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CVG 2132 LABORATORY REPORT EVALUATION CRITERIA

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Lab Title: Nutrients in Water (Ammonia, Nitrate, and Phosphorous)

Group Number: Group 5 & Group 7 (combined as permission)

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University of Ottawa

Fundamentals of Environmental Engineering - CVG2132

Nutrients in Water (Ammonia, Nitrate, and Phosphorous)

Due Date: Monday, Dec 3, 2018
Members:

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OBJECTIVES

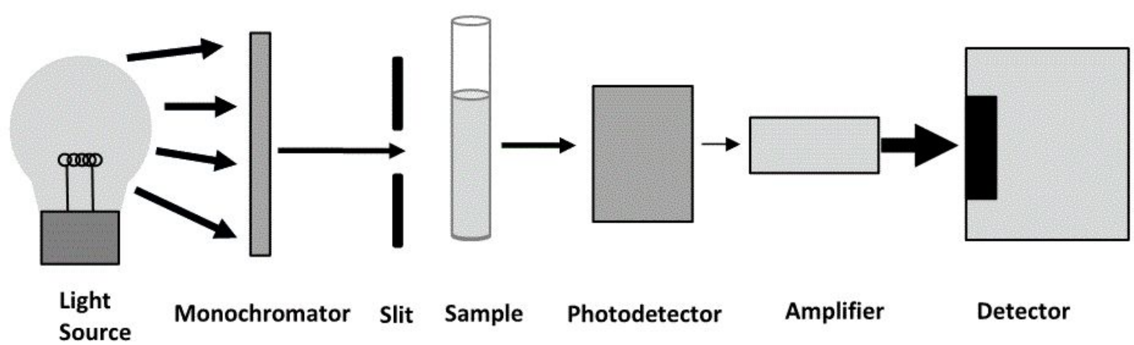
The objective of this laboratory experiment is to explore the nutrient concentrations of Ammonia, Nitrate, and Phosphorus in wastewater samples. The concentrations are analyzed using Direct Nesslerization method for Ammonia, Dimethylphenol method for Nitrate, and Stannous Chloride method for Phosphate. Also, the experiment enlightens that sources and effects of nutrients in water human should pay attention and deal with are significant points

THEORY

Certain water nutrients are essential for plant-based lives. However, some lakes and rivers have been ravaged from eutrophication due to excess nutrients in water system. High nutrient concentration affects aquatic lifeforms and this phenomenon caused by the bloom of algae on the surface of the water generates a barrier that reduces sunlight exposure for deep-water producers. The result of eutrophication of water system is minimize photosynthesis from producers so that the low dissolved oxygen (DO) level occurs. It is important to control the quantity of nutrients and avoid the eutrophication in the environment.

In this laboratory, the key nutrients that will be analyzed are ammonia, nitrate, and phosphorous. The analysis of the nutrients consists of colorimetric analysis through the use of a spectrophotometer. The working theory can refer to lab manual and shows in following figure:

Figure 1. Schematic diagram of spectrophotometers



Spectrophotometers measures the absorbance, a calibration curve is generated by plotting a graph of absorbance vs. concentration. Also, a trendline created as a linear function along the calibration curve can obtain certain unknown concentrations of nutrients through given absorbances.

Ammonia (NH3-NH4) is the first nutrient analyzed and it is a compound present in countless industrial chemicals; produced both naturally and through human manufacturing. Ammonia exists in two different states, depending on factors such as pH and temperature. It is typically found in its un-ionized form, NH3, and ionized form, NH4. Two states are related through the following equilibrium equations:

$$NH_4 + OH^- \leftrightarrow NH_3 + H_2O$$

$$NH_3 + H_3O^+ \leftrightarrow NH_4 + H_2O$$

To measure ammonia content, this experiment uses the Nesslerization process, which involves Nessler's reagent (K_2HgI_4) and a mineral stabilizing salt. Under strong alkali conditions, Nessler's reagent (K2HgI4) will react with the un-ionized ammonia, resulting in colour development within the sample, ranging from clear to dark brown. This change in colour is directly proportional to the quantity of ammonia present. A stabilizing salt solution, in this laboratory Rochelle's salt was used, which consists of potassium sodium tartrate tetrahydrate is also typically added to reduce chemical interferences such as the precipitation of Nessler's reagent due to hardness. Once the reaction is complete, a spectrophotometer is utilized to establish the absorbance of the sample at a wavelength of 425nm.

Nitrate is the second nutrient analyzed and results from the bond between one nitrogen and three oxygen atoms. Natural waterways would contain minimal nitrate quantities through nitrification reactions. The excessive nitrates, as low as 30 mg/L, in the water have a wide range of side effects on the environment and humans themselves, from impeding fish growth and immune system development, to being linked to "blue-baby syndrome" in infants. Nitrate testing in this lab will involve HACH TNT835 commercial test kits where the dimethylphenol method is used. The dimethylphenol method will allow for nitrate concentration measurements between 0.23 and 13.50 mg/L for NO3-N, limited by the presence of organic matter in the water. The dimethylphenol method is the result of nitric acid reacting with 2,6-xylenol (dimethylphenol) in order to form 4-nitro-2,6-xylenol. The resulting reaction will present a green color shift that is proportional to constituent concentration of nitrate and it is indicated by a spectrophotometer.

Figure 2. HACH TNT835 nitrate determination kit

Phosphorous is the third nutrient analyzed and can occur naturally under three forms; orthophosphates, condensed phosphates and organic phosphates. Kinds of Phosphorous include PO_4^{3-}, HPO_4^{2-} , $H_2PO_4^{-}$, and H_3PO_4. Sources of orthophosphates in water include boiler water treatment, laundering and cleansing products, and most significantly, surface runoff containing agricultural fertilizers. The analysis of phosphorous usually entails the following two steps: first all available phosphorous forms of interest must be converted to dissolved orthophosphates and second, the orthophosphates are measured through colorimetric analysis. Interferences includes presence of arsenic, hexavalent chromium, and nitrite
*Refer to (CVG 2132 Lab Manual, 2018)

MATERIALS AND EQUIPMENT

- Sample
- Dilution water
- Nessler's reagent
- Rochelle's salt solution
- 100 mL volumetric flask
- 50 mL volumetric flask
- 30 mL beaker
- 10 mL pipettor
- 1 mL pipettor
- Pipette tips (1 and 10 mL)
- Parafilm squares
- Glass test tubes
- Test tube rack

- Nitrate HACH TNT835 + Nitrate test vial
- Ammonium molybdate (ammonium molybdate, 28% v/v H2SO4)
- Dropper bottle with stannous chloride solution
- 100 mg/L nitrogen ammonia cuvettes
- Yellow square (VIS) glass cuvettes
- Spectrophotometers
- Waste beakers
- Soft duty wipes
- Heating blocks
- 100 mg/L phosphate standard solution

METHODS AND PROCEDURE

Changes for lab 3:
-replace to 1000 ppm stock solution and 50 ml standards
-1ml sample and 0.2 ml solution are pipetted to HACH TNT835.

As seen in the laboratory manual

Results:

Direct Nesslerization for Ammonia Analysis:

Before consulting the test, calibration standards are set by calculating the dilutions required to make 50.00 mL of standard solution using 1000 ppm stock solution. The following table shows how much the volume of 1000 ppm stock solution is required with corresponding standard solution.

Table 1. Volume of stock solution for ammonia calibration standards

Standard	1 ppm	2 ppm	3 ppm	4 ppm	7 ppm	8 ppm	10 ppm
Volume of 1000 ppm stock solution required (mL)	0.05	0.10	0.15	0.20	0.35	0.40	0.50

Sample calculation

$$V_1 = \frac{C_2 \times V_2}{C_1}$$

$$V_1 = \frac{8ppm}{1000ppm} \times 50ml = 0.40ml$$

Calculation:
Part A: Ammonia:

A1:

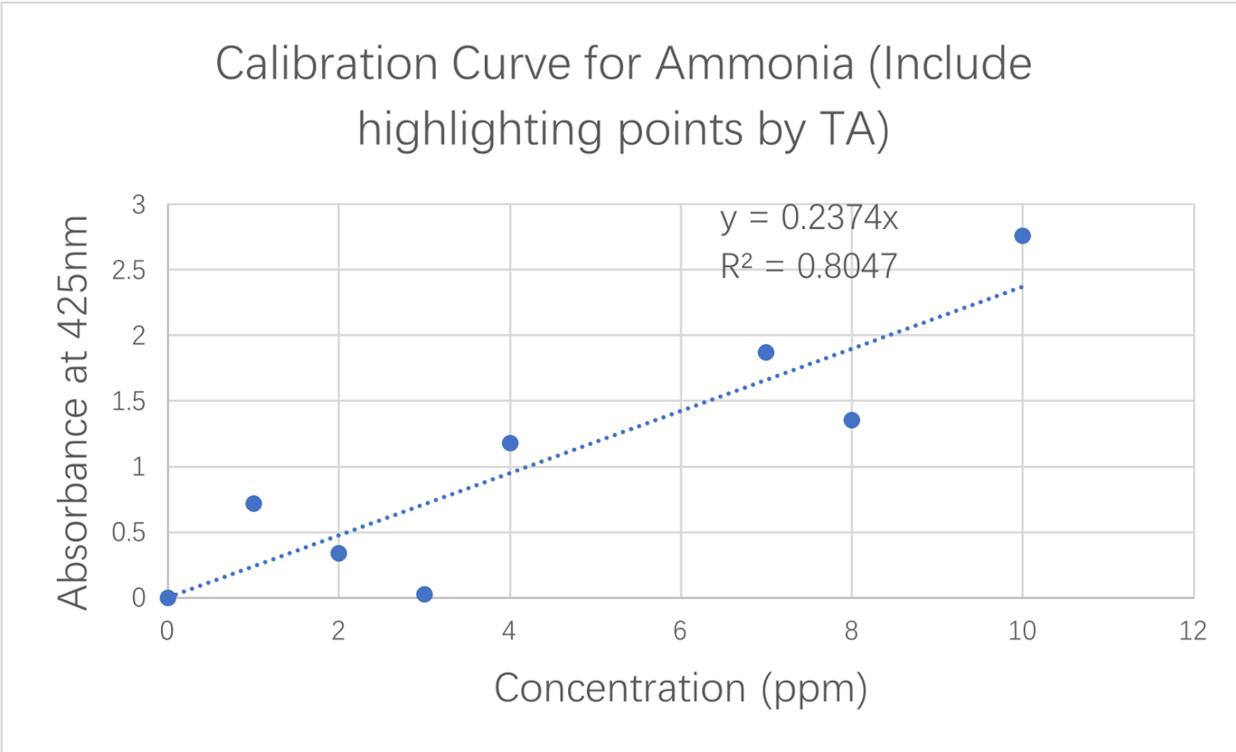
The corresponding absorbances of different wastewater samples measured for plotting a calibration curve of concentration vs. absorbance are listed in table 2:

Table 2. Absorbance values for calibration standards

Group #	**Sample**	**Con. (ppm)**	**Abs at 425 nm**
8	Casselman	1	0.717

6	Stormwater	2	0.34
4	Post Nitrification	3	0.025
1	Casselman	4	1.176
6	Beau's (Dilution 1:10)	7	1.87
Lobo	Stormwater	8	1.352
5	Post Nitrification	10	2.761

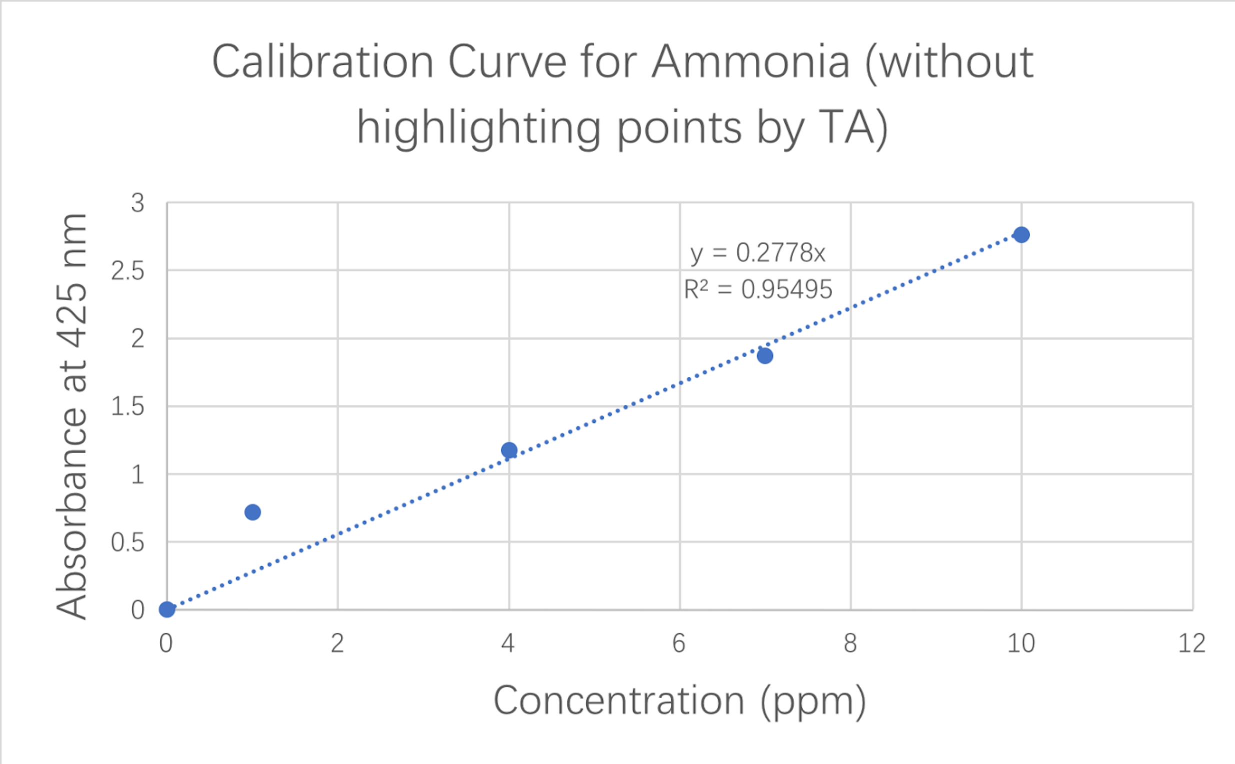
Figure 3. Calibration curve for Ammonia (Include highlighting points by TA)



The absorbance values are supposed to be getting higher as the concentration standards increase and has to be close to zero. The data recorded by different groups in the lab are various and they do not follow the principle so that the graph in figure 1. plotted by absorbance at 425 nm vs. concentration standards is not accurate as theoretical one.

The following figure is a plot without lighting points that generate more accurate trendline than the previous one:

Figure 4. Calibration curve for Ammonia (without highlighting points)



***note:** figure 2. Shows a trendline plotted without highlighting points that follows the principle of higher absorbance along higher concentration. The R square 0.9549 in figure 2. is closer to zero than that of figure 1., which means the equation is more linear. It is important to note that the calibration curve should be an ideal linear line*

A2:

Table 3. Recorded absorbance values with corresponding concentration from different groups

Group	Sample	Sample Dilution	Blank Abs	Sample Abs
8	Casselman	/	0	0.011
6	Stormwater	/	0	0.018

4	Post Nitrification	/	0	0.553
1	Casselman	/	0	0.04
6	Beau's	1:10	0	1.648
Lobo	Stormwater	/	0	0.004
5	Post Nitrification	/	0	0.018

A3:

The linear relationship between absorbance and concentration can be determined through:

$$Absorbance = 0.2778 \times concentration$$

And our group #Lobo absorbance of stormwater is 0.004

$$\text{Concentration} = \frac{\text{Absorbance}}{0.2778} = \frac{0.004}{0.2778} = 0.0144ppm$$

Ammonia concentration of each sample measured by different group is listed in Table below:

Table 4. Ammonia concentration of each sample

Group	Sample	Sample Abs	Concentration (ppm)
8	Casselman	0.011	0.0396
6	Stormwater	0.018	0.0648

4	Post Nitrification	0.553	1.9906
1	Casselman	0.04	0.1440
6	Beau's **(dilution required 1:10)**	0.1648	59.323
Lobo	Stormwater	0.004	0.0144
5	Post Nitrification	0.018	0.0648

Nitrate:

The results of nitrate concentration for each sample measured by different groups is listed in Table 3 below.

Table 5. Nitrate Concentration for each sample

Group #	**Water Source**	**Nitrate Concentration (mg/L)**
8	Casselman	1.3
6	Stormwater	3.17
4	Post Nitrification (Dilution 1:10)	2.81
1	Casselman	1.17
6	Beau’s Brewery	0.492
Lobo	Stormwater	3.07

5	Post Nitrification (Dilution 1:10)	1.97

Phosphorus:

C1:

The calibration curve measurements represented by absorbance at 425nm were measured by different group shown below.

Table 6. Calibration curve measurements for Phosphorus

Group #	Standard cent. (ppm)	Absorbance at 425 nm
8	0.05	0.017
6	0.1	0.097
4	0.2	0.021
1	0.4	0.183
6	0.6	0.259
Lobo	0.8	0.368
5	1	0.69

Figure 5. Calibration curve for phosphorus (include highlighting points)

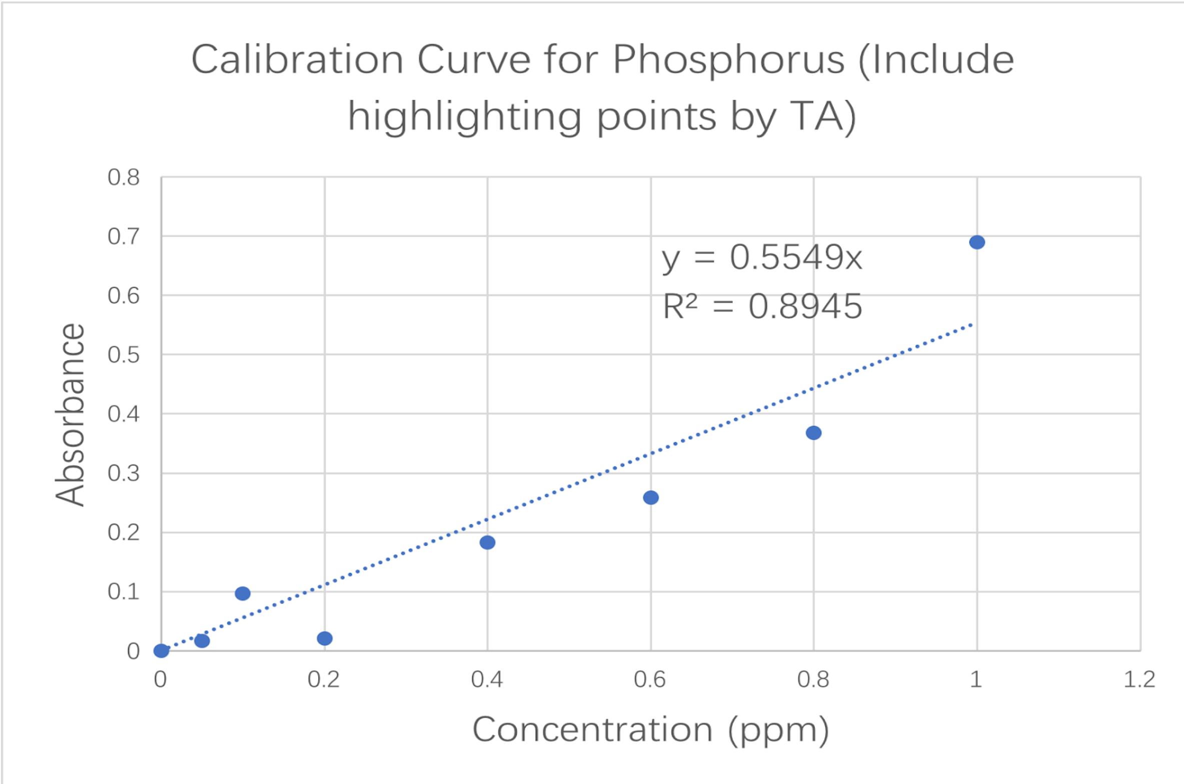
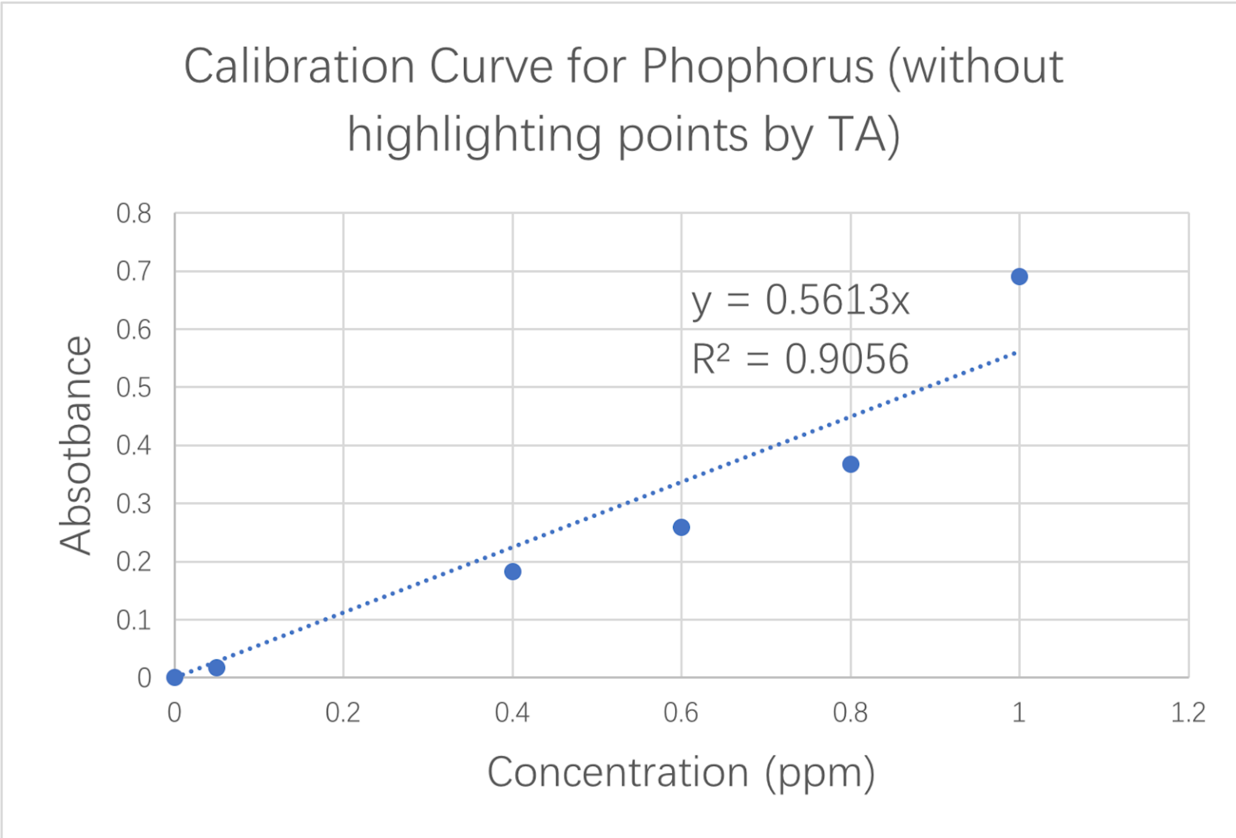


Figure 6. Calibration curve for phosphorus (without highlighting points)



C2:

Table 7. Recorded absorbance values with corresponding concentration from different groups

Group	Sample	Sample Dilution	Blank Abs	Sample Abs
8	Casselman	/	0	0.045
6	Stormwater	/	0	0.135
4	Post Nitrification	1:100	0	0.171
1	Casselman	/	0	0.048

6	Beau's	1:100	0	0.027
Lobo	Stormwater	/	0	0.101
5	Post Nitrification	1:100	0	0.26

C3:

The linear relationship between absorbance and concentration can be determined by

$$\text{Absorbance} = 0.5613\times concentration$$

And our group #Lobo absorbance of stormwater is 0.101

$$\text{Concentration} = \frac{\text{Absorbance}}{0.5613} = \frac{0.101}{0.5613} = 0.1799ppm$$

Phosphorus concentration of each sample measured by different group is listed in Table below:

Table 8. phosphorus concentration of each sample

Group	Sample	Sample Abs	Concentration (ppm)
8	Casselman	0.045	0.0802
6	Stormwater	0.135	0.2405
4	Post Nitrification **(dilution required 1:100)**	0.171	30.4650
1	Casselman	0.048	0.0855
6	Beau's **(dilution required 1:100)**	0.259	4.8103

Lobo	Stormwater	0.101	0.1799
5	Post Nitrification**(dilution required 1:100)**	0.26	46.3210

Discussion

1. The Canadian Wastewater Systems Effluent Regulations state that the maximum concentration of un-ionized ammonia in wastewater effluent is 1.25mg/L. Therefore, the results of the lab state that only Casselman 8, Stormwater 6, Casselman 1, Stormwater Lobo, and Post-Nitrification 5 (ammonia concentrations of 0.0396mg/L, 0.0648mg/L, 0.144mg/L, 0.0144mg/L, 0.0648mg/L, respectively) could be discharged into the environment without treatment. Samples Post-Nitrification 4 and Beau's 6 with ammonia concentrations of 1.9906mg/L and 59.323mg/L, respectively do not meet the regulation. These could not be discharged directly into the environment without undergoing treatment first.
   It is important to note that extremely low concentrations of ammonia in waterways can kill off fish populations as it is toxic. Additionally, the toxicity is amplified when the pH or temperature is increased. For example, if Beau's 6 wastewater with an extremely high ammonia concentration of over 50 mg/L were to be discharged into a given river, the river would experience great ecological upset. Immediately fish can begin to die, and any eggs which are in contact with high concentrations can experience defects. This will negatively influence generations of fish in the area, and the immediate decrease in population could take a long time to re-establish.
2. Ammonia can be treated from wastewater by means of a conventional activated sludge system. These systems include the complete mix, plug flow, and step flow treatment reactors. All of these reactors have similar processes in which the ammonia is converted in the reactors aeration basins.
   The advantage of treating ammonia by sludge systems is that it is a proven method for wastewater treatment that can treat multiple types of wastewater. Sludge streams are also simpler to use as compared to other methods of treatment such as extended aeration, and sequencing batch reactors. The disadvantages to these systems is cost of the reactors and systems, as they are generally made using concrete and the mechanical equipment such as the pumps and the clarifier mechanisms are expensive. Additionally, bulking sludge is more likely to occur in the sludge systems, which requires the addition of an anoxic step or zone to treat.

3. The domestic average for wastewater effluents are as follows: Nitrate concentration of 40 mg/L, Ammonia concentration of 25 mg/L, and phosphorous concentration of 15 mg/L. When looking at the samples from the lab, it is clear that most of the samples are below the average in terms of concentrations of all nutrients. It is worth noting that both Post-Nitrification samples' phosphorus concentrations are much higher than the average at 30.464mg/L and 46.321mg/L. If these results are accurate, then the water would most likely need to undergo treatment processes to ensure that it is safe for excretion into the environment. Additionally, one of the Beau's Brewery samples presented an ammonia concentration of 59.323mg/L which is much higher than all of the other values obtained as well as any standard.
   For the average brewery, nitrate concentrations dwell around 50 mg/L, and phosphorus concentrations are often near 60 mg/L. These high values are most likely due to the high amount of waste material leftover from the beer making process. A large plant such as a brewery also produces a lot more wastewater than domestic systems. With these additions to the wastewater complexity, the wastewater treatment process also becomes much more complex. Often breweries have separate arrangements in order to treat their effluent. The values obtained in the lab from Beau's are 59 mg/L ammonia, 0.492mg/L nitrate, and 4.8163mg/L phosphorus. Both the phosphorus and nitrate concentrations are lower than the average brewery effluent concentrations. These unexpected results could have been due to an incorrect dilution.

4. In addition to blue baby syndrome, high exposure to nitrate can lead to cancer, disruption of thyroid functions, and birth defects. After ingestion, nitrates are converted to nitrites which react with both natural and organic compounds inside of the stomach. These reactions produce N-nitroso compounds, which many of them are carcinogenic in humans. Additionally, few studies have suggested that ingestion of water with high levels of nitrate is linked to enlargement of the thyroid, which can have negative effects on its function of regulating hormonal functions.
   The EPA set the maximum contaminant level for nitrate at 10 ppm as Nitrogen, or 45ppm as Nitrate. According to this standard, none of these wastewater samples used during the lab would have adverse effects or lead to the problems mentioned above, as none of the samples nitrate concentration were above 10 ppm.

Conclusion

In this lab, it is important to note that eutrophication caused by high concentration of nutrients damages to environment and results in healthy concerns. The results of ammonia, nitrate, and phosphorous can be various related to where the samples are collected. The concentration of ammonia and phosphorus are calculated to be 0.0144 ppm and 0.1799 ppm in stormwater respectively. After comparing the Canadian Wastewater Systems Effluent Regulations, the wastewater sample of stormwater analyzed from our group Lobo meets the requirement to be discharged into the environment directly without water treatment and has minimal direct exposure to fertilizers or fossil fuel burning in the atmosphere. It is the only sample of 4 without alarming nutrient concentrations.

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