV4 bottoms

701 BV5 condensate



TITLE Wine Dosing Project No. 12963 121

From Page No. Book No.

Purpose: spike wines to find desireable guaiacol and syringol concentrations in wine

Procedure:
① aim for 75 ppb guaiacol using BV6
② dose separated syringol + guaicol (from rotovapped samples on p. 119 and 120) based on aroma in 100 mL of pinot noir
③ Cut the dosing in ½
④ Taste testing with AJ, Satish, Thomas Logan

BV6 (OD44817) → 4103 4.4 ppb guaiacol

volume	ppb (guaiacol)	Taste Testing Feedback
0.18 mL	74	• intense on mouth, too much smoke
.09 mL	37	• good level of smoke but a little bretty

BV5 (OD 67903) → ~~288~~ 12.1 ppb guaiacol

volume	ppb	Taste Testing Feedback
0.3 mL	86	• too much guaiacol, dominant food flavor, note of chocolate + raisin
0.15 mL	43	• dark, jammy, just right * *

(OD 67962) → 144 965 ppb syringol

Volume	ppb	Taste Testing Feedback
1.3 mL	1885	• pencil shavings, little ashy (sweetness upfront, ashy after taste)
0.65 mL	942	• front attack: clove/spices, slight charred flavor ↳ dried fruit and herb characteristics

BV4 (OD 67901) → 21226.1 ppb guaiacol

volume	ppb	Tasting Feedback
0.5	106	- char-like taste, soody/ashy finish
0.25	53	- has a menthol mouthfeel (good), vanilla & woody notes but not intense

OD 67900 → 94148 ppb syringol

Volume	ppb	Tasting Feedback
1.4 mL		• thicker mouthfeel than the 0.7 mL
0.7 mL		• better than 1.4 mL, but ashy

To Page No.

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