Abstract

The Environmental Protection Agency's, otherwise known as the EPA, stated purpose is to ensure that all Americans are protected from significant risks to human health and the environment where they live, learn and work. As they fulfill their duties they require Environmental Laboratories that test the environment for harmful chemicals by performing several methods or tests in order to determine whether that environment is safe for humanity. In the project performed, The Effect of Surfactants in NPM Analysis, the two methods used were Hexane Extractable Material (HEM; EPA Method 1664) and Non-Polar Material (NPM; EPA Method 1664). These methods are performed on certain contaminants, like industrial waste water, along with organic substances like soil. The main purpose of the HEM method and the NPM method is to determine if the chemicals in these substances are safe enough to be able to be released into the environment. The HEM method is applicable to measurement of n-hexane extractable petroleum hydrocarbons from surface waters, industrial and domestic wastes by using Hexane, also a colorless, flammable liquid, derived from the distillation of petroleum, as a solvent and it aids in filtering and extracting the substance. The HEM method is practically a test looking for all organic chemicals (not metals) in water such as cooking oils and petroleum products. Once the weight of the initial flask is taken and the weight of the same flask after the HEM method is preformed it is recorded and a equation is used to determine the HEM result. HEM has no detection limit due to the fact that it is quantitative and after doing the HEM method the NPM method is required which gives the finishing results on the analysis. The NPM method, on the other hand, is a test

looking for only non-polar materials such as petroleum and citrus based cleaners. The NPM analysis main difference to the HEM method is the treatment of silica gel to the sample as a way to remove all polar materials. The project performed followed the procedures and preparations of the HEM method and the NPM method in order to test the validity for EPA compliance in surfactants. The main purpose of surfactants in this study is to infer whether or not the surfactants play any role in the HEM and NPM results of a sample. Since surfactants are found in various companies industrial waste it is important to see if the surfactants play any role in the analysis and see if the surfactants can cause a company to be fined due to results exceeding the detection limit. Two types of surfactants were used in this experiment, one being commercial laundry soap supplied by the environmental laboratory and the other being the popular detergent “TIDE”.

After this experiment was completed we continued by testing the other chemicals found in industrial waste water aside from surfactants like oils, grease, and fats.

INTRODUCTION

In the year 1970, the personals in congress and the white house worked together to establish the EPA in response to the growing public demand for cleaner land, water and air. Prior to the establishment of the EPA, the federal government was not equipped to make a coordinated attack on the pollutants that harm human health and degrade the environment leading to the EPA was assignment and daunting task of repairing the damage already done to the natural environment and to establish new criteria to guide Americans in making a cleaner environment a reality. The Environmental Protection Agency (EPA) works to assess environmental conditions and to identify, understand, and solve current and future environmental problems. As their job to help the environment they must regulate the substances going into the environment by requiring tests to be done by environmental labs as mentioned once before. The HEM method and the NPM method are two methods required by the EPA as a way to test sample water and soils. The EPA required the test to be one once on each sample, which leads to the problem in our experiment. When a silica gel treatment is done during the NPM method the results is suppose to remain the same no matter how many times the experiment is performed, only that with some samples that doesn't occur. Since the detection limit for the NPM method is 50 mg/L the company can be fined if it exceeds this limit and often sample do exceed the detection limit. Another problem we are encountering while performing this experiment is the fact that silica gel doesn't always remove one hundred percent of the polar material in a sample as its suppose to. Polar Materials, which the NPM method removes, can be described as materials that is related to or characterized by a dipole. A non-polar material, which the NPM method

detects, can be described as a compound that does not have concentrations of positive or negative electric charge. Our project demonstrates that the test is flawed due to shortcomings in the analytical procedure which can lead to fines for industrial clients.

MATERIALS

- Step Saver- to do experiment in
- Analytical Balance- to weigh flasks
- Desiccator- to remove water from flasks
- 125 mL flasks- put samples in
- Beakers- stir samples with silica gel in
- Stirring plats and magnetic stirring bars- used to stir sample

- Silica gel- remove polar material
- Detergent- variable
- Standard- composed of hexadecane stearic acid and acetone
- DI Water and sulfuric acid- Reagent and Stock Standard
- Sodium sulfate cartridges- HEM Method
- P5 Filters- filter through sample with silica gel
- Oil and grease disks- capture the contents of the sample
- Hexane- extract the material from the sample
- Methanol- part of standard
- Water Bath- used to evaporate the hexane from the sample
- Acetone or Metholyne Chloride- used to clean flasks
- 50 mL and 10 mL graduated cylinder- measure out methanol and hexane
- Funnel- used to place filter in
- Aluminum Weighing Disks- used to measure the silica gel
- Micropipettes- used to measure out aliquot of sample
- 1000 mL glass flask- used to prepare standard in
- Plastic pipette - used to fix up measurements

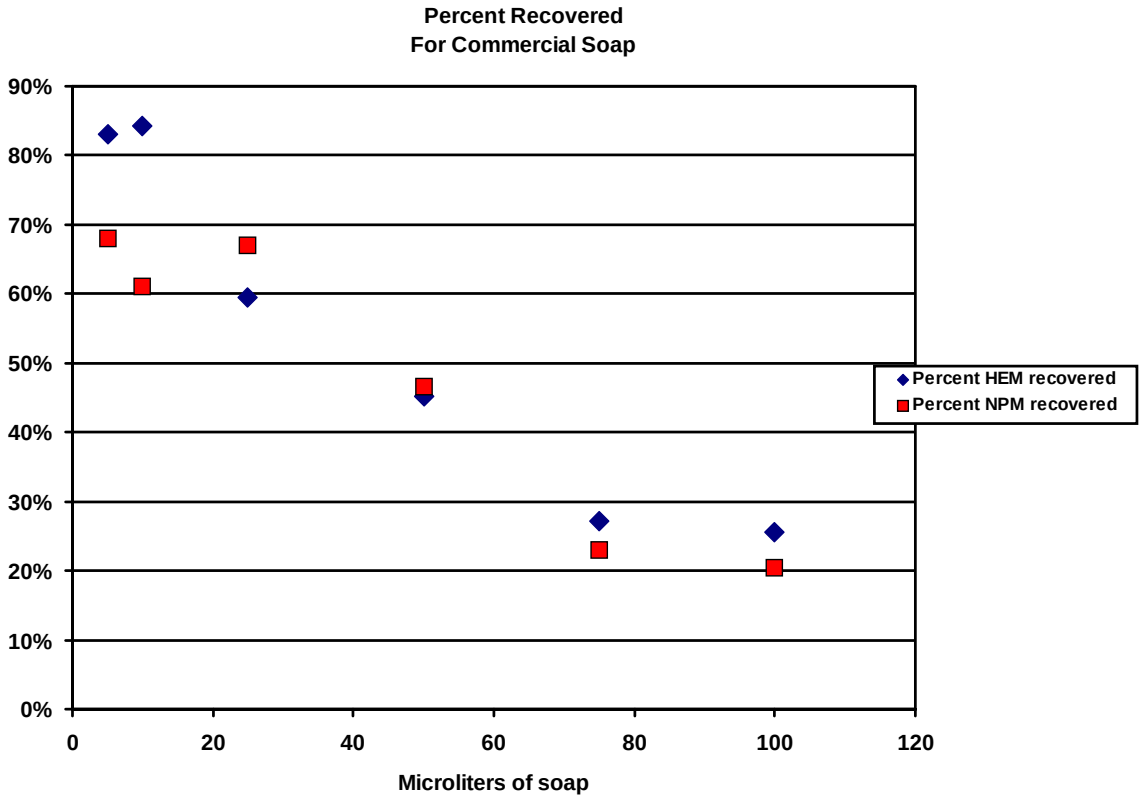
METHODS

For HEM method: Prepare the Step Saver by centering the funnels onto the head of each section on the Step Saver. Make sure that the oil and grease disks are inside the gasket of the step saver and that the rough side of the filter is facing upwards while the

smooth side is facing downwards. Then Place the gasket and filter together onto the stainless steel screen, and center the funnel on the head make sure to squeeze the clamp into place. Attach a flask to collect waste solvent to collection arm of StepSaver with Keck clip. Turn the upper stopcock with the red handles so that flow will be toward the collection flask. Position the valve on the manifold to the off position. Wash the disk and walls of the funnel with 10-15 ml of n-hexane. Quickly turn the manifold valve to the on position and the quickly back to off position. This should draw a small amount of hexane through the disk. Allow it to soak. Apply vacuum and pull remaining hexane through disk into collection flask. Allow it to dry. Repeat hexane rinse if necessary. Turn Off the valves. Turn the StepSaver stopcock (red handles) to the waste position. Add 10-15 ml of methanol to the reservoir. Let it soak and then let methanol flow through the disk. Add a small amount of methanol and don't dispose of it. Add sample on top of methanol and immediately turn vacuum the on position. Invert bottle in reservoir when empty in order to empty any remaining sample into the container. After sample extraction is complete, maintain full vacuum for 10-20 minutes to strip as much water as possible from the oil and grease disk. Turn manifold valve to off position. Measure the actual volume of the sample used in the analysis. Record the true volume in your data book.

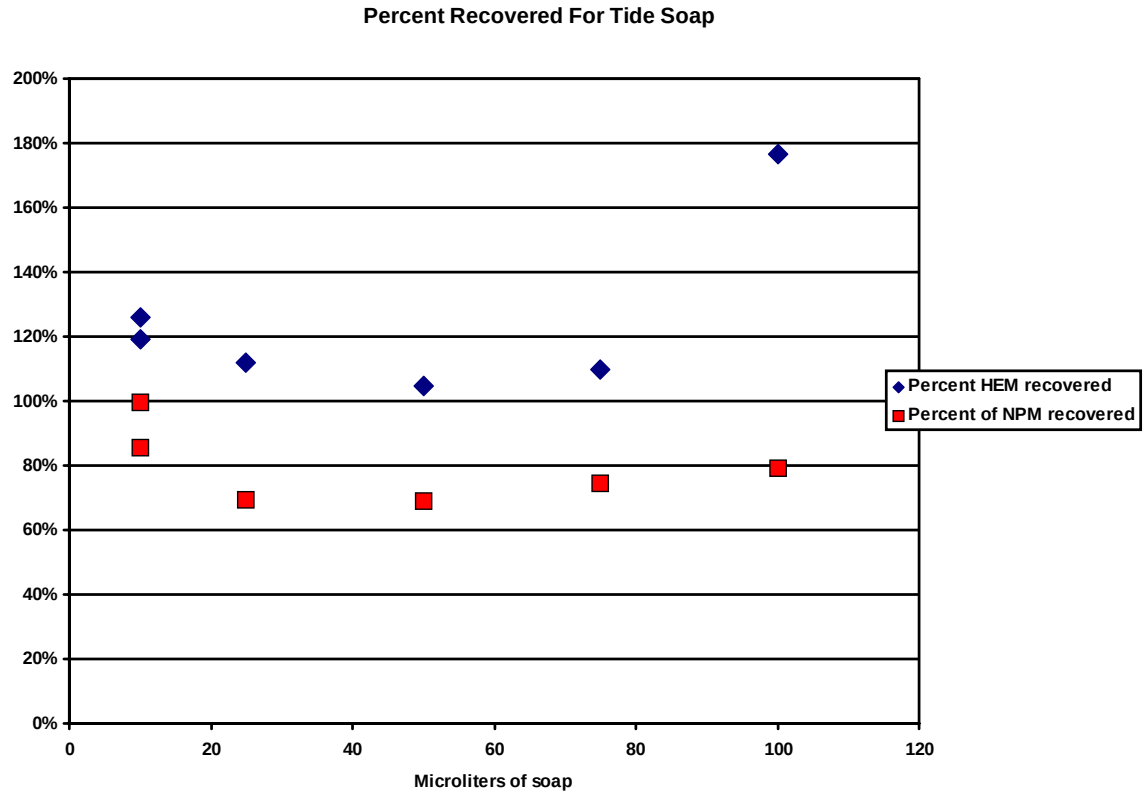
NPM: The method for the NPM analysis just requires the addition of silica gel added to the hexane soluble material along with the sample in order to remove the polar materials. The hexane is then filtered to remove the silica gel. The solvent is evaporated from the extract and the residue is weighed.

CONCLUSION


Percent Recovered
For Commercial Soap
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
0
20
40
60
80
100
120
Microliters of soap
Percent HEM recovered
Percent NPM recovered

Microliters of soap	HEM	NPM	Percent HEM recovered	Percent NPM recovered
5	33.2	13.6	83%	68%
10	33.7	12.2	84%	61%
25	23.8	13.4	60%	67%
50	18.1	9.3	45%	47%
75	10.9	4.6	27%	23%
100	10.2	4.1	26%	21%

This graph demonstrates that commercial soap does interfere with a sample. It demonstrates that the less of that commercial soap there is the better recovery there is. While, on the other hand, the more commercial soap is present in the sample, the worse the recovery is.



Microliters of soap	HEM	NPM	Percent HEM recovered	Percent NPM recovered
10	47.6	19.9	119%	100%
10	50.3	17.1	126%	86%
25	44.8	13.9	112%	70%
50	41.8	13.8	105%	69%
75	43.9	14.9	110%	75%
100	70.7	15.8	177%	79%

Contrastingly the Tide Detergent demonstrates that the less soap is present in a sample the worse the recovery is. While the more the detergent is in a sample the better the recovery appears.

After these results it was concluded that…

- Surfactants have a varying interference with the results of the analysis depending on how much and type of surfactant.
  -Industrial soap interferes with the analysis, giving a smaller amount of analyte than is truly there.
  -Tide is opposite of industrial soap because it gave a positive interference with larger amounts

- Depending on the type of contamination, multiple silica gel treatments may be required to obtain valid results.

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