SEGREGATION OF WASTE SOLVENTS QUANTITATIVE ANALYSIS OF HALOGEN CONTENT

Andrew Dusevic, Hanika Rahman, Marc Zinman, Peng Yao Wang, Tyler Tsui

University of British Columbia

CHBE 464

March 28, 2014

THE UNIVERSITY OF BRITISH COLUMBIA

CHBE 464 PROBLEM-BASED-LABORATORY FINAL REPORT

SEGREGATION OF WASTE SOLVENTS

QUANTITATIVE ANALYSIS OF HALOGEN CONTENT

Submission Date – March 28, 2014

Instructor: Dr. Christina C. Gyenge

Submitted by

Group 5

Hanika Rahman (Project Manager)
Peng Yao Wang (Process Engineer)
Andrew Dusevic (Design Engineer)
Tyler Tsui (Quality Assurance Engineer)
Marc Zinman (Safety & Environmental Engineer)

Hanika Rahman	Peng Yao Wang	Andrew Dusevic	Tyler Tsui	Marc Zinman

1020 West 43rd Avenue
Vancouver, BC V6M 2B9
March 28, 2014

Dr. Christina C. Gyenge, CHBE 464 PBL Instructor
UBC Chemical and Biological Engineering Department
2360 East Mall
Vancouver, BC V6T 1Z3

Dear Dr. Gyenge:

Please find attached the Final Report of the CHBE 464 Problem-Based Laboratory (PBL) project entitled "Segregation of Waste Solvents – Quantitative Analysis of Halogen Content". This report describes the final section of a year-long project (September 2013 – April 2014) funded in part by a SEEDS grant from the AMS Sustainability Projects Fund.

The overall goal of this project was to develop a simple in-house protocol for the proper segregation of halogenated and non-halogenated waste solvents collected from different laboratories at the University of British Columbia (UBC) by the Environmental Services Facility (ESF). The goal of this section of the project was to develop a method for quantifying halogen content in waste solvents. The goal was achieved by completing the following tasks, which are described in the report:

- Carried out calibration tests with known concentrations of NaCl
- Applied Volhard's method in quantifying halogen content in waste sample
- Analyzed halogenated samples using Dexsil HydroCLOR-Q test kit

The results of the calibration tests showed that organic liquids do not interfere with the Volhard's method. The calibration tests also helped establish error margins and sources of error that could be expected in later experimentation, which would allow results to be better understood and consistent. From the quantitative analysis of halogens in waste solvents via Volhard's method, it was observed that incorrect labeling was still a major barrier in the proper segregation of the waste samples. Halogenated samples were also tested using Dexsil HydroCLOR-Q test kits, for which it was determined that the results from this kit were conclusive within 200-4000ppm halogen content.

A total of four laboratory sessions (totaling 20 hours) were spent in carrying out necessary experiments. If you require any further information regarding this project, please contact me at hanika.rahman@gmail.com.

Sincerely,
Hanika Rahman
Team 5 Project Manager
UBC Chemical and Biological Engineering, Year 4
Encl. [Final Report]

EXECUTIVE SUMMARY

The purpose of this project is to develop an easy cost effective in-house protocol to quantify the halogen content of waste solvents collected from different laboratories at the University of British Columbia (UBC) for the proper segregation by the Environmental Services Facility (ESF). During this project, two methods were tested. The first method used to determine halogen content was Volhard titration, which was applied to a total of 23 waste samples labeled as non-halogen and halogen. This method quantifies dissolved chloride content by first precipitating silver chloride using silver nitrate, then back titrating excess dissolved silver with potassium thiocyanate using an iron (III) indicator. This method was modified by adding amyl alcohol to segregate precipitate into a separate layer to inhibit the back dissolving of silver chloride. Calibration tests were carried out using standard concentrations of NaCl ranging from 500 to 1500ppm in the presence of the organic liquids, such as amyl alcohol and acetone. It was observed that organic compounds do not interfere with this method. The second method used a Dexsil HydroCLOR-Q test kit. This method is similar to the first as it uses the Volhard's method; however, it also includes several steps in which the samples are cleaned of organics using sulphuric acid and dewatering compounds. This kit was applied to 12 samples and the results were compared against the original proposed Volhard's method outlined above. For scale-up, supplies such as reagents glass ware and other compounds will be scaled up linearly by the number of samples. In addition, it is suggested that an automatic potentiometery titrator be used to minimize employee labor.

The results of the analyzed chloride concentrations from both methods show that many of the samples labeled as halogen and non-halogen received by the ESF are mislabeled. Both methods agreed on which samples were mislabeled; however, the calculated chloride concentrations differed between the two methods. This discrepancy can be attributed to the error of the Volhard's method, which arises from unknown compounds, dissolved solids, and suspended solids within the waste samples. These impurities may have affected the precipitation of AgCl or the reaction between iron (III) and potassium thiocyanate, and can inhibit the ability of the technician to clearly observe the equivalence point. The HydroCLOR-Q test kit cleans samples of dissolved solids and suspended solids, and, therefore, its results are not affected by these compounds.

All experimentation were carried out under a fume hood, with every member of the team familiar with the MSDS of each compound they were handling. Proper PPE for this experimentation included nitrile gloves, googles, and a lab coat. Analyzed samples and reagents were disposed of into halogen waste containers. For the Dexsil HydroCLOR-Q test kit, a reagent was included that rendered the entire kit components within regulatory waste disposal guidelines outlined by the EPA.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

1.0 INTRODUCTION

The UBC Environmental Services Facility (ESF) manages the transportation, storage, disposal and recycling of hazardous materials generated on campus. The waste includes biological waste and hazardous chemicals such as halogenated and non-halogenated solvents (UBC LSC, 2012). Based on the halogen content, these solvents are treated differently in terms of disposal and the disposal costs of non-halogenated and halogenated wastes are 1.25 CAD per jerry can and 1.65 CAD per jerry can, respectively. While non-halogenated solvents can be used as fuel additives in cement kilns, halogenated solvents cannot be reused and are incinerated at high temperatures. Thus, proper segregation of the halogenated and non-halogenated samples minimizes the overall waste disposal cost. Therefore, it is very important to segregate these waste solvents according to their class (UBC LSC, 2012). Figure 1(a) shows the inside of an ESF pick-up truck which collects waste samples from different laboratories across the UBC campus and Figure 1(b) illustrates the ESF storage facility of the segregated halogenated and non-halogenated wastes.

(a) (b)

Figure 1 – Waste Sample (a) collection truck and (b) storage facility at the ESF

Solvents are considered halogenated when the halogen (i.e. chlorine, bromine, or iodine) content is higher than 1000 ppm. However, at the ESF some solvents that were labeled 'non-halogenated' contained more than the maximum allowable halogen content and some halogen samples contained less than the allowable limit. At present, the ESF does not have an in-house protocol to quantify halogen concentration in all of the waste samples. As a result, all non-halogenated waste samples are required to be sent to a halogenated waste disposal facility. This increases the annual disposal costs incurred by UBC. Therefore, a need to develop an in-house protocol to screen the non-halogenated samples from the halogenated ones in order to minimize the disposal costs arises. This also allows the ESF to identify the generators responsible for the mislabeling of waste samples and hence rectifying the segregation problem. Hence, the objectives of this project are to develop a protocol to calculate the halogen content in waste solvents and determine if organics interfere with the developed method. In order to do so, a total of 23 samples, collected from the ESF, were tested and the halogen concentrations in waste samples were quantitatively determined using Volhard’s method and Dexsil HydroCLOR-Q® test kits. Volhard’s method is based on argentometric analysis and requires a laboratory setting for the titration. Dexsil HydroCLOR-Q® is a pocket-sized test kit which contains necessary reagents and glassware to perform the experiments. The reagents are sealed in glass ampule and the kit is highly portable for on-site use.

2.0 THEORY

In this PBL, Volhard's method and Dexsil HydroCLOR-Q® test kits are used in quantifying concentrations of halogens in waste solvents. This section describes the theory behind the methods used.

2.1 Volhard's Method

With the exception of fluoride ions, all of the halides (Cl^-, I^-, and Br^-) as well as the pseudohalides (OCN^-, SCN^-, and CN^-) form insoluble precipitates with Ag^+. Volhard's method is one of the widely used argentometric analyses which are based on the reaction with Ag^+. This method is applicable to acidic solutions with a pH lower than 6.5.

Volhard's method uses a back titration with potassium thiocyanate to determine the concentration of chloride ions in a solution. Before the titration is carried out, an excess volume of a silver nitrate solution is added to the solution containing chloride ions, forming a precipitate of silver chloride, as indicated in Equation 1. The term 'excess' is used as the moles of silver nitrate added are known to exceed the moles of sodium chloride present in the sample so that all chloride ions present react (Volhard's Method, UC, 2014).

$$Ag^+{}_{(aq)} + Cl^-{}_{(aq)} \rightarrow AgCl_{(s)} \quad \text{……………………..Eqn. 1}$$

The indicator Fe^{3+} (ferric ion) is then added and the solution is titrated with the potassium thiocyanate solution. The titrant remains pale yellow as the excess (unreacted) silver ions react with the thiocyanate ions to form a silver thiocyanate precipitate, shown in Equation 2.

$$Ag^+{}_{(aq)} + SCN^-{}_{(aq)} \rightarrow AgSCN_{(s)} \quad \text{……………………..Eqn. 2}$$

Once all the silver ions have reacted, the slightest excess of thiocyanate reacts with Fe^{3+} to form a dark red complex, $[FeSCN]^{2+}$, as indicated in Equation 3. The concentration of chloride ions is determined by subtracting the titration findings of the moles of silver ions that reacted with the thiocyanate from the total moles of silver nitrate added to the solution.

$$Fe^{3+}{}_{(aq)} + SCN^-{}_{(aq)} \rightarrow [FeSCN]^{2+}{}_{(aq)} \quad \text{…………………..Eqn. 3}$$

However, silver thiocyanate is less soluble than silver chloride (Chemistry 311 Course Material). If titration of the excess silver was carried out in the presence of silver chloride precipitate, the thiocyanate would therefore react with the AgCl particles according to the reaction, mentioned in Equation 4:

$$AgCl_{(s)} + SCN^{-}{}_{(aq)} \rightarrow AgSCN_{(s)} + Cl^{-}{}_{(aq)} \quad \text{………………Eqn. 4}$$

This reaction must be prevented for the endpoint to be meaningful. A common method of circumventing this problem is to add a small quantity of organic liquid such as amyl alcohol or nitrobenzene to the solution. The organic liquid coats the colloidal silver chloride particles, preventing them from reacting with thiocyanate. Precipitate solubility is not a problem during determination of I^- and Br^-, as both AgBr and AgI have much lower solubilities than AgSCN.

2.2 Dexsil HydroCLOR-Q® - Quantitative Test Kit

Dexsil HydroClor-Q is a quantitative field test kit designed to measure organic chlorine contamination in oil/water mixtures (HydroClor-Q, 2014). HydroClor-Q measures "organic" chlorine, therefore inorganic chloride from seawater or other sources does not interfere with the test. One kit can be used in testing 12 samples and all premeasured reagents are sealed in glass ampules. Each test takes less than 10 minutes to run and quantifies total organic chlorine in the range of 200 ppm to 4000 ppm. If water concentration in the sample is greater than 70%, the result can be read directly from the titration syringe. This syringe is labeled from 0 to 4000ppm. The reading from the syringe depends on the amount of titrant used to reach the titration end point. For the samples containing less than 70% water and more oil, the following correction is used (Suggestions for HydroClor-Q, 2007) for adjusting the experimental results:

$$\text{True Concentration} = \text{Reading from Syringe} \times \frac{10 + \text{mL oil in sample}}{10} \quad \text{..Eqn. 5}$$

Samples containing more than 80% oil can be tested using Clor-D-Tect Q4000.

3.0 EXPERIMENTAL APPARATUS AND TECHNIQUES

In this PBL, a total of 23 samples were tested using the Volhard's method in three lab sessions (totaling 15 hours). Out of 23, 7 samples were labeled as halogenated while the remaining 16 were labeled as non-halogenated. Another lab session (5 hours) involved testing 12 out of 16 non-halogenated samples using the Dexsil HydroCLOR-Q test kit. These samples were collected from the ESF. All experimental procedures were carried out in the fume food.

3.1 Sample Preparation

A pH of less than 6.5 is necessary for Volhard's method. Therefore, the pH of each waste samples must be tested to determine whether Volhard's method is appropriate for the given sample. The pH of a waste sample was determined by immersing a piece of litmus paper into the sample, as shown in Figure 2. If the pH was found to be above 6.5, a few drops of 3M nitric acid were added to the sample for pH adjustment.

Figure 2 – Litmus paper

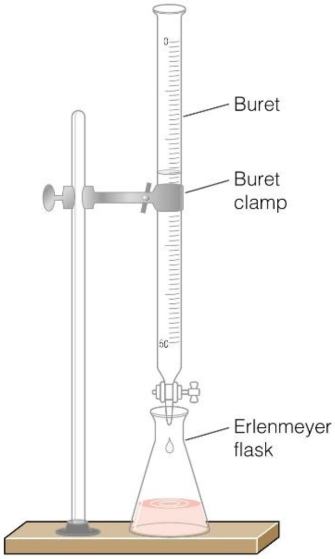


Figure 3 – Experimental apparatus for titration

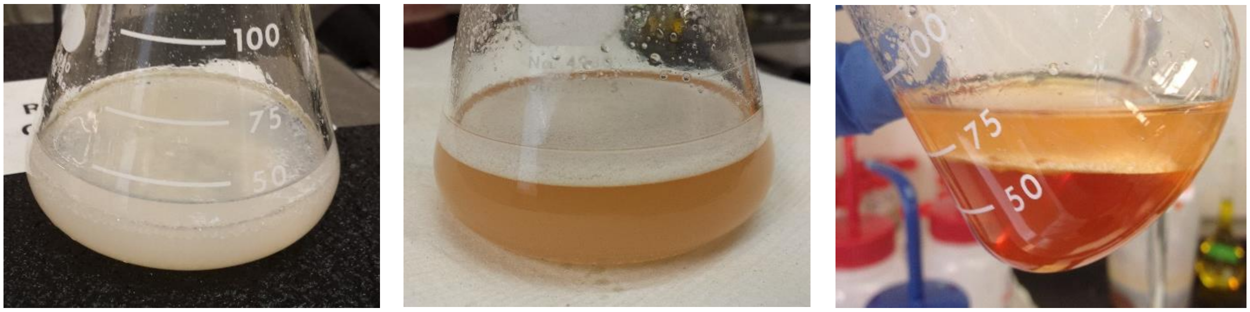
3.2 Titration

Titration was used in carrying out calibration tests and quantitative analysis of halogen content in waste samples using Volhard's method. Figure 3 illustrates the experimental set-up required for the titration.

3.2.1 Calibration

First, 50:50 mixtures of water and acetone, and water and amyl alcohol at different concentrations of NaCl were tested for accuracy. For these calibration tests, the Volhard's method was applied for a quantitative analysis of halogen content in known concentrations of NaCl solutions. To prepare samples for titration, a 50 mL burette was filled with titrant, 0.1 M potassium thiocyanate, and placed in a burette stand inside the fume hood. Next 10 mL of sample was transferred to a 100 mL Erlenmeyer flask and 10 – 15 mL of 0.1 M silver nitrate solution was added to the sample until no more precipitation was formed. Then, 10 mL of amyl alcohol was added to the sample as an organic liquid to prevent the precipitates dissolving back to the solution. Approximately 3 – 5 drops of the indicator, saturated ferric ammonium sulfate solution, was added to the sample. The Erlenmeyer flask containing sample was then placed under the burette and allowed to settle for few minutes so that two immiscible layers (organic and aqueous) were formed. After recording the initial burette volume, incremental volumes of titrant was added to the waste sample solution. The Erlenmeyer flask containing waste sample was periodically shaken by hand after each addition of a small volume of titrant. This was continued until the titration reached the end-point, at which time the solution became a uniform pale peach colour. The final volume of the burette was then recorded and used for chloride concentration calculations. Duplicate titrations (if necessary triplicates) were performed for each waste sample. Figure 4(a) shows an NaCl sample in a water/amyl alcohol mixture before titration, Figure 4(b) depicts an NaCl sample when the titration end point was achieved and Figure 4(c) shows the NaCl sample if it was over titrated.

The concentrations of NaCl solution for the calibration tests were selected such that they were close to the target chlorine concentration (i.e.: 1000ppm) in waste solvent. The concentrations used for NaCl/water/amyl alcohol mixture were 500, 1000 and 1500 ppm. The NaCl/water/acetone samples had chlorine concentrations of 500, 1000, and 1200 ppm.



(a) (b) (c)

Figure 4 – Known concentration of one NaCl sample in water/amyl alcohol mixture (a) before titration, (b) after titration and (c) over titrated

3.2.1 Volhard's Method – In-house Protocol

The detailed step-by-step experimental protocol for the quantitative analysis of halogen content in waste samples via Volhard Titration is included in Appendix C. Figure 5 describes one non-halogenated sample before and after the Volhard titration.

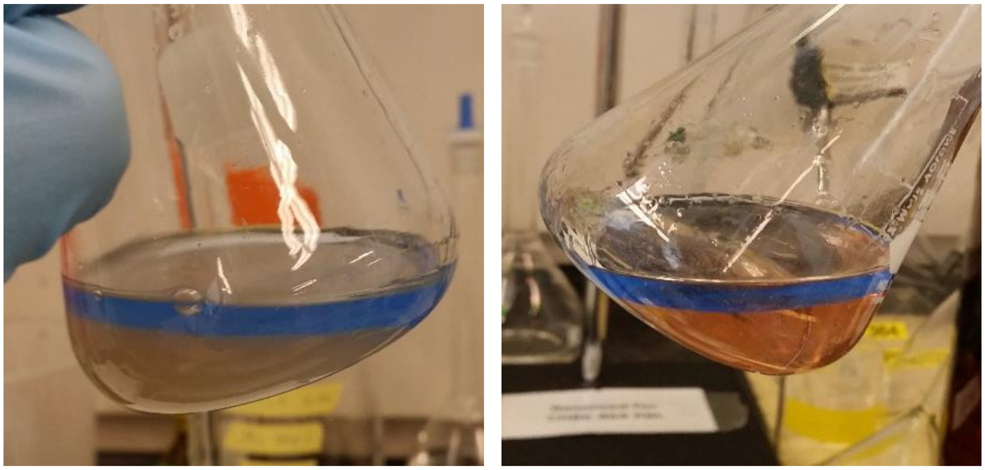


Figure 5 – A non-halogenated waste sample, collected from the ESF, before titration (left) and after titration (right)

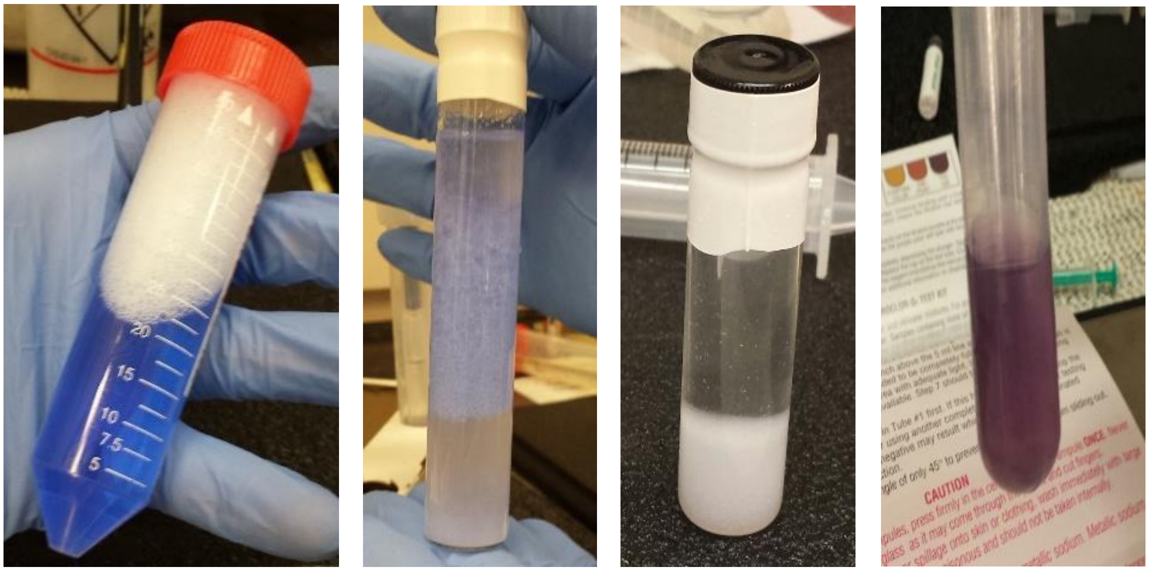
3.3 Dexsil HydroCLOR-Q® - Quantitative Test Kit

A pack of Dexsil HydroCLOR-Q contained 12 test kits which were used for testing 12 waste samples. Each kit included an ampule containing 1 ml of mixing reagent, a large glass vial containing extraction solvent & a white powdered emulsion breaker, a 10 cc plastic sampling syringe, a plastic pipette, a small glass vial containing a white drying agent, a 1 ml polypropylene sampling syringe with a preset volume, a plastic filtration funnel, a plastic titration burette attached to a plastic screw cap, a plastic syringe plunger rod and a glass ampule contained in a cardboard sleeve and plastic tube designated as “Disposal Ampule”. The kit also contained two plastic tubes. Tube #1 was equipped with a white dispensing cap and contained a colourless ampule (bottom) and a yellow-dotted, gray ampule (top). Tube #2 had a clear cap and contained 7 ml of buffer solution and a red-green ampule. Figure 6 includes the experimental set-up for the HydroCLOR kit.

First experimental step was the addition of a mixing reagent (surfactant) to the waste sample to remove any immiscible layers. Using a syringe, 10 mL of the sample was tranferred to the large vial containing extraction solvent. The sample was vigorously mixed and allowed to settle for 3 minutes so that two distinct layers were formed. One-third of the top layer was then transferred to the small vial containing white drying agent. Using 1 mL syringe, 1 mL of sample was then transferred to a vial containinig two ampules. First, the bottom ampule, then the top ampule were broken and the sample was vigorously mixed with the reagents. Clear buffer was then added to the sample and two phases were allowed to separate for 2 minutes. Using a funnel, 5 mL of the clear phase was poured into another vial and titrant was slowly dispensed into the solution. Once the end point was obtained, the chlorine ppm was read off the burette (i.e.: syringe).

Figure 6 – Dexsil HydroCLOR-Q® Test Kit

Figure 7(a), (b), and (c) show a halogenated sample, collected from the ESF, after the addition of the mixing agent, extraction agent and drying agent respectively. Figure 7(d) illustrates the sample after the titration end point was reached. The detailed step-by-step experimental protocol on how to use Dexsil HydroCLOR-Q test kit is included in Appendix C.



(a) (b) (c) (d)

Figure 7 – A halogenated sample after the addition of the (a) mixing agent, (b) extraction agent, (c) drying agent and (d) at titration end point

4.0 RESULTS AND DISCUSSION

The experimental results obtained from the calibration tests, quantitative analysis of halogen content in waste samples via Volhard's method and Dexsil HydroCLOR-Q kit are discussed in this section.

4.1 Calibration Test Results

Calibration tests are carried out in order to study the unknown organic liquid's effects on titration results obtained from the Volhard's method. The experimental results regarding calibration tests with NaCl/water/amyl alcohol are included in Table A1. The concentrations of chloride in the samples are determined based on the calculation included in Appendix B. The errors indicate the percentage differences between the calculated chloride concentrations and the expected values. In this experiment, most of the percentage errors are smaller than 10%, which indicates that amyl alcohol has no effect on the titration result. Therefore, amyl alcohol is a viable option for the use as an organic liquid to isolate AgCl precipitates. Table A3 shows the average titration results and the expected values and Figure A1 shows the calibration curve of amyl alcohol. The small gap between the two curves indicates the negligible effect of Amyl Alcohol on the titration result.

Since acetone is widely used in laboratories for various purposes, a calibration test for an acetone/water mixture was performed. However, since acetone is miscible with water, samples were required to be prepared differently to account for the dilution factor. To make 500 ppm sample and accounting for dilution factor, 5 ml of 1.5 g/L NaCl solution was mixed with 5 ml acetone and 5 ml water. To make 1000 ppm sample, 10 ml of 1.5 g/L NaCl solution was mixed with 5 ml acetone. To make 1200 ppm sample, 12 ml of 1.5 g/L NaCl solution was mixed with 3 ml acetone. The samples were then tested using the Volhard's method. Table A2 shows the titration results for the acetone calibration. The percentage differences of Cl^- concentrations and the expected values are under 15%. Figure A2 shows the calibration curve of acetone. The small difference between the two curves shows that the effect of acetone on the titration result is insignificant. Table A4 shows the average titration results and the excepted values. The differences between them are relatively large compared to previous calibration. The main factor is the absence of the organic layer which restrains AgCl precipitates from dissolving back to the aqueous phase. More details on improving titration accuracy can be found in the quality assurance section of the report.

4.2 Results from Volhard Titration

The titration results for the samples (ID - S081202548, S0111009683, S081201325, S081201327), labeled as halogenated, are shown in Table A5. The titration results for the chloride concentration of samples S081203416, S0881201557 are found to be 0.11 and 0.07 g/L respectively. These concentrations indicate a slight presence of halogens within the solutions, but are well below ESF standards for the classification of halogen. Standards indicate that samples labeled halogenated should have more than 1000ppm, or 1.0g/L, halogen concentration. Thus, samples S081202548, S0111009683, S081201325, S081201327, S081203416, and S0881201557 should be labeled as non-halogenated based on this standard. Only sample S011104038 is found to have chloride concentrations high enough for the classification of halogenated at 7.06 g/L. These results do not come at a complete surprise considering that during chemical disposal, for safety reason, users always dispose samples of unknown composition into the halogenated tank. Thus, most of the halogenated tanks in ESF contain no halogens or relatively small amount of halogens.

Table A6 shows the titration results for non-halogenated samples. Samples S081203194, S081201147, S081203070, S081203180, S081203824, S081203838, S081203823, S081203062, S081203066, S081203189, S070703422, S0111006739, and S081202078 are found to have a chloride concentration lower than 0.1 g/L. Thus, by ESF standard, they are non-halogenated. Samples S020703420 and S081201140 are found to have a Cl^- concentration of 0.82 and 0.96 g/L respectively. Their concentrations are still lower than 1.0 g/L; however, they are very close to the limit. Due to the error in the results found from the calibration curves (which come close to 20%), it is suggested that samples S020703420 and S081201140 should be labeled halogenated. Sample S081203060 is found to have 2.06 g/L chloride concentration which is higher than the ESF standard. Thus, it should be labeled as halogenated. The labels on the non-halogenated samples are more accurate than ones labeled as halogenated. Out of the sixteen non-halogenated samples tested, only one sample is labeled wrong. On the other hand, for the halogenated sample, out of the seven tested samples, only one sample is labeled correctly.

4.3 Results from Dexsil HydroCLOR-Q Test Kit

Table A7 provides experimental results obtained from the experimentation of 12 non-halogenated samples using Dexsil HydroCLOR-Q test kit. The results of the testing kit are dependent on the water percentage in the samples. The water percentages in the samples are listed in Table 1, and the Cl^- concentrations determined from the test kit and Volhard's method are compared.

Table 1 - Chloride Testing Kits Results

Sample ID	Water Content (%)	Halogen Concentration using HydroCLOR Test Kit (g/L)	Halogen Concentration vis Volhard Titration (g/L)	% Difference
S081203060	3.80	3.14	1.91	39.15
S011106739	13.00	0.19	0.07	62.57
S081203189	16.00	7.36	0.00	>100
S081203194	27.00	6.91	0.00	>100
S081203824	51.00	4.17	0.00	>100
S081203823	52.00	5.92	0.00	>100
S081203838	66.10	3.75	0.00	>100
S081203066	70.00	1.20	0.04	96.67
S070703422	70.00	0.00	0.00	0.00
S081201147	70.00	0.20	0.21	5.00
S020703420	75.25	2.40	0.82	65.83
S081203070	87.78	0.00	0.04	4.00

The HydroCLOR-Q kit is designed for testing water and oil/water mixtures. For accuracy, the sample should contain more than 70% water (i.e.: less than 30% oil). From Table 1, the water percentages for most of the samples are less than 70%; therefore, the test results are adjusted using the correction included in Appendix B. In general, the differences between the results obtained from the testing kit and the Volhard titration are significant. Sample (ID

S081203189, S081203194, S081203824, S081203823, S081203838, S081203066) are found to be non-halogenated by the Volhard's method. However, by using the testing kit, the results suggested that all the samples tested have high halogen concentrations (>1000ppm or 1.0g/L). By comparing the percentage differences between the testing kit results and titration results, a general trend is obtained. The percentage differences between the results are significantly affected by the percentage of water in the samples; the lower the percentage of water the bigger the difference. Samples (ID - S070703422, S081201147, S020703420, S081203070) that have high water percentage (>70%) have lower percentage differences between the testing kit and titration results of 0%, 5%, 65% and 4% respectively. The relatively small differences between the results indicate that, Hydroclor-Q testing kit provides accurate Cl^- concentration for the samples with water percentage (>70%). It is also observed that the results become extremely inconclusive when the halogen content is below 200 ppm (which is most of the cases since the samples contain almost no halogens). Thus, Hydroclor-Q testing kit is inaccurate for samples with low water percentage and with halogen content below 200ppm. To improve Hydroclor-Q testing kit method, it is suggested that calculated amount of water is added to the samples being tested to ensure the water percentage is above 70% before using the kit. Thus, the more accurate results can be obtained.

Table A8 summarizes the quantitative analysis of halogen content in waste samples collected from the ESF. Most samples are determined to be non-halogenated. Out of 23 samples, 6 samples are labeled incorrectly.

5.0 SAFETY AND ENVIRONMENAL ISSUES

With the hazardous nature and unknown composition of the chemical samples from ESF and chemical reagents used during experimentation, lab safety was paramount. During sample collection at the ESF facility it was ensured that all members wore proper equipment including safety goggles and lab coats. When transferring the chemical samples from their ESF containers to the required sample vials a fume hood was used in order to mitigate the fumes inhaled. During experimentation it was ensured that all members wore safety goggles, nitrile gloves and lab coats for the entirety of the lab. A fume hood was also used for the experiments to allow any vapours to escape without being inhaled. MSDS sheets for the chemical reagents used for experimentation were made available during all lab sessions and safety audits were completed for both the original Volhard titration experiment and the Dexsil chlorination titration kit. These audits were meant to examine various aspects of the experiment including the quantity of chemicals used, potential hazards (risk of explosions, toxic materials, etc.) and safety features of the laboratory. After each lab period the experiment area was cleaned and all experimental apparatus was disassembled and put away while all used glassware was sent to be cleaned. Used chemicals were emptied into the proper halogen/non-halogen waste containers for disposal following ESF guidelines.

Due to the scale of the experiment only the overall chemical reagent quantities would increase in comparison to the ESF procedure. This is due to the quantity of waste sample used per titration being relatively the same however the total amount of samples being increased from the experiment to ESF operations. This might result in greater waste products from used chemical reagents initially. However once the ESF has the experimental methodology, they should begin to address where the halogenated waste samples are originating from. They would do this by first screening the samples using the experimental methods set out in this report. Then using the results, they will notify the generators of their halogenated waste and set up new rules to better avoid future problems regarding mislabeling by reducing halogenated waste being sent to ESF from generators. The effluent treatment of the waste would fall into two categories, the first is the sample waste and the second is the reagent waste. The sample waste is disposed of based on the halogen cutoff content of 1000 ppm. Any sample that are above 1000 ppm are considered halogenated and must be incinerated while any sample below 1000 ppm are considered non-halogenate and can be recycled as fuel additives. The chemical reagent waste treatment will follow ESF guidelines in terms of recycle and/or disposal.

One of the main benefits of using Dexsil HydroCLOR kit is that the chemical reagent quantities are very small in relation to Volhard's method as well as all the reagents being stored in their own closed vials. This makes disposal extremely easy and safe as there is minimal glass that can be accidentally broken. However, this kit includes small glass ampules that must be broken inside plastic vials. It is possible that while breaking these ampules the plastic breaks as well causing a personal injury or chemical spill. Caution should be taken during these steps to ensure safe experimental operation. One of the main disadvantages of the Dexsil test kit is that there is no control over what the kit actually determines. This is a problem when the contents of the waste samples are completely unknown in terms of composition and make the results determined by the kit extremely difficult to interpret.

6.0 PROPOSED DESIGN

To meet the demands of the ESF, the proposed method will be scaled up with the minimization of labour and the ability to simultaneously process multiple samples in mind. For the operational scale of the ESF, the proposed method is carried out under the same conditions as before with the only change being an increased number of samples that need to be processed. For this reason, supplies such as reagents glass ware and other compounds will be scaled up linearly by the number of samples. According to the process, 10 mL of each sample is tested with 10 mL of amyl alcohol with 15 mL of 0.1 M $AgNO_3$. The amount of 0.1 M KSCN needed is typically nearly 15 mL. If 10 samples were tested daily, then on a 5 day work week 12.5 grams of $AgNO_3$, 100 mL of amyl alcohol, and 150 mL of KSCN would be needed. Testing each day would also require 10 Erlenmeyer flasks, which can be cleaned on a daily basis, and a titration stand and a glass burette.

In addition to supplies, labour will also scale linearly with the number of samples. Volhard’s method requires tedious labour which can be from 10-15 minutes per sample, including sample preparation, experimentation, and analyses. However, it is proposed to use an automatic titrator to minimize labour. For the Volhard’s method, an automatic potentiometric titrator is suggested. This titrator would be fully automatized and can process multiple samples per hour, minimizing the labor requirement of the employees. An automatic potentiometric titrator functions by measuring the electric potential across an electrolyte solution using two electrodes: a reference electrode and indicator electrode. The indicator electrode forms an electrochemical half-cell with the ions of interest while the reference electrode holds a constant electrical potential and forms the other half cell according to Equation 6. A potentiometric titrator is applicable for use by the ESF since the electric potential of the solution increases with increased resistance due to the decrease of ions present in the solution at the equivalence point according to ohms law. With an automatic titrator, many samples can be investigated automatically, with very little labour from employees. It should also be noted that with an automatic titrator the indicator iron (III) would not be required.

$$E_{Cell} = E_{Ind} - E_{Ref} + E_{Sol} \qquad \text{..............................Eqn. 6}$$

The design for the proposed protocol follows the Volhard's method as outlined previously. The method is designed to be applicable to all liquid waste samples, non-halogenated and halogenated, received by the ESF. These liquid wastes may contain precipitates, oils, and suspended solids. For this reason, the design of this PBL employs measures such as the addition of amyl alcohol to separate precipitates and suspended solids. Further design specifications are the reagents used, the apparatus used, and application of additional processes to the Volhard's method to ensure accurate results. Since the Volhard's method operates in a low pH range, reagents are chosen so that no undesirable changes in pH result. This is because an acidic environment prevents the precipitation of the indicator, iron (III), as iron hydroxide. If samples are basic, nitric acid is added to lower the pH. Nitric acid lowers the pH of the sample without having any interference from the dissolved NO_3^- ion. In addition, reagents are also selected based on their solubility and their unlikeliness to have undesired reactions.

The advantages of the proposed design are that it is simple and results can be obtained quite quickly with reasonable accuracy. In addition, the method uses relatively safe reagents and poses minimal risks to the technician. A disadvantage of this method is that the chloride ion is assumed to be the most prolific halogen contained in the liquid waste streams from UBC laboratories and, therefore, this method does not measure any halogens other than chloride. It is not entirely known how often this assumption is valid, and, hence, it may be possible to have false negatives for halogen content.

The economic breakdown per sample is as follows: $1.75 $AgNO_3$, $0.81 KSCN, and $0.48 amyl alcohol. Thus, the total material costs per sample amount to approximately $3.04. The cost of glassware is $4.00 for a 250mL Erlenmeyer flask and $64.95 for a titration burette and stand. Safety considerations such as fume hood, lab coats are not included, as they vary in cost and are common in all laboratories including the one at ESF. The cost for use of an automatic titrator for scale up varies from $1,200 to $6,000. The cost of disposal for non-halogenated samples is $0.80 while the cost of disposal of halogenated samples is $1.65. During the experimentation with 7 supposedly halogenated samples from ESF, only one sample was found to contain over 1000ppm chloride ions (the minimum concentration required for halogenated classification). If these samples were disposed of as halogenated samples the ESF would have lost $5.95, with the extra cost of $0.85 per sample. Seeing as the ESF received 22791 kg of solvents in 2013 (Risk Management, 2013), it is worthwhile for the investment of the proposed design. Even if it is assumed that only one quarter of these solvents are determined to not be halogenated, the savings would pay for the materials and equipment costs. In addition, based on the amount of waste solvents received, and assuming a 280 day a year work period, roughly 8-10 samples would be processed a day.

Alternatively, another method that may be employed is the chloride testing kit. This kit costs approximately 25$ per sample, much more expensive than the proposed method. However, this kit may provide more accurate results within 200-4000ppm of halogen content due to its method of cleaning up the sample of interfering particles, precipitates, water, and suspended solids. Other than this kit, there are not any other cost effective alternatives.

Table 2 - Economic Breakdown for the Proposed Protocol

Reagent	$/sample	Glassware	$
$AgNO_3$	1.75	250mL Erlenmeyer Flask x8	32.00
KSCN	0.81	Titration Burette and Stand	64.95
Amyl Alcohol	0.48	Automatic Titrator	1,200-1,600
Total:	$3.04	Total:	1,296.95 - 1,696.95

7.0 QUALITY ISSUES

Quality control is the design procedure in which accuracy and consistency is thoroughly reviewed so that optimal results can be both maintained and achieved. This section discusses the challenges encountered during the experimentation and the techniques utilized to overcome those challenges. It also details the steps taken to ensure that the results obtained are of the highest accuracy.

To attain precise and accurate results, each sample is tested in duplicates. If the two test results for the same sample are consistent, this implies that both tests yield precise results. Furthermore, if the two tests do not agree with each other a third test is performed.

One major constraint of Volhard's method is that the sample pH must be below 6.5 for the method to be valid. Thus, every sample needs to be tested for their pH levels before any experimental procedures begin. If necessary, the pH of the samples is adjusted using nitric acid.

Volhard's method is a titration based process. Like all other titration processes, complications arise mainly due to the operator overshooting the titration end point. This occurs due to the inability of the operator to clearly distinguish a change in colour as the equivalence point is reached due to the cloudiness or colour of the sample or due to the small amount of chloride ions present. In order to avoid overshooting, a diluted titrant is used so that the operator may have better control over the amount of titrant being added to the sample. Although the diluted titrant increases the overall time required for each trial to conclude, it increases the accuracy of the results. This step does introduce another degree of error as the accuracy of the dilution must be considered. In addition, to decrease the overshooting done by the operator, the use of prepared samples, which have been perfectly titrated, are used as a basis to compare samples to. These comparison samples are created using a known amount of NaCl which is titrated to its equivalence point by adding its respective amount of AgCl and KSCN to create a perfect titration.

Initially a magnetic stirrer was used during titration so that the titrant would be uniformly mixed. However, this step was ceased once it was observed that the precipitate would cause the sample to become cloudy, inhibiting the technician's ability to distinguish when the equivalence point was reached. Therefore, the magnetic stirrer was not continued to be used during the titration and the sample was allowed to settle for few minutes so that two distinctive layers (aqueous and organic layers) were formed. As a result, the indicator colour was easily visible in the clear aqueous layer.

As with overshooting being an issue, undershooting can also be a possibility. This possibility arises due to the indicator, ferric ammonium sulfate, which has a strong orange colour to mislead one to believe an end point has been reached. The end point of the titration is usually marked when the indicator forms a dark peachy colour, as shown in Figure 4. However, the end point can sometimes be confused because the waste sample colour varies. To prevent undershooting, the use of comparison samples are used, similarly to how they are used to combat the overshooting issue.

It is almost impossible to know what kind of chemicals exist in waste samples and if they have any effect on the results obtained through the Volhard's method. Therefore, two calibration tests are carried out to observe what effects organic compounds have on the result of the Volhard's Method. Acetone was chosen since it is a common cleaning agent used throughout the campus. Amyl Alcohol was chosen because it was an integral part of the experimental procedure.

8.0 CONCLUSION AND RECOMMENDATIONS

In order to determine the halogen content of ESF waste samples, two titration techniques were employed. The main experimental technique utilized was Volhard's method while the other was a chlorine detection testing kit provided by a chemical company known as Dexsil. Volhard's method is used to determine the chlorine content in a sample and is applicable at pH <6.5. The Dexsil testing kit is applicable in a range of water/oil mixtures with an oil content <80% and where the water percentage is known. A total of 23 samples were collected for testing from the ESF, 7 labeled halogenated and 16 labeled non-halogenated. All 23 samples were tested using the Volhard's method while only 12 non-halogenated samples were tested using the Dexsil kit.

The ESF samples contained oils and other organic compounds that might skew titration results, therefore a series of calibration curves were produced from standard chlorinated samples. A mixture at 50:50 of water/acetone and amyl alcohol/water were tested. The results of these tests show that there is a negligible interference by amyl alcohol and acetone and the results typically have deviations from the true concentrations of 10% to 15%. It is therefore determined that organics do not affect titration results in any significant way.

According to the Volhard's method, of the 7 ESF samples labeled as halogenated, four have chlorine concentrations of 0.00 g/L, two have concentrations of 0.11 and 0.07 g/L, and one has a concentration of 7.06 g/L (which is above the ESF cutoff of 1000 ppm). It is determined that the six samples below the 1000 ppm cutoff should not have been labeled as halogenated. Of the sixteen ESF samples labeled non-halogenated, thirteen have chlorine concentrations below 0.1 g/L, two have chlorine concentrations of 0.82 and 0.96 g/L, and one has a chlorine concentration of 2.06 g/L. It is determined that due to their proximity to the ESF cutoff of 1000 ppm and according to the error found by the calibration curves, the two samples with chlorine concentrations of 0.82 and 0.96 g/L are recommended to be labeled as halogenated. The sample with the chlorine concentration of 2.06 g/L should also be labeled as halogenated as it is clearly above the ESF cutoff.

Using the Dexsil testing kit, chlorine concentrations of 3.14, 0.19, 7.36, 6.91, 4.17, 5.92, 3.75, 1.20, 0.00, 0.20, 2.40, and 0.00 g/L are found for the twelve samples. These results show eight of the twelve samples are halogenated while the remaining four are non-halogenated. This differs greatly with the results from the previous Volhard's method, which show that for the same samples only one of the twelve are halogenated with the remaining eleven being non-halogenated.

It is recommended that more calibration curves be produced with varying chemical additives to better understand how they may affect titration results. It is also recommended that more testing be done to understand why there is such a large variance between the two titration techniques. An increase in sample size is suggested as it may yield more accurate results. It is recommended that safety equipment be worn at all times and a fume hood is used while conducting these experiments as harmful vapours are present. Safety audits, MSDS sheets and proper documentation should be made readily available to any individual involved with the experiment as toxic chemicals are used regularly. It is also recommended that the ESF contact waste generators known to mislabel wastes in order to inform them of their malpractice. This would save the ESF money and would reduce the work done for the ESF in correcting future samples. Finally, it is recommended that the Volhard’s method should be employed by the ESF in order to lower their disposal costs of halogenated waste and improve overall waste turnover efficiency.

9.0 ACKOWLEDGEMENT

Our Group wishes to acknowledge Mr. Bang Dang at the UBC Environmental Services Facility (ESF) for providing us information regarding waste samples, giving us a tour of the facility and demonstrating how silver nitrate can be used to precipitate halogens from waste samples. We are thankful to everyone at the ESF who provided guidance and helped us collect samples for our project. We would also like to thank Ms. Noga Levit (Manager, UBC Environmental Services) for her guidance in understanding the requirements of the project. We wish to kindly thank the AMS Sustainability Projects Fund Committee for awarding the project a Social Ecological Economic Development Studies (SEEDS) Grant in the amount of $3570. We also thank the previous groups who worked on this project and helped us gather experimental data. Finally, we thank our instructor, Dr. Christina Gyenge, for her guidance and support on this project.

NOMENCLATURE

Term	***Definition***
AMS	Alma Mater Society
Ag^+	Silver ion
$AgBr_{(s)}$	Silver bromide precipitate
$AgCl_{(s)}$	Silver chloride precipitate
$AgI_{(s)}$	Silver iodide precipitate
$AgSCN_{(s)}$	Silver thiocyanate precipitate
Br^-	Bromide ion
Cl^-	Chloride ion
ESF	Environmental Services Facility
Fe^{3+}	Ferric ion
$[FeSCN]^+$	Ferric thiocyanate complex
l^-	Iodide ion
PBL	Problem-Based Laboratory
SCN^-	Thiocyanate ion
SEEDS	Social Ecological Economic Development Studies
SPF	Sustainability Projects Fund
UBC	University of British Columbia

REFERENCES

Chemistry 311 Course, University of Calgary (n.d.). Experiment 6 – Determination of Soluble Chloride: The Volhard Titration. Retrieved from http://www.chem.ucalgary.ca/courses/ f13/chem311/ Volhard.pdf

College of Science, University of Canterbury (n.d.). Determination of Chloride Ion Concentration by Titration (Volhard's Method). Retrieved from http://www.outreach. canterbury.ac.nz/chemistry/ documents /chloride_volhard.pdf

Dexsil HydroCLOR-Q (HY-DRO) (2014). Retrieved from http://www.dexsil.com/products/ detail.php?product_id=32

Dexsil HydroCLOR-Q® (2006, July 13). Instructions for HydroCLOR-Q® - Quantitative Test Kit for Organic Chlorine Contamination in Water and Water/Oil Mixtures. Retrieved from http://www.dexsil.com/uploads/docs/hydroclorq.pdf

Dexsil HydroCLOR-Q (2007, January 1). Suggestion for Using the HydroCLOR-Q Test Kit. http://www.dexsil.com/uploads/docs/hydrocon.pdf

UBC Life Science Centre (2012, January). Laboratory Hazardous Waste Management. Retrieved from http://facilities.med.ubc.ca/files/2011/12/LSC-Hazardous-Waste-Management-Procedures.pdf

UBC Risk Management Services (2013). Hazardous Waste Reduction & Recycling at UBC http://riskmanagement.ubc.ca/environment/hazardous-waste-management/hazardous-waste-reduction-recycling

APPENDICES

APPENDIX A

RAW DATA & WORKED DATA

Table A1 - Calibration Results for NaCl/ Water/ Amyl Alcohol

Trial Number	NaCl Concentration		Sample Volume (mL)	Volume of 0.1M AgNO3 added (mL)	KSCN titrant concentration used (mol/L)	Initial burette volume (mL)	Final burette volume (mL)	volume of KCSN titrant added (mL)	KCSN required (moles)	Excess AgNO3 (moles)	Calculated NaCl/ chloride ion concentration (g/L)	Percent error
	ppm	g/L										
1	500	0.5	15	10	0.1	37.3	45.9	8.6	8.60E-04	1.00E-03	0.55	9
2			15	10	0.1	4.5	13.1	8.6	8.60E-04	1.00E-03	0.55	9
3			15	10	0.1	13.1	21.6	8.5	8.50E-04	1.00E-03	0.58	17
1	1000	1	10	10	0.1	0	8.2	8.2	8.20E-04	1.00E-03	1.05	5
2			10	10	0.1	17.1	25.2	8.1	8.10E-04	1.00E-03	1.11	11
3			15	10	0.1	17	24.6	7.6	7.60E-04	1.00E-03	0.94	6
4			15	10	0.1	25.3	33	7.7	7.70E-04	1.00E-03	0.90	10
1	1500	1.5	10	15	0.1	4.5	16.9	12.4	1.24E-03	1.50E-03	1.52	1
2			10	15	0.1	24.6	37.1	12.5	1.25E-03	1.50E-03	1.46	2
3			10	15	0.1	17.5	29.8	12.3	1.23E-03	1.50E-03	1.58	5

Table A2 - Calibration Results for NaCl/ Water/ Acetone

Trial Number	NaCl Concentration		Sample Volume (mL)	Volume of 0.1M AgNO3 added (mL)	KSCN titrant concentration used (mol/L)	Initial burette volume (mL)	Final burette volume (mL)	volume of KCSN titrant added (mL)	KCSN required (moles)	Excess AgNO3 (moles)	Calculated NaCl/ chloride ion concentration (g/L)	Percent error
	ppm	g/L										
1	500	0.5	15	10	0.1	5.7	14.2	8.5	0.00085	0.001	0.58	17
2			15	10	0.1	14.6	23.4	8.8	0.00088	0.001	0.47	6
1	1000	1	15	10	0.1	31.7	39.4	7.7	0.00077	0.001	0.90	10
2			15	10	0.1	23.4	31.3	7.9	0.00079	0.001	0.82	18
1	1200	1.2	15	15	0.1	7	19.1	12.1	0.00121	0.0015	1.13	6
2			15	15	0.1	34.1	46.5	12.4	0.00124	0.0015	1.01	16

Table A3 - Amyl Alcohol Calibration Result Summary

NaCl Concentraion		Titration NaCl Concentration	Percentage Difference
ppm	g/L	g/L	%
500	0.5	0.56	11.70
1000	1	1.00	0.15
1500	1.5	1.52	1.31

Table A4 - Acetone Calibration Result Summary

NaCl Concentraion		Titration NaCl Concentration	Percentage Difference
ppm	g/L	g/L	%
500	0.5	0.53	5.21
1000	1	0.86	14.27
1200	1.2	1.07	10.70

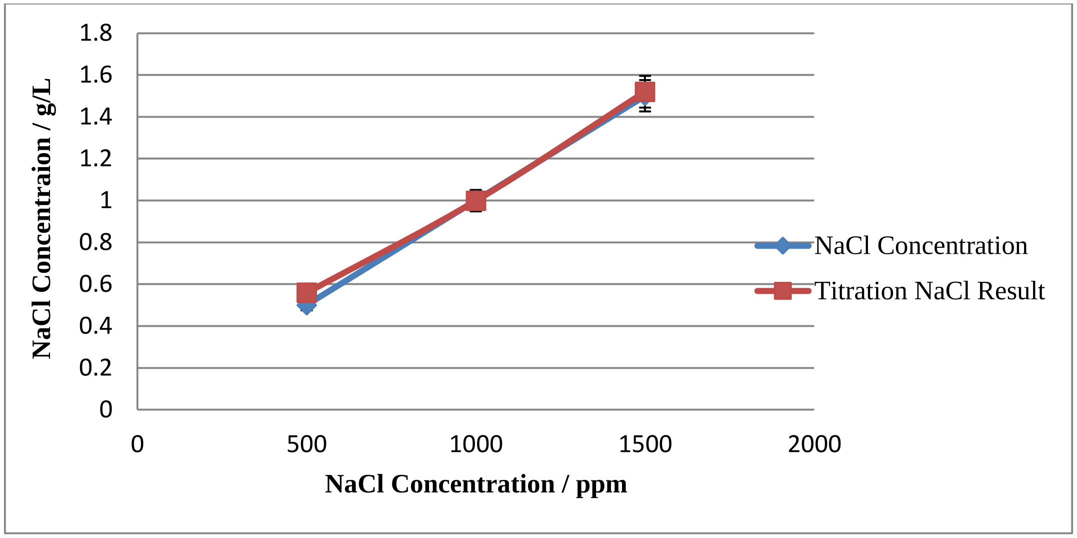


Figure A1 - Amyl Alcohol Calibration

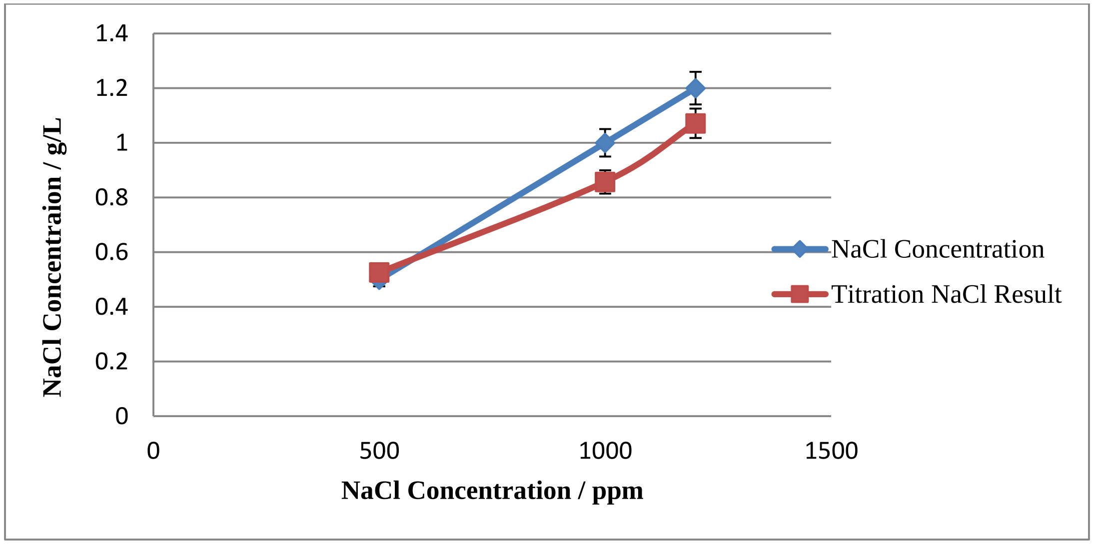


Figure A2 - Acetone Calibration

Table A5 - Halogenated Sample Titration Results

Sample ID	Trial Number	Sample Volume (mL)	Volume of 0.1M AgNO3 added (mL)	Burette Volume (mL)		volume of 0.1M KCSN titrant added (mL)	KCSN required (moles)	Excess AgNO3 (moles)	Calculated Cl$^-$ concentration (g/L)
				Initial	Final				
S081202548	1	5	10	19.3	29.3	10.0	1.00E-03	1.00E-03	0.00
	2	10	10	21.6	32.0	10.4	1.04E-03	1.00E-03	0.00
S0111009683	1	5	5	0.8	5.8	5.0	5.00E-04	5.00E-04	0.00
	2	10	10	18.7	30.0	11.3	1.13E-03	1.00E-03	0.00
	3	5	5	4.4	8.9	4.5	4.50E-04	5.00E-04	0.35
S081203416	1	10	10	5.8	15.6	9.8	9.80E-04	1.00E-03	0.07
	2	10	10	8.9	18.7	9.8	9.80E-04	1.00E-03	0.07
S0881201557	1	10	10	17.0	26.7	9.7	9.70E-04	1.00E-03	0.11
	2	10	10	32.7	42.6	9.9	9.90E-04	1.00E-03	0.04
S011104038	1	10	15	15.6	16.0	0.4	4.00E-05	1.50E-03	5.18
	2	10	20	16.7	16.8	0.1	1.00E-05	2.00E-03	7.06
	3	10	20	16.9	17.0	0.1	1.00E-05	2.00E-03	7.06
S081201327	1	10	10	27.2	37.2	10.0	1.00E-03	1.00E-03	0.00
	2	10	15	9.5	24.6	15.1	1.51E-03	1.50E-03	0.00
	3	10	15	10.6	25.5	14.9	1.49E-03	1.50E-03	0.04
S081201325	1	10	10	24.7	39.4	14.7	1.47E-03	1.00E-03	0.00
	2	10	10	13.0	27.4	14.4	1.44E-03	1.00E-03	0.00
	3	10	10	39.4	49.6	10.2	0.00102	0.001	0.00

Table A6 - Non-Halogenated Sample Titration Results

Sample ID	Trial Number	Sample Volume (mL)	Volume of 0.1M AgNO3 added (mL)	Burette Volume (mL)		volume of 0.1M KCSN titrant added (mL)	KCSN required (moles)	Excess AgNO3 (moles)	Calculated Cl$^-$ concentration (g/L)
				Initial	Final				
S081203194	1	10	10	26.4	38.0	11.6	1.16E-03	1.00E-03	0
	2	10	10	27.0	38.3	11.3	1.13E-03	1.00E-03	0.00
S081201147	1	10	10	7.5	16.9	9.4	9.40E-04	1.00E-03	0.21
	2	10	10	5.6	15.4	9.8	9.80E-04	1.00E-03	0.07
S081203070	1	10	15	16.9	31.8	14.9	1.49E-03	1.50E-03	0.04
	2	10	15	15.5	30.4	14.9	1.49E-03	1.50E-03	0.04
S081203180	1	10	10	31.8	41.5	9.7	9.70E-04	1.00E-03	0.11
	2	10	10	30.7	40.4	9.7	9.70E-04	1.00E-03	0.11
S020703420	1	10	15	5.1	17.8	12.7	1.27E-03	1.50E-03	0.82
	2	10	15	6.3	19.2	12.9	1.29E-03	1.50E-03	0.74
S081203824	1	10	10	18.0	28.7	10.7	1.07E-03	1.00E-03	0.00
	2	10	10	19.2	29.5	10.3	1.03E-03	1.00E-03	0.00
S081203838	1	10	10	29.5	39.5	10.0	1.00E-03	1.00E-03	0.00
	2	10	10	28.7	38.7	10.0	1.00E-03	1.00E-03	0.00
S081203060	1	5	10	39.5	46.8	7.3	7.30E-04	1.00E-03	1.91
	2	5	10	38.7	45.8	7.1	7.10E-04	1.00E-03	2.06
S081203823	1	10	10	3.2	13.4	10.2	1.02E-03	1.00E-03	0.00
	2	10	10	13.6	23.6	10.0	1.00E-03	1.00E-03	0.00
S081201140	1	10	10	5.3	13.3	8.0	8.00E-04	1.00E-03	0.71
	2	10	10	23.6	30.9	7.3	7.30E-04	1.00E-03	0.96
	3	10	10	33.3	42.2	8.9	8.90E-04	1.00E-03	0.39
S081203062	1	10	10	13.3	23.4	10.1	1.01E-03	1.00E-03	0.00
	2	10	10	30.9	41.0	10.1	1.01E-03	1.00E-03	0.00

Table A6 - Non-Halogenated Sample Titration Results Cont'd

Sample ID	Trial Number	Sample Volume (mL)	Volume of 0.1M AgNO3 added (mL)	Burette Volume (mL)		volume of 0.1M KCSN titrant added (mL)	KCSN required (moles)	Excess AgNO3 (moles)	Calculated Cl^- concentration (g/L)
				Initial	Final				
S081203066	1	10	10	23.4	33.3	9.9	9.90E-04	1.00E-03	0.04
	2	10	10	29.5	39.4	9.9	9.90E-04	1.00E-03	0.04
S081203189	1	10	10	7.5	20.9	13.4	1.34E-03	1.00E-03	0.00
	2	10	10	6.9	19.6	12.7	1.27E-03	1.00E-03	0.00
	3	10	10	19.6	31.4	11.8	1.18E-03	1.00E-03	0.00
S070703422	1	10	10	14.4	24.7	10.3	1.03E-03	1.00E-03	0.00
	2	10	10	2.6	13.0	10.4	1.04E-03	1.00E-03	0.00
S0111006739	1	10	10	31.3	41.6	10.3	1.03E-03	1.00E-03	0.00
	2	10	10	4.3	14.1	9.8	9.80E-04	1.00E-03	0.07
S081202078	1	10	10	31.4	41.5	10.1	1.01E-03	1.00E-03	0.00
	2	10	10	21.0	31.3	10.3	1.03E-03	1.00E-03	0.00

Table A7 - Dexsil HydroCLOR-Q Testing Kit Results

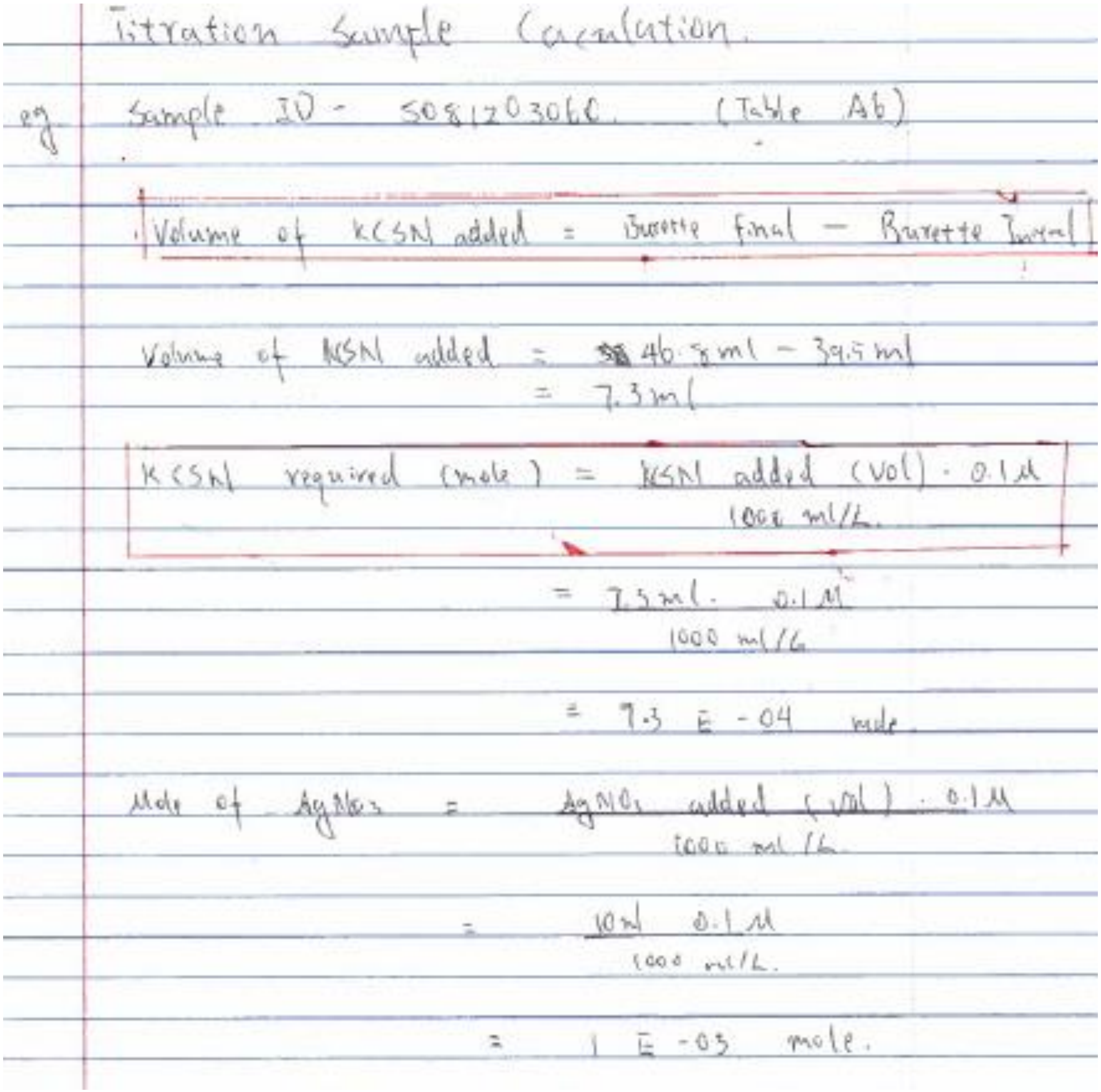
Sample ID	Water Percentage (%)	Halogen Concentration using Test Kit (PPM)	Halogen Concentration using Test Kit (g/L)
S081203060	3.80	3139	3.14
S011106739	13.00	187	0.19
S081203189	16.00	7360	7.36
S081203194	27.00	6910	6.91
S081203824	51.00	4172	4.17
S081203823	52.00	5920	5.92
S081203838	66.10	3749	3.75
S081203066	70.00	1200	1.20
S070703422	70.00	0	0.00
S081201147	70.00	200	0.20
S020703420	75.25	2400	2.40
S081203070	87.78	0	0.00

Table A8 - Summary of Quantitative Analysis of Halogen Content in Waste Solvents

Sample ID	Halogenated	Labeled as halogenated
S081202548	No	Yes
S0111009683	No	Yes
S081203416	No	Yes
S081201557	No	Yes
S011104038	Yes	Yes
S081201327	No	Yes
S081201325	No	No
S081203194	No	No
S081201147	No	No
S081203070	No	No
S081203180	No	No
S020703420	No	No
S081203824	No	No
S081203838	No	No
S081203060	Yes	No
S081203823	No	No
S081201140	No	No
S081203062	No	No
S081203066	No	No
S081203189	No	No
S070703422	No	No
S0111006739	No	No
S081202078	No	No

APPENDIX B

SAMPLE CALCULATIONS



Titration Sample Calculation.

eg. Sample ID - S08120306C. (Table A6)

Volume of KCSN added = Burette final − Burette Initial

Volume of KCSN added = 46.8 ml − 39.5 ml

= 7.3 ml

KCSN required (mole) = $\frac{\text{KCSN added (vol)} \cdot 0.1M}{1000\ \text{ml/L}}$

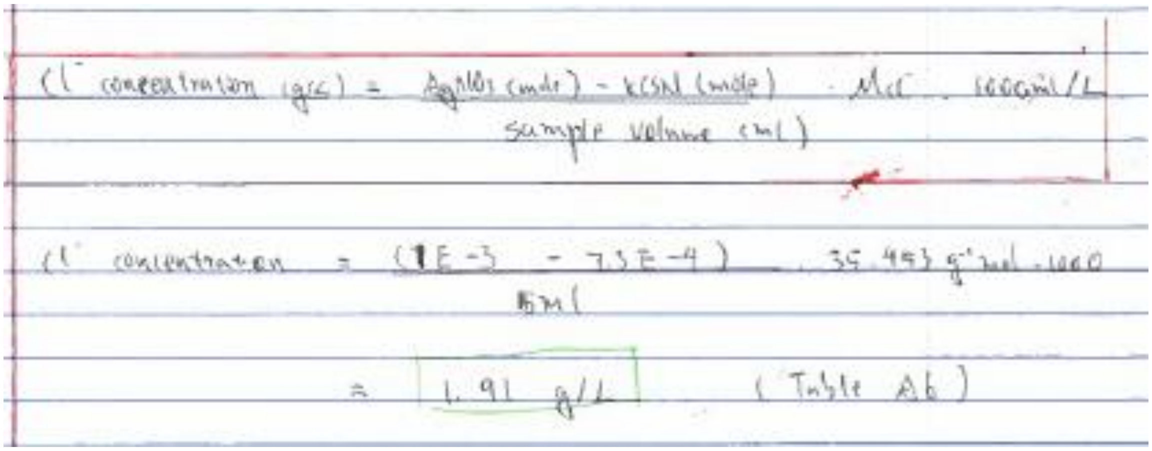
= $\frac{7.3\ \text{ml} \cdot 0.1M}{1000\ \text{ml/L}}$

= 7.3 E - 04 mole.

Mole of $AgNO_3$ = $\frac{AgNO_3\ \text{added (vol)} \cdot 0.1M}{1000\ \text{ml/L}}$

= $\frac{10\ \text{ml} \cdot 0.1M}{1000\ \text{ml/L}}$

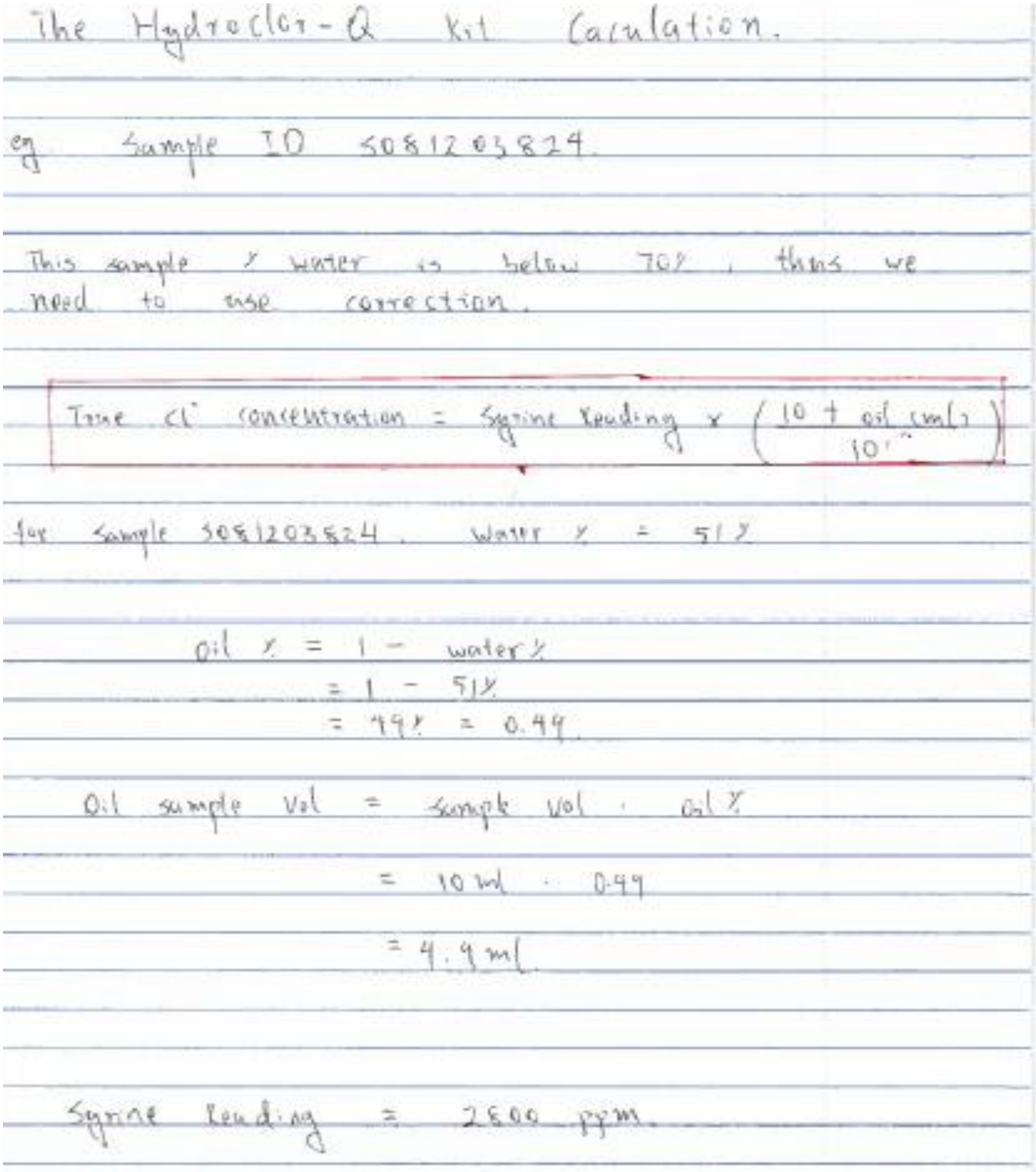
= 1 E - 03 mole.



Cl^- concentration (g/L) = $\frac{AgNO_3\ \text{(mole)} - \text{KCSN (mole)}}{\text{sample volume (ml)}} \cdot M_{Cl} \cdot 1000\ \text{ml/L}$

Cl^- concentration = $\frac{(1E-3 - 7.3E-4)}{5\ \text{ml}} \cdot 35.453\ \text{g/mol} \cdot 1000$

= $\boxed{1.91\ \text{g/L}}$ (Table A6)



The Hydroclor-Q kit Caculation.

eg Sample ID 5081203824.

This sample % water is below 70%, thus we need to use correction.

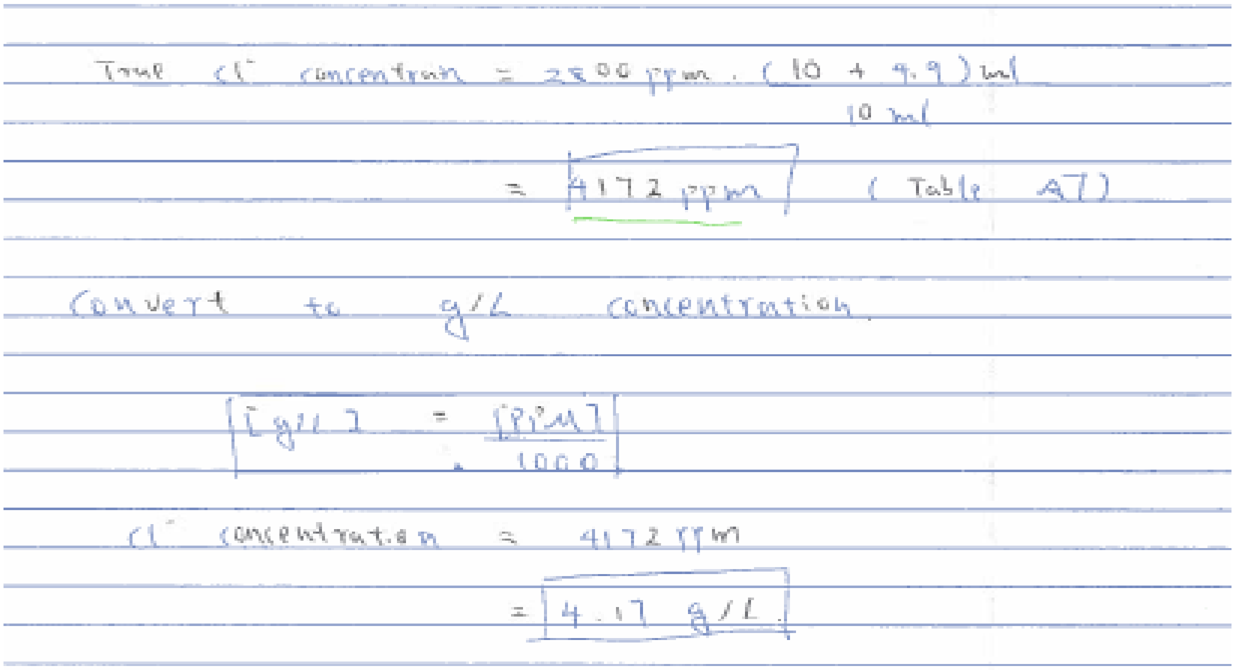
$$\boxed{\text{True } Cl^- \text{ concentration} = \text{Syrine Reading} \times \left(\frac{10 + \text{oil (mL)}}{10}\right)}$$

for sample 5081203824. Water % = 51%

$$\text{oil } \% = 1 - \text{water } \%$$
$$= 1 - 51\%$$
$$= 49\% = 0.49$$

$$\text{Oil sample vol} = \text{sample vol} \cdot \text{oil } \%$$
$$= 10 \text{ ml} \cdot 0.49$$
$$= 4.9 \text{ ml}$$

Syrine Reading = 2800 ppm.



True Cl^- concentratn $= 2500 \text{ ppm} \cdot \frac{(10 + 9.9) \text{ ml}}{10 \text{ ml}}$

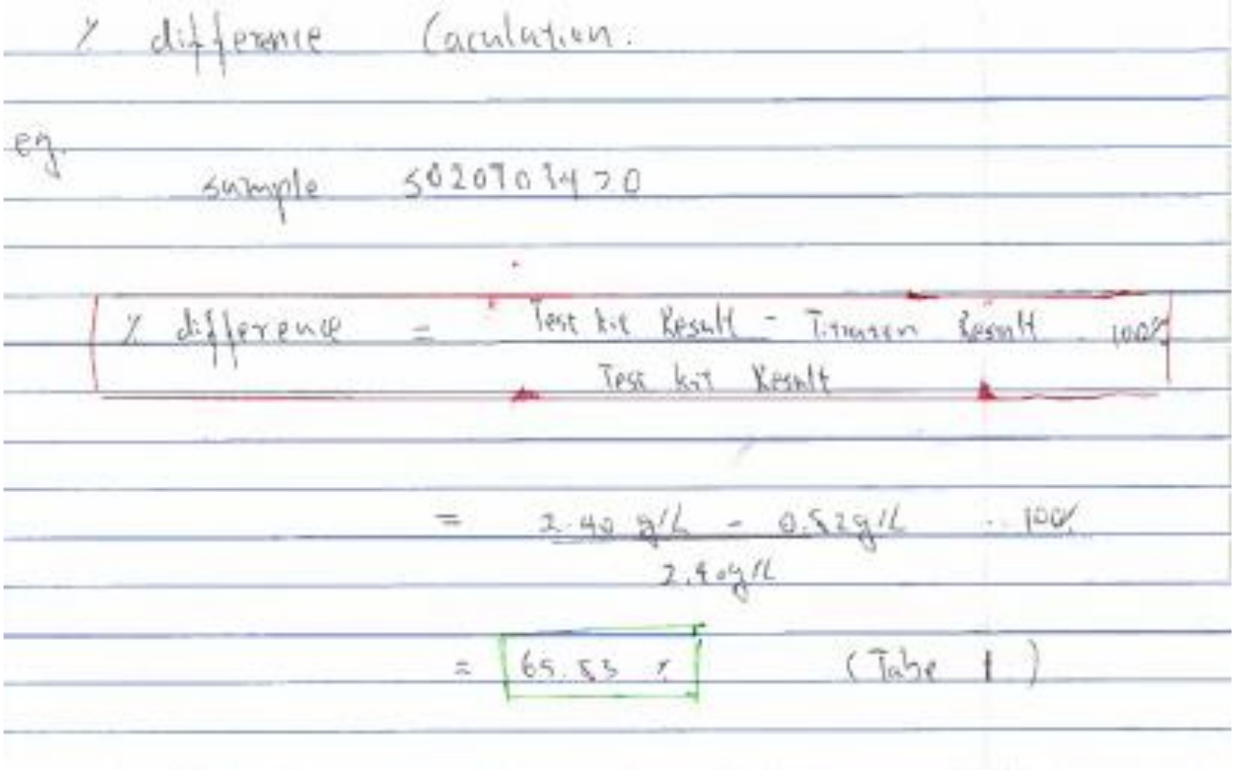
$= \boxed{4172 \text{ ppm}}$ (Table A7)

Convert to g/L concentration.

$$\boxed{[g/L] = \frac{[PPM]}{1000}}$$

Cl^- concentration $= 4172$ ppm

$= \boxed{4.17 \text{ g/L}}$



% difference Caculation:

eg.

sample 5020703470

$$\boxed{\% \text{ difference} = \frac{\text{Test kit Result} - \text{Titration Result}}{\text{Test kit Result}} \cdot 100\%}$$

$= \frac{2.40 \text{ g/L} - 0.52 \text{ g/L}}{2.40 \text{ g/L}} \cdot 100\%$

$= \boxed{65.53 \%}$ (Tabe 1)

APPENDIX C

EXPERIMENTAL PROTOCOL

PROTOCOL FOR VOLHARD TITRATION

***All steps must be carried out in the FUME HOOD.**

1. Immerse a piece of litmus paper into the conical tube of waste sample to determine the pH.
2. If necessary, add few drops of 3M nitric acid to make the sample acidic (pH < 6.5).
3. Transfer 5 – 10 mL of waste sample from the conical tube to a 50 mL Erlenmeyer flask using a disposable pipette.
4. To 10 mL waste sample, add 10 - 15 mL of 0.1 M silver nitrate solution depending on the observed precipitation.
5. Add amyl alcohol as an organic liquid and mix the sample vigorously.
6. Allow the sample to settle until two distinguished immiscible layers are observed.
7. Add ~5 drops of indicator, saturated ferric ammonium sulfate.
8. Ensuring the stopcock of the burette is closed, use a funnel to fill a 50 mL burette with 0.1 M potassium thiocyanate solution.
9. Clamp burette in place on the burette stand.
10. Record the initial burette volume.
11. Place the Erlenmeyer flask containing waste sample underneath the filled burette.
12. By hand rotation of the burette stopcock, add titrant into the flask drop-by-drop until the solution turns a pale red colour.
13. Record the final burette volume.
14. Repeat for the remaining waste samples.

PROTOCOL FOR HYDROCLOR-Q® - QUANTITATIVE TEST KIT

***All steps must be carried out in the FUME HOOD.**

1. *Set-up:* Remove contents from box. Check contents to ensure that all items are present and intact. Place the two plastic tubes and the large glass vial into the holder at the front of the box.
2. *Sample Preparation:* Carefully snap off the top of the break-top ampule containing the mixing reagent and pour the contents into the jar containing approximately 500-1000 mL of the sample to be analyzed. Cap the jar and shake thoroughly until a uniform sample is observed.
3. Remove the cap from the large glass vial. Place the tip of the 10 mL sampling syringe into the test sample and slowly pull back on the plunger until it reaches the 10 mL mark. Dispense the entire contents of the syringe into the large glass vial and replace the cap tightly.
4. Shake the mixture continuously for 1 minute. Allow the mixture to settle until it has separated into distinct phases (approximately 3 minutes). Remove the cap from the large glass vial.
5. Remove the cap from the small glass vial containing the white drying agent. With the plastic pipette, transfer approximately one-third of the top layer (phase) from the large glass vial into the small vial. Do NOT remove any of the bottom layer. Shake the solution in the small vial containing the drying agent for 15 seconds. Let stand, allowing the drying agent to settle to the bottom of the vial.
6. *Sample Introduction:* Unscrew the white dispensing cap from Tube #1. Work the plunger on the empty 1 mL sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the small vial and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. Do NOT remove any of the white powder. Remove the syringe from the sample and wipe any excess liquid from the outside of the syringe with the enclosed tissue. Place the tip of the syringe into Tube #1 and dispense the entire contents by depressing the plunger. Replace the white dispensing cap securely on tube #1.
7. *Reaction:* Break the bottom (colourless) ampule in the tube by compressing the sides of the tube. Mix thoroughly by shaking the tube vigorously for about 30 seconds. Break the top (gray) ampule in the tube and shake thoroughly for about 20 seconds. Allow the reaction to proceed for an additional 40 seconds (total of one minute), while shaking intermittently several times.

8. *Extraction:* Remove the caps from both tubes and pour the clear buffer solution from Tube #2 (clear cap) into Tube #1. Replace the white dispensing cap tightly on Tube #1 and shake vigorously for about 10 seconds. Vent the tube carefully by partially unscrewing the dispensing cap. Close securely and shake well for an additional 10 seconds. Vent again, tighten cap and stand tube upside down on its cap. Allow the phases to separate for a full two minutes.
9. *Analysis:* Place the plastic filtration funnel into Tube #2. Position Tube #1 over funnel and open nozzle on the dispensing cap. Be sure to point the nozzle away from the operator while opening it, and check that the nozzle is open completely before dispersing the clear solution. Dispense 5 ml of the clear solution through the filter into Tube #2 (up to the 5 mL line) by squeezing the sides of Tube #1. Close the nozzle on the dispensing cap on Tube #1 and remove the filter funnel from Tube #2.
10. Place the plunger rod into the titration burette and press until it snaps into place. Break off (do not pull off) the tip on the titration burette, insert the burette into Tube #2 and tighten the cap. Break the coloured ampule and shake gently for 10 seconds. Do NOT zero the plunger prior to analyzing the sample.
11. Dispense titrant slowly by pushing down on the white plunger rod. Shake the tube continuously while adding titrant to mix the titrant with the solution. Continue adding titrant until the solution turns from yellow to light purple. An intermediate pink colour may develop in the solution but should be disregarded. Continue titrating until a true light purple colour is obtained. A dark purple colour means the titration has been carried too far. Read the total chlorine concentration of the original oil sample directly on the titration burette at the black tip of the plunger rod. Record the total chlorine concentration immediately as the purple colour will fade with time.
12. *Disposal:* Empty the titration burette into Tube #2 by completely depressing the plunger. Open the “Disposal Ampule” container and drop the ampule into Tube #2. Replace the cap on the test tube. Crush the ampule by squeezing the sides of the tube. Shake for 5 seconds. This reagent immobilizes the mercury so that the kit passes the EPA’s TCLP test.

APPENDIX D

MATERIAL SAFETY DATA SHEETS & SAFETY DOCUMENTS

DEPARTMENT OF CHEMICAL AND BIOLOGICAL ENGINEERING

2360 East Mall, Vancouver BC, V6T 1Z3 *http://www.chml.ubc.ca/safety/safety.html*

LABORATORY EQUIPMENT SAFETY INSPECTION CHECKLIST

HAZARD GRADE: High Y Medium Low

Equipment	Fume hood, glassware	Previous Inspection Date *(m/d/yyyy)*	NA
Room #	CHBE 464	Inspection request date *(m/d/yyyy)*	March.6/2014
Experimenter name(s)	Group 5	Signature(s)	Marc Zinman
Advisor(s)	Prof. Gyenge	Advisor's Signature	

1. POTENTIAL HAZARDS:

Hazard:		**Specific hazard information and recommended actions to minimize safety risk:**
High pressure	N	
High temperature	N	
High voltage	N	
Risk of explosion	Y	ESF Chemical samples are unknown
Toxic materials	Y	Chemical samples contain harmful vapors
Cryogenics	N	
Solvents	Y(?)	Organic solvents possibly present
Loss of services:		
-air	Y	Fume hood would stop operating
-water	N	
-electricity	Y	Fume hood would stop operating
-other	NA	

2. SPECIFY POTENTIAL LEAK-THROUGH HAZARDS:

-ESF chemical sample vial may fall and open.
-plastic/glass vials holding reagent chemicals may break.
-fume hood sash may be left open, allowing vapors to leak into lab.
-

3. SPILL CONTROL:

Mop and pail	Y -near first aid kit and lab exit			
Spill kits – required	Acid Y	Base N	Solvent Y(?)	Mercury N
Catch pan	No			
Other	NA			

4. PRINCIPAL CHEMICAL SUBSTANCES USED:

(If additional chemicals are involved, please attach a list to this form)

Item	**Quantity**	**MSDS available:**
sodium dispersed in oil	20-50 ml	Y
napthalene in ethyl diglyme solution	50-100 ml	Y
ethanol	20-50 ml	Y
organo-sulfur compound	10-20 ml	Y
mercuric nitrate in water	10-20 ml	Y
sulfuric acid in water	20-50 ml	Y
butyl diglyme/emulsion breaker	20 50 ml	Y
florisil	20-50 ml	Y
triton X-100	20-50 ml	Y

5. WASTE GENERATION:

(If additional waste is involved, please attach a list to this form)

Waste	**Quantity**	**Disposal**
ESF Titration samples		Chemical waste bins
Left over chemicals from test kit		Chemical waste bins

6. SAFETY FEATURES:

Emergency contact sheet posted *(please specify):*		
on equipment:	no	
in hallway:	In room near first aid kit	
Emergency shutdown procedures posted:	N	
All MSDS posted:	N	
Fire extinguisher location:	Near exit door	
Eye wash fountain location:	Next to fume hood	
Emergency shower location:	Next to fume hood	
First aid kit location:	Near exit door	
Eye protection: Y	Apron: Y	Respirator: N
Other personal protection:	Nitrile gloves	

7. PAST SAFETY PROBLEMS:

chemical Samples from ESF produce harmful vapors that caused coughing and irritation to the nasal cavity and eyes. Harmful vapor produced during titration using Volhard’s method.

8. RECOMMENDATIONS:

a) Mandatory: wear safety glasses/ lab coat and nitrile gloves at all times. All titrations performed in fume hood. All titration chemicals are to be covred when not in use. Proper Labeling of all titration chemicals to ensure safety and proper disposal. All ESF samples should be properly labeled to ensure proper disposal and safety as contents of samples are unknown.

b) Optional: Ensure all used glassware is placed into the proper bin to be cleaned and the fume hood and surrounding area is cleaned and prepped for the next day of experiments

9. OTHER REMARKS:

This experiment involves the use of chemicals that have unknown components due to their origin. Therefore it will be assumed that they are very hazardous and extreme caution will be taken during handling and experimentation.

NOTE: The equipment/set-up must be operated within the conditions specified in this form/document. Any modifications in the conditions and/or change of the operations would require re-inspection of the set-up.
All future significant changes in the equipment inspected that affect its operation, such as new components, re-piping, or more stringent operating conditions, must be reported to the Safety Committee before they are implemented.

Date: *(m/d/yyyy)* March.6/2014

Material Safety Data Sheet

To comply with OSHA's Hazard Communication Standard, 29 CFR 1910.1200.

HYDROCLOR Q4000 CHLORINE/HALOGEN TEST KIT

SECTION 1 — COMPANY IDENTIFICATION

Manufacturer: Dexsil Corporation
One Hamden Park Drive
Hamden, CT 06517

Emergency Telephone Number:
USA (800) 424-9300 (CHEMTREC)
INT'L (703) 527-3887

Date Prepared: 08-14-2013
Date Reviewed: 08-14-2013

Telephone Number: (203) 288-3509

Email: info@dexsil.com

Product Description: The HYDROCLOR Q4000 product line is a field test kit for organic chlorine determination in oil/water mixtures and used antifreeze/coolant.

SECTION 2 C HAZARDS IDENTIFICATION

Inhalation	Harmful if inhaled. May cause respiration tract irritation
Skin	May be harmful if absorbed through skin. May cause skin burns.
Eyes	May cause eye irritation.
Ingestion	Will cause burns of the gastrointestinal tract if swallowed.

For additional information on toxicity, please refer to Section 11.

SECTION 3 C COMPOSITION/INFORMATION ON INGREDIENTS

The HYDROCLOR Q4000 Chlorine/Halogen Test Kit consists of one test tube containing two ampules, one test tube containing one ampule and an aqueous solution, a separate ampule, a filled plastic syringe, a glass vial containing extraction solvent & powdered emulsion breaker and a vial containing a drying agent.

Component	Contents	CAS#	EC#	% liquid
Ampule 1 (gray color yellow dot)	Sodium	7440-23-5	231-132-9	0.27%
	dispersed in oil	64742-46-7	265-148-2	1.10%
Ampule 2 (large, colorless)	Naphthalene in	91-20-3	202-049-5	0.49%
	Ethyl Diglyme Solution	112-36-7	203-963-7	4.92%
Ampule 3 (red-green color)	Ethanol	64-17-5	200-578-6	1.56%
Ampule 4 (colorless)	Organo-Sulfur Compound	128-04-1	204-876-7	2.41%
Plastic Syringe	Mercuric Nitrate in water	10045-94-0	233-152-3	0.02%
	in water	7732-18-5	231-791-2	4.35%
Aqueous Solution	Sulfuric Acid in water	7664-93-9	231-639-5	0.94%
	in water	7732-18-5	231-791-2	31.26%
Extract Solvent	Butyl Diglyme/ Emulsion Breaker	112-73-2	204-001-9	47.71%
Drying Agent	Florisil	1343-90-4	NA	0.99%
Surfactant	Triton X-100	9036-19-5	NA	3.98%
				100.00%

SECTION 4 C FIRST AID MEASURES

First Aid: In case of contact with reagents, rinse well with water.

Eye Contact: For all kit components, flush eyes with large amounts of water for 15 minutes. Seek medical attention.

Skin contact: Flush with large amounts of water. Use soap and water to wash away organic components.

Inhalation: In case of inhalation, remove to fresh air.

Ingestion: If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

SECTION 5 — FIRE AND EXPLOSION HAZARD

Flash Point	Ampule 1	138°F	58.9°C
	Ampule 2	162°F	72.2°C
	Ampule 3	48°F	8.89°C
	Ampule 4	N/A	N/A
	Plastic Syringe	N/A	N/A
	Extraction Solvent	100°F	37.8°C
	Drying Agent	None	None
	Surfactant	N/A	N/A

Flammable Limit	Unknown
Extinguishing Media	DO NOT USE WATER ON A SODIUM FIRE. Dry chemical, foam, CO_2
Special Fire Fighting Procedures	Do not use water. Wear SCBA. Avoid breathing sodium oxide fumes that will form on combustion.

SECTION 6 C ACCIDENTAL RELEASE MEASURES

Personal Protection	Chemical safety glasses and rubber gloves
Spills and Leaks	*Ampule 1 - Sodium Ampule* -Cover with dry soda ash or salt. Store in a wellventilated area away from moisture. *Ampule 2 - Naphthalene/Ethyl Diglyme Ampule* -Absorb completely and dispose of as organic waste. *Ampule 3 - Ethanol Ampule* -Solvent absorbent recommended for spills. Flush area with water. *Ampule 4 - Disposal Ampule* - Absorb completely and flush area with water. *Plastic Syringe - Mercuric Nitrate Solution* - Absorb completely and flush area with water. *Aqueous Solution* - Absorb completely and flush area with water. *Extraction Solvent* - Absorb completely and flush area with water. *Surfactant* - Contain spill and absorb with inert material (e.g. sand, earth).

SECTION 7 C HANDLING AND STORAGE

Protective equipment	Wear appropriate safety equipment when performing the test on site.
Storage	Store test kits in a cool, dry place. Check expiration date prior to performing test.

SECTION 8 C EXPOSURE CONTROLS/ PERSONAL PROTECTIVE EQUIPMENT

Engineering Controls	None required
Personal Protective Equipment (PPE)	
Respiratory protection	None required during normal use.
Ventilation	Perform test only in a well-ventilated area.
Protective gloves	Always wear rubber gloves when performing the HYDROCLOR Q4000 test.
Eye protection	Wear safety glasses.
General Hygiene Measures	Avoid contact with skin, eyes and clothing. Wash hands after running tests.

SECTION 9 — PHYSICAL/CHEMICAL CHARACTERISTICS

Property	*Ampule 1*	*Ampule 2*	*Ampule 3*	*Ampule 4*	*Syringe*	*Aq. Sol=n*	*Ext. Sol=n*	*Drying Agent*	*Surfact.*
Boiling Pt.^{o}C	N/A	185	78	100	100	>100	256	N/A	>100
Vapor Pressure mm Hg @ 21^{o}C	N/A	0.5	44	18	18	16	<0.01	N/A	N/A
Solubility in Water	reacts	91%	miscible	miscible	complete	complete	0.3%	none	complete
Specific Gravity	0.86	0.91	0.79	1.17	1.02	1.09	0.88	1.2	1.06
Percent Volatile	none	100	99	none	none	none	100	0	none
Evaporation Rate Butyl Acetate =1	N/A	0.04	2.7	N/A	N/A	N/A	<0.001	0	<0.01
Appearance	gray	colorless	red-green	colorless	colorless	colorless	colorless	white cryst.	colorless
Odor	none	ether-like	pleasant	N/A	none	none	ether-like	none	none

N/A = not available

SECTION 10 C REACTIVITY DATA

Chemical Stability All components are stable.

Conditions to Avoid Do not expose broken sodium ampule to moisture. Keep from strong oxidizers.

Hazardous Decomposition Products Sodium will form hydrogen and sodium oxide when moisture is present. Ethyl diglyme may form peroxides on exposure to air. Other solutions are stable.

Hazardous Polymerization Will not occur

SECTION 11 C TOXICOLOGICAL INFORMATION

Carcinogenicity
NTP: Yes IARC: Yes Z List: No OSHA Reg: No

This product contains Cadmium, which is listed as a NTP anticipated human carcinogen and an IARC probable human carcinogen (Groups 2A and 2B). Ethyl diglyme may cause teratogenic or mutagenic effects and cause harm to the unborn child.

Toxicity Data

COMPONENT	CONTENTS	TLV / (PEL)
Ampule 1(gray color, yellow-dot)	[Sodium] dispersed in oil	[2 mg/m3 / (2 mg/m3)]
Ampule 2 (large, colorless)	[Naphthalene] in Ethyl Diglyme Sol'n	[50 mg/m^3 / (50 mg/m^3)] 33 mg/m^3 / (33 mg/m^3)
Ampule 3(red-green color)	Ethanol	1900 mg/m^3 / (1900 mg/m^3)
Ampule 4(colorless)	Organo-Sulfur Comp'd	1 mg/m^3 / (1 mg/m^3)
Plastic Syringe	[Mercuric] Nitrate in water	[0.1 mg/m^3 / (0.1 mg/m^3)
Aqueous Sol'n	[Sulfuric Acid] in water <0.05% Cadmium	[1 mg/m^3 / (1 mg/m^3)] 0.05 mg/m^3 / (0.2 mg/m^3)
Extraction Sovent	[Butyl Diglyme]/ Emulsion Breaker	[N/A] / Not Regulated
Drying Agent	Florisil	N/A
Surfactant	Triton X-100	N/A

SECTION 12 C ECOLOGICAL INFORMATION

Naphthalene: Ecotoxicity in water (LC50): 305.2 ppm 96 hour(s) [Trout].

Ethanol: Ecotoxicity: Fish: Rainbow trout: LC50 = 12900-15300 mg/L; 96 Hr; Flow-through @ 24-24.3°CFish: Rainbow trout: LC50 = 11200 mg/L; 24 Hr; Fingerling (Unspecified)Bacteria: Phytobacterium phosphoreum: EC50 = 34900 mg/L; 5-30 min; Microtox test When spilled on land it is apt to volatilize, biodegrade, and leach into the ground water, but no data on the rates of these processes could be found. Its fate in ground water is unknown. When released into water it will volatilize and probably biodegrade. It would not be expected to adsorb to sediment or bioconcentrate in fish.

Organo-Sulfur Compound: Ecotoxicity: Fish: Rainbow trout: LC50 = 2.6 mg/L; 96 Hr.; Unspecified
Bacteria: Phytobacterium phosphoreum: EC50 = 0.508 mg/L; 15 minutes; Microtox test.

Mercuric Nitrate: An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.
Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

SECTION 13 C DISPOSAL CONSIDERATIONS

Sampling Syringe: Dispose of in accordance with all applicable federal, state and local environmental regulations.

Test Tube 1: Contains reacted oil sample and organic liquid. Dispose of as an organic waste in accordance with all applicable federal, state and local environmental regulations.

Test Tube 2: Upon completion of test including the addition of ampule 4, contents pass US EPA TCLP test. Dispose of in accordance with all applicable federal, state and local environmental regulations.

Extraction Solvent Vial & Drying Agent Vial: Contains oil sample and extract solvent. Dispose of as an organic waste in accordance with all applicable federal, state and local environmental regulations.

SECTION 14 C TRANSPORTATION INFORMATION

DOT		
Proper Shipping Name:	None	
Domestic Highway or Rail Transport Only:	As originally packaged by Dexsil Corporation, under Competent Authority Approval CA-1996100010, packages are approved for transportation by domestic highway or rail transport only. Packaging conforms to conditions and limitations specified in 49 CFR 173.4.	
IATA		
Proper Shipping Name:	Alkali Metal Dispersion	Ethanol
UN Number:	1391	1170
Hazard Class:	4.3	3
Packing Group	I	II

SECTION 15 C REGULATORY INFORMATION

SARA Listed: Mercuric nitrate, cadmium, naphthalene/ethyl diglyme, Organo-sulfur compound, and ethanol are identified on SARA Sec. 313.

TSCA Inventory Items: Mercuric nitrate, cadmium, naphthalene/ethyl diglyme, Organo-sulfur compound, and ethanol.

CERCLA Listed: Mercuric nitrate, cadmium, naphthalene/ethyl diglyme, Organo-sulfur compound, and ethanol.

EINECS: Components of this product are on the European Inventory of Existing Commercial Chemical Substances.

SECTION 16 C OTHER INFORMATION

The information in this Material Safety Data Sheet meets the requirements of the United States OCCUPATIONAL SAFETY AND HEALTH ACT and regulations promulgated thereunder (29 CFR 1910.1200 et. seq.) and the Canadian WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEM. This document is intended only as a guide to the appropriate precautionary handling of the material by a person trained in, or supervised by a person trained in, chemical handling. The user is responsible for determining the precautions and danger of these chemicals for his or her particular application. Depending on usage, protective clothing including eye and face guards and respirators must be used to avoid contact with material or breathing chemical vapors/fumes.
Exposure to this product may have serious adverse health effects. These chemicals may interact with other substances. Since the potential uses are so varied, Dexsil cannot warn of all of the potential dangers of use or interaction with other chemicals or materials. Dexsil warrants that the chemicals meet the specifications set forth on the label. DEXSIL DISCLAIMS ANY OTHER WARRANTIES; EXPRESSED OR IMPLIED WITH REGARD TO THE PRODUCT SUPPLIED HEREUNDER, IT'S MERCHANTABILITY OR ITS FITNESS FOR A PARTICULAR PURPOSE. The user should recognize that this product can cause severe injury and even death, especially if improperly handled or the known dangers of use are not heeded. READ ALL PRECAUTIONARY INFORMATION. As new documented general safety information becomes available, Dexsil will periodically revise this Material Safety Data Sheet.
CHEMTREC emergency telephone number is to be used ONLY in the event of CHEMICAL EMERGENCIES involving a spill, leak, fire, exposure, or accident involving chemicals. All non-emergency questions should be directed to the Customer Service Dept. at 1-203-288-3509.

For additional information, contact Dexsil. Rev. 10