

**Quantifying Water Content for
Non-halogenated Waste Solvents**

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University of British Columbia

CHBE 464

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CHBE 464

Problem-Based Laboratory: Final Report

Quantifying Water Content for Non-halogenated Waste Solvents

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Introduction

The Environmental Services Facility (ESF) located at the southern tip of campus is responsible for gathering and disposing chemical wastes from a wide range of laboratories and research centers across UBC. The facility operates in compliance and within the framework of the by-laws and regulations provided by the municipal, provincial and federal government. Specifically, UBC follows a Chemical Waste Disposal Program which is approved by the Ministry of Environment. Treatment of the chemical wastes are based on the chemical classification, and are organized into three main categories: solvent/oil waste (halogenated, non-halogenated and oils), non-regulated contaminated solid waste and photographic waste. The main problem this Problem Based Laboratory focuses on is the non-halogenated solvent wastes in the facility.

Currently, the ESF operates in contract with Sumas Environmental Services Inc. for treatment of the solvent wastes. Upon collection of the solvents from generators across campus, Sumas Environmental Services ships the wastes to an analytical lab (such as Exova or Powertech) to determine if the solvent is halogenated or non-halogenated. The halogenated materials are then sent to Nisku, Alberta, where treatment costs approximately \$1.65 for one litre of solvent waste. The non-halogenated solvents then undergo a flammability test. If the results are positive and the solvent is flammable, the solvent can be mixed with other flammable materials and can be used as a fuel additive. If the solvent is not flammable, the contractor treats the non-halogenated solvents for about \$0.85 per litre of waste.

Due to the lack of acknowledgement, many of the samples collected at the ESF (Figure A1) contain various amount of water, and some halogenated solvents are mislabeled as non-halogenated. The price for treatment is volume based, and excess water in the solvents will significantly increase the costs for treatment. Moreover, when a solvent is primarily composed of water, it will likely fail the flammability test and cannot be converted into a usable fuel additive, thus limiting the ability of this solvent to be converted. Any non-halogenated solvents predominantly composed of water (>95%) can be tested for toxicology via a bioassay fish test by independent laboratory, Maxxam Analytical. If the toxicology test yields negative results, the non-halogenated waste is considered safe for drain disposal. In this case, the contaminated water waste costs \$0.31/L to treat, rather than \$0.85/L. Therefore, the water content of the waste

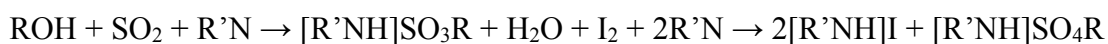
solvents and correct labelling are important factors affecting the operating cost of the ESF. Since the ESF does not have a method to determine the water content, they previously planned to hire BC Hydro to test samples. However due to high cost, the plan was suspended.

In order to minimize the cost for waste solvent treatment, the objectives of this PBL are to develop a method that is both practical and economically feasible to examine non-halogenated streams collected at the ESF (See Table A1). That is, to qualitatively determine the water content using the Karl Fischer titration, evaluate its feasibility at the ESF, and to qualitatively analyze these samples for presence of halogens. Being able to accomplish this will ultimately allow the ESF to better manage their waste solvents by reducing treatment costs

Group six has selected the Hanna Instruments HI903 Karl Fischer titration apparatus to run the Karl Fischer volumetric titration to determine water content. The apparatus utilizes Hydranal-composite-5 as the titrant, and dry methanol as the solvent and provides a moisture analysis with precision to the thousandths. The analysis is advantageous in that it only requires a small sample of 4 μ L. To test the samples for halogens, the silver nitrate test is selected and conducted for all the collected samples labeled non-halogenated. The two portions of the experiment were conducted simultaneously over the timeframe of six weeks.

Theory

Karl Fischer Titration is a method for determining moisture or water content of a target solution. The Karl Fischer Titration is a titration method by which the reagent or titrant reacts with water and converts water into a non-conductive chemical (CSC, 2014). The method of conversion is as follows:



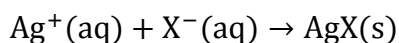
From the above equation, alcohol (ROH) reacts with sulfur dioxide (SO₂) and base (R'[']N) to form an intermediate alkylsulfite salt ([R'[']NH]SO₃R) which is then oxidized by iodine to an alkylsulfate salt ([R'[']NH]SO₄R) and a hydroiodic acid salt (2[R'[']NH]I), thereby reducing the electrical conductivity of the solution (EMD, 2014). The water content present is calculated by analyzing the amount of solvent added to reduce the solution down to the base electrical conductivity. The oxidation reaction consumes water, reducing the initial solvent back to a state where another sample can be analyzed using the same process (EMD, 2014). For this experiment, the alcohol used is dry methanol while the reagent used which contains sulfur

dioxide is Hydranal-composite 5. Two types of Karl Fischer titration tests are available that manipulate this theory to determine water content; they are volumetric or coulometric titration.

In Coulometric Karl Fischer Titration, the reagent and a solvent are first combined in the titration cell. As a sample is introduced and dissolved in the titration cell, the reagent is released by the induction of an electric current. The amount of moisture in the sample is proportionally determined by the amount of current required. This method is mainly suitable for samples containing less than 1% moisture and inherently has the ability to determine low water content sample to a greater degree of accuracy (CSC, 2014).

By contrast, in Volumetric Karl Fischer Titration, the sample is added into a titration cell containing the solvent (methanol) at the base electrical conductivity. Upon injection, the solution in the test vessel's electrical conductivity increases. Titrant is added to return the conductivity level to the initial value. -The recorded amount of reagent required is then used to determine the amount of water in the sample. This method provides more flexibility in the determination of water content, ranging from 100 ppm to 100% water by volume. The Volumetric Karl Fischer Titration was ultimately selected to determine water content for the non-halogenated samples collected in this Problem-Based laboratory (CSC, 2014).

A silver nitrate test is allowed for evaluation of halides and halogens in the collected samples of interest. The specific ions of interest in the solution are fluorides, chlorides, bromides and iodides. The reaction pathway for the silver nitrate test is as shown below (Test, 2014):



Where X represents halide ions. According to the above equation, when the silver nitrate solution is added to a sample consisting of bromide, chloride or iodide ions, the silver ions react and form silver halide. A solid precipitate forms as silver chloride, silver bromide and silver iodide are insoluble in water as shown in Figure A3 ("Test", 2014). However, it should be noted that samples containing fluoride ions cannot be identified as silver fluoride is soluble in water. Since the non-halogenated samples collected are of unknown composition, the solution is first acidified with dilute nitric acid first to ensure the removal of all carbonate and hydroxide ions which also form precipitates with silver nitrate ("Test", 2014).

Experimental Apparatus and Techniques

The major equipment used in this Problem Based Learning Lab is the Hanna Instruments HI903 Karl Fischer Volumetric Titrator. It has a capacity to measure water content of liquid or solids ranging from 100 ppm to 100% (by volume), with resolution down to 1 ppm (0.0001%). As shown in Figure A2, the main body of Karl Fischer unit consists of an analyzing block, a Karl Fischer test vessel and a burette system. The Karl Fischer beaker is connected to two containers - one for dry methanol, used as solvent, and another bottle, was used to collect waste purged the test vessel.

Dry methanol was added to the beaker via an air pump, setting up the testing solution so that the sample could be injected to evaluate for water content. The test vessel maintains uniform mixing via a magnetic digital stirrer and the conductivity was measured by a dual platinum pin polarization electrode placed inside the beaker. The burette system was connected to a bottle of Hydranal-composite 5 titrant. The titrant was injected into the Karl Fischer beaker through the burette which has a resolution of 1/40000 of the burette volume. The analyzing block calculated the water content of the injected sample automatically with preset sample volume and calculating method. Moreover, each of the Karl Fischer test vessel, stock methanol, titrant and the waste solvent, possessed a desiccant attachment to their respective lids to prevent water vapor from entering into the system.

A centrifuge placed on the fourth floor lab in the Chemical and Biological Engineering Building was used to effectively segregate solids in the collected waste samples. Prior to Karl Fischer titration, Group 6 treated all the samples with the centrifuge and extracted the upper clear liquid with a pipette to run the test. The samples were centrifuged at the speed of 3250 rpm for 10 min. Given that there was no pretreatment of the waste samples, samples were centrifuged to effectively remove out any precipitates or other impurities, therefore providing a homogeneous solution that can be easily transferred into the test environment for Karl Fischer Titration.

To inject correct amount of sample into the system, optimum sample volume and an appropriate syringe were needed. Group 6 has determined the optimum sample volume to be 4.0 μL through calibration, with the result is shown in Table A2. The Department of Chemical and Biological Engineering provides syringes with varying volumes, from 0.5 μL to 100 μL . However, due to transportation and order issues, the 10 μL syringe was not supplied to the lab

within the 6 week time frame for this laboratory, so a Hamilton 50 μL syringe was chosen instead.

Prior to the sample analysis, Karl Fischer unit was used to test samples with known water content at different sample volume in order to determine the optimum sample size.

Prior to experimentation, Group 6 checked all the connections of the Karl Fischer unit and then turned it on. The burette was rinsed with the Hydranal-composite 5 titrant three times, then the Karl Fischer beaker was emptied by the air pump. Group 6 then added 50 ml of dry methanol and inputted operating method. The operating method consists of the information the system requires in order to analyze the sample. This includes titrant type, titrant concentration, initial solvent volume, format of the results and the maximum time length for each trial. The "start titration" button located on the control panel was then pressed to undergo a pre-titration meant to rid of any water content inside the system. This procedure took approximately 15-20 minutes until the system entered standby mode, indicating that the Karl Fischer unit was ready to run samples. Initial problems arose due to human error resulted in wasted solvent and titrant and delays in the testing process. The Fischer titration method is very sensitive to water content and may require excessive titrant in order to restore back to the baseline testing conditions.

Samples were prepared with known water content (See Table A2). Different sample volumes (1 μL ,2 μL ,4 μL ,10 μL) for each sample were injected into the Karl Fischer unit by syringes. Initially, Group 6 used a 0.5 μL syringe to inject twice for 1 μL sample volume and the 100 μL syringe for the remaining volume due to the absence of more appropriate syringes at the time of testing. 50 μL and 10 μL syringes were ordered upon realization, but took approximately two weeks for delivery. Before each trial, the "Sample Analysis" button on the panel was pressed and the volume of injected samples was inputted. Sample was then injected and the "Sample Analysis button" was pressed again to start analyzing the sample. When the unit finished analyzing, the result was shown on the screen, the titrant volume used, time length and the water content was recorded. To increase the accuracy, Group 6 completed triplicate trials for each sample.

With the optimum sample volume determined (4.0 μL), Group 6 split into two groups: one to perform the quantitative analysis of waste solvent samples, and the second to conduct silver nitrate test to ensure the experiment is completed in an effective manner within the scheduled time frame.

To quantitatively analyze the waste solvent samples to determine the water content, the samples were labeled and centrifuged to rid of salt precipitates. While two group members were centrifuging the samples, the other two were preparing the Karl Fischer unit as mentioned above. After centrifugation, the upper clear liquid was collected by a pipette, transferred into a separate vial and labeled with the Sample ID. Most of the samples were used for the silver nitrate test (~7 mL) but only a fraction (4.0 μ L) of the sample was required for the Karl Fischer Titration. The samples were shaken so that the separating layers within the sample were evenly mixed. 4.0 μ L of sample was collected by the 50 μ L and 10 μ L syringes. When extracting samples, the syringe was filled and emptied several times in order to rid of the air bubbles inside. Because the analysis of the water content is entirely based on the volume, the presence of air bubbles would be associated with errors in the results. The 50 μ L syringe was used for first two weeks until the 10 μ L syringe arrived. Similar to the optimum volume determination procedure, the "Sample Analysis" button was pressed prior to any injection of the 4.0 μ L samples. The button was pressed again after the injection was complete. Triplicates of each sample were performed, and the resulting titrant volume, time required for titration and the water content were recorded.

To qualitatively identify of the presence of halogens, preparation for the silver nitrate test began on the second week of the Problem-based Lab on January 23, 2014. The qualitative nature of this portion of the laboratory indicates that neither volume of the halogens present nor the specific halogen components present in the waste streams are identified. Since the non-halogenated samples are potentially toxic upon inhalation and may be volatile, the experiment was conducted under a fume hood for the duration of the experiment.

During this time, the group decided to investigate the impact (if any) of centrifuging the samples on the silver nitrate test. To do this, two centrifuged and non-centrifuged samples were prepared to be tested with silver nitrate test. The silver nitrate test was carried out by mixing 5.0 mL of centrifuged sample with 1.0 mL of dilute nitric acid (0.1M) in a 15 mL vials. The mixtures were shaken vigorously before letting it settle for approximately 5.0 minutes. The sample's cover was tightened tightly to prevent vapor escape. Afterwards, an additional 1.0 mL of silver nitrate (0.1M) was added. The mixture was again shaken vigorously mixing the solution into the sample and allowing for settling (0.5 hr). Upon comparing the initial silver nitrate tests, it was determined that there was no difference between centrifuging and not centrifuging the samples.

The remainder of the silver nitrate test was carried out with all non-halogenated labeled waste solvents once more dilute nitric acid was available. The test on the waste samples is described previously. If there were any formation of precipitate, it would be observed at the bottom of the test tube, similar to that of Figure A3. This indicates that the non-halogenated samples tested contained chloride, bromide or iodide. All of the observations were recorded and listed in the Table A4.

This experiment utilized four analytical syringes (0.5 μL , 30 μL , 50 μL , 100 μL) with errors of $\pm 0.1 \mu\text{L}$, $\pm 0.5 \mu\text{L}$, $\pm 1 \mu\text{L}$, and $\pm 2 \mu\text{L}$ respectively.

Results and Analysis

Twenty non-halogenated waste samples were collected from the ESF on January 9th, 2014 from various generators across campus (see Table A1). The generators vary in locations - some generators are from the Pharmaceutical Sciences Building, Life Sciences Building or Food Sciences Building. The samples collected varied in homogeneity and transparency demonstrating the need for a flexible procedure in determining water content.

Prior to analyzing the samples, an effective procedure needed to be developed to evaluate all samples consistently and within the available time frame. Using the Karl Fischer Volumetric Titrator, various volumes of pure distilled water were analyzed to obtain an estimate of the upper time limit associated when testing samples.

Titrant volume (mL), time (s) and final water content (%) were recorded to observe a correlation between the three values and gauge the accuracy of water content with what was outputted from the device. The density of the water was taken to be $1 \text{ g}\cdot\text{mL}^{-1}$ as it was a requirement for the system.

Upon experimentation in the preliminary stages, water content analysis for smaller pur water sample volumes was found to be associated with greater error ($>100\%$ water content) given its greater potential to deviate from the target volume. Hence it can be determined that volumes are underestimated when entering it into the automated titrator. When observing the values, note that three syringes of varying volume were used for this initial analysis, 0.5 μL , 50 μL , and 100 μL . The 50 μL and the 10 μL were not ordered until much later into the experiment. The 0.5 μL syringe, typically used for gas chromatography, was found to be irrelevant to this experiment for the injection of 1.0 μL . Upon injection, the sample from the 0.5 μL syringe would

adhere to the needle tip therefore, not ever entering the test solvent. The 100 μL syringe was the second smallest syringe available to the lab but was associated with error in total volume injected due to its lack of precision for much smaller volumes. Ultimately, 4.0 μL was selected as the sample size because the 100 μL syringe was able to obtain a consistent degree of precision based on the titrator results and was able to completely inject the sample into the test solvent. The experiment was later tested with a 50 μL syringe set to 4.0 μL to obtain even more accuracy from the equipment.

Alternatively, volume was also entered according to mass transferred to test the effectivity of this method. Prior and after injection, the syringe used for transfer was weighed using an analytical balance, and assuming the density of the solution to be that of water, 1.0 g/cm^3 , the true volume injected was calculated. Using this method however led to inaccurate readings for water resulting water content greater than 100% which is impossible. Therefore, this procedure was deemed unfit for the remainder of the experiment resorting to volume determination based on aligning the meniscus to the target volume of the syringe. Contributors to errors associated with the mass difference method could be attributed to the density assumption of 1.0 g/cm^3 .

In addition to evaluating the time dependency of varying sample sizes of pure water, constant sample sizes of varying compositions of water was also analyzed for the accuracy of water content. The raw data recorded (Table A2) from these results can be taken as insignificant as a 100 μL syringe was used to process these results whereas a 50 μL and 30 μL syringe was used to evaluate the water content of the various waste samples.

Twenty non-halogenated waste samples from the ESF were analyzed for water content using the same method used to analyze the pure water sample previously. Locations of these generators were distributed across the Food Science Building, the Pharmaceutical Sciences Building and Life Sciences Centre. As previously, water content, titrant volume and time was recorded for each trial run for the twenty samples with three trials were samples to ensure consistency.

The water content of the tested samples varied from 0.13% to 88.69% water demonstrating that there is a great deviation of water content of all samples being processed at the ESF. It took an average of 8.06 minutes for the titrator to analyze 4 μL of sample. Three trial runs for each sample were performed to assess the precision of this method. An average

standard deviation of 0.924% water was determined for each sample showing that this method of determining water content is indeed precise for the needs required by the ESF. The average titrant volume is 0.248 mL is used to roughly calculate the feasibility of this experiment as discussed further within this report.

A distribution plot can be observed in Figure A6. As shown, a majority of the samples collected (35%) fall in the range of 0-5% water, whereas the other samples tend to be at either ends of highly diluted (50-90% water content) or low water content (15-30% water content). The ESF should take this distribution into consideration when evaluating potential options for handling samples with greater water content.

In addition to sample findings, the method for determining water content was also evaluated. As shown in Figure A6, both time and titrant volume can be related to the actual water content present in a sample. Considering titrant volume, a linear correlation ($R^2=0.999$) is found following an expression:

$$\text{titrant volume (mL)} = 0.0083 \times \% \text{ water content} + 0.0053$$

The linear correlation is expected as Karl Fischer Volumetric Titration relies on volume added to in turn calculate the total water content in the analyte. Moreover, the time dependence appears to correlate logarithmically with a line of best fit ($R^2=0.915$) equating to:

$$\text{time} = 148.22 \times \ln(\% \text{ water content}) + 122.18$$

Note that this correlation is only valid for the current default settings of injection method for the Karl Fischer Volumetric Titrator used for this experiment. The sampling settings can be found in Table A3.

The method proves precise but provide limitations given the time constraints given the time it takes to analyze a sample correlated with the water content which is unknown. The control experiment shows that the method is indeed accurate, but with the unknown nature of each waste sample, the true water content cannot be for certain given the limitless side reactions that could potentially allow for errors in the water uptake calculation the titrator preforms.

To improve this experiment, one can incorporate a more automated way to inject a fixed volume of sample into the test environment to reduce human error. Moreover, the actual density of the sample can be determined to provide further accuracy to the experiment. All of the water content data collected is associated to some other due to the assumption that all solvent waste possesses the density of water, which is not true.

As a control measure, the silver nitrate test aims to test for halogens in the samples labeled non-halogenated. Only eighteen out of the twenty were tested. The two samples that were not tested had some technical issues pertaining to the nature of the samples, such as lack of transparency and opaqueness, and side reactions within the solution, leaving the solution difficult to qualitatively observe precipitate formation. According to Table A4, eight of the samples labeled non-halogenated contained chloride, bromide or iodide halide ions. However, the silver nitrate test cannot identify fluoride ions in the samples since silver fluoride does not precipitate due to its solubility in water. Various samples were also observed to contain an immiscible layer of solution. It is possible this layer is oil, and may be present due to inappropriate disposal of oily wastes through non-halogenated containers by the generators. In attempt to minimize the waste to be treated, the volume of sample, dilute nitric acid and silver nitrate used were scaled down to 5:1:1 mL. In addition, a comparison test between centrifuged and non-centrifuged samples found that the results were the same. However, to simplify the procedure and improve the fluidity of the experiment, all samples were centrifuged prior to the silver nitrate test as centrifugation was required to remove impurities for titration.

Experimental errors may be caused by few assumptions which were made during the experiment. Firstly, this experiment assumed the only substance that can be react with Hydranal-composite 5 titrant in the samples was water. As the composition of the samples were unknown during the experiment, the possibility of other compounds present which may oxidize with iodine were neglected. Such compounds include MnSO_3 , metal oxides and triphenylphosphines. The presence of any of the aforementioned compounds in the waste samples have the potential to compromise the accuracy of the test. Secondly, samples used in the experiment were all assumed to be uniform and well mixed. Prior to the experiment, all the samples were centrifuged to get rid of precipitates and impurities. How well is the water distributes itself among the sample was unclear. During the experiment, all the samples were shaken before injection to minimize the error. Given that 4.0 μL was taken from a solution of 30 mL, homogeneity poses as a significant factor contributing to the titrator's precision when determining the sample's water content. For example, one of the sample (S08120363) possessed two layers naturally which reformed quickly. Although the collection was done as soon as possible (5-10 seconds was needed to ensure there is no air bubbles inside the syringe), the water distribution was still associated with large variances. Moreover, human error during the sample collection and injection will also contribute

to result in inaccuracies. With triplicate trials performed for each sample, the errors were minimized, variance observed and the reproducibility of this experiment was evaluated.

Scale-up and Proposed Design

As a chemical engineer, scale-up is often a consideration when designing processes that derive from lab scale experiments. For the scope of this experiment, optimal scale-up would require designing a feasible process that would be able to handle the numerous samples being processed at the ESF (725 samples collected over three months). Currently in place at the ESF, samples are selected at random and undergo a silver nitrate test to identify for halogens but do not attempt to identify water content. From the proposal, the intent of the performed experiment was to identify whether the Karl Fischer volumetric titration method would be a feasible procedure to be implemented in in the ESF lab.

The experiment uses an HI 903 Karl Fischer Volumetric Titrator estimated at (\$6,000-7,000 CAD). A 10 μ L syringe was purchased for this experiment (\$40 CAD) to sufficiently inject the sample into the titration vessel. Moreover, consumables for this experiment, Hydranal-composite 5 and methanol, were found to cost \$130 and \$40 respectively on a basis of 1L for each. Assuming the above mentioned experimental conditions, each sample costs approximately \$0.10 in consumables with an initial capital cost of \$7,000.

Several factors should be recognized for this study prior to assessing the feasibility. Firstly, the sample costs were calculated (See Appendix B) with the assumption that 15 samples can be analyzed over a period of four hours requiring approximately 16 minutes for sample preparation and testing using the Karl Fischer Volumetric Titrator for each respective sample. It is likely that the lab technician will not be attending to a series of samples for four hours seeing as they would have additional responsibilities to pertain to throughout their shift. However, with the automation of the Karl Fischer Titrator, one would easily be able to multitask while awaiting water content results from the equipment. Moreover, the degree of accuracy as analyzed by the equipment is far beyond what is required by ESF standards. Investigating alternative means to acquire a rougher estimate of water content could potentially streamline the time required per sample.

Based on the design requirements to scale this procedure to one that would be used at the ESF on a regular basis, the proposed design seems unlikely to be feasible given the time

constraints and equipment costs required to analyze the water content of water. Given that the ESF is particularly interested in samples with 95% and none of the samples collected possessed a water content greater than 95%, the ESF would not benefit from this method as way of reducing waste treatment costs.

However, the performed experiment has provided further insight to efficiently perform a Karl Fischer Titration to sample water content on an unknown waste sample. An alternative HYDRANAL® Moisture Test Kit (\$210 CAD) seems like a viable option for the ESF given that both methods utilize similar chemical mechanisms to determine the water content (“HYDRANAL”, 2014). The proposed test kit lacks the automation and accuracy associated with the Karl Fischer Volumetric Titrator because the test kit utilizes a colour indicator for determining water content rather than electrical conductivity. However, further investigation would be necessary to evaluate whether the proposed test kit would be a viable option for the ESF to use at their test facility.

Experience from the previously performed experiment would be useful in developing an efficient procedure for determining water content, knowing approximately how much titrant (250 μL) would be required for the recommended sample size (4 μL). Samples of particular transparency, as the ESF is particularly interested in >95% water content, could be individually tested quickly. As a result, the ESF would have a means of determining the water content distribution of the waste that they process and act accordingly.

Additional non-destructive methods have been investigated to assess potential options that would be feasible for the ESF. Infrared spectroscopy is a flexible means of detecting water content by assessing the solution’s absorbance to light and relating it to water content. However, this method would pose difficulties given the diverse and unknown nature of the samples processed to determine a water content to absorbance relationship. Refractometry is a method that measures moisture content of oils and syrups based on its refraction of light. From the previous experiment, experience has shown that not all of the waste solutions processed are fully transparent or are solely composed of oils and/or syrups which is ideal conditions for refractometry.

Other methods were not considered for recommendation as they were often specific to a particular type of solution (inorganic or organic) or often dealt with determining moisture

content of vapour solutions or through oven drying which would not be recommended due to the nature of the solutions processed.

Safety and Environmental Issues

This problem-based laboratory involves testing samples with unknown compositions. The samples, labeled non-halogenated, may be contaminated and may carry volatile, corrosive and carcinogenic properties. As a chemical engineer, safety and environmental issues are always considered in any project design and operation. If the operation does not meet the regulatory framework provided by the municipal, provincial, and federal governments, there are potential liabilities to human health, the environment, economic risks or charges, and may result in plant shut-down.

Prior to the experiment, members of Group 6 familiarized themselves with the MSDS of the known chemicals used in the experiment (including silver nitrate, ethyl acetate, dilute (0.1M) nitric acid, methanol and Hydranal-composite 5) - which can be found in Appendix D3 (“Chemicals”, 2014). In addition, the team reviewed standard lab safety protocol, such as understanding emergency shut-down procedures, the location of the emergency eyewash stations, the first aid kits, and the fire extinguishers. The chemicals are handled with proper Canadian OHSA (Occupational Health and Safety Act) approved personal protective equipment (PPE) including chemical-resistant nitrile gloves, cotton knee-length lab coats, safety goggles/glasses and under a fume hood to prevent inhalation of toxic vapors from the samples (“Safety”, 2014). Safety audits were conducted during each laboratory session and was updated and compiled over the course of the problem-based lab (see Appendix D1 & D2).

Twenty non-halogenated samples were collected from the storage facility at the ESF by members of group 6 under a fume hood using proper personal protective equipment. The samples were labeled and stored in separate vials, a plastic bag under the fume hood. One of the samples that was believed to be sealed tightly lost a considerable amount of volume several days after the sample collection (see Figure D2) , indicating that the sample is not only volatile and the cap was not sealed tightly for that sample, but there is always an element of human error in the laboratory. As a result of the loss in volume, the sample volume required for the silver nitrate test was reduced, and thus the silver nitrate test was scaled down proportionally as it is only a qualitative test.

The silver nitrate test required a dilute solution of nitric acid (~0.1 M). However, the laboratory only had a stock of strong nitric acid (~0.95 M) and upon reviewing the MSDS sheets for nitric acid, it was discovered that water cannot be added to the strong acid for dilution purposes as it results in a reaction that produces heat and toxic, corrosive and flammable vapors. As a result, the team requested for Mr. K. Wong to prepare dilute nitric acid, resulting in a delay in the silver nitrate test. The delay resulted in a slight change in previously planned laboratory procedures - two random non-halogenated samples were selected and the silver nitrate test was conducted and compared for two trials: the raw sample and the centrifuged sample. The Karl Fischer titration required the samples to be centrifuged to remove suspended salts in the solution, greatly reducing the risk of internal corrosion in the Karl Fischer titration apparatus. This test allowed for a qualitative analysis to compare the differences between centrifuging and not centrifuging the samples. The results found that there was no apparent difference between the two, and all samples were centrifuged in the remaining laboratory time to prepare for the Karl Fischer titration.

The silver nitrate test proposed utilized 15 mL of the sample stock in a 1:1:5 ratio of nitric acid to silver nitrate, to the non-halogenated sample. However, the silver nitrate test is identified as a qualitative test, and a smaller volume for the test would suffice as the expected results would be the same. A smaller total volume of 7.0 mL was selected (1 mL nitric acid, 1 mL silver nitrate, and 5 mL of the non-halogenated sample) because a reduction of the total volume of waste generated for the test means there will be less volume to treat at the ESF. This is also important in terms of scale-up of the silver nitrate test at the ESF. The ESF handles more generators than the scope of this laboratory, so the facility would need to treat much more waste for the same qualitative test.

The samples and their wastes generated in the experiment are to follow UBC Hazardous Waste Management Procedures outlined by the ESF. The wastes are disposed of by segregating based on chemical classification (halogenated and non-halogenated) in the corresponding tagged red-jerry cans (“Non-hazardous”, 2014). The cans are retrieved by the ESF and shipped to the contracted Sumas Environmental Services, who sends the solvents out for testing for appropriate treatment based on chemical classification. If the solvent is halogenated, it is sent to Nisku, Alberta for treatment costing about \$1.65/L. If the solvent is non-halogenated, and a flame test yields positive results, the solvent can be used as a fuel additive as an environmental alternative

to treat the solvent. However, solvents with high water content are typically non-flammable, and inhibit the ability for the solvent to make a good agent for the blend. Therefore, the water content for non-halogenated streams influences not only the disposal costs of the ESF, but also the method of treatment for the non-halogenated solvent. Should the water content of the non-halogenated stream be greater than 95% and is proven non-toxic in the Bioassay fish test (conducted by Maxxam Analytical Labs), the stream is considered safe for disposal via sewage and is treated as contaminated waste water at \$0.31/L. The non-halogenated blends that fall into neither of the above mentioned categories for non-halogenated wastes are treated by Sumas Environmental Services directly at the cost of \$0.85/L.

The titrant, Hydranal-composite-5, used in the Karl Fischer titration, consists of specific chemical components and is disposed of separately, using the Chemical Waste Inventory System (CWIS). The chemical composition and components are entered into the system by Mr. K. Wong, the laboratory technician. The CWIS ensures that the disposal of the wastes operate in compliance with the municipal bylaws, and provincial and federal regulations and policies. The ESF then trucks the segregated generated waste streams to the treatment facility, and sends the solvent streams to be analyzed at a lab to determine the proper method of disposal or treatment.

In addition to solvent and solution wastes produced in the laboratory, the pipette tips, syringes and vials used are stowed in a labeled plastic bag in the lab. The used equipment is labeled with contaminants and is to be sent off-site for incineration to ensure there is no environmental contamination.

If the Karl Fischer titration method used in this problem-based laboratory to determine water content is implemented at the ESF, the treatment of the waste streams would be similar to the above discussed treatment methodologies. Though only a small volume of sample is required for the Karl Fischer titration, a fairly large volume of titrant solution is required for the test. Since the ESF handles significantly larger quantities of generator waste than that of the scope of this laboratory, and the titrant waste generated will proportionally increase. The moisture kit (discussed in the scale-up section) is an alternative to the apparatus used in this PBL, produces similar wastes to the titration method selected (“HYDRANAL”, 2014), but without further testing, the kit may not necessarily produce fewer wastes to treat post-testing.

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Nomenclature

Symbol	Definition	Units
t	Time	s
V	Titration volume	mL
ρ	Density	g/cm^3
M	Molarity	mol/L

Appendix A: Raw Tables and Figures



Figure A1: Non-halogenated solvent storage at ESF



Figure A2: Karl Fischer Volumetric Titrator Apparatus

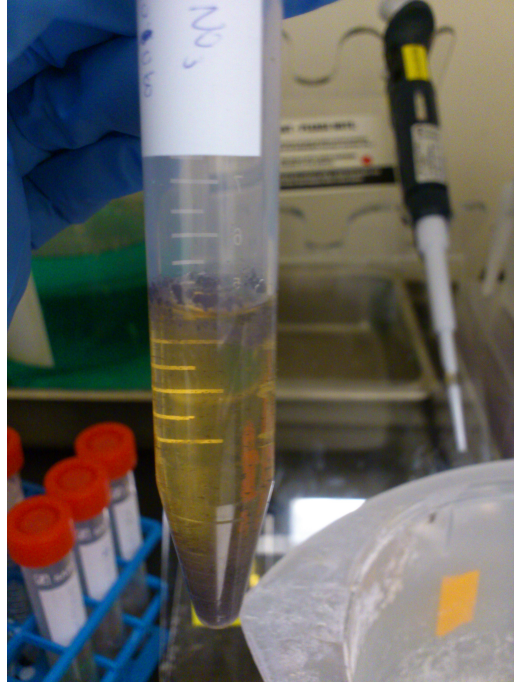


Figure A3: Sample S081203060 Forming Small Sand Like Solid Deposit at The Bottom of The Vial (precipitation) for Silver Nitrate Test

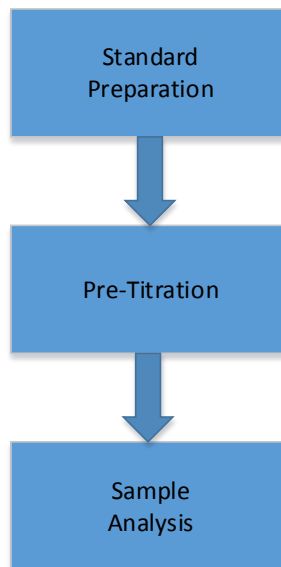


Figure A4: Flow Chart for Procedure Development

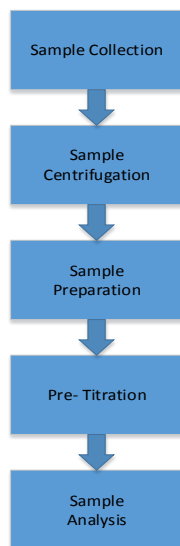


Figure A5: Flow Chart for Sample Water Content Determination

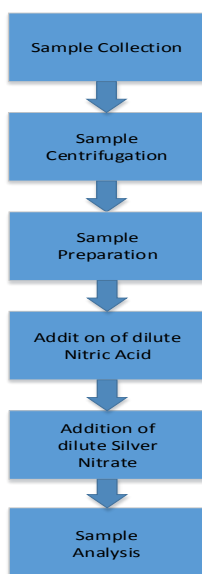


Figure A6: Flow Chart for Qualitative Determination of Halogen Content

Table A1: Generator ID and Sample ID

Generator ID	Sample ID
2350 Health Sciences Mall 4.450 (Brooks Lab)	S081201108
2350 Health Sciences Mall, 4.450 (Brooks Lab)	S081201147
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203823
2350 Health Sciences Mall 4.450 (Brooks Lab)	S081201146
2350 Health Sciences Mall 4.450 (Brooks Lab)	S081203802
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203824
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203070
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203838
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203063
Not found	S081203180
Family & Nutritional Science Building, 2205 East Mall Vancouver	S081203194
Not found	S081202078
4 th Floor, Life Sciences Centre, 2350 Health Sciences Mall Vancouver	S020703422
Life Sciences Centre 2.320, 2.359	S0111006739
4 th Floor, Life Sciences Centre, 2350 Health Sciences Mall Vancouver	S020703420
Food Science Building, 6650 N.W. Marine Drive Vancouver	S081203189
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203062
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203061
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081203060
Pharmaceutical Sciences Building 2405 Wesbrook Mall	S081201140

Table A2: Karl Fischer Titration

Sample Composition (%)		Volume Size (µL)	Titrant Volume (mL)			Time (s)			Concentration of Water Detected (%)		
Water	Ethanol		Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3
100	0	4	0.9914	0.8869	0.8294	808	768	718	121.396	108.459	101.428
50	50	2	0.3198	0.219	0.2617	597	491	525	76.2605	50.5561	60.9533
30	70	4	0.2675	0.2611	0.3149	455	466	510	32.0095	31.1845	37.7658

Table A3: Percentage of Water Content, Titrant Volume and Time for Different Non-Halogens Sample

Generator ID	Trial Number	Titrant Volume (mL)	Time (s)	Water Content (%)
S081201108	1	0.0081	96	0.8207
	2	0.0096	106	0.9050
	3	0.0090	89	0.7588
S081203823	1	0.4269	729	50.7396
	2	0.4439	717	52.9077
	3	0.4521	724	51.9138
S081203189	1	0.1455	446	16.5847
	2	0.1456	452	16.5787
	3	0.1430	428	16.3368
S081203802	1	0.0028	59	0.1317
	2	0.0033	61	0.1870
	3	0.0035	45	0.2758
S020703420	1	0.6431	828	76.4661
	2	0.6324	799	75.2598
	3	0.6132	772	72.9972
S081203061	1	0.0197	199	2.2960
	2	0.0227	239	2.6363
	3	0.0197	201	2.6081
S081203062	1	0.0299	240	3.1344
	2	0.0315	231	3.3680
	3	0.0284	228	2.9769
S081202078	1	0.4588	690	55.6188
	2	0.4533	715	54.8688
	3	0.4781	658	58.1206

Generator ID	Trial Number	Titrant Volume (mL)	Time (s)	Water Content (%)
S081203180	1	0.1796	474	21.2681
	2	0.1777	465	21.0562
	3	0.1807	470	21.4187
S081203060	1	0.0396	290	3.6882
	2	0.0406	285	3.8351
	3	0.0379	241	3.2281
S0111006739	1	0.1292	429	13.6537
	2	0.1162	406	12.1629
	3	0.1242	440	14.0416
S020703422	1	0.6532	844	77.9352
	2	0.5904	761	69.7975
	3	0.6720	809	80.4450
S081203838	1	0.5535	918	67.2272
	2	0.5405	932	66.1062
	3	0.5352	900	64.6691
S081203194	1	0.2304	601	26.7936
	2	0.2384	610	27.7636
	3	0.2394	610	27.8886
S081203063	1	0.0225	210	2.1125
	2	0.0212	214	1.9429
	3	0.0263	233	2.5046
S081201140	1	0.0991	422	10.9839
	2	0.0985	417	10.9225
	3	0.1012	412	11.2829
S081203824	1	0.4209	751	50.7319
	2	0.4283	766	51.6163
	3	0.4241	749	51.1432
S081201147	1	0.1830	536	21.5350
	2	0.1770	520	20.5541
	3	0.1770	502	20.6085
S081203070	1	0.6989	891	83.7858
	2	0.7377	880	88.6894
	3	0.7309	864	87.8941
S081201146	1	0.0105	102	0.8078
	2	0.01	127	0.8697
	3	0.013	116	1.1991

Table A3: Karl-Fischer Titration testing parameters

Sample Size	0.0040	mL
Sample Density	1.000	g/L
Sample Type	Volume	-
Sample Determination	Normal	-
Maximum Duration	3600	S
Maximum Titrant Volume	50	mL
Termination Criterion	Relative Drift	-
Relative Drift	10.0	µg/min

Table A4: Precipitation of different non-halogens samples

Generator ID	Precipitate Presence	Observation
S081201108	Y	
S081201147	N	
S081203823	N	
S081201146	N	
S081203802	N	
S081203824	N	
S081203070	*cannot do test*	The solution appear to be milky (lack of transparency)
S081203838	Y	
S081203063	Y	
S081203180	Y	
S081203194	Y	
S081202078	N	
S020703422	N	
S0111006739	*cannot do test*	The solution reacts with dilute nitric acid and form precipitate before the addition of dilute nitrate acid
S020703420	N	
S081203189	N	
S081203062	Y	
S081203061	N	
S081203060	Y	
S081201140	Y	

Table A5 Karl Fischer Titration Sample Results, Averaged

Sample ID	Titrant volume		Sampling Time		Water Content	
	Average Vol (mL)	STD	Average Time (s)	STD	Average Water Content	STD
S081201108	0.0089	0.0008	97	8.5440	0.8282	0.0734
S081203823	0.4410	0.0129	723	6.0277	51.8537	1.0853
S081203189	0.1447	0.0015	442	12.4900	16.5001	0.1414
S081203802	0.0032	0.0004	55	8.7178	0.1982	0.0727
S020703420	0.6296	0.0152	800	28.0060	74.9077	1.7611
S081203061	0.0207	0.0017	213	22.5389	2.5135	0.1889
S081203062	0.0299	0.0016	233	6.2450	3.1598	0.1968
S081202078	0.4634	0.0130	688	28.5715	56.2027	1.7027
S081203180	0.1793	0.0015	470	4.5092	21.2477	0.1821
S081203060	0.0394	0.0014	272	26.9629	3.5838	0.3167
S0111006739	0.1232	0.0066	425	17.3494	13.2861	0.9918
S020703422	0.6385	0.0427	805	41.6693	76.0592	5.5661
S081203838	0.5431	0.0094	917	16.0416	66.0008	1.2823
S081203194	0.2361	0.0049	607	5.1962	27.4819	0.5994
S08120363	0.0233	0.0027	219	12.2882	2.1867	0.2881
S081201140	0.0996	0.0014	417	5.0000	11.0631	0.1928
S081203824	0.4244	0.0037	755	9.2916	51.1638	0.4426
S081201141	0.1790	0.0035	519	17.0098	20.8992	0.5513
S081203070	0.7225	0.0207	878	13.5769	86.7898	2.6317
S081201146	0.0112	0.0016	115	12.5300	0.9589	0.2103

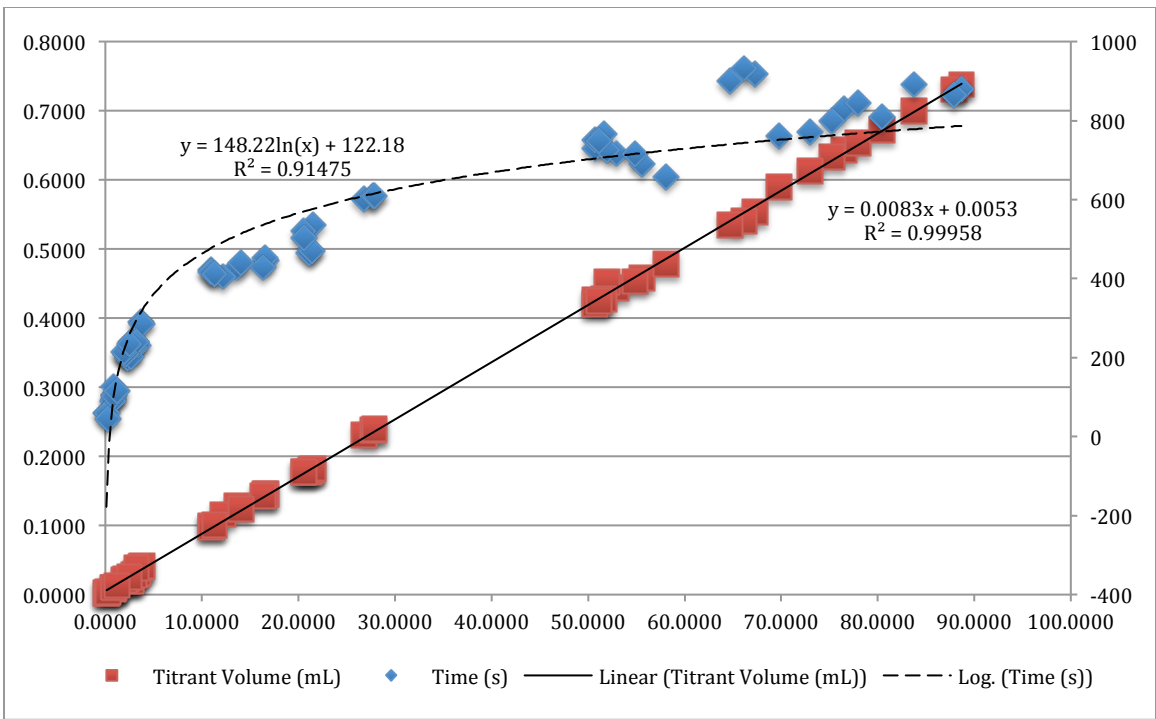


Figure A6: Karl Fischer Titration Sample Results Plot

Appendix B: Raw Calculations

Determination of Silver Nitrate Powder Weight

To determine the silver nitrate powder weight needed to prepare 0.1M silver nitrate solution with 250 ± 0.02 mL of volumetric flask, the equation below is used.

$$\text{Molarity (M)} = \frac{\text{moles of solute (mole)}}{\text{litres of solution (V)}}$$

$$\text{mole} = M \times V$$

$$\begin{aligned} \text{Weight (W)} &= M \times V \times \text{Mw} \\ &= \frac{\text{mol}}{\text{L}} \times \text{L} \times \frac{\text{g}}{\text{mol}} \end{aligned}$$

Where: Mw = molecular weight (AgNO_3 Mw = 169.8731 g/mol).

Sample calculation:

$$\begin{aligned} \text{Weight of AgNO}_3 \text{ powder} &= \frac{0.1 \text{ mol}}{\text{L}} \times 250 \pm 0.02 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times \frac{169.8731 \text{ g}}{\text{mol}} \\ &= 4.25 \pm 0.02 \text{ g} \end{aligned}$$

Handwritten Sample Calculation. $\sum x = 5028.0 = n \bar{x}$

Standard Deviation: $(2825.0 + 0229.0 + 5028.0) = n \bar{x}$

Standard Deviation shows how much variation or dispersion from the average. A high standard deviation indicates that the data points are spread out over a large range of values. We use standard deviation to test the reliability of the results.

$\sum x = 5028.0$ $\sum x^2 = 1422123.0$

The equation used to calculate the standard deviation is shown below:

$$\text{Standard Deviation} = \sqrt{\frac{n \sum x^2 - (\sum x)^2}{n(n-1)}}$$

x: value of each data.

n: number of data.

We use this method to calculate the standard deviation for titrant volume, time, and water content.

As an example; we the standard deviation for water content of Sample ID: 5081201108.

Test result:	Trial.	Water Content (%)
	1	0.8207
	2	0.9050
	3	0.7588

$$\text{Average Water Content} = \frac{0.8207 + 0.9050 + 0.7588}{3} = 0.8282 \%$$

$n = 3$.

$$\sum x^2 = 0.8207^2 + 0.9050^2 + 0.7588^2 = 2.068351$$

$$(\sum x)^2 = (0.8207 + 0.9050 + 0.7588)^2 = 6.17274$$

$$STD = \sqrt{\frac{2 \times 2.068351 - 6.17274}{2 \times (3-1)}} = 0.0734\%$$

The result in the report is shown as

0.8282% with STD of 0.0734%

$$\frac{(\sum x^2) - \frac{(\sum x)^2}{n}}{(n-1)}$$

$\sum x^2$: value of each data
 n : number of data

Water Content (%)	Time
0.8207	1
0.9050	2
0.7588	3

$$\therefore \frac{0.8207^2 + 0.9050^2 + 0.7588^2 - \frac{(0.8207 + 0.9050 + 0.7588)^2}{3}}{3} = 0.0734\%$$

Appendix B: Calculations

B3 Capital Cost

Karl Fischer Titrator	\$ 7000
10 μ L syringe	\$ 40

Consumables

Hydranal-composite-5(1L)	\$ 130
Methanol (1L)	\$ 40

15 samples tested in 4 hrs

\therefore $\frac{1 \text{ sample}}{16 \text{ minutes}}$

1 sample on average consumes: 0.25 mL

$$\therefore \frac{\$ 130}{1000 \text{ mL}} \times 0.25 \text{ mL} = \$ 0.0325$$

One day of testing requires 50 mL methanol

$$\therefore \frac{\$ 40}{1000 \text{ mL}} \times 50 \text{ mL} = \$ 2$$

15 samples cost

$$\underbrace{\$ 0.0325 \times 15}_{\text{Hydranal used}} + \underbrace{\$ 2}_{\text{Methanol used}} = \$ 2.48 \approx \$ 3.00$$

rounded up
for potential
of experimental
error & mistakes

Appendix C: Error Analysis

Sources of Errors

The list shown below is a summary of all potential sources of errors that need to be accounted for during the experiment.

- The four analytical syringes utilized in this experiment (0.5 μL , 30 μL , 50 μL , 100 μL) are associated with their measuring errors of $\pm 0.01 \mu\text{L}$, $\pm 0.5 \mu\text{L}$, $\pm 1 \mu\text{L}$, $\pm 2 \mu\text{L}$ respectively.
- Unwell mixing leading to a concentration difference between different parts of the sample.
- Potential existence of substance that could be reacted with iodine, these substances including metal oxides, triphenylphosphine can lead to a higher water content result than the real value.
- Tiny air bubbles in the syringe while collecting the samples during Karl Fischer Titration, the volume of sample injected into the Karl Fischer beaker will be smaller than the input volume into the machine.
- Possibility of dropping tiny part of the sample onto the wall of Karl Fischer beaker, making the volume of samples reacted with titrant smaller than it should be.
- The accuracy of the silver nitrate test was limited, as it is based solely on human observation. Given the variability in each sample's appearance and the subjective nature of identification procedure, it is possible that the test may result in errors.
- Some sample was evaporating really fast even being kept in the fridge with caps, the rate of evaporation of organic substance and the water inside may not be the same, thus after one or two weeks, the composition of the samples may be different from the original sample.
- Other human error during the sample collection and injection may also cause errors in the results.

Appendix D: Laboratory Safety Documents

Table D1: MSDS for Chemicals Used

Chemical Name	Physical State	Health	Fire	Reactivity	Personal Protective Equipment	Notes
silver nitrate	Solid	3	0	0	Splash goggles, lab coat, vapor respirator, gloves	-non, flammable, stable
						-reactive with reducing agents, combustible material, organics, alkalis
						-very hazardous if ingested (irritant), hazardous skin contact (permeator) upon inhalation
						-slightly corrosive upon skin hazard
methyl alcohol	Liquid	2	3	0	Splash goggles, lab coat, vapor respirator, gloves	-use in proper ventilation, may damage lungs & mucous membranes
						-hazardous in case of skin & eye contact (irritant)
						-flammable liquid, keep away form heat
ethyl alcohol	liquid	2	3	0	Splash goggles, lab coat, vapor respirator, gloves	-violent reaction with oxidizers, metals, acids
						-reactive with oxidizing agents, acids, alkalis
						-highly flammable in presence of open flames & sparks , fire hazard
hydranal-composite 5	Liquid	2	2	-	Splash goggles, lab coat, vapor respirator, gloves	-irritant (skin contact), slightly hazardous - may permeate skin
						-composed of iodine, sulfur dioxide, 2-methylimidazole, imidazole, 2-ethanol, 1H-imidazole, monohydriodide
						-combustible liquid, avoid heat, flames, spares, direct sunlight
						-if inhaled, may cause respiratory tract irritation
nitric acid	liquid	3	0	0	Splash goggles, lab coat, vapor respirator, gloves	-may cause skin & eye irritation, skin allergic reaction and may be absorbed through skin
						-reactive with strong oxidizing agents
						-strong oxidizer, reacts violently with alcohols
						-react explosively with reducing agents, arsine, phosphine, tetraborane
						- incompatible with water (do NOT add), reducing agents, combustible materials, organic materials, metals, acids, alkali, moisture
						-reacts with water/steam to produce toxic, corrosive & flammable vapors + heat
-extremely corrosive in presence of aluminum, copper						
-very toxic material, combustible with fire, corrosive,						



Figure D1: Plastic Bag Storing Used Pipette Tips, Syringes and Vials used in the Experiment

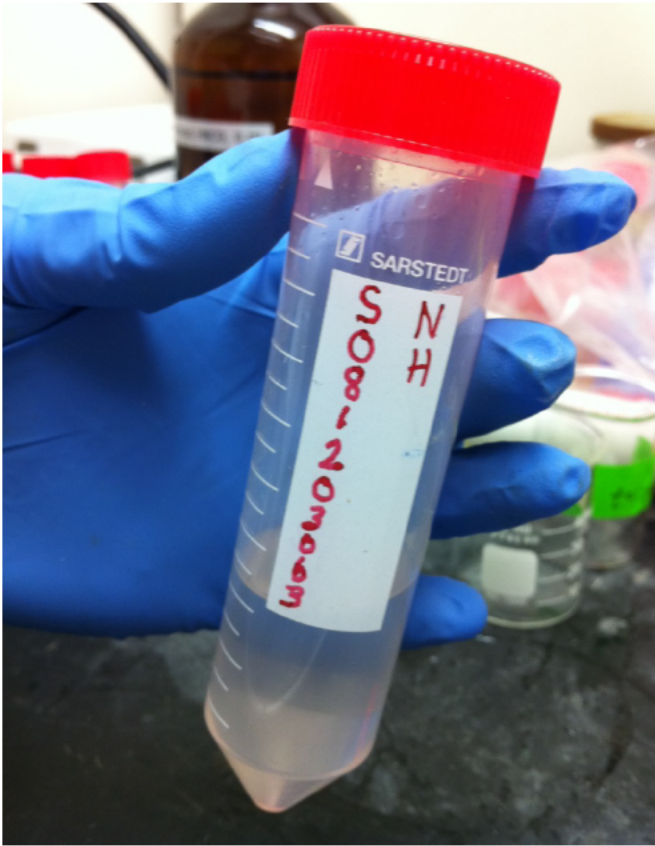


Figure D2: Large Volume of Sample S081203063 Vaporized due to Improper Securing of the Cap

University of British Columbia

Department of Chemical and Biological Engineering

Laboratory Safety Audit Checklist

Emergency/Hazard Information	Yes	No	Comments
Emergency shut-down procedures known for titrator	√		Shut down titrator apparatus & turn the power off
Fire extinguishers present and operational	√		
Fire alarms locations are known	√		
First aid kit present and accessible	√		
Emergency eye-wash stations and showers present and operational	√		
No food or drink in the laboratory	√		
Laboratory supervisor present at all times during experiment	√		Laboratory supervisor: Dr. C. Gyenge
Emergency exits and fire evacuation procedures known and clear	√		
Emergency contacts are known	√		Ivan Leversage (CHBE Safety Officer) - 822-3857 First Aid (UBC Campus) - 822-4444 Fire/Police/Ambulance - 911 Poison Control - 682-5050

General Laboratory Safety	Yes	No	Comments
Work benches kept tidy and clear of spills	√		
Laboratory equipment stowed properly	√		
Aisles in laboratory kept unobstructed	√		
Laboratory samples sealed and stowed properly in secondary containment (either in fumehood or wrapped in foil and stowed in sealed plastic bag in fridge)	√		Prevent toxic vapors from entering laboratory air
Be aware of external events that may occur during the laboratory - particularly incidents that are not caused by this lab	√		Lab environment is a shared workspace, and the work environment may be affected by others using the lab space
Dispose of wastes according to the outlined by the CHBE waste disposal protocol	√		
Chemicals sealed and used stowed in proper cabinets	√		
Chemicals/samples are properly labelled	√		
Proper disposal of used equipment	√		Stowed in large plastic bag with labels identifying contaminants
Workspace is properly lit	√		

Operator Safety	Yes	No	Comments
Proper PPE (splash goggles, nitrile gloves, lab coat)	√		
Closed toed shoes, long hair tied back	√		Not applicable for all group members
Tests conducted/samples tested under functional fumehood/Benchtop Fume Extractor	√		
Review the known chemical MSDS sheets	√		See Appendix D-3
Understand the large margin of possible risks affiliated with chemicals	√		
Understand laboratory procedure prior to entering lab	√		
Students have completed basic Laboratory Safety Training	√		
Flammable liquids kept away from sources of heat	√		