# HALOGENATED ORGANICS IDENTIFICATION: QUALITATIVE ANALYSIS

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**CHBE 464**

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January 27, 2014

Dear Dr. Gyenge:

Attached is the final report for the CHBE 464 PBL (Problem Based Lab) project, titled "Halogenated Organics Identification: A Qualitative Analysis".

Group 4 began the project in October 2013 and concluded in January 2014. The overall goal of the project is to create a procedure that will allow the UBC Environmental Services Facility to easily test non-halogenated waste samples for the presence of halogens. This is in an effort to reduce the amount of non-halogenated waste that is being disposed of as halogenated solvents, which comes at a greater price.

Six teams of five students each are assigned to this project. The second team developed a qualitative method for determining the halogen composition through the use of silver nitrate and nitric acid. Our team has continued from the progress of the previous group by performing a qualitative analysis on a larger pool of waste halogenated and non-halogenated samples using the silver nitrate test. The tasks completed by our group are as follows and were completed between October 24, 2013 and November 28, 2013:

- Performing a silver nitrate test on 36 different non-halogenated samples and 20 halogenated samples and subsequently determining the halogen precipitate mass of each sample.
- Generating a list of sample IDs of non-halogenated samples that contain halogens and halogenated samples that did not contain halogens.

This final report contains an analysis of the results from the experiments as well as background theory, an experimental apparatus overview, safety and environmental findings, scale up methods, and quality issues. For any further inquiries, please contact derekgfong@gmail.com.

Sincerely,

Derek Fong
Group 4 Project Manager
UBC Chemical and Biological Engineering, Year 4

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AMS SUSTAINABILITY

*UBC SOCIAL ECOLOGICAL ECONOMIC DEVELOPMENT STUDIES (SEEDS) STUDENT REPORT*

*CHBE 464: PROBLEM BASED LABORATORY REPORT*

# HALOGENATED ORGANICS IDENTIFICATION: QUALITATIVE ANALYSIS

## CHBE 464: PBL FINAL REPORT

*EXPERIMENT PERIOD: OCTOBER 24, 2013 TO NOVEMBER 28, 2013*

*DATE SUBMITTED; JANUARY 27, 2014*

*INSTRUCTOR: DR. CHRISTINA GYENGE*

*PREPARED BY GROUP 4*


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## Summary

The overall objective of this Problem Based Laboratory (PBL) project is to use a silver nitrate test to determine if halogens are present in non-halogenated waste samples collected by the Environmental Services Facility (ESF).

The silver nitrate test was carried out on 36 out of the 50 non-halogenated samples collected. It was not possible to test all samples because samples appearing to have significant amounts of suspended solids or foreign materials were excluded in order to ensure accurate results. In addition, the Karl Fischer titration apparatus was set up for the subsequent group to complete the water content test.

Silver nitrate was used because it reacts with chlorine, bromine and iodine ions in the waste solvent to form precipitates. Nitric acid was then added to each sample so the precipitates not containing halogens are dissolved.

From the average mass of the precipitate sample and its duplicate, 11 out of the 36 samples non-halogenated samples contained no precipitate or gave a negative mass reading, 17 out of the 36 samples contained a precipitate mass between 0 g and 0.05 g, and the remaining 8 out of 36 samples contained a precipitate mass greater than 0.05 g. While the results from the test do not give a direct indication to the parts-per-million halogen concentration in each sample, it does show that approximately 61% of the “non-halogenated” samples were in fact halogenated. There is therefore a significant problem with the improper filling of non-halogenated waste containers in laboratories across campus.

Because safety during the experiment is of paramount importance, safety inspections were carried out during each laboratory period. Due to the volatile and corrosive nature of many of the chemicals used, all tests were performed under a fume hood with proper protective equipment such as gloves, lab coats, and safety glasses. To dispose correctly of these chemicals, all used samples were disposed of in halogenated waste containers.

## Nomenclature

| Term | Definition |
|---|---|
| $Ag^+$ | silver ion |
| AgBr | silver bromide |
| AgCl | silver chloride |
| $Ag_2CO_3$ | silver carbonate |
| AgI | silver iodide |
| $AgNO_3$ | silver nitrate |
| $Br^-$ | bromide ion |
| $Cl^-$ | chloride ion |
| $CO_2$ | carbon dioxide |
| $CO_3^{2-}$ | carbonate ion |
| ESF | Environmental Services Facility |
| $HNO_3$ | nitric acid |
| $H_2O$ | water |
| $I^-$ | iodide ion |
| $NO_3^-$ | nitrate ion |
| PBL | Problem Based Laboratory |
| UBC | University of British Columbia |

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# 1.0 Introduction

The major issue faced by Environmental Services Facility (ESF) is the identification and segregation of halogenated waste from non-halogenated solvent waste due to the improper disposal of chemicals by the generators. The excess water content in the halogenated waste is also problematic as too much water in the waste stream makes it difficult for the solvent to be used as a fuel additive. The main objective of the second stage of the Problem Based Laboratory (PBL) is to qualitatively determine the presence of halogens in non-halogenated waste samples. The project was started on October 24, 2013 and 4 laboratory sessions have been held in order carry out the experiment.

During the first laboratory period, sample vials and duplicates were prepared. In the second laboratory session, silver nitrate and nitric acid were added to each sample and the duplicates. These samples were then centrifuged to settle the halogen precipitates and the supernatant of each sample was removed using a vacuum set-up. The samples were then placed open in the fume hood until the next laboratory session in order to evaporate any moisture remaining in the vials. The samples and duplicates were weighed the following week, and through a simple calculation, the mass of the precipitate in each vial was determined. These results gave qualitative insight into the presence of halogens in the ‘non-halogenated’ waste solvent samples and the absence of halogens in the halogenated samples. In the final laboratory session, the automatic titration assembly was set up to allow the subsequent group to carry out the water content tests.

## 2.0 Theory

The silver nitrate test qualitatively determines the presence of halogens in a solution. It gives a measure of the amount of halogens in the solution, but does not allow for the determination of halogen concentration. Silver nitrate solution, $AgNO_3$, is reacted with the waste solvent and precipitates with chlorine, bromine and iodine ions as shown in equations 1-3 (Clark 1):

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s) \quad (1)$$

$$Ag^+(aq) + Br^-(aq) \rightarrow AgBr(s) \quad (2)$$

$$Ag^+(aq) + I^-(aq) \rightarrow AgI(s) \quad (3)$$

Silver fluoride is soluble hence no precipitate is formed. The three precipitates are white or yellow in color and they darken when exposed to light as the light stimulates the reduction of silver ions to silver atoms ("Identification of Halide Ions Using Silver Nitrate" 1). The silver ions are added to the solvent with diluted nitric acid. The nitric acid decomposes the carbonate ions presented in the solvent, forming a white precipitate of silver carbonate as shown in equation 4 (Clark 1):

$$2Ag^+(aq) + CO_3^{2-}(aq) \rightarrow Ag_2CO_3(s) \quad (4)$$

Nitric acid reacts with and removes other ions that might form precipitates with silver nitrate as shown in equation 5:

$$CO_3^{2-}(aq) + 2HNO_3(aq) \rightarrow CO_2(g) + H_2O(l) + NO^{3-}(aq) \quad (5)$$

The addition of nitric acid confirms that the precipitates formed indeed consist of halogens (Clark 1).

# 3.0 Experimental Apparatus and Techniques

## 3.1 Experimental Apparatus

The experimental apparatus for the silver nitrate test consists of a magnetic stirrer, automatic pipettes, analytical balance, centrifuge, and a vacuum set-up. The magnetic stirrer is used to mix silver nitrate in 95% ethanol to make 2wt% silver nitrate solution and the automatic pipettes are used to transfer samples and chemicals for analysis.

The analytical balance is used to determine the chemical and precipitates weights. The centrifuge is used to separate the solid precipitate and the supernatant and is shown in Figure 1.

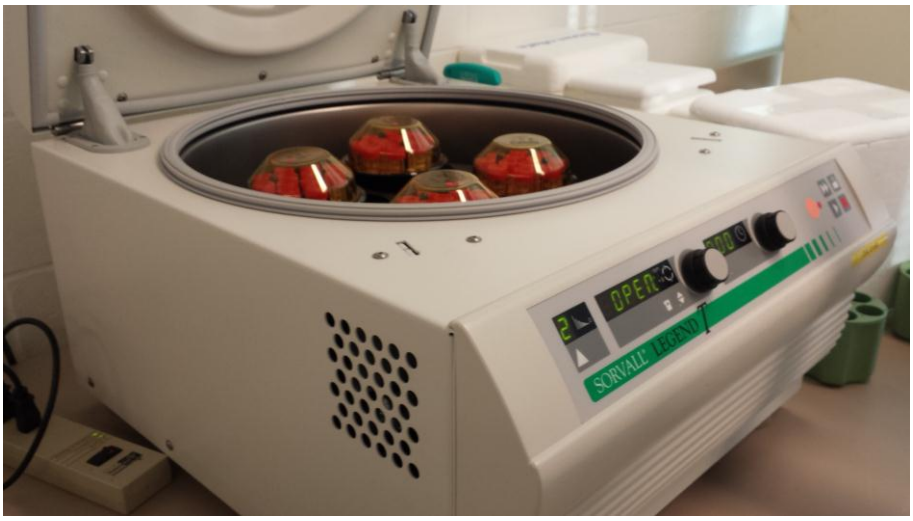


**Figure 1: Thermo Scientific™ Sorvall™ Legend™ T Plus Centrifuge**

Finally, the laboratory vacuum set-up consists of a vacuum flask connected to an aspirator with rubber tubing to create a vacuum in the flask as shown in Figure 2. The top of the flask is sealed and is connected to a tube, through which the supernatant is collected in the flask.

**Figure 2: Laboratory Vacuum Set-Up**

### 3.2 Experimental Techniques

5 mL of each sample were first transferred to 15 mL conical vials. This is followed by addition of 1 mL of silver nitrate and 1 mL of nitric acid. Once the precipitates were formed, the samples were then centrifuged and the supernatant was removed using the vacuum set-up. The test samples were prepared in the first lab period and during the next week it was observed that some of the volatile chemicals in each sample had evaporated. In order to avoid any miscalculations, the volume of the samples was filled back to 5 mL with a 30% ethanol mixture. The dried precipitate samples were then weighed to determine the presence and amount of halogens in each sample.

The chemicals used in the silver nitrate test are 2% silver nitrate solution in ethanol and 5% nitric acid. The detailed documentation on the experimental procedure can be found in Appendix B.

## 4.0 Results and Discussion

The silver nitrate test to determine the presence of halogens was carried out on 36 out of the 50 non-halogenated samples and 20 out of the 50 halogenated samples collected. The halogenated samples were tested for the presence of halogens in order to verify that the waste in the halogenated containers is indeed halogenated. The samples that contained significant amounts of suspended solids or foreign particles were excluded from the testing as the solids can interfere with the results of the experiment. In addition, the result accuracy is highly dependent on the formation of solid precipitates.

1 mL of silver nitrate and 1 mL of nitric acid was added to 5 mL of each sample in weighed vials. After the addition of chemicals, samples were first centrifuged at a rate of 3000 rpm for 5 minutes. However some of the samples still had solid particles floating in the supernatant liquid; therefore, the settings of the centrifuge were changed to 4000 rpm for 10 minutes to ensure complete settling of the solid precipitates. The supernatant was then removed from the vials using a vacuum set-up leaving the solid precipitates at the bottom of the vials. The open vials with wet precipitates were then placed under fume hood for a week to remove any moisture in the precipitates before weighing the vials.

The samples were visually observed for the presence of any precipitates in each vial. Based on the visual observations, 5 out of 20 halogenated samples did not have a precipitate formation whereas out of 21 out of 36 non-halogenated samples showed the formation of precipitates. However, it should be noted that the 5 of the halogenated samples that did not form any precipitates may contain fluoride ions which can not be detected from the silver nitrate test as described in Section 2. After the drying of the precipitates under a fume hood, the vials were then weighed to obtain accurate qualitative results of the experiment. A simple calculation of the difference between the weight of the vials after the experiment and before the experiment gives the mass of precipitate in each vial.

Based on the weight difference results, 22 out of 36 non-halogenated samples contain halogens and 2 out of the 20 halogenated samples contained no precipitate or gave a negative mass reading. Among the non-halogenated samples that tested positive for the presence of halogens, 8 samples contain a precipitate mass greater than 0.05 g and the remaining samples have a precipitate mass less than 0.05 g. 5 of the non-halogenated samples, namely, NH9, NH11,

NH28, NH36 and NH41 are inconclusive for the presence or absence of halogens as the mass results of the sample and its duplicate are inconsistent with each other. Some samples that did not contain precipitates gave a negative mass value when weighed out. These negative mass results can be attributed to inconsistent experimental conditions such as humidity. While the silver nitrate test does not give an accurate indication of the concentration of the halogens in the samples, it does show that 61% (Figure 3) of the samples that were collected from non-halogenated container do in fact contain halogens and 10% samples from halogenated waste containers do not contain any halogens. Raw data tables can be found in Appendix A.

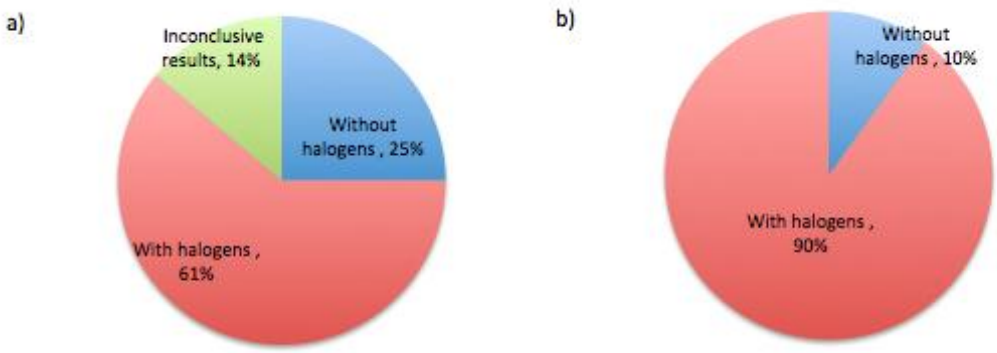


**Figure 3: a) Non-halogenated Test Results, b) Halogenated Test Results**

## 5.0 Safety and Environmental Findings

To ensure safety of team members during the experiment, safety audits were conducted before, during and after every experiment with the safety checklist in Appendix C. Any safety and environmental issues found were properly addressed by team members with the instructor. Team members were also reminded of emergency procedures: instruction and location of emergency eye-wash, shower, spill kit, and fire extinguisher, before conducting experiments.

The major risks of this experiment are the exposure to hazardous samples while collecting samples from ESF and conducting the experiment. To prevent exposure, members wore adequate personal protective equipment. In addition, all experiments were conducted either under the fume hood or an elephant trunk to prevent the inhalation of chemical vapors.

The limited work space in the fume hood presented a safety concern as it increased the risk of chemicals, transferring samples, or adding nitric acid or silver nitrate to small vials. To address this safety hazard, further work was conducted in a larger fume hood with only two members working at any given time. Also, team members were rotating work shift every thirty minutes to prevent any incidence due to the lack of concentration or fatigue.

Furthermore, during the centrifugation stage, lab members were instructed on the operation of the centrifuge by the instructor before using the apparatus. The importance of balancing the centrifuge was understood by all lab members, resulting in no incidences with the centrifugation section of the experiment.

Proper disposal of waste products and samples were audited to ensure that no environmental contamination was caused. Used micropipette tips were collected in a beaker and disposed of in plastic bags provided by the instructor. All liquid wastes were collected and disposed of in a red halogenated waste container. No solutions were disposed of in drains to ensure that any contamination of halogenated waste cannot occur.

If this project is scaled up to facilitate ESF's needs, using disposable vials and pipettes will produce an excessive amount waste and a small fume hood will not provide an adequate amount of space due to highly volatile and toxic chemicals in the waste. A small and well-ventilated room is one possible solution for these safety and environmental issues.

# 6.0 Proposed Design

One of the objectives of our project is to design a feasible protocol for ESF's halogenated waste segregation. The proposed design will qualitatively identify the waste samples containing halogens and allow ESF to identify laboratories practicing incorrect labeling and disposal of halogenated waste.

## 6.1 Experiment Overview

The silver nitrate test qualitatively analyzes the presence of halogen ions in waste solvents. As discussed in the Section 2.1, addition of the silver nitrate solution into the waste sample forms precipitates in presence of chlorine, bromine and iodine. The presence of halogen ions is then confirmed by further reacting the sample with 1N nitric acid. The experiment was conducted on a sample population of 56 waste samples and it is proposed to scale up for ESF's needs.

## 6.2 Advantages and Disadvantages

The major advantage of the proposed method is that it provides a quick visual analysis of the presence or absence of halogens. ESF technicians can perform the experiment on a large sample population in a short period of time and identify the laboratories incorrectly disposing of halogenated wastes. Another advantage is that the experiment does not need to be performed to a high degree of accuracy.

The major disadvantage is that this method cannot detect the presence of fluoride ion as it was described in section 2. Furthermore, the method does not determine the halogen ion concentration.

## 6.3 Scale-Up and Cost Analysis

The cost of disposal of halogenated waste and non-halogenated waste are $1.65/L and $0.80/L, respectively. Thus, ESF can save up to $0.85/L of waste by correctly segregating halogenated and non-halogenated waste. The following cost analysis was performed to ensure the cost savings of the proposed design.

To perform the analysis on a larger scale, it is recommended that the ESF invest on automatic bottle top dispensers (Figure 4) to dispense 1 mL of silver nitrate and nitric acid into

the waste sample. This will greatly reduce the time and labour required to conduct the experiment. Two dispensers can be purchased for a total of $708.44. Alternative options such as automatic burettes and multichannel pipettes were explored but they were rejected due to high capital cost and subsequent operating cost, such as the cost of pipette tips.

**Figure 4: Labmax Universal Bottle Top Dispenser** (LW0801 1)

The total cost of silver nitrate, 1N nitric acid, and 95% ethanol for the test is $0.35 per sample, including duplicates. For the experiment, conical sterile vials were used, which can be bought for $0.42 per sample. However, use of such vials is not recommended in a scale-up as it is very costly and creates unnecessary waste. Instead, it is recommended that ESF invest in reusable, 8mL glass vials (Figure 5). This will limit the operating cost at $0.35 per sample. The total capital cost including 2 bottle top dispensers and 200 glass vials sums up to $865.21.

**Figure 5: Clear Glass Vial** (Glass Vials 1)

Capital costs of various equipment and operational cost per waste sample are summarized in Tables 1 and 2. Non-recommended options are in italicized text and recommendations and required chemicals are in bold text.

**Table 1: Various Capital Cost Options**

| Equipment | $/Unit | Quantity Needed | Total Capital Cost (C$) |
|---|---|---|---|
| *Automatic Burette*[1] | *680.22* | *2* | *1360.44* |
| *Multichannel Pipette*[2] | *1,297.62* | *1* | *1297.62* |
| **Automatic Bottle Top Dispensers**[3] | **354.22** | **2** | **708.44** |
| *Centrifuge*[4] | *5,653.63* | *1* | *5653.63* |
| **8mL Glass Vial**[5] | **0.78** | **200** | **156.77** |

**Table 2: Operating Cost**

| Equipment or Chemical | Cost | Quantity Needed/ Sample | Operating Cost (C$/ Sample) |
|---|---|---|---|
| *15mL Sterile Conical Vials*[6] | *$0.21/vial* | *2* | *$0.42* |
| **Silver Nitrate**[7] | **$2.75/g** | **0.0398g** | **$0.11** |
| **1N Nitric Acid Solution**[8] | **$34.10/L** | **0.002L** | **$0.07** |
| **95% Ethanol**[9] | **$70.40/L** | **0.0024L** | **$0.17** [10] |

[1] (Buret 1)
[2] (50-1200µl ELINE Pipette, 8-Channel 1)
[3] (0.25-2.5ml Labmax Universal Bottle Top Dispenser 1)
[4] (Thermo Scientific Heraeus Megafuge16 Centrifuge 1)
[5] (Glass Vials 1)
[6] (15ml APEX Essential Centrifuge Tube, Loose, Sterile 1)
[7] (Silver Nitrate | Sigma-Aldrich 1)
[8] (Nitric Acid | Sigma-Aldrich 1)
[9] (Ethanol | Sigma-Aldrich 1)

## 7.0 Quality Control Issues

Cross contamination is a concern during the experiment as both halogenated and non-halogenated samples are being tested. To ensure that accurate results are obtained, halogenated samples and non-halogenated samples obtained from the facility are stored in separate boxes and each sample has a duplicate to ensure that experimental outcomes agree with each other.

To minimize the possibility of sample contamination during the experiment, automatic pipettes were used and a new pipette tip was used for every sample. The same pipette tip was used to transfer the sample and the duplicate into separate vials. A batch of non-halogenated samples was first transferred, which was then followed by the transfer of halogenated sample. Contamination is not a great concern while adding nitric acid and silver nitrate to the samples. However, to ensure the quality of the results, pipette tips were discarded for every few samples taken.

To avoid errors, all vial bodies and caps were clearly labeled. The list is found in Tables A1 and A2 in Appendix A. To ensure consistency in the weight measurements, all vials were weighed using the same balance. The original settings of the centrifuge were changed to 4000 rpm for 15 minutes to confirm the settling of all the precipitate at the bottom of the vials. Some samples contained precipitates that were floating at the top or stuck to the walls of the vial. To ensure that these precipitates were not removed in the process of discarding the supernatant, great care was taken during the separation of the experiment. To ensure that no moisture remains in the samples after vacuuming out the supernatant, the vials were stored in a fume hood in order to dry out those samples.

## 8.0 Conclusions and Recommendations

The project, *Halogenated Organics Identification: Qualitative Analysis,* described in this report indicates the presence of halogens in the waste samples collected from ESF. This is to ensure that non-halogenated waste is not being disposed of as halogenated waste. The results from the silver nitrate test shows that approximately 61% of the non-halogenated samples contain halogens. The economic analysis concluded that the cost of chemical for testing one sample is $0.77. Therefore, the silver nitrate test was found to be a feasible and simple method for testing the presence of halogens in waste containers collected by the ESF. The results of this experiment can be used to identify the laboratories that do not dispose of non-halogenated and halogenated waste properly and warn them.

The error in measuring the mass of precipitate occurred due to inconsistent calibration of the scale from the time when empty vials were weighed and loss of precipitates during the vacuum stage of experiment. To ensure reproducible results from the experiment, contamination and loss of precipitates during silver nitrate test should be minimized and completing the silver nitrate test and measuring the mass of precipitate on the same day possible by using a drier or an oven to dry up the precipitate in a short period of time. To get more accurate and precise results, larger sample population should be tested and it is recommended that ESF invest on automatic burette and centrifuge for a more efficient analysis.

As a major safety concern in this experiment is the to harmful waste chemicals, a small and well ventilated room can minimize the chance of vapor inhalation and maximize the working space compared to working in a fume hood. In addition, work should be completed in shifts in order to avoid excess fatigue. Furthermore, to minimize the waste from the experiment, glass vials are recommended.

To further improve the quality of the results of this experiment, it is recommended to increase the sample volume in order to generate greater mass of the precipitate that can be accurately weighed by the available analytical balances. In a scale-up analysis, it was concluded that the centrifugation step will significantly increase the capital cost, operating cost and the time required for the experiment. Hence, for a scale-up, it is recommended that ESF limit the objective of the analysis only to qualitatively determining the presence of halogens through

visual observation. The per sample operating cost will be reduced to $0.35 which is just the cost of chemicals as opposed to $0.77.

With the data that our group has collected, a subsequent lab group will contact the generators of the waste samples that have been incorrectly disposing of their waste to ensure that the problem does not persist. Subsequent groups will also continue the project by testing the water content of non-halogenated samples. This is to be done because non-halogenated wastes containing excessive water cannot be used as fuel additives. In addition, another group will further explore a method of determining the exact halogen concentration in a waste sample.

By completing the PBL project, chemical engineering students are contributing to a more sustainable campus by promoting sustainable practices in laboratories across campus. In order to create a large impact on the environment and economy, small steps must first be taken. Raising the awareness of correct waste disposal on campus is the first step that we have taken to create a truly significant impact.

## 9.0 Acknowledgements

Our team would like to extend our gratitude towards everyone who provided assistance in our project. In particular, we would like to thank Mr. Bang Dang and all members of the UBC Environmental Services Facility (ESF) for providing us with guidance and cooperation throughout the course of our investigation. We also would like to acknowledge our colleagues in team 1 and 2 for their hard work in providing us with the assistance needed to initiate the project. Finally, we are greatly thankful to Dr. Christina Gyenge for the opportunity, guidance and support during this project.

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# Appendix A- Raw Data

**Table A1: Non-halogenated Waste Sample Analysis**

| Sample Name | Replicate # | ESF Sample Identity | Visual precipitation observed (Y/N) | Weight of Empty vial, g | Weight of vial and precipitate, g | *Weight of Precipitate, g* | Precipitation (Y/N) |
|---|---|---|---|---|---|---|---|
| NH1 | 1 | S0111009077 | Y | 6.2300 | 6.2209 | *-0.0091* | N |
| | 2 | | | 6.2596 | 6.2437 | *-0.0159* | |
| NH2 | 1 | S081201504 | Y | 6.2623 | 6.2318 | *-0.0305* | N |
| | 2 | | | 6.2707 | 6.2498 | *-0.0209* | |
| NH3 | 1 | S081201529 | Y | 6.2010 | 6.1952 | *-0.0058* | N |
| | 2 | | | 6.2312 | 6.2119 | *-0.0193* | |
| NH5 | 1 | S0111009794 | N | 6.2457 | 6.2369 | *-0.0088* | N |
| | 2 | | | 6.2246 | 6.2083 | *-0.0163* | |
| NH6 | 1 | S0111009799 | N | 6.2528 | 6.2404 | *-0.0124* | N |
| | 2 | | | 6.1754 | 6.1541 | *-0.0213* | |
| NH7 | 1 | S0111009798 | N | 6.2264 | 6.1937 | *-0.0327* | N |
| | 2 | | | 6.1909 | 6.1511 | *-0.0398* | |
| NH8 | 1 | S0111008672 | N | 6.2477 | 6.2223 | *-0.0254* | N |
| | 2 | | | 6.1907 | 6.1468 | *-0.0439* | |
| NH9 | 1 | S081201513 | Y | 6.2444 | 6.2437 | *-0.0007* | Inconclusive |
| | 2 | | | 6.2456 | 6.2584 | *0.0128* | |
| NH10 | 1 | S081201117 | Y | 6.1971 | 6.3082 | *0.1111* | Y |
| | 2 | | | 6.2705 | 6.3618 | *0.0913* | |
| NH11 | 1 | S020705673 | Y | 6.2192 | 6.2515 | *0.0323* | Inconclusive |
| | 2 | | | 6.3689 | 6.2226 | *-0.1463* | |
| NH16 | 1 | S081200812 | Y | 6.2160 | 6.2790 | *0.0630* | Y |
| | 2 | | | 6.2706 | 6.3311 | *0.0605* | |
| NH17 | 1 | S081200813 | N | 6.3417 | 6.4064 | *0.0647* | Y |
| | 2 | | | 6.2111 | 6.2734 | *0.0623* | |
| NH18 | 1 | S0309004908 | Y | 6.2270 | 6.2364 | *0.0094* | Y |
| | 2 | | | 6.2076 | 6.2190 | *0.0114* | |
| NH19 | 1 | S081203083 | N | 6.2387 | 6.2394 | *0.0007* | Y |
| | 2 | | | 6.2319 | 6.2333 | *0.0014* | |
| NH21 | 1 | B060814481 | N | 6.2286 | 6.2622 | *0.0336* | Y |
| | 2 | | | 6.2315 | 6.2608 | *0.0293* | |
| NH22 | 1 | S020703419 | Y | 6.2566 | 6.2566 | *0.0000* | N |
| | 2 | | | 6.2334 | 6.2300 | *-0.0034* | |
| NH23 | 1 | S0111006735 | Y | 6.2471 | 6.2947 | *0.0476* | Y |
| | 2 | | | 6.2356 | 6.2819 | *0.0463* | |
| NH24 | 1 | S020703669 | Y | 6.2410 | 6.2448 | *0.0038* | Y |
| | 2 | | | 6.2094 | 6.2150 | *0.0056* | |
| NH25 | 1 | S020703279 | Y | 6.3429 | 6.3503 | *0.0074* | Y |
| | 2 | | | 6.2524 | 6.2630 | *0.0106* | |
| NH26 | 1 | S000058499 | Y | 6.2451 | 6.4045 | *0.1594* | Y |
| | 2 | | | 6.2064 | 6.3588 | *0.1524* | |

*Table A1 continued on page A2*

| Sample Name | Replicate # | ESF Sample Identity | Visual precipitation observed (Y/N) | Weight of Empty vial, g | Weight of vial and precipitate, g | *Weight of Precipitate, g* | Precipitation (Y/N) |
|---|---|---|---|---|---|---|---|
| NH27 | 1 | S0111007890 | Y | 6.2056 | 6.2095 | *0.0039* | Y |
| | 2 | | | 6.2092 | 6.2119 | *0.0027* | |
| NH28 | 1 | S0111007892 | N | 6.1952 | 6.1948 | *-0.0004* | Inconclusive |
| | 2 | | | 6.2138 | 6.2155 | *0.0017* | |
| NH30 | 1 | S081201623 | N | 6.1747 | 6.1746 | *-0.0001* | N |
| | 2 | | | 6.1984 | 6.1950 | *-0.0034* | |
| NH31 | 1 | S0111009451 | N | 6.2387 | 6.3458 | *0.1071* | Y |
| | 2 | | | 6.2316 | 6.3414 | *0.1098* | |
| NH33 | 1 | S0111006740 | Y | 6.2060 | 6.2949 | *0.0889* | Y |
| | 2 | | | 6.2301 | 6.3191 | *0.0890* | |
| NH34 | 1 | S0111006741 | Y | 6.2318 | 6.2896 | *0.0578* | Y |
| | 2 | | | 6.2116 | 6.2958 | *0.0842* | |
| NH36 | 1 | S0111003883 | N | 6.2131 | 6.2152 | *0.0021* | Inconclusive |
| | 2 | | | 6.2239 | 6.2204 | *-0.0035* | |
| NH37 | 1 | S081203100 | N | 6.2279 | 6.2299 | *0.0020* | Y |
| | 2 | | | 6.2050 | 6.2064 | *0.0014* | |
| NH39 | 1 | S020706225 | Y | 6.2131 | 6.2332 | *0.0201* | Y |
| | 2 | | | 6.2319 | 6.2523 | *0.0204* | |
| NH40 | 1 | S0111009073 | Y | 6.1987 | 6.2300 | *0.0313* | Y |
| | 2 | | | 6.2376 | 6.3952 | *0.1576* | |
| NH41 | 1 | S081203093 | Y | 6.2523 | 6.2561 | *0.0038* | Inconclusive |
| | 2 | | | 6.2450 | 6.2422 | *-0.0028* | |
| NH42 | 1 | S081201119 | N | 6.1510 | 6.2351 | *0.0841* | Y |
| | 2 | | | 6.2375 | 6.3289 | *0.0914* | |
| NH43 | 1 | S0309009803 | N | 6.2195 | 6.2222 | *0.0027* | Y |
| | 2 | | | 6.1986 | 6.2030 | *0.0044* | |
| NH45 | 1 | S081202393 | N | 6.1429 | 6.1497 | *0.0068* | Y |
| | 2 | | | 6.2402 | 6.2443 | *0.0041* | |
| NH48 | 1 | S081202393 | Y | 6.2036 | 6.2302 | *0.0266* | Y |
| | 2 | | | 6.1931 | 6.2113 | *0.0182* | |
| NH49 | 1 | S0309009802 | Y | 6.2314 | 6.2354 | *0.0040* | Y |
| | 2 | | | 6.2325 | 6.2380 | *0.0055* | |

**Table A2: Halogenated Sample Analysis**

| Sample Name | Replicate # | ESF Sample Identity | Visual precipitation observed (Y/N) | Weight of Empty vial, g | Weight of vial and precipitate, g | *Weight of Precipitate, g* | Precipitation (Y/N) |
|---|---|---|---|---|---|---|---|
| H1 | 1 | S0309002811 | N | 6.2200 | 6.3056 | *0.0856* | Y |
| | 2 | | | 6.1713 | 6.2557 | *0.0844* | |
| H4 | 1 | S000039108 | Y | 6.3644 | 6.3708 | *0.0064* | Y |
| | 2 | | | 6.2477 | 6.2542 | *0.0065* | |
| H8 | 1 | S0111000299 | Y | 6.2374 | 6.3291 | *0.0917* | Y |
| | 2 | | | 6.2253 | 6.3089 | *0.0836* | |
| H9 | 1 | S081200946 | Y | 6.2209 | 6.2676 | *0.0467* | Y |
| | 2 | | | 6.2316 | 6.2751 | *0.0435* | |
| H10 | 1 | S001004450 | Y | 6.3534 | 6.4241 | *0.0707* | Y |
| | 2 | | | 6.2421 | 6.3098 | *0.0677* | |
| H13 | 1 | S020701976 | N | 6.2152 | 6.2125 | *-0.0027* | N |
| | 2 | | | 6.2529 | 6.2481 | *-0.0048* | |
| H14 | 1 | S081201044 | Y | 6.2413 | 6.3185 | *0.0772* | Y |
| | 2 | | | 6.2528 | 6.3314 | *0.0786* | |
| H20 | 1 | S081200939 | Y | 6.2561 | 6.3061 | *0.0500* | Y |
| | 2 | | | 6.1933 | 6.2404 | *0.0471* | |
| H21 | 1 | S0111004033 | N | 6.2078 | 6.2378 | *0.0300* | Y |
| | 2 | | | 6.2465 | 6.2841 | *0.0376* | |
| H24 | 1 | S081201304 | N | 6.2377 | 6.2351 | *-0.0026* | N |
| | 2 | | | 6.2430 | 6.2418 | *-0.0012* | |
| H25 | 1 | S081201042 | Y | 6.2066 | 6.2847 | *0.0781* | Y |
| | 2 | | | 6.2485 | 6.3284 | *0.0799* | |
| H26 | 1 | S0111009659 | Y | 6.2690 | 6.3305 | *0.0615* | Y |
| | 2 | | | 6.2563 | 6.3174 | *0.0611* | |
| H28 | 1 | S081202579 | Y | 6.1674 | 6.2496 | *0.0822* | Y |
| | 2 | | | 6.2182 | 6.3149 | *0.0967* | |
| H29 | 1 | S0111009159 | Y | 6.2325 | 6.2786 | *0.0461* | Y |
| | 2 | | | 6.2275 | 6.2712 | *0.0437* | |
| H33 | 1 | S081201052 | Y | 6.1695 | 6.2345 | *0.0650* | Y |
| | 2 | | | 6.2684 | 6.3339 | *0.0655* | |
| H36 | 1 | S081201057 | Y | 6.2449 | 6.2682 | *0.0233* | Y |
| | 2 | | | 6.2088 | 6.2141 | *0.0053* | |
| H38 | 1 | S0111006811 | N | 6.3697 | 6.4500 | *0.0803* | Y |
| | 2 | | | 6.2461 | 6.3220 | *0.0759* | |

*Table A2 continued on page A4*

| Sample Name | Replicate # | ESF Sample Identity | Visual precipitation observed (Y/N) | Weight of Empty vial, g | Weight of vial and precipitate, g | *Weight of Precipitate, g* | Precipitation (Y/N) |
|---|---|---|---|---|---|---|---|
| H41 | 1 | S081201081 | Y | 6.2684 | 6.3270 | *0.0586* | Y |
| | 2 | | | 6.2177 | 6.2737 | *0.0560* | |
| H46 | 1 | S001005076 | Y | 6.2317 | 6.2492 | *0.0175* | Y |
| | 2 | | | 6.2130 | 6.2251 | *0.0121* | |
| H49 | 1 | S0111009158 | Y | 6.2331 | 6.2682 | *0.0351* | Y |
| | 2 | | | 6.2309 | 6.2619 | *0.0310* | |

# Appendix B - Experimental Procedure

## Silver Nitrate Test Experimental Procedure

The following procedure outlines the laboratory-scale Silver Nitrate test used in the experiment to qualitatively analyze halogenated waste samples. *This document was prepared by Team 2 on October 31, 2013 and revised by Team 4 on January 14, 2014.*

Solutions:

- 1N Nitric Acid
- 2 wt% Silver Nitrate solution (see solution preparation)
- 95% Ethanol

Equipment:

- 15mL conical centrifuge tubes
- 1mL automatic pipette & tips
- 10mL automatic pipette & tips
- analytical balance
- magnetic stirrer
- Parafilm
- centrifuge
- laboratory vacuum apparatus:1000mL Erlenmeyer flask with vacuum port, rubber stopper, tubing, glass pipette tip

Solution Preparation:

*2wt% Silver Nitrate Preparation*

1. Weigh 0.9961 g of silver nitrate powder in a 50 mL beaker
2. Add 48.81 g of 95% ethanol to the beaker
3. Place a magnetic stir bar in the beaker
4. Cover the beaker with a Parafilm and place it on magnetic stirrer; stir at high speed for 15 minutes until the silver nitrate is completely dissolved

Procedure

1. Label and weigh empty 15 mL vials with their cap on using analytical balance; 2 per waste sample, label both cap and vial
2. From the original waste sample collected from ESF, transfer 5 mL of sample to each vial using 10 mL automatic pipette
3. Add 1 mL of silver nitrate solution using 1 mL automatic pipette to the vials
4. Add 1 mL of 1N nitric acid to the vials
5. Close the cap and mix well to allow precipitation
6. Centrifuge the vials at 4000 rpm for 10 minutes; ensure that all precipitates are collected at the bottom of the conical vial, repeat centrifugation if necessary
7. Using the laboratory vacuum apparatus, carefully remove the supernatant
8. Allow for the collected solids to dry for a week
9. Weigh the vials (with caps on) to find the amount of solids accumulated

***Note:*** *Perform all procedure in fume hood*

## Appendix C – Laboratory Safety Inspection Checklist

Date: _____ Performed by:_____

Time: _____ Room: _____

| General Laboratory Safety | Yes | No | Comments |
|---|---|---|---|
| Working area clean and clear | | | |
| First aid kit in close proximity | | | |
| Fire extinguisher in close proximity | | | |
| Eyewash and showers in close proximity | | | |
| Personal protective equipment equipped | | | |
| Emergency contacts available | | | |
| Emergency shut-down procedure available | | | |
| **Chemical Safety** | **Yes** | **No** | **Comments** |
| MSDS of chemicals available | | | |
| Chemicals properly stored and labeled | | | |
| Containers compatible with the chemicals | | | |
| Flammable chemicals away from heat source | | | |
| Titration performed within fume hood | | | |
| **Waste Disposal** | **Yes** | **No** | **Comments** |
| Waste container labeled | | | |
| Waste container compatible with waste | | | |
| Waste disposed to specific container | | | |
| **Other** | **Yes** | **No** | **Comments** |
| | | | |
| | | | |