An Analysis of Methods for Detecting Triclosan and Removal of Triclosan from Water Using Activated Carbon and Zeolites

Major Qualifying Project completed in partial fulfillment
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Abstract

With the growing popularity of triclosan in consumer products, the levels of wastewater contamination have heightened concerns about potentially harmful effects to public health and the environment. The goal of this project was to determine if adsorption onto zeolites or activated carbon is a viable method of removal of triclosan from wastewater. HPLC was used for determination of the concentration of triclosan in water both before and after adsorption with various zeolites and activated carbons. Many of the tested adsorbents, such as zeolite Y1, zeolite beta, and activated carbon, were found to be effective at the removal of triclosan.

Acknowledgements

We would like to thank our advisors, Professor Robert W. Thompson and Professor Drew R. Brodeur, for their guidance and support throughout the duration of this project. We would also like to thank the Chemistry and Chemical Engineering department for providing us with the materials necessary to complete our research.

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Chapter 1: Introduction

With the growing concern about bacterial infection, the use of antimicrobial agents has become widespread. This increase has led to rising levels of these compounds in the environment, especially in our fresh water supply. As these antimicrobial agents are used, they ultimately contaminate the water either through the sewage system or runoff from chemical plants. Lately, the emergence of these contaminants in water has been presenting many complications. One of the major concerns is that overuse of antimicrobial agents will cause bacterial resistance. The emergence of strains of multiple drug resistant bacteria has already been attributed to the overuse of several antibiotics[1,2]. These drug resistant strains can cause serious illness and even be fatal.

Currently, wastewater treatment policies do not regulate the concentrations of many pollutants that are not seen to be immediately harmful[3]. Triclosan is one of these unregulated compounds. Triclosan is a common antimicrobial agent found in many commercial products. It is used in antibacterial soaps, plastics, toys, and many other household items. Due to the relatively recent increase in the use of triclosan, there is a growing concern about the safety of this chemical. Triclosan, like many other antimicrobial agents, can lead to bacterial resistance. As triclosan becomes more prevalent in the environment, it can kill off susceptible bacteria, leaving the resistant strains to grow and multiply[4].

The aquatic environment is disrupted by the presence of triclosan. In particular, triclosan can kill off algae, which is important to the health of the ecosystem[5]. There have also been several other health concerns associated with triclosan. Triclosan levels can build up in the human body over time and may cause long-term health risks[6,7]. It can also degrade in the aquatic environment to make more deadly products. The most concerning degradation products are dioxins, which are known carcinogens and have also been found to mutate DNA and cause birth defects in offspring[8].

Growing interest in triclosan's possible negative effects on humans and the environment has prompted the need to evaluate methods of detection and removal. The main goal of this study was to determine if adsorption onto various zeolites or activated carbons is a viable method for

removal of dissolved triclosan in water. Two different brands of activated carbon and many zeolites with varying characteristics were used in the analysis to determine which adsorbent, if any, was most suitable for the removal of triclosan. A list of these zeolites can be seen in Table 4 in Appendix C: Table of Zeolite Properties. Additionally, given access to only basic undergraduate equipment, it was necessary to determine which machine would function best for the needs of this study. Fluorescence spectroscopy, gas chromatography, and high-pressure liquid chromatography were tested as methods of detection. These methods were used to determine the concentration of triclosan in samples both before and after adsorption onto the zeolites or activated carbons.

Chapter 2: Background

The following chapter summarizes the literature. This information was gathered prior to the execution of the experiments in this project. The properties and functions of triclosan are discussed, and its environmental occurrence and potential hazards to the public and environment are analyzed. Degradation pathways and byproducts of triclosan are examined, and potential methods of removing triclosan from water are discussed. Finally, fluorescence spectroscopy, gas chromatography, and high-pressure liquid chromatography are described. Potential methods of increasing triclosan solubility in water are also explained.

2.1 Triclosan

Triclosan (5-chloro-2-(2,4-dichlorophenoxy)phenol) is a strong broad-spectrum anti-microbial agent used in many consumer products, such as anti-bacterial soaps, toothpastes, cosmetics, clothing, kitchenware and toys [9]. The structure of triclosan can be seen below in Figure 1. Triclosan is a relatively small molecule, with a molecular weight of 289.54 g/mol and a diameter of about 7.4 Å[10]. It is a white solid at standard temperature and pressure, with a boiling point in the range of 280-290° C and a melting point in the range of 56-58° C. According to Sigma Aldrich, the exact solubility of triclosan in water is unknown. However, other sources have reported a solubility of $<10^{-6}$ g/mL in water[11]. Triclosan has a low partition coefficient (log P_{ow} = 4.7), suggesting that it is lipophilic[12]. The partition coefficient is a ratio of solubility between two liquids, typically octanol and water. Values of its pKa have been reported in the range of 7.9-8.1[13].

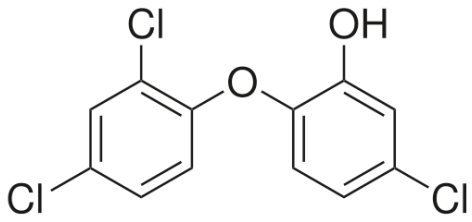

Figure 1: Structure of Triclosan

2.2 Potential Hazards of Triclosan

Triclosan enters the environment when antimicrobial products are used and disposed of. In recent years, triclosan has been detected in influent and effluent bodies of water throughout the U.S. In a study performed by the U.S. Geological Survey (USGS), triclosan was found in 57.6% of the 139 streams tested across the U.S., with an average concentration of 1.4×10^{-8} g/mL[14]. A 2006 study in California measured the concentrations of triclosan in raw drinking water, reclaimed drinking water, and influent wastewater during the dry season (August-November) and the wet season (January – June). They reported an average concentration of triclosan between 0.28 and 2.00×10^{-9} g/mL[15]. The growing concentrations of triclosan have raised concern about potentially harmful effects to public health and the environment.

2.2.1 Bacterial Resistance

Triclosan works as an antimicrobial agent by inhibiting the synthesis of fatty acids in bacteria. Triclosan is similar to the natural substrate that binds to the enzyme enoyl-acyl carrier protein reductase (ENR), which is involved in lipid synthesis[16]. Therefore, triclosan acts as a competitive inhibitor, which means it can bind to the enzyme in place of the substrate and prevent the synthesis of lipids used to create the cell walls of bacteria[17]. Without a functional cell wall, bacteria will lyse and die[18].

ENR is encoded by the gene fabI, and mutations in fabI can result in resistance to triclosan. Bacteria with this mutation become resistant to broad spectrum antibiotics. Triclosan becomes increasingly present in the environment, it can kill off susceptible bacteria, leaving the resistant strains to grow and multiply[4]. Bacterial resistance is a very serious concern, as some of the most deadly illnesses are caused by antibacterial-resistant strains of bacteria.

2.2.2 Accumulation of Triclosan from Consumer Products

In low concentrations, triclosan has yet to be proven harmful to humans. Due to its lipophilicity and long half-life, however, it has been found to accumulate in the body; this may potentially cause long-term health risks[6,7]. Animal studies suggest that triclosan can have a harmful effect on the endocrine system[19]. Other studies have shown that triclosan can act as an inhibitor to biological processes such as estradiol and estrone sulfonation and glycolysis[20].

A Swedish study in 2006 tested the blood plasma and breast milk of new mothers. For the mothers exposed to triclosan in their daily lives (through the use of triclosan-based toothpaste, deodorant, soap, etc.), average concentrations of 11.4 ng triclosan/g plasma and 0.44 ng triclosan/g breast milk were determined. This was much higher than mothers who did not use products containing triclosan; these women had an average concentration of 0.07 ng triclosan/g plasma and 0.02 ng triclosan/g breast milk[6]. This demonstrates that triclosan is absorbed into the human body when triclosan-based products are used regularly. Even though these products only contain small concentrations of triclosan, the chemical can build up in the body.

A study from the University of Rochester demonstrated that triclosan can inhibit glycolysis in streptococcus mutants, a bacterium found in the human mouth. This study also found that, at neutral pH, triclosan was an irreversible inhibitor of the enzymes pyruvate kinase, lactic dehydrogenase, aldolase, and the phosphoenolpyruvate:sugar phosphotransferase system (PTS), also used in glycolysis. This inhibition prevents glycolysis in dental plaque, which in turn reduces the number of cavities formed[23]. Because of this, triclosan is commonly found in toothpaste. Although this is important to dental health, the risks of triclosan accumulation in the body, as noted above, outweigh the benefits.

2.2.3 Health Concerns

Thyroxine, a hormone secreted by the thyroid gland, is used by the body to control the rates of metabolic processes[19]. In 2007, a study was conducted on Long-Evans rats to examine the effect of short-term exposure to triclosan on the disruption of thyroxine. In this study, female rats were exposed to triclosan in concentrations between 0 and 1000 mg/kg per day for 4 days. The concentrations of thyroxine decreased by 28, 34, and 53% in rats given 100, 300, and 1000 mg/kg doses, respectively. Triclosan was found to inhibit thyroxine reception by the pregnane-X receptor, therefore disrupting proper endocrine functions that should be carried out by this enzyme. Inhibition is caused by the similarity in shape and size of the triclosan and thyroxine molecules (Figure 2). This study concluded that triclosan is responsible for interrupting thyroid homeostasis in rats[21]. When thyroxine reception is inhibited, the body is no longer capable of controlling metabolic rates. This condition is known as hypothyroidism, which can lead to other

health concerns such as “tiredness, lethargy, weight gain, cold intolerance, hoarseness of the voice, and dryness of the skin[20].”

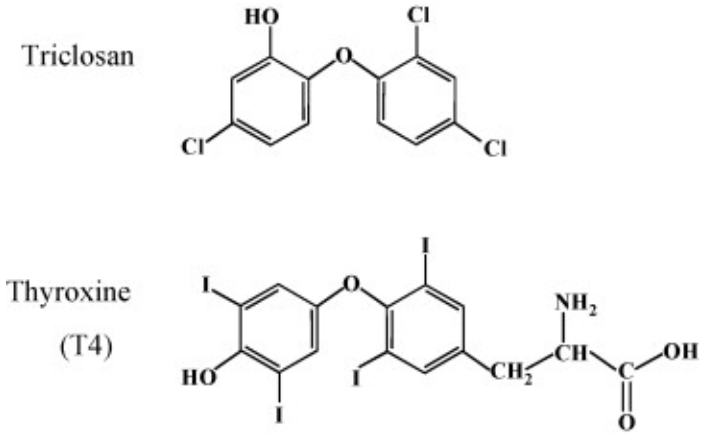

Figure 2: Structure of triclosan and thyroxine molecules

Estrogen is a group of compounds that are the primary female sex hormones used by the body to control several processes related to pregnancy. In fetal blood, estrogen is found mostly in the sulfonated forms of estradiol and estrone. A study at the University of Florida observed the effects of triclosan on sheep placenta during pregnancy. This study found that triclosan acted as a competitive inhibitor for the enzyme placental estrogen sulfotransferase. This restricted the delivery of estrogen from the placenta to the fetus[22].

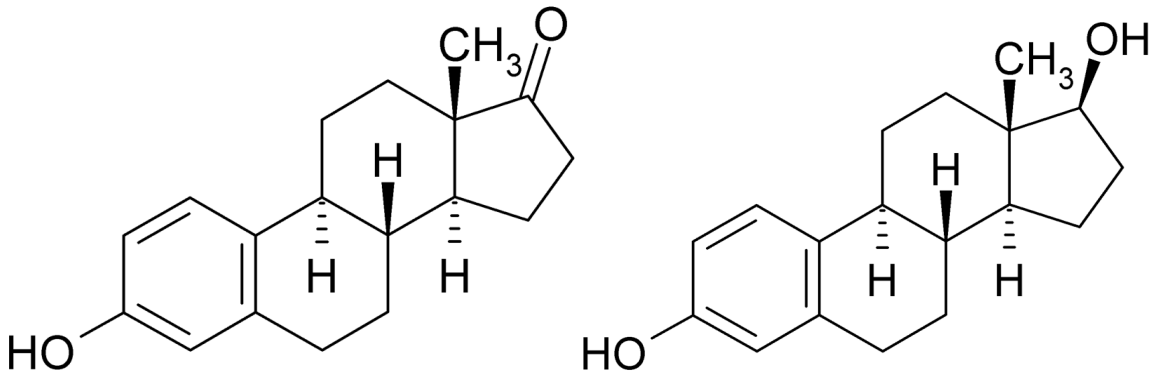

Figure 3: Structure of esterone (left) and estradiol (right)

2.2.4 Triclosan in the Aquatic Environment

Triclosan is an environmental hazard to aquatic life when it enters the ecosystem via wastewater. An Australian study in 2005 found triclosan in surface water samples from rivers located near wastewater treatment plants. Triclosan was found in concentrations ranging from 14 to 60 ng/L in these rivers[24]. A 2003 study found that triclosan is highly toxic to aquatic life, especially to algae. As mentioned above, triclosan affects a specific enzyme, ENR; this enzyme is found in bacteria, fungi, and some plants. Algae are affected by triclosan indirectly; triclosan is highly toxic to the bacteria that interact with algae in the biofilm. The lack of bacteria inhibits the photosynthesis process of the algae, causing it to perish[25]. This is particularly harmful because algae are a "first-step producer," meaning any disruption to algal life will affect the rest of the ecosystem.

2.3 Degradation Products of Triclosan

Under certain conditions, triclosan has been found to degrade into four different byproducts: 2,7-dibenzodichloro-*p*-dioxin, 2,8-dibenzodichloro-*p*-dioxin, 2,4-dichlorophenol, and 2,4,6-trichlorophenol. These byproducts have been found to pose health risks to humans when they accumulate in the body. This will be discussed in further detail below.

2.3.1 UV Degradation

In the presence of UV light (sunlight), triclosan has been shown to degrade into two dioxins: 2,7-dibenzodichloro-*p*-dioxin and 2,8-dibenzodichloro-*p*-dioxin. A study at the University of Almeria, Spain, found that the concentrations of these dioxins varied with the pH of the solution – at higher pH levels, the dioxins had a higher concentration[26]. A study at the University of Minnesota found that above pH 8, the dioxins made up between 1 and 12% of the original triclosan concentration[27].

While these concentrations are low, dioxin byproducts are still of primary concern because of the health threat that they pose. These dioxins have been found to accumulate in the human body due to their lipophilicity. They are known carcinogens, and have also been found to mutate DNA and cause birth defects in offspring[8].

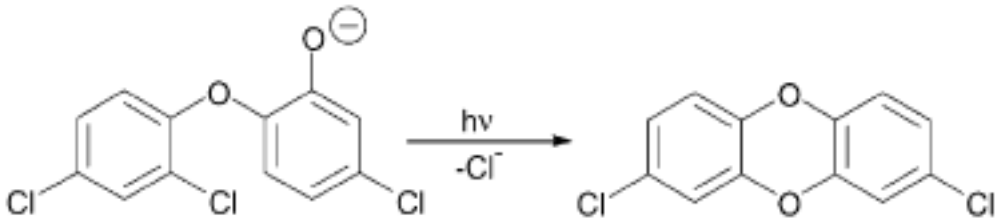

Figure 4: UV degradation of triclosan to 2,8-dibenzodichloro-p-dioxin

2.3.2 Degradation in Presence of Free Chlorine

When free chlorine is present in water, triclosan degrades into 2,4-dichlorophenol (2,4-DCP) and 2,4,6-trichlorophenol (2,4,6-TCP). The degradation pathway of triclosan involves chlorination, which forms either a tetraclosan or pentaclosan, and then cleavage of the bicyclic structure to form 2,4-DCP and 2,4,6-TCP. The degradation pathway can be seen in Figure 5. A 2005 study found that, in chlorinated water at pH 7.3, approximately 9% of triclosan was converted to 2,4-DCP, while only 2.5% was converted to 2,4,6-TCP. This study also found both byproducts in wastewater, which implies the degradation of triclosan in the natural environment[28]. Both 2,4-DCP and 2,4,6-TCP are more stable than triclosan, which means that, while triclosan can degrade into these byproducts naturally, it cannot reform from these products spontaneously.

2,4,6-TCP is a known endocrine disrupter, meaning it interferes with human glands and blocks proper reception of hormones by these glands. This may cause cancer, or birth defects and developmental disorders in offspring. 2,4-DCP may be fatal if large amounts are absorbed by the body.

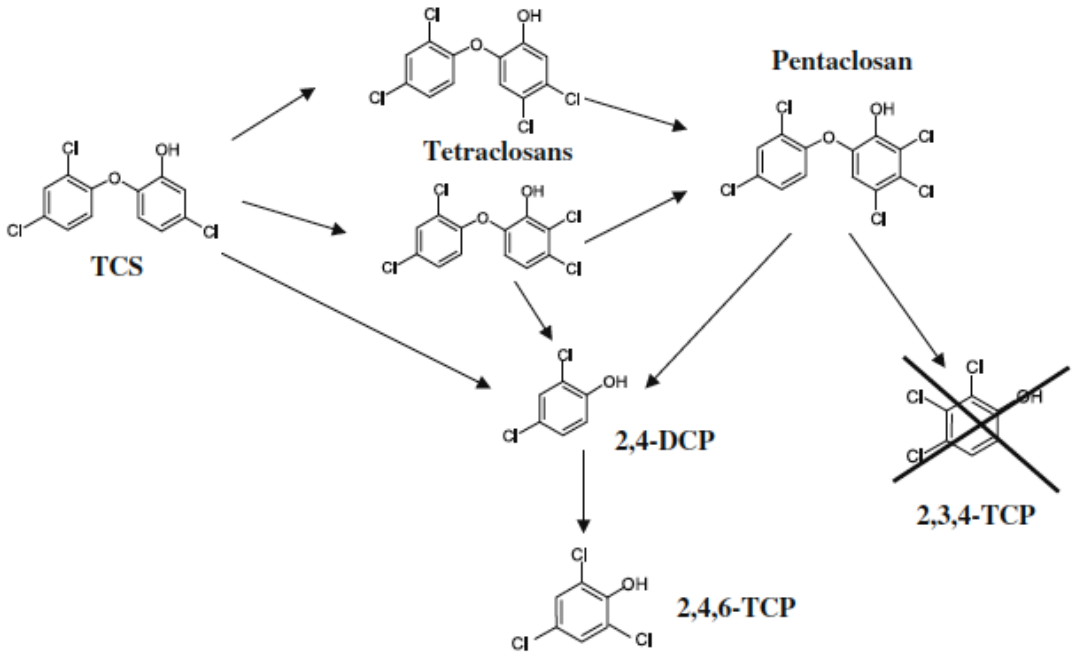

Figure 5: Degradation pathway of triclosan in the presence of free chlorine[8]

2.4 Methods of Removal

2.4.1 Adsorption

A common method for removing contaminants from a liquid is adsorption. Adsorption is the process by which a target substance accumulates on the surface of a solid at the interface between the two phases. The target substance is known as the adsorbate and the solid on which the adsorbate accumulates is called the adsorbent. Adsorbents are generally very porous materials. The high porosity creates a large total surface area, so that the adsorbate primarily adheres to the pore walls or to designated sites inside the particle. Various types of chemical forces are responsible for holding adsorbates on the surface of the adsorbent. These forces include hydrogen bonds, Van der Waals forces and dipole-dipole interactions. Separation occurs in adsorption when a difference in molecular weight, polarity, or shape causes some molecules to bind to the surface more strongly than others or when the adsorbent pores are too small to admit particles larger than the targeted adsorbate[29,30].

There are different factors that can affect how much of the adsorbate can accumulate on the surface of the adsorbent. These factors include the physical and chemical properties of the adsorbate and the adsorbent, as well as the type of bond, pH, and temperature. The operating temperature and pH are generally held constant for a process. It is important to consider the adsorbate's solubility, pKa, concentration and structure. The solubility is particularly important; typically, lower solubility will result in greater adsorption. A good adsorbent has a higher affinity for the adsorbate than for water. Thus, a low adsorbate solubility would result in an easier separation[31]. For the purposes of this project, we reviewed two different types of adsorbents, zeolites and activated carbon, to remove triclosan from water.

2.4.2 Zeolites

Zeolites are crystalline, microporous solids that are composed of aluminum, silicon and oxygen. They have open three-dimensional structures that contain channels and cavities. Their structures are composed of tetrahedra of silicon (SiO_4) and aluminum (AlO_4). These tetrahedra can combine in various ways to form about 800 different crystal structures; however, less than a quarter of these possible structures have been found in nature or synthesized[32].

When used as adsorbents, the most important considerations are the pore size and the Si/Al ratio. The intercrystalline volume and affinity for water are highly dependent on this ratio. Both the volume and the affinity for water increase as the amount of aluminum in a zeolite increases. In contrast, dealuminated zeolites are hydrophobic, so they repel water[33]. The pore size and structure are also important to consider, as they limit the size of the molecule that can enter the zeolite. The pore sizes and structures of two common zeolites can be seen in Figure 6 below. The variation in structure and pore size demonstrates the adsorption limitations that can occur for a particular zeolite.

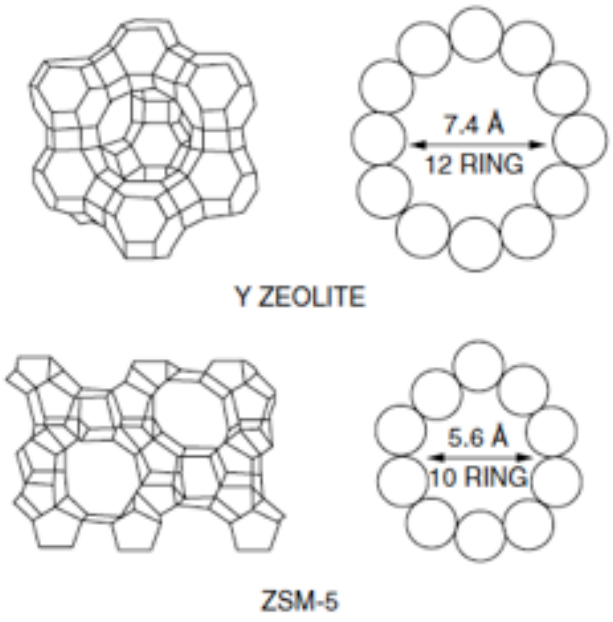

Figure 6: Structures of Zeolite Y and ZSM-5[32]

A 2009 study in Nevada tested removal of several "emerging contaminants of concern (ECCs)" through the use of various adsorbents. When tested with Ambersorb® 563, a carbonaceous resin, triclosan was almost completely removed from solution. Activated carbon from coconut shells was also found to remove almost all of the triclosan in solution. When tested with mordenite, no triclosan appeared to be removed; however, zeolite Y removed about 50% of the triclosan in solution. This is likely due to triclosan's diameter compared to the zeolite's pore size. Triclosan has a diameter of about 7.4 Å. Zeolite Y has circular pore openings with a diameter of 7.4 Å, while mordenite has elliptical pore opening with dimensions ranging from 6.5 to 7 Å. The study attributed the high adsorption by Ambersorb® 563 to the hydrophobicity of triclosan and to the activated carbon's broader micropore size distribution, which allows compounds of different sizes and shapes to be accommodated more easily[10].

2.4.3 Activated Sludge

There are many wastewater treatment processes used both industrially and municipally worldwide. In the secondary step within the treatment process, the sewage removes dissolved and suspended particles. Triclosan is one of the dissolved particles commonly found in water sources. There are two primary methods of removal in the secondary step: attached and

suspended growth. The attached growth process is a series of filters or a surface coated in a microbial growth that removes particles as sewage filters through the tank. In suspended growth, the sewage is mixed and aerated with activated sludge[34].

Activated sludge is composed of bacteria and protozoans, both dead and alive, as well as other materials. The living microorganisms are aerobic and react positively to the oxygen that is mixed in with the wastewater. Generally, the microorganisms will consume various particles and nutrients; however, in the case of triclosan, the bacteria will absorb the triclosan and die from the collapse of their cell walls[34]. Activated sludge in wastewater treatment is the most efficient method of removing triclosan because the antimicrobial is attracted to the lipophilic nature of the bacteria[35].

2.4.4 Activated Carbon

Another material used for water filtration is activated carbon. This type of carbon is created by oxidizing the char of an organic compound. Large pores define the structure of oxidizing carbon, or charcoal. These pores greatly increase the surface area of the material, thus increasing the amount of adsorption potential. When water is filtered, it permeates through the pores and across the surface of the carbon, creating attractive forces with the contaminants, which traps them in the carbon.

Like zeolites, the pore sizes of activated carbons are important. If the pores are too small, then there is not as much surface area. However, if the pores are too large, the carbon is very brittle and cannot withstand the purification process of water. Another downfall to activated carbon is that not all compounds will bind to the carbon. Certain molecules, such as fluorine, hold no attractive forces with carbon[36].

2.5 Methods of Detection

In this study, three methods of detection were analyzed. High-pressure liquid chromatography, fluorescence spectroscopy, and gas chromatography can all be used to detect the presence of certain compounds in solution. The results can then be used to determine the concentration of the compound, based on a calibration curve. The calibration curve is a linear plot that is produced by

running samples of the compound of interest at a known concentration. The slope of this line can be used to determine the concentration of the compound present a sample.

2.5.1 High-Pressure Liquid Chromatography

High-pressure liquid chromatography (HPLC) is a type of column chromatography; however, the column is subjected to high pressures, rather than allowing the sample to flow through the column by gravity alone. Because of the high pressure, a packing material with a greater surface area can be used. This allows for much better separation and detection of materials in the sample than gravity column chromatography. The sample is allowed to flow through a silica column at pressures up to 400 bar, using a mixture of two solvents, typically water and a polar solvent. Compounds with different polarities will have a different retention time within the column, allowing for separate peaks. With these different peaks, one can determine the compounds present as well as the concentration. The peaks are created when a compound reaches the UV detector at the end of the column. This detector is set to an excitation wavelength specific to the compound; in the case of triclosan, the excitation wavelength is 290 nm. The height of this peak can then be used to determine the concentration of the compound present in solution when compared to a calibration curve[37]. A diagram of this process can be found in Figure 7.

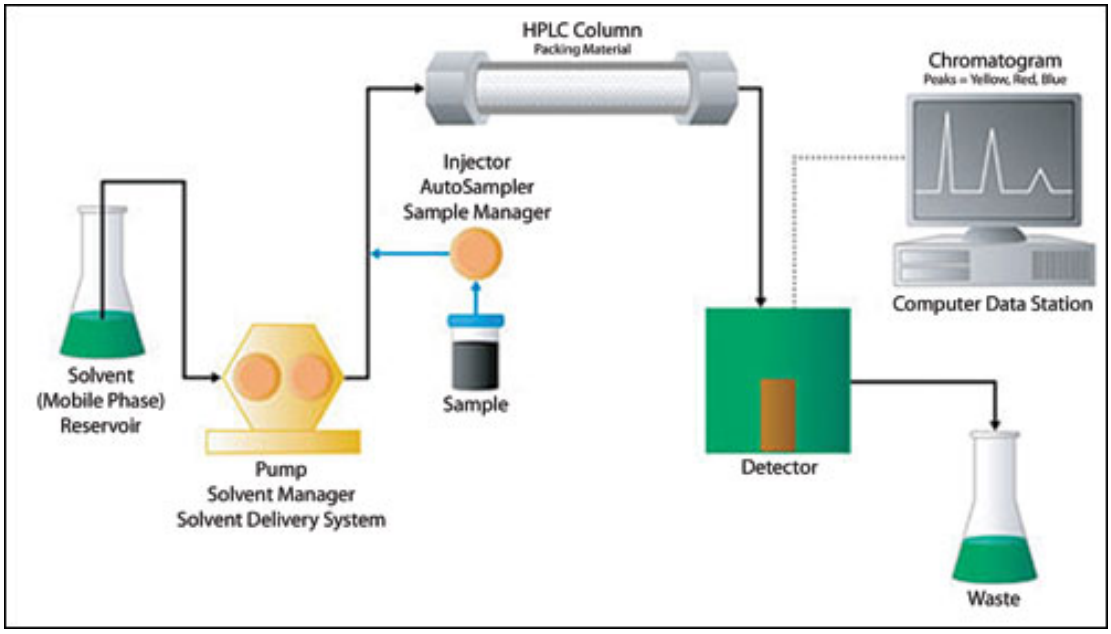

Figure 7: Schematic of a High-Pressure Liquid Chromatograph[38]

2.5.2 Fluorescence Spectroscopy

Fluorescence spectroscopy is a technique that excites a molecule by irradiating it with light of a certain frequency. The molecule absorbs a photon, which causes an increase in its energy level. When the molecule relaxes and returns to the ground state, it emits light of a different frequency. The intensity of this emission can be used to determine the concentration of the molecule in the sample by comparing the emission intensity to a calibration curve. Figure 8 outlines this process. Triclosan has a known excitation wavelength of 290 nm and emission wavelength of 340 nm[40]. The byproducts of triclosan, 2,8-dibenzodichloro-p-dioxin, 2,4-dichlorophenol, and 2,4,6-trichlorophenol, have excitation wavelengths of 266, 280, and 488 nm, respectively[40,41,42].

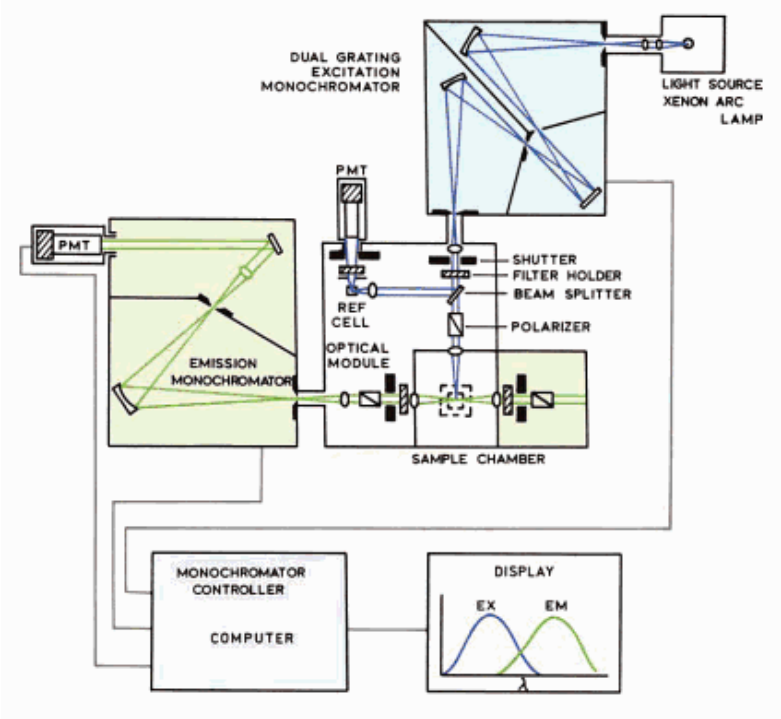

Figure 8: Schematic of a Fluorescence Spectrometer[43]

2.5.3 Gas Chromatography

Gas chromatography (GC) is a method of separating and analyzing volatile compounds. In GC analysis, the test sample is injected into the heated injection port. Once vaporized, the sample is carried to the column by an inert carrier gas, such as helium. The test sample separates into two phases, a mobile phase and a stationary phase. The mobile phase is the carrier gas, while the

stationary phase is where the sample is adsorbed onto the column. Components in the test sample react differently with the walls of the column. This leads to compounds being separated and carried to the detector at different times. This is known as the retention time and is what allows the components to be identified by the GC. The detector measures the quantity of a particular substance and then sends the information to a computer, which plots the data[44]. This process is shown in Figure 9.

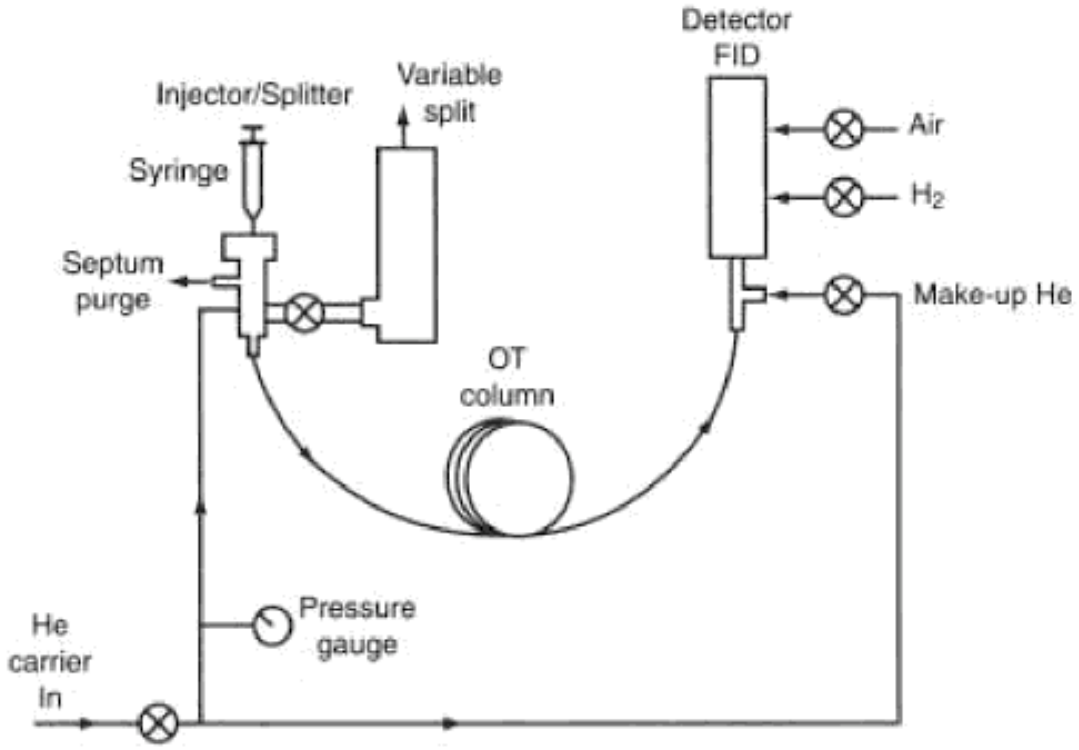

Figure 9: Schematic of a common Gas Chromatograph[44]

A previous study isolated triclosan from various hand soaps and determined the concentrations using GC. In order to create a calibration curve, triclosan was dissolved in isopropanol, with an initial concentration of 10,000 ppm. Dilutions from this stock were used for the calibration, and the concentrations of the soap samples were determined by comparison to this calibration curve. The study concluded that GC was a very effective and straightforward analytical technique to determine concentrations of triclosan[45].

While gas chromatography can give decent readings, the quality of these results can be significantly improved by the addition of mass spectroscopy. In the USGS study previously mentioned, the USGS laboratory used GC/MS analysis to determine the concentration of various organic water contaminates, including triclosan. They were able to yield accurate results using this method[14]. Unfortunately a GC/MS was not available for this study.

2.6 Co-Solvents

Triclosan has a solubility of approximately 10^{-6} g/mL in water. A study from the *Journal of Cosmetic Science* found that the solubility of triclosan could be greatly increased with the use of various co-solvents. Three of the more effective co-solvents were found to be β-cyclodextrin (βCD), hydroxypropyl β-cyclodextrin (HPβCD), and sodium lauryl sulfate (SLS). βCD and HPβCD are seven-sugar ring molecules; their structures can be seen in Figures 10, 11, and 12. They attract hydrophobic molecules, such as triclosan, to the center of the ring, which causes a 2,000-4,000 times increase in the solubility of triclosan. SLS was slightly more effective, with a solubility increase of 3,000-6,000 times the original amount. SLS forms micelles, which are spherical and have a hydrophilic "head" facing the solution and a hydrophobic "tail" inside the sphere, protected from the aqueous solution. The hydrophobic triclosan molecules get trapped in the core of the micelle, allowing the solubility to increase[13]. All three co-solvents have excitation wavelengths in the 400 nm range, so they do not interfere with the determination of triclosan concentration in the samples.

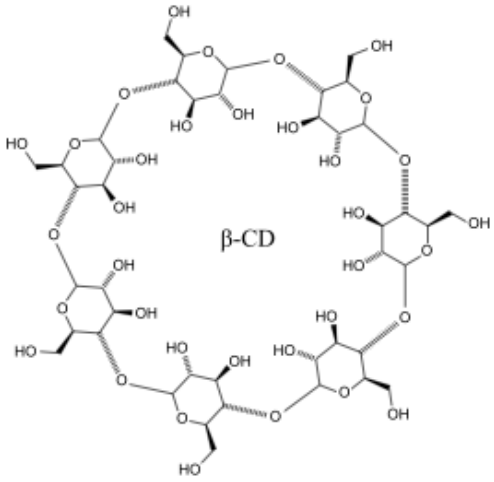

Figure 10: β-cyclodextrin

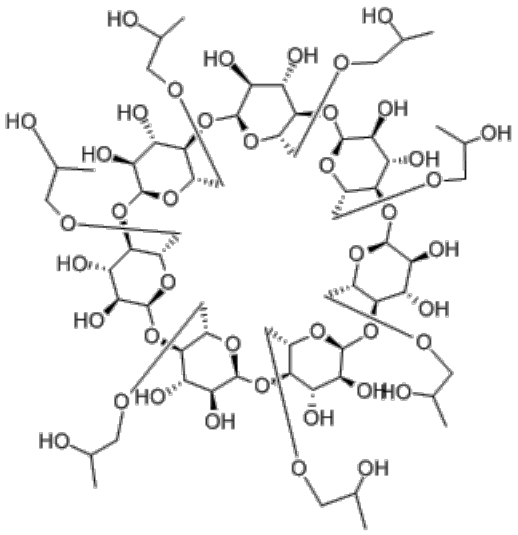

Figure 11: Hydroxypropyl β-cyclodextrin

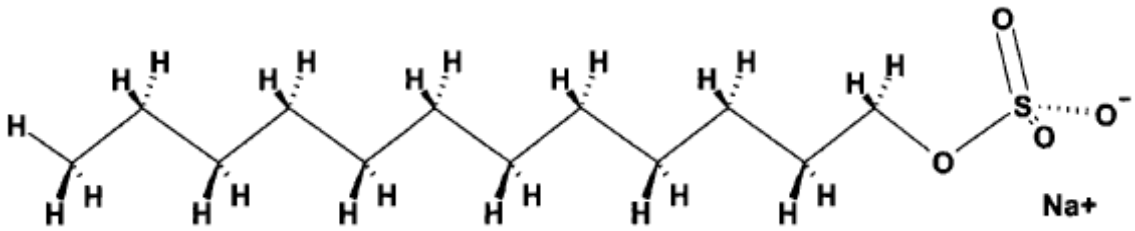

Figure 12: Sodium lauryl sulfate

2.7 Summary

Triclosan is a commonly used antimicrobial agent that is growing in popularity. Triclosan contamination in wastewater is of rising interest, as there are many concerns of its effects on human health and the environment. Under certain conditions, the antimicrobial can degrade into other products that are harmful to humans and the environment. While some methods of triclosan removal have been examined, it is necessary to do more research in this area. The goal of this study was to determine the effectiveness of various zeolites and activated carbons at adsorbing triclosan from wastewater. Due to limited equipment available, this study was only able to achieve semi-quantitative results, however it did prove that zeolites and activated carbons are a viable method for adsorption of triclosan.

Chapter 3: Methodology

The following is a description of the steps taken to achieve the goal of this study. It is important to note that the triclosan used was from Alfa Aesar. The MSDS for this particular brand of triclosan can be seen in Appendix B. Before experimentation, a calibration curve was needed in order to determine the concentration both before and after adsorption. Three different methods of detection were tested to determine the best method. All three methods are described below, but ultimately HPLC was used for experimentation.

3.1 Fluorescence Spectroscopy

Samples were run on a Perkin Elmer LS 55 Fluorescence Spectrometer. Triclosan was dissolved in DI water to create a 6.91×10^{-5} M solution. Seven serial dilutions were made from this solution by adding 5 mL of DI water to 1 mL of the previous solution, with concentrations as seen in Table 1.

Table 1: Concentrations of Triclosan Solutions for Fluorescence

Sample	Concentration (M)
Original Solution	6.91×10^{-5}
Dilution 1	1.15×10^{-5}
Dilution 2	1.92×10^{-6}
Dilution 3	3.20×10^{-7}
Dilution 4	5.33×10^{-8}
Dilution 5	8.88×10^{-9}
Dilution 6	1.48×10^{-9}
Dilution 7	2.47×10^{-10}

Each sample was added to a plastic cuvette and run in the spectrometer. The excitation wavelength was set to 290 nm, and the emission wavelength was found to be 340 nm. The excitation and emission slits were both set to 5 nm, and the scan speed was set to 100 nm/sec.

Solutions of the same concentrations were also prepared using the process above with various other solvents: methanol, ammonium hydroxide, sodium hydroxide, and acetone. Due to the low concentrations, however, fluorescence was not a viable option for detection of triclosan.

3.3 Gas Chromatography

An Agilent series 6850 GC system was used with an Agilent HP-1 30 m x 0.53 mm x 2.65 µm column. The GC system was prepared for an oven temperature of 140 °C. Helium carrier gas was used at a constant flow rate of 7.5 mL/min. A serial dilution was made using dichloromethane as a solvent and concentrations can be seen below in Table 2.

Table 2: Concentrations of Triclosan Solutions for GC

Sample	Concentration (M)
Original Solution	1.38×10^{-4}
Dilution 1	2.30×10^{-5}
Dilution 2	3.83×10^{-6}
Dilution 3	6.39×10^{-7}
Dilution 4	1.06×10^{-7}
Dilution 5	1.77×10^{-8}

On each run, 0.5 µL of the solution was injected into the system with an injection port temperature of 250 °C. The first sample was run for 45 minutes to determine the retention time of the solution. The retention time was found to be approximately 1.75 minutes. When trying to create a calibration curve, it was determined that GC was not sensitive enough to measure the needed concentration levels of triclosan.

3.4 High-Pressure Liquid Chromatography

An HP series 1100 HPLC was used in this study with a PEPMAP C18 4.6X250 mm silica C18 (5 mm, 300 Å) column. The diode array detector (DAD) was set to an adsorption wavelength of 290 nm, which is the excitation wavelength for triclosan. A 50/50 solution of acetonitrile and DI water was used as a carrier solution at a flow rate of 1.5 mL/min.

A calibration curve was obtained using five solutions with known concentrations. These solutions were generated as a serial dilution from a 1.84 x $10^{=2}$ M solution of triclosan in methanol. Methanol was used due to the fact that triclosan has a higher solubility in methanol than in water. A serial dilution was made by taking 1 mL of the previous solution and adding that to 5 mL methanol in a dram vial. These five solutions were run through the HPLC and the peak height recorded. The peak height was plotted as a function of concentration to yield the calibration curve. Concentrations for the serial dilution for the calibration curve can be found below in Table 3.

Table 3: Concentrations of Triclosan Solutions for HPLC

Sample	**Concentration (M)**
Original Solution	1.84×10^{-2}
Dilution 1	3.07×10^{-3}
Dilution 2	5.11×10^{-4}
Dilution 3	8.52×10^{-5}
Dilution 4	1.42×10^{-5}

Upon each use of the HPLC, the system was allowed to run for approximately 30 minutes to clear out the system and the column. For each sample, a small amount was put into HPLC sample vials, and 10 µL from these vials are injected into the system. Each run lasted about 10 minutes to ensure the detection of triclosan, which was found to have a retention time between four and five minutes. After each run, a peak height was measured and compared to the calibration curve in order to determine the concentration of the sample.

3.4 Adsorption

Zeolites and activated carbons of differing characteristics were used to determine what characteristics would best support the adsorption of triclosan. A table with the adsorbents used and their properties can be seen in Table 5 in Appendix C: Table of Zeolite Properties. Each adsorbent was measured out by volume to fill about 1/3 of a dram vial. Three vials were made for each adsorbent and weighed for consistency. A saturated solution of triclosan in DI water was

prepared. The concentration of the saturated solution was determined using the HPLC and the calibration curve. For each sample, 9 mL of the solution was added to the adsorbents. The dram vials were then placed in a rotating machine to provide constant agitation. The samples were agitated for approximately a week. Once the cycle was complete, the samples were places in a centrifuge to separate the solids from the liquid for about 20 minutes. After separation the liquids were run through the HPLC to determine the post-adsorbent concentration.

Chapter 4: Results and Discussion

The following section is a compilation of the findings of this study. First, the results and analysis of the three methods of detection are presented. These methods were fluorescence spectroscopy, gas chromatography, and high-pressure liquid chromatography. The calibration curve used to determine the concentrations of each sample is presented and discussed. Finally, the results of the adsorption testing are discussed and analyzed.

4.1 Methods of Detection

Fluorescence spectroscopy, gas chromatography and high-pressure liquid chromatography were analyzed as possible methods of detection. After two months of running tests on the fluorescence spectrometer, it was determined that the results were not viable. The intensity of each sample with varying concentration appeared to follow no clear trend. It is believed that the concentrations of the samples were outside of the detectable range.

Gas chromatography did yield a peak for the most concentrated of the calibration solutions; at lower concentrations, however, the peaks were too small to be read. It was determined that the machine was not able to detect triclosan at the low concentrations needed to successfully accomplish the goals of this study.

Considering the undesirable results of the fluorescence spectroscopy and the GC, high-pressure liquid chromatography (HPLC) was tested. Although the HPLC software was unable to integrate all the peaks, there was a distinguishable difference in peak height that we were able to use to interpret the differences in concentration. Thus, the HPLC was able to yield quantitative results to a certain extent.

4.2 Calibration Curve

A calibration curve was successfully achieved from the HPLC. The peak height of each calibration solution was recorded and plotted against the solution's molar concentration. The resulting calibration curve can be seen below in Figure 13. The peaks for each calibration run can be seen Appendix D: Raw Data. The most concentrated solution gave a point that did not follow the trend of the rest of the samples, so it was disregarded. The remaining points were able to yield a best fit line with an R^2 value of 0.999, as seen in Figure 13: Calibration Curve for

Triclosan and Methanol on the HPLC. Given such a high R^2 value, the equation for this curve was used to determine the concentration of samples in later runs.

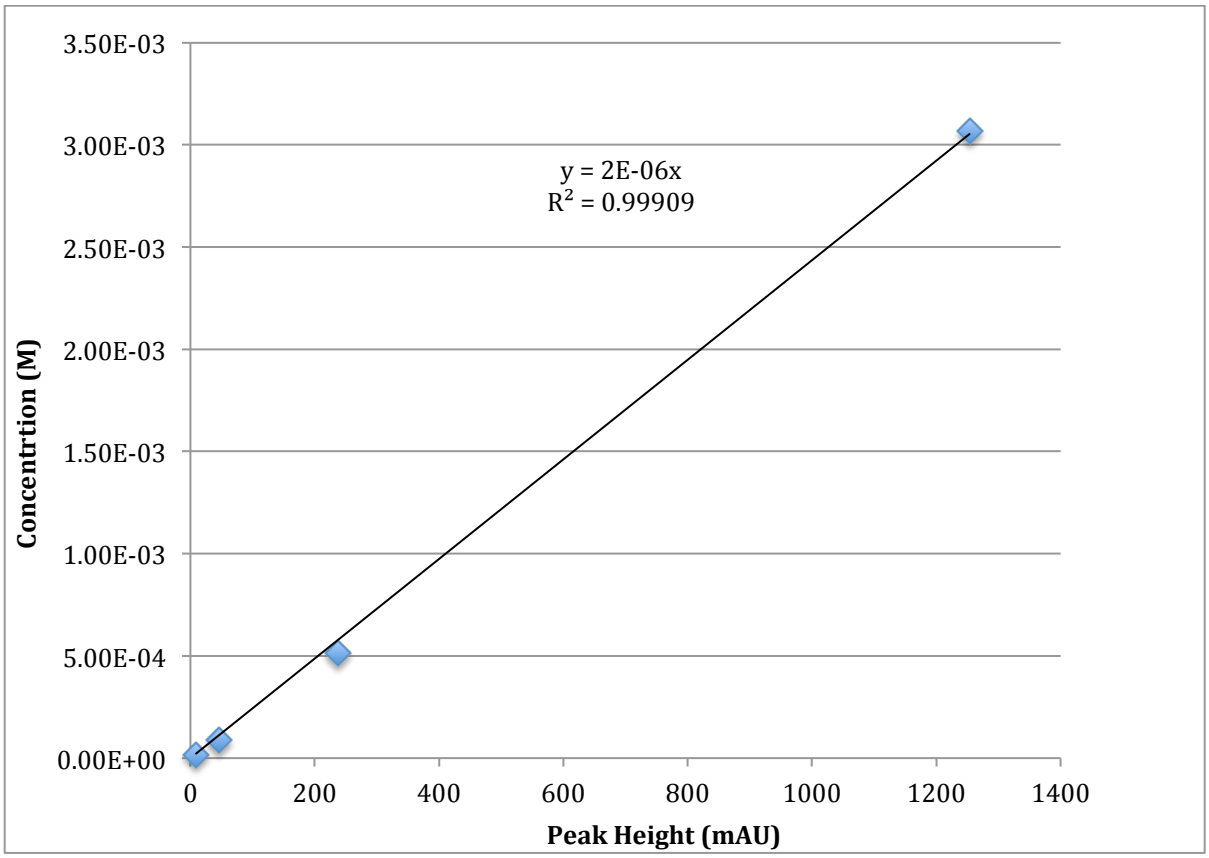

Figure 13: Calibration Curve for Triclosan and Methanol on the HPLC

4.3 Adsorption

After the saturated solution of triclosan (3.04×10^{-5} M) was allowed to adsorb onto the various zeolites and activated carbons for approximately 4 days, all samples were run on HPLC. Once all the HPLC data were gathered, the peak heights were measured. The software calculated some of the peak heights, while other heights had to be measured manually. These peak heights were inserted into the calibration equation to determine the concentration of each solution. The average percentage of triclosan removed was calculated and is presented in Figure 14.

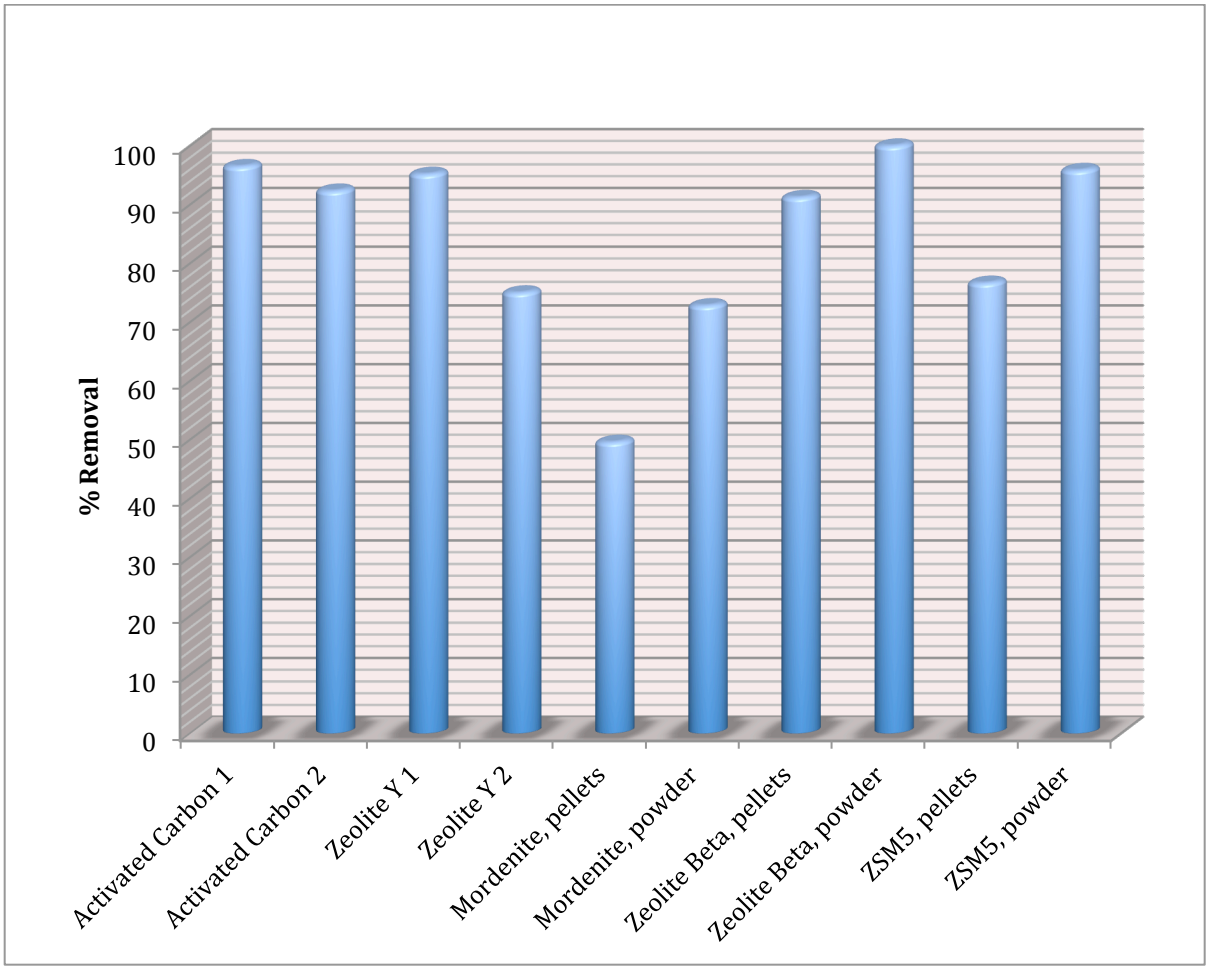

Figure 14: Percentage Triclosan removed after exposure to zeolites and activated carbons

It can be seen that zeolite β powder appeared to be the most effective at removing triclosan, while mordenite pellets appeared least effective based on the data obtained. There are several contributing factors that could have led to these results. Zeolite β powder was likely an effective adsorbent due to its high Si/Al ratio and its larger pore diameter, which has the ability to better accommodate triclosan. A high Si/Al ratio alone was not enough to achieve good adsorption. For example, ZSM-5 powder has a very high Si/Al ratio, but its pore diameter was far too small to accommodate triclosan molecules. The pore size on ZSM-5 is either 5.1 x 5.5 or 5.3 x 5.6 Å, while the diameter of triclosan is 7.4 Å. It is possible that the triclosan adsorbed only onto the surface of the ZSM-5, and not into the pores. These findings are consistent with the poor results for mordenite, which has a low Si/Al ratio and small pore diameters (6.5 x 7.0 Å). Zeolite β has a

high Si/Al ratio as well as large enough pores (6.6 – 7.7 Å) to accommodate triclosan, which could account for the large adsorption seen.

The powder form of every zeolite appeared to perform better than their pellet counterparts. Triclosan has a diameter of approximately 7.4 Å, which is close to or slightly higher than the pore diameters of most of the zeolites. From this, it can be inferred that the triclosan was primarily adsorbed onto the external surface of the zeolites. Since the powder has more surface area, it would follow that the powder form would adsorb better than the pellet form.

The test of adsorption onto the two different types of zeolite Y yielded unexpected results. Y1 was only 9% zeolite, whereas Y2 was 14% zeolite. In theory, zeolite Y2 should have adsorbed better than Y1. The data shows, however, that Y1 was a better adsorbent for triclosan than Y2. These are consistent with Brian Momani's findings in his study on the adsorption of estrone[46].Since the Si/Al ratios of the two zeolite Ys is unknown, it is likely that Y1 has a higher ratio than Y2.

Both forms of activated carbon used in this experiment appeared to remove a majority of the triclosan in solution. It was expected that the activated carbons would have a high adsorption. Unfortunately, very little information on the characteristics of these two activated carbons could be found, and the manufacturers could not be contacted during the course of this study.

Chapter 5: Conclusions and Recommendations

Throughout the course of this study, three methods of detecting triclosan and determining the concentration in various samples were tested. In doing so, it was determined that fluorescence spectroscopy was not a viable method for determining triclosan concentrations. Although there were peaks that were read by the spectrometer, they were not consistent when the same sample was run multiple times, even if done in immediate succession. Also, when creating the calibration curve, there was no clear trend with respect to concentration and the fluorescence readings. Because of this, it was determined that fluorescence spectroscopy could not be used for this study.

Many studies in the past have used gas chromatography (GC) as a method for detecting triclosan, and have had success. After the failure of fluorescence spectroscopy, this method was analyzed. Unfortunately, the equipment available at WPI is not sensitive enough. All the previous studies had used either gas chromatography that was coupled with a mass spectrometer (GC/MS) or an atomic emission detector (GC-AED). At WPI, the only GC machine available had a flame ionization detector (GC/FID). The GC/FID was able to detect triclosan at high concentrations, but it was unable to detect the low concentrations needed. The calibration curve was designed to operate within the concentrations found in nature; however, the samples were outside of the detectable limits of the machine. Both GC/MS and GC-AED are more sensitive than GC/FID and thus better able to detect triclosan at low concentrations. It is believed that more accurate results could be obtained for this study if a GC/MS was available. If this project is continued in the future, the GC/MS should best be able to detect triclosan at the low concentrations found in nature.

The final detection method tested was high-pressure liquid chromatography (HPLC). This gave the best results, and from these a calibration curve was generated. This was then used to determine the concentrations before and after adsorption onto the various zeolites and activated carbons. After adsorption, most samples showed a very small peak, but some samples showed no peak. It was assumed that adsorption for these samples was 100%; however, this may not have always been the case. It is possible that the concentration post-adsorption was outside of the detectable limits of the machine. Again, it is believed that performing this study on a GC/MS could greatly improve the accuracy of these results and provide a much more accurate efficiency

of the various adsorbents. Additionally, it is believed that a study of the amount of zeolite necessary for full adsorption would provide a deeper understanding of the effectiveness of each zeolite. Due to time constraints, this study did not examine the zeolite to triclosan ratio and would be a useful study to complete in the future.

Prior to the adsorption testing, it was hypothesized that the best zeolite would be one with a high Si/Al ratio, pore size closest to the diameter of triclosan (7.4 Å), and high surface area. The Si/Al ratio affects adsorption because zeolites with a high ratio (containing more silica) attract more hydrophobic molecules, such as triclosan. The surface area is much larger in the powdered form compared to the pellet form, and it was found that all powdered zeolites adsorbed more triclosan than their pellet counterparts. This finding confirmed the idea that a larger surface area increases adsorption.

During the tests, zeolite β powder was found to be the most effective at removing triclosan from water. Zeolite β has a high Si/Al ratio, so it easily attracts triclosan. The pore size in zeolite β is almost identical to the diameter of triclosan; therefore, triclosan was able to fit tightly into the pore. We also found that mordenite pellets were the least effective adsorbent tested. The Si/Al ratio in mordenite is low (contains less silica), so it does not attract the hydrophobic triclosan. The pore size in mordenite is smaller than the diameter of triclosan, so triclosan was only able to adsorb onto the surface, and not into the pores. The powdered form of mordenite was able to adsorb slightly more triclosan, but overall, mordenite was not the best adsorbent for triclosan.

Based on previous studies, we expected that the activated carbons would be very effective in adsorbing triclosan. Many studies have had success with activated carbons, and we believe that our results support these findings. Both activated carbons tested adsorbed most of the triclosan, so we believe that these are a feasible method for adsorption. Based on the results, however, zeolite β powder is the best adsorbent for triclosan.

One aspect of our study that was not able to be accomplished was a time-dependent study of the adsorbents. Previous studies concluded that maximum adsorption was reached between 24-48 hours. Originally, this study had intended to create a time-dependent test on the adsorbents by determining the concentration of the samples over the course of several days. Due to time constraints, however, this was not possible. It is believed that this information would be useful in

a future study. This could also be expanded to use compounds other than triclosan to determine if there is any difference in the time required for maximum adsorption for each adsorbent.

Another aspect that this study was unable to complete was determining if the use of co-solvents with triclosan changed the amount adsorbed. Prior studies indicated that the co-solvents would increase the solubility of triclosan in water, but the review of the literature did not reveal any studies that analyzed adsorption using co-solvents with the triclosan. It would be helpful to conduct a study in this manner in the future.

Overall, it was determined that the results of this study would be much more accurate if the testing had been performed on more sensitive equipment. If it is possible to repeat this study using a GC/MS as a method of detection, the results would be much more significant, as it would be more effective at detecting triclosan in lower concentrations.

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Appendix A: Glossary of Terms

Å: Angstrom

βCD: beta-cyclodextrin

DAD: Diode array detector

ENR: Enzyme enoyl-acyl carrier protein reductase

ECC: Emerging contaminants of concern

GC: Gas Chromatography

GC-AED: Gas chromatography atomic emission detector

GC/FID: Gas chromatography flame ionization detector

GC/MS: Gas chromatography, Mass spectrometry

HPβCD: hydroxypropyl beta-cyclodextrin

HPLC: High pressure liquid chromatography

M: Molar, moles per liter

ml: Milliliter

µl: Microliters

mol: moles

MSDS: Material safety data sheet

nm: Nanometers

R^2 value: coefficient of determination

Si/Al ratio: ratio of silicon dioxide to aluminum oxide in a zeolite

SLS: sodium lauryl sulfate

USGS: United States Geological Survey

Appendix B: Triclosan Material Safety Data Sheet

Material Safety Data Sheet
acc. to OSHA and ANSI

Printing date 11/11/2008 *Reviewed on 11/11/2008*

1 Identification of substance:

Product details:

Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

Stock number: L18655

Manufacturer/Supplier:
Alfa Aesar, A Johnson Matthey Company
Johnson Matthey Catalog Company, Inc.
30 Bond Street
Ward Hill, MA 01835-8099
Emergency Phone: (978) 521-6300
CHEMTREC: (800) 424-9300
Web Site: www.alfa.com

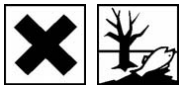
Information Department:
Health, Safety and Environmental Department
Product Safety Department
Emergency information:
During normal hours the Health, Safety and Environmental Department.
After normal hours call Chemtrec at (800) 424-9300.

2 Composition/Data on components:

Chemical characterization:
Description: (CAS#)
5-Chloro-2-(2,4-dichlorophenoxy)phenol
(CAS# 3380-34-5): 100%
Identification number(s):
EINECS Number: 222-182-2
Index number: 604-070-00-9

3 Hazards identification

Hazard description:



Xi Irritant
N Dangerous for the environment

Information pertaining to particular dangers for man and environment
R 36/38 Irritating to eyes and skin.
R 50/53 Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment
Classification system
HMIS ratings (scale 0-4)
(Hazardous Materials Identification System)

HEALTH	1
FIRE	1
REACTIVITY	1

Health (acute effects) = 1
Flammability = 1
Reactivity = 1

4 First aid measures

After inhalation
Supply fresh air. If required, provide artificial respiration. Keep patient warm.
Seek immediate medical advice.

(Contd. on page 2)

Material Safety Data Sheet
acc. to OSHA and ANSI

Printing date 11/11/2008 Reviewed on 11/11/2008

Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

(Contd. of page 1)

After skin contact
Immediately wash with water and soap and rinse thoroughly.
Seek immediate medical advice.
After eye contact
Rinse opened eye for several minutes under running water. Then consult a doctor.
After swallowing Seek immediate medical advice.

5 Fire fighting measures

Suitable extinguishing agents
Carbon dioxide, extinguishing powder or water spray. Fight larger fires with water spray or alcohol resistant foam.
Special hazards caused by the material, its products of combustion or resulting gases:
In case of fire, the following can be released:
Carbon monoxide and carbon dioxide
Hydrogen chloride (HCl)
Protective equipment:
Wear self-contained respirator.
Wear fully protective impervious suit.

6 Accidental release measures

Person-related safety precautions:
Wear protective equipment. Keep unprotected persons away.
Ensure adequate ventilation
Measures for environmental protection:
Do not allow material to be released to the environment without proper governmental permits.
Measures for cleaning/collecting: Pick up mechanically.
Additional information:
See Section 7 for information on safe handling
See Section 8 for information on personal protection equipment.
See Section 13 for disposal information.

7 Handling and storage

Handling
Information for safe handling:
Keep container tightly sealed.
Store in cool, dry place in tightly closed containers.
Ensure good ventilation at the workplace.
Information about protection against explosions and fires:
Keep ignition sources away.

Storage
Requirements to be met by storerooms and receptacles:
No special requirements.
Information about storage in one common storage facility:
Store away from oxidizing agents.
Further information about storage conditions:
Keep container tightly sealed.

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Material Safety Data Sheet
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Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

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Store in cool, dry conditions in well sealed containers.

8 Exposure controls and personal protection

Additional information about design of technical systems:
Properly operating chemical fume hood designed for hazardous chemicals and having an average face velocity of at least 100 feet per minute.

Components with limit values that require monitoring at the workplace:
Not required.
Additional information: No data

Personal protective equipment
General protective and hygienic measures
The usual precautionary measures for handling chemicals should be followed.
Keep away from foodstuffs, beverages and feed.
Remove all soiled and contaminated clothing immediately.
Wash hands before breaks and at the end of work.
Avoid contact with the eyes and skin.
Breathing equipment:
Use suitable respirator when high concentrations are present.
Protection of hands:
Check protective gloves prior to each use for their proper condition.
Impervious gloves
Material of gloves
The selection of suitable gloves not only depends on the material, but also on quality. Quality will vary from manufacturer to manufacturer.
Eye protection: Safety glasses
Body protection: Protective work clothing.

9 Physical and chemical properties:

General Information	
Form:	Crystalline powder
Color:	White
Odor:	Not determined
Change in condition	
Melting point/Melting range:	56-58°C (133-136°F)
Boiling point/Boiling range:	Not determined
Sublimation temperature / start:	Not determined
Flash point:	Not applicable
Ignition temperature:	Not determined
Decomposition temperature:	Not determined
Danger of explosion:	Product does not present an explosion hazard.
Explosion limits:	
Lower:	Not determined
Upper:	Not determined
Vapor pressure:	Not determined

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Density:	Not determined

10 Stability and reactivity

Thermal decomposition / conditions to be avoided:
Decomposition will not occur if used and stored according to specifications.
Materials to be avoided: Oxidizing agents
Dangerous reactions No dangerous reactions known
Dangerous products of decomposition:
Carbon monoxide and carbon dioxide
Hydrogen chloride (HCl)

11 Toxicological information

Acute toxicity:

LD/LC50 values that are relevant for classification:		
Oral	LD50	3700 mg/kg (rat)
Irritation of skin	mild	750 µg/3D-I (human) 10 % (rabbit)

Primary irritant effect:
on the skin: Irritant to skin and mucous membranes.
on the eye: Irritating effect.
Sensitization: No sensitizing effects known.
Other information (about experimental toxicology):
Reproductive effects have been observed on tests with laboratory animals.
Mutagenic effects have been observed on tests with bacteria.
Subacute to chronic toxicity:
Subacute to chronic toxicity:
The Registry of Toxic Effects of Chemical Substances (RTECS) reports the following effects in laboratory animals:
Reproductive - Effects on Embryo or Fetus - fetal death.
Additional toxicological information:
To the best of our knowledge the acute and chronic toxicity of this substance is not fully known.
No classification data on carcinogenic properties of this material is available from the EPA, IARC, NTP, OSHA or ACGIH.

12 Ecological information:

Ecotoxical effects:
Remark: Very toxic for fish
General notes:
Do not allow product to reach ground water, water course or sewage system.
Danger to drinking water if even small quantities leak into the ground.
Also poisonous for fish and plankton in water bodies.
Do not allow material to be released to the environment without proper governmental permits.
Very toxic for aquatic organisms

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Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

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13 Disposal considerations

Product:
Recommendation
Consult state, local or national regulations to ensure proper disposal.

Uncleaned packagings:
Recommendation: Disposal must be made according to official regulations.

14 Transport information

DOT regulations:

Hazard class:	9
Identification number:	UN3077
Packing group:	III
Proper shipping name (technical name):	ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (5-Chloro-2-(2,4-dichlorophenoxy)phenol)
Label	9

Land transport ADR/RID (cross-border)

ADR/RID class:	9 (M7) Miscellaneous dangerous substances and articles
Danger code (Kemler):	90
UN-Number:	3077
Packaging group:	III
Description of goods:	3077 ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (5-Chloro-2-(2,4-dichlorophenoxy)phenol)

Maritime transport IMDG:

IMDG Class:	9
UN Number:	3077
Label	9
Packaging group:	III

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Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

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Proper shipping name:	ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (5-Chloro-2-(2,4-dichlorophenoxy)phenol)

Air transport ICAO-TI and IATA-DGR:

ICAO/IATA Class:	9
UN/ID Number:	3077
Label	9
Packaging group:	III
Proper shipping name:	ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID, N.O.S. (5-Chloro-2-(2,4-dichlorophenoxy)phenol)

15 Regulations

Product related hazard informations:

Hazard symbols:
Xi Irritant
N Dangerous for the environment

Risk phrases:
36/38 Irritating to eyes and skin.
50/53 Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment

Safety phrases:
26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
39 Wear eye/face protection.
46 If swallowed, seek medical advice immediately and show this container or label.
60 This material and its container must be disposed of as hazardous waste.
61 Avoid release to the environment. Refer to special instructions/Safety data sheets

National regulations
All components of this product are listed in the U.S. Environmental Protection Agency Toxic Substances Control Act Chemical substance Inventory.
All components of this product are listed on the Canadian Domestic Substances List (DSL).

Information about limitation of use:
For use only by technically qualified individuals.

16 Other information:

Employers should use this information only as a supplement to other information gathered by them, and should make independent judgement of suitability of this information to ensure proper use and protect the health and safety of employees. This information is furnished without warranty, and any use of the product not in conformance with this

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Product name: 5-Chloro-2-(2,4-dichlorophenoxy)phenol

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Material Safety Data Sheet, or in combination with any other product or process, is the responsibility of the user.

Department issuing MSDS: Health, Safety and Environmental Department.
Contact: Paul V. Connolly

USA

Appendix C: Table of Zeolite Properties

Table 4: Summary of Zeolite Samples Used[47]

Sample name	Size (in)	SiO_2/Al_2O_3	Zeolite %	Company name	Surface area (m^2/g) [35]	Micropore area (m^2/g) [35]	External area (m^2/g) [35]	Pore dimensions (Å) [14]
Zeolite Y1	0.15		9	Engelhard	158.6	73.4	85.2	7.4 × 7.4
Zeolite Y2	0.15		14	Engelhard	158.3	58.7	99.6	7.4 × 7.4
Mordenite, pellets	0.0625	50	80	Engelhard	472.6	304.3	168.3	6.5 × 7.0
Mordenite powder	0.0625	90	80	Zeolyst				6.5 × 7.0
Zeolite β, pellets	0.0625	35	80	Engelhard	533.7	266	267.6	6.6–7.7
Zeolite β, powder	0.0625	360	80	Zeolyst				6.6–7.7
ZSM-5, pellets	0.0625	280	80	Zeolyst	390.8	141.8	249	5.1 × 5.5, 5.3 × 5.6
ZSM-5 powder		1000		Zeochem				5.1 × 5.5, 5.3 × 5.6

Appendix D: Raw Data

Table 5: Triclosan Concentration in Treated Water

Adsorbent	Run A	Run B	Run C
Activated Carbon 1	0	3.20E-6	0
Activated Carbon 2	8.00E-7	6.20E-6	0.
Zeolite Y1	1.60E-6	1.80E-6	1.00E-6
Zeolite Y2	4.20E-6	3.20E-6	1.54E-5
Mordenite, pellets	1.80E-5	3.40E-6	2.46E-5
Mordenite, powder	0	2.60E-6	2.22E-5
Zeolite β, pellets	0	0	8.00E-6
Zeolite β, powder	0	0	0
ZSM-5, pellets	7.20E-6	7.00E-6	Over limit
ZSM-5, powder	2.40E-6	4.00E-7	1.00E-6

Table 6: Percent Triclosan Removed

Adsorbent	Run A	Run B	Run C
Activated Carbon 1	100.00	89.47	100.00
Activated Carbon 2	97.37	79.61	100.00
Zeolite Y1	94.74	94.08	96.71
Zeolite Y2	86.18	89.47	49.34
Mordenite, pellets	40.79	88.82	19.08
Mordenite, powder	100.00	91.45	26.97
Zeolite β, pellets	100.00	100.00	73.68
Zeolite β, powder	100.00	100.00	100.00
ZSM-5, pellets	76.32	76.97	Over limit
ZSM-5, powder	92.11	98.68	96.71

High-Pressure Liquid Chromatography Data

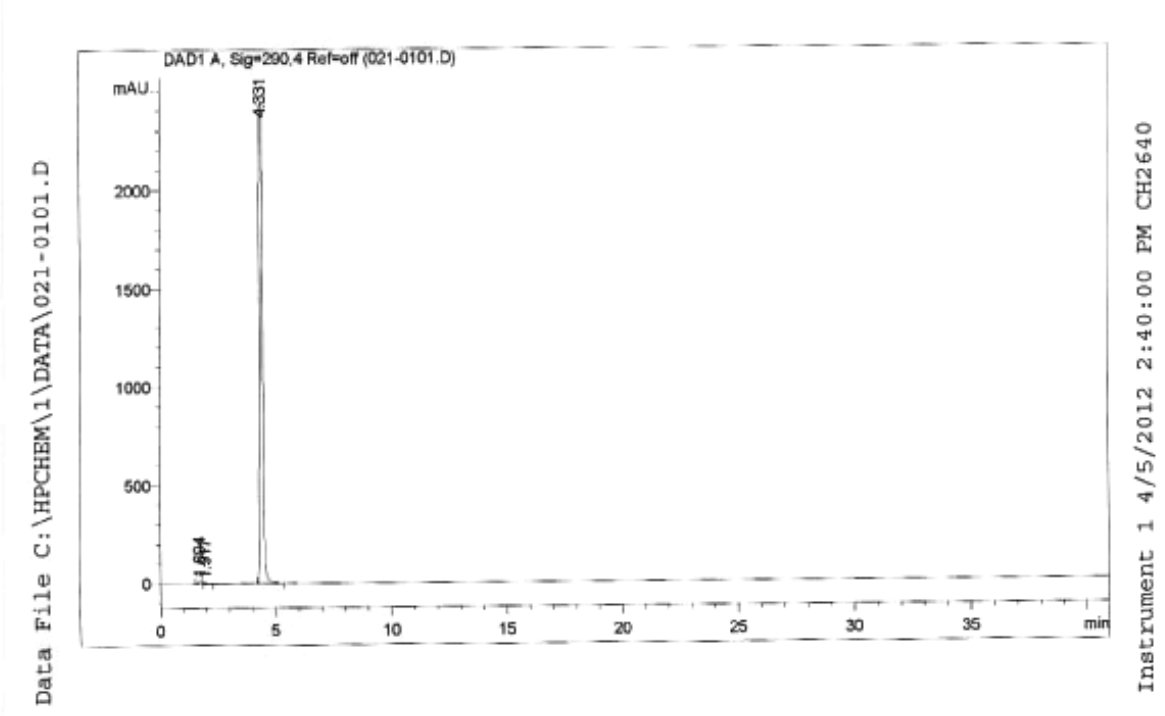

Figure 15: Stock Solution Peak for Calibration Curve

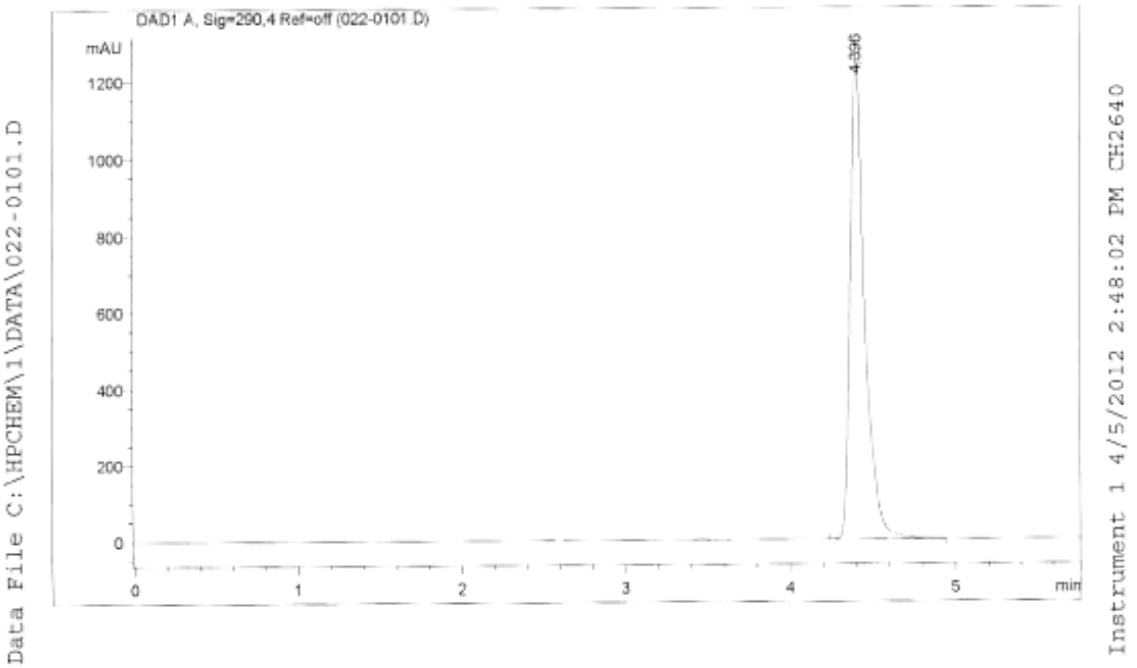

Figure 16: Dilution One Peak for Calibration Curve

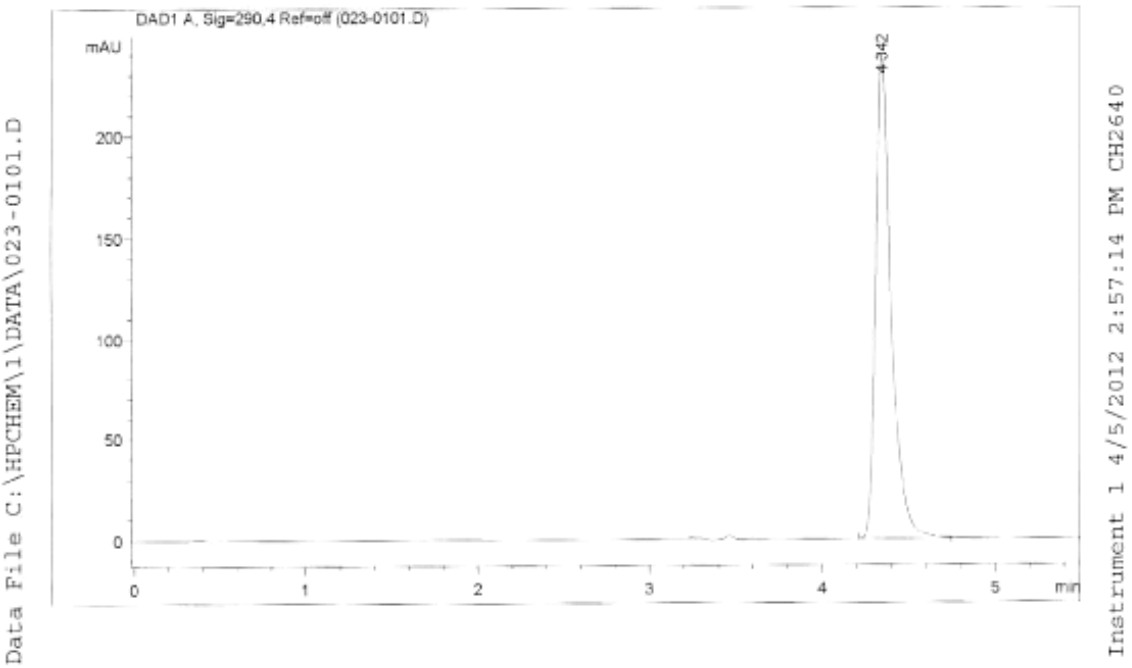

Figure 17: Dilution Two Peak for Calibration Curve

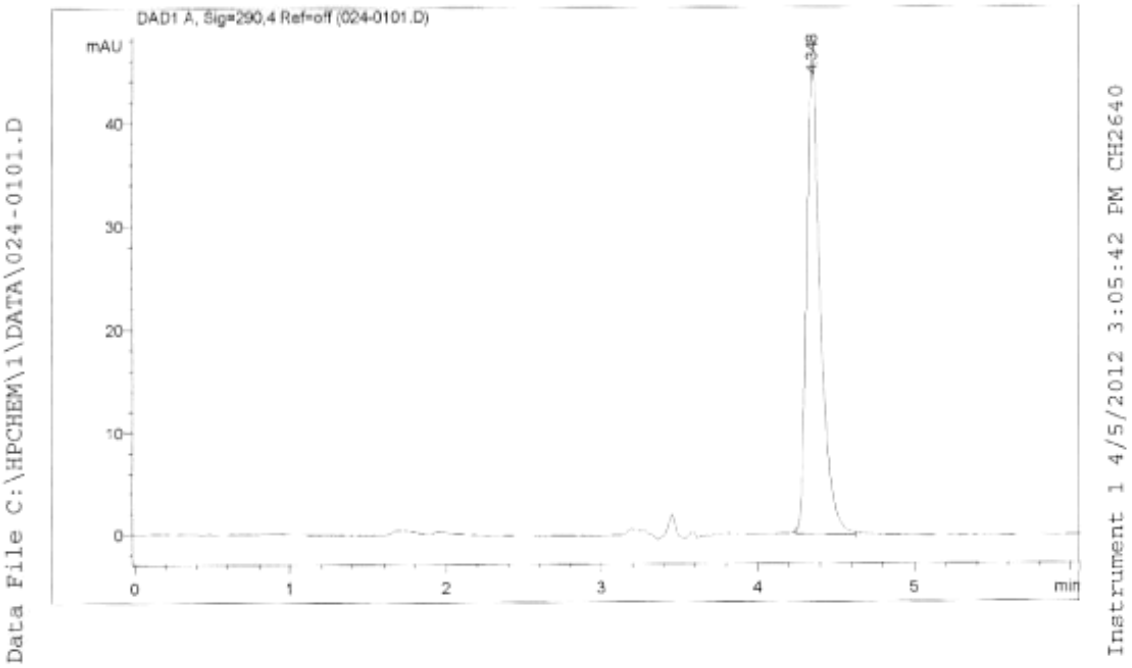

Figure 18: Dilution Three Peak for Calibration Curve

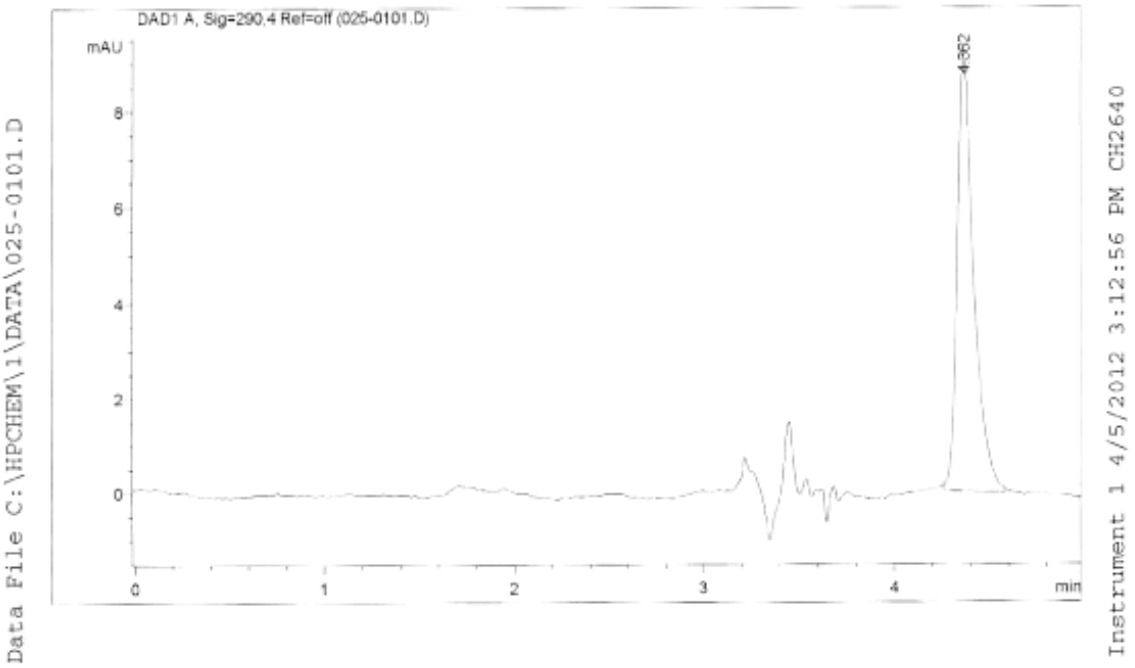

Figure 19: Dilution Four Peak for Calibration Curve

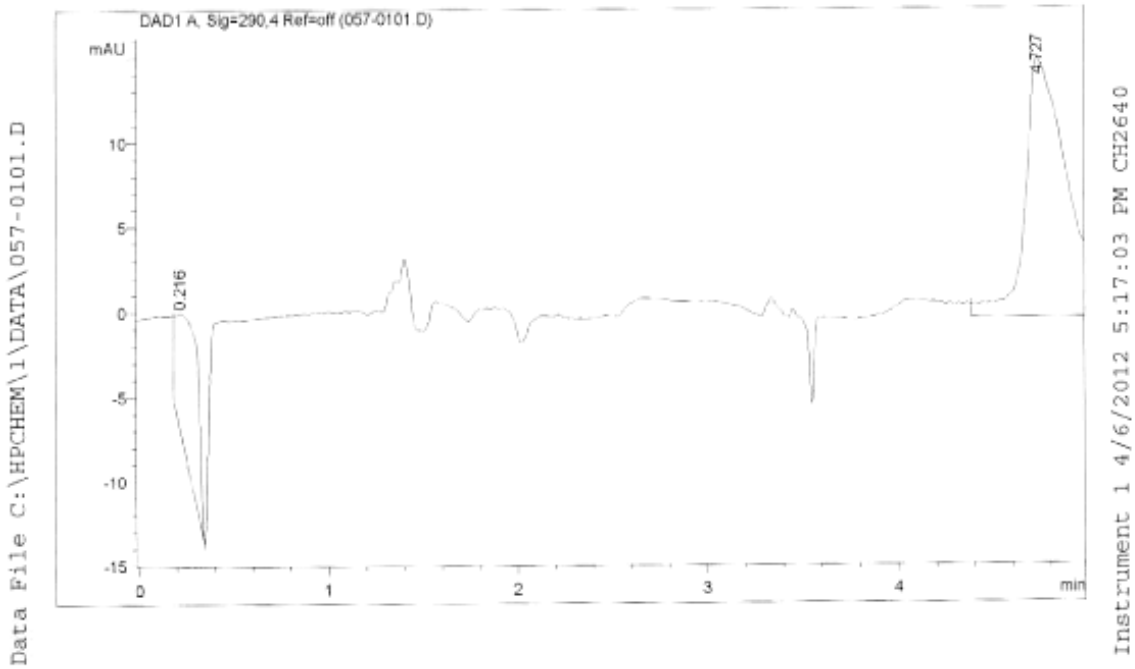

Figure 20: Saturated Solution Peak for Initial Concentration

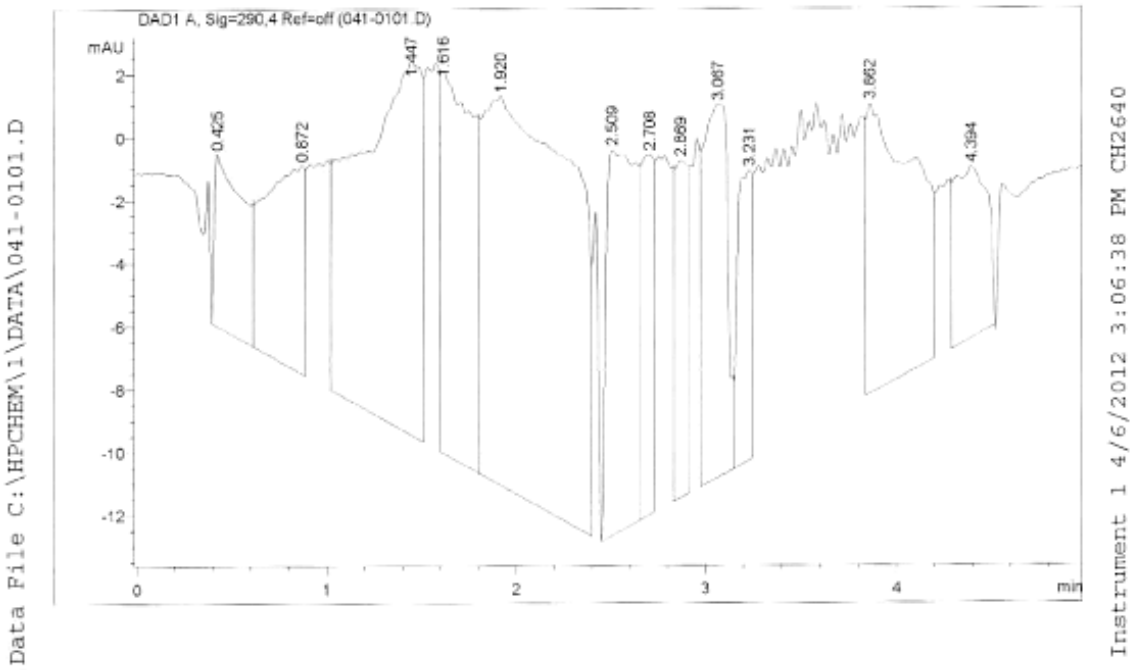

Figure 21: Activated Carbon One Peak A

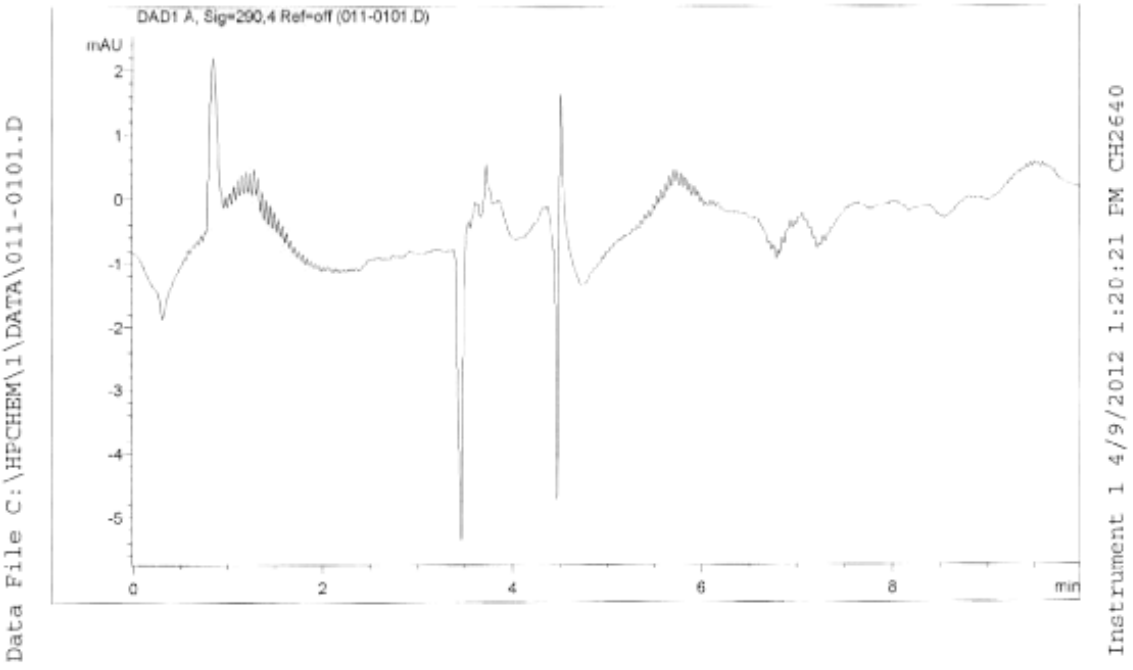

Figure 22: Activated Carbon One Peak B

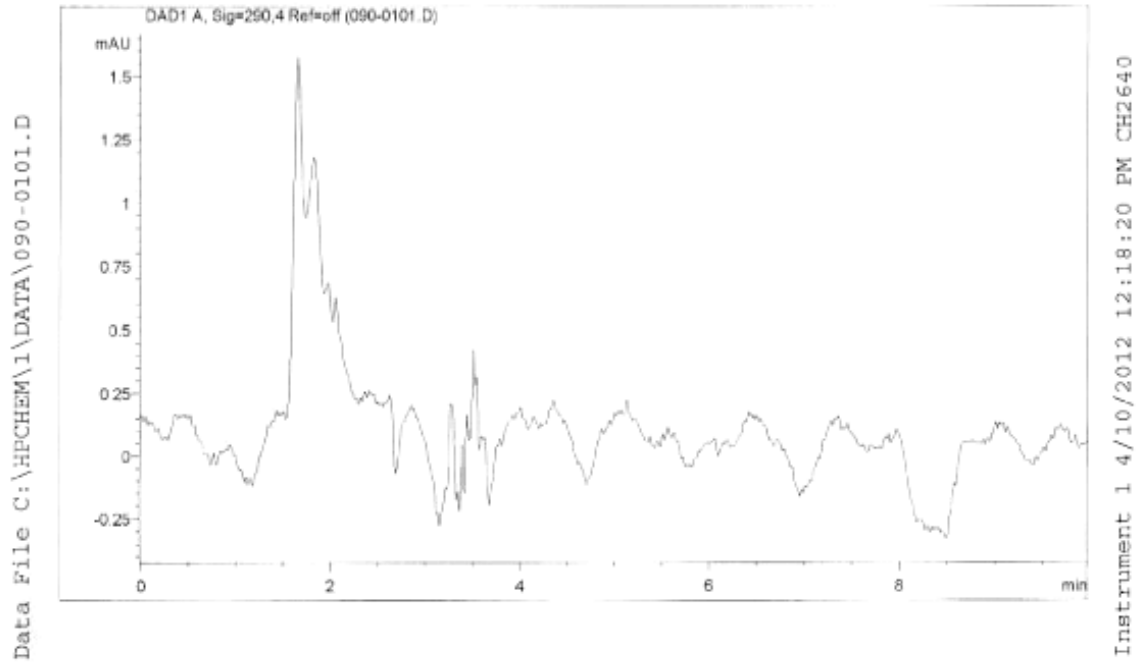

Figure 23: Activated Carbon One Peak C

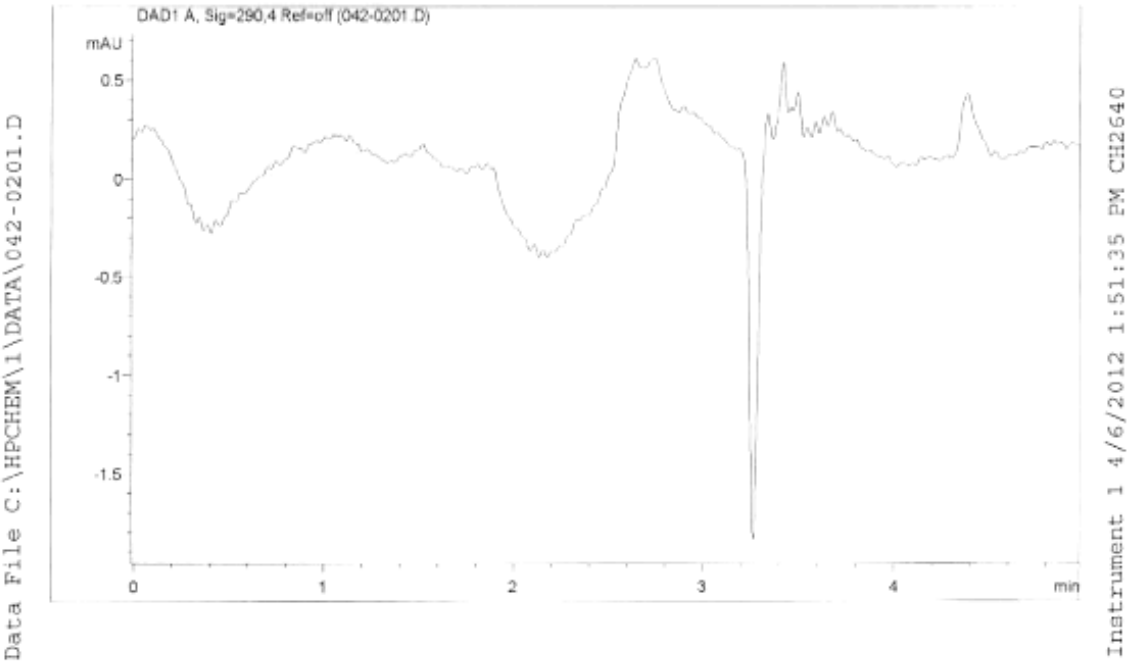

Figure 24: Activated Carbon Two Peak A

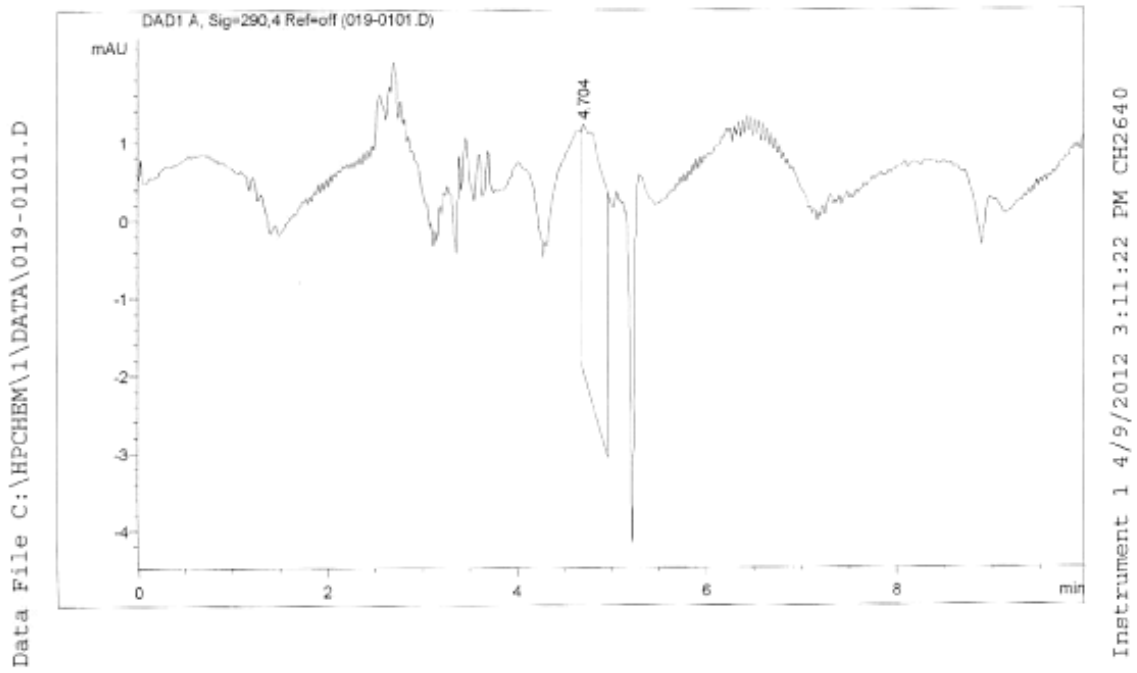

Figure 25: Activated Carbon Two Peak B

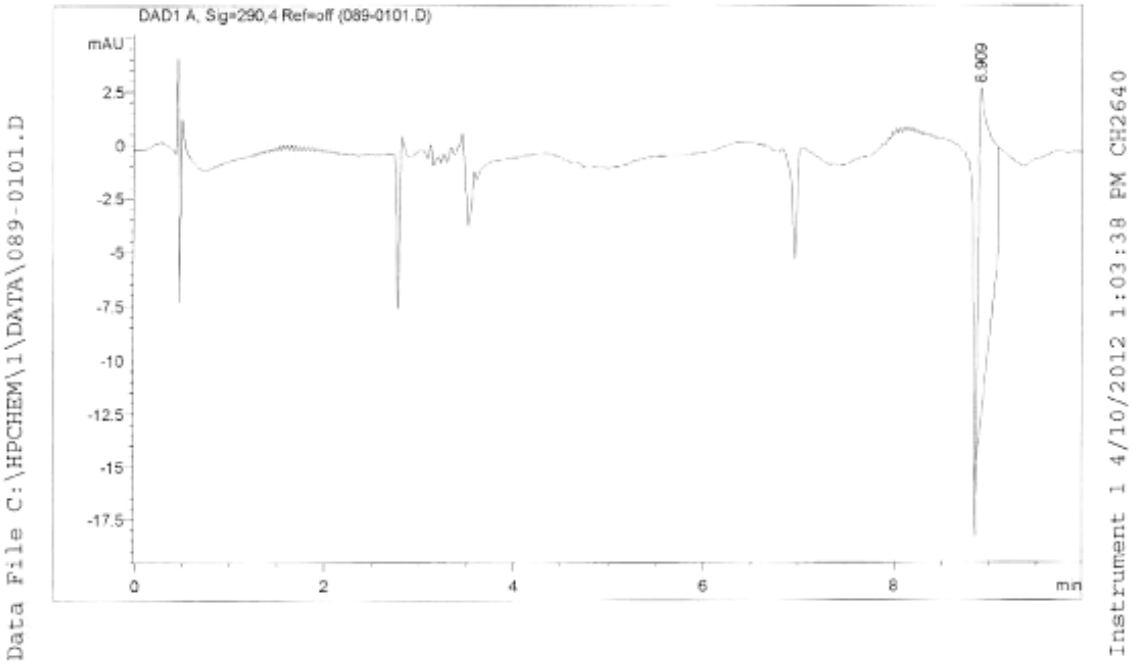

Figure 26: Activated Carbon Two Peak C

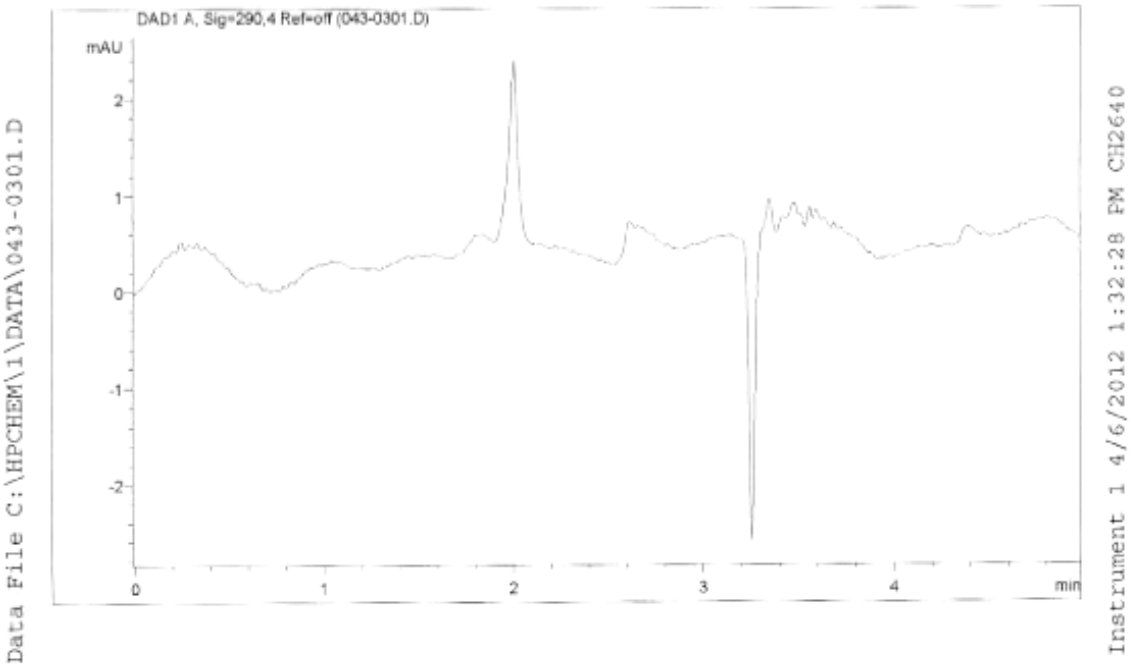

Figure 27: Zeolite Y1 Peak A

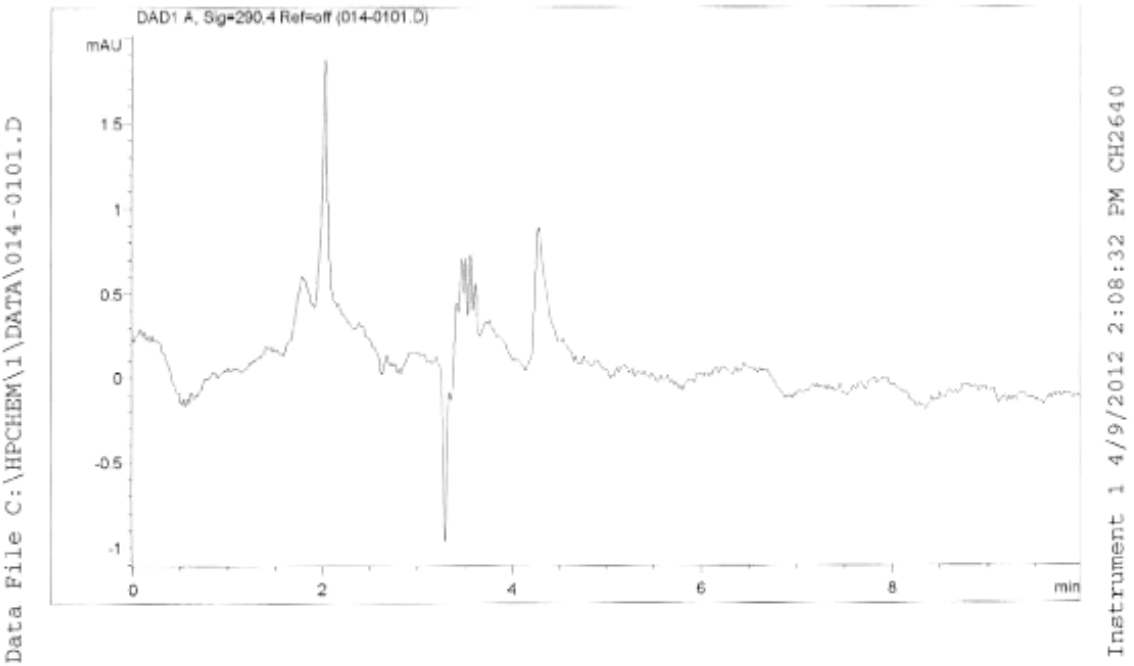

Figure 28: Zeolite Y1 Peak B

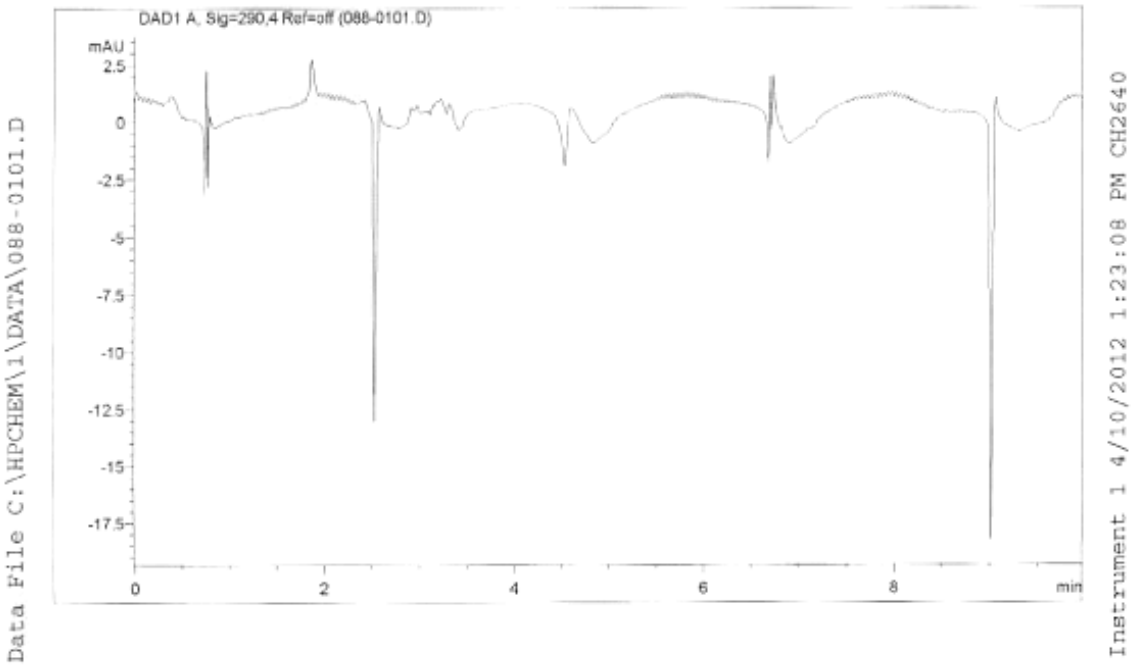

Figure 29: Zeolite Y1 Peak C

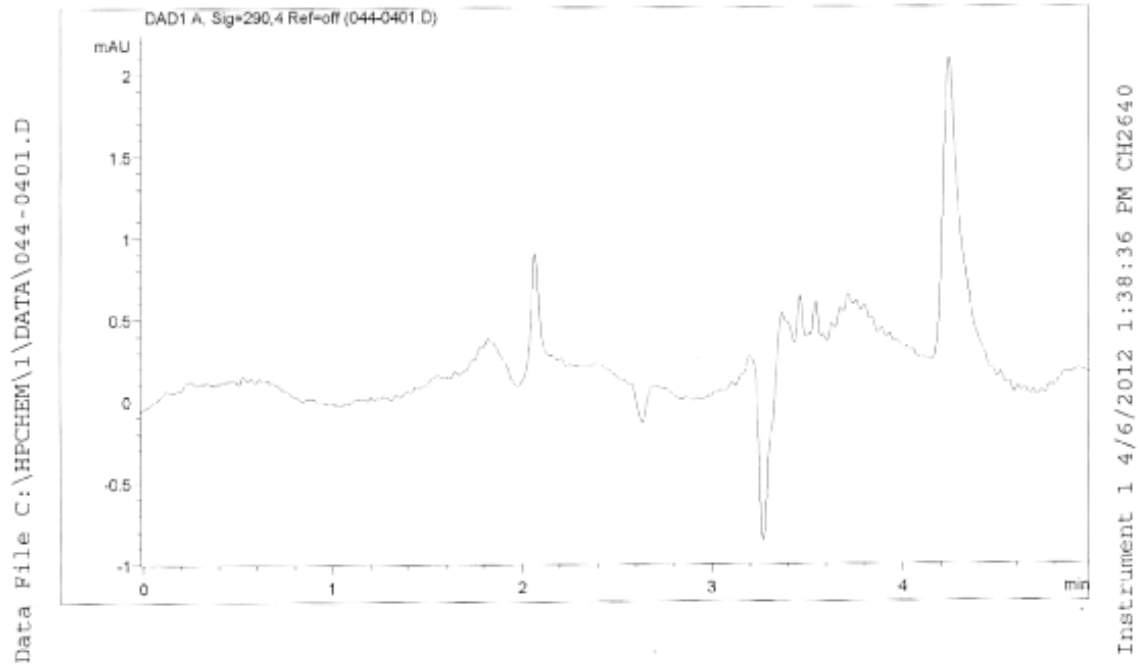

Figure 30: Zeolite Y2 Peak A

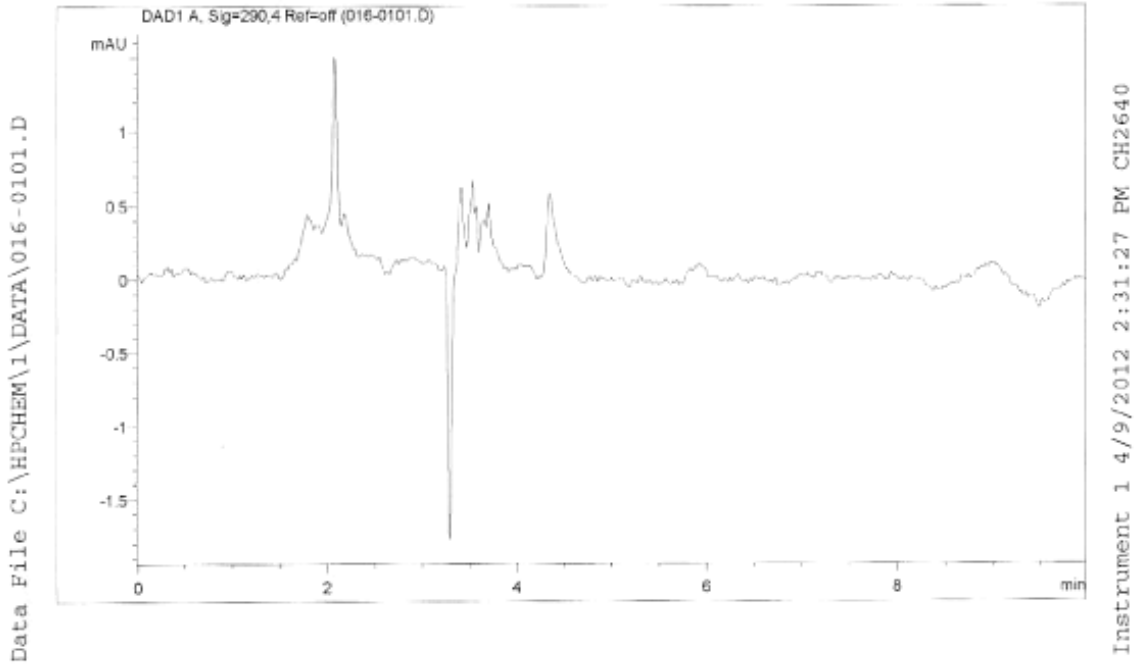

Figure 31: Zeolite Y2 Peak B

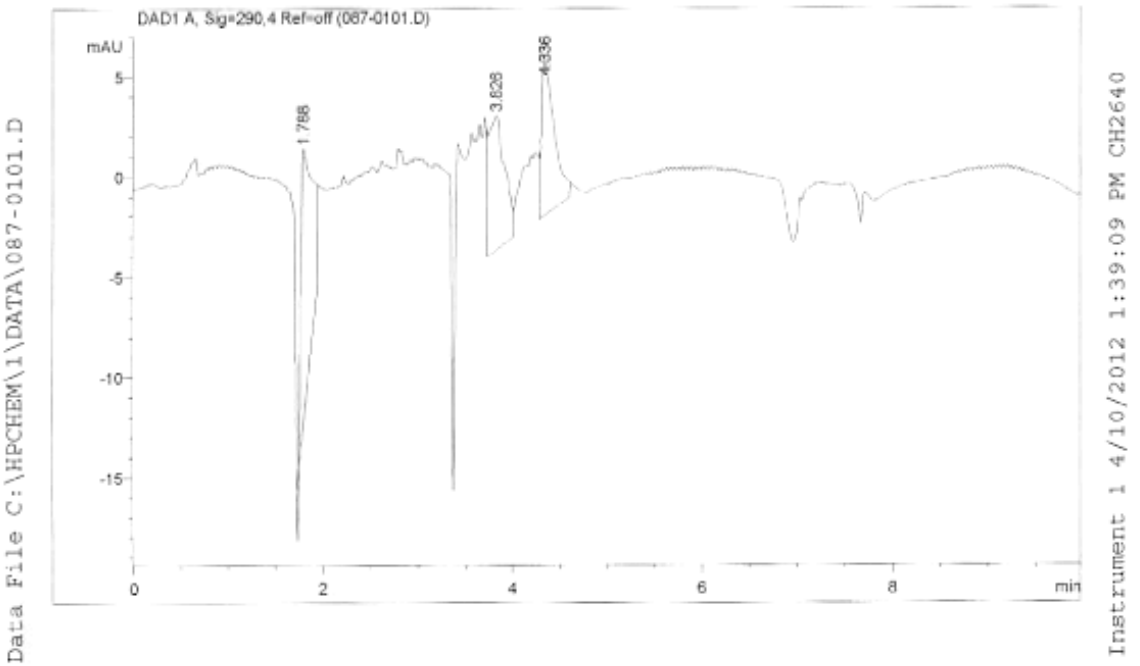

Figure 32: Zeolite Y2 Peak C

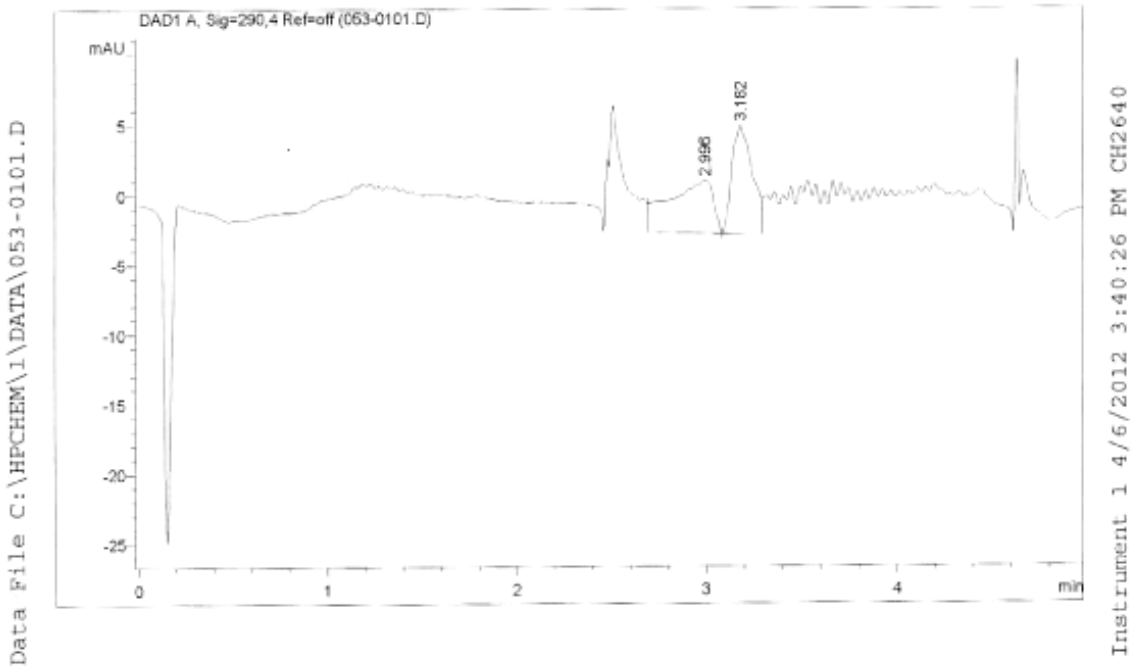

Figure 33: Mordenite (Pellet) Peak A

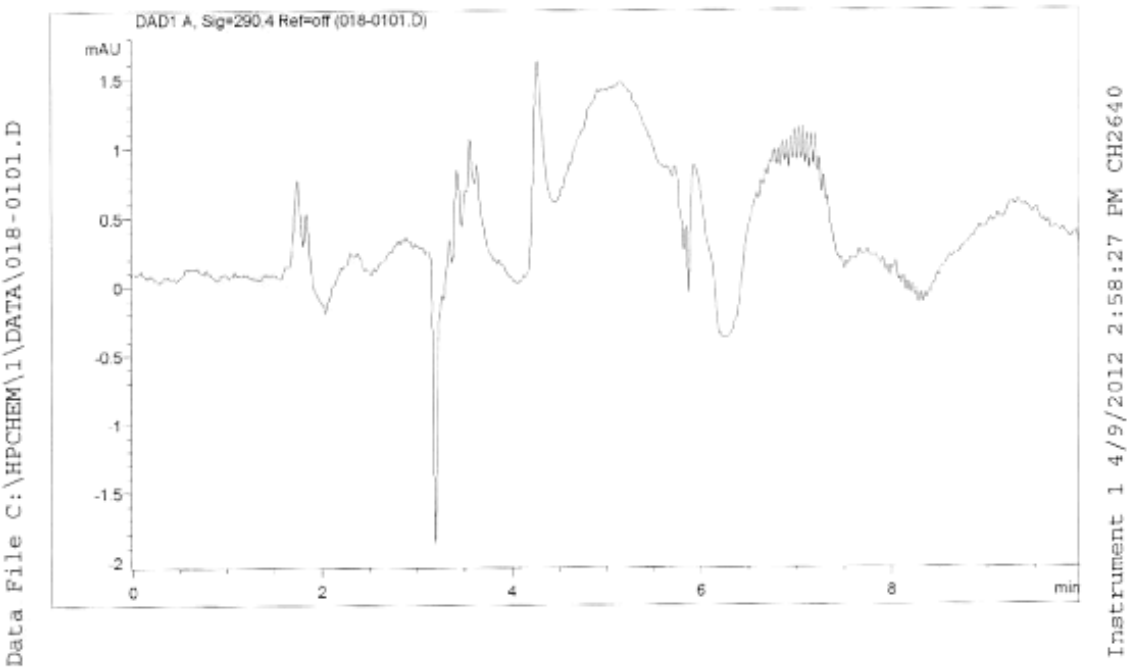

Figure 34: Mordenite (Pellet) Peak B

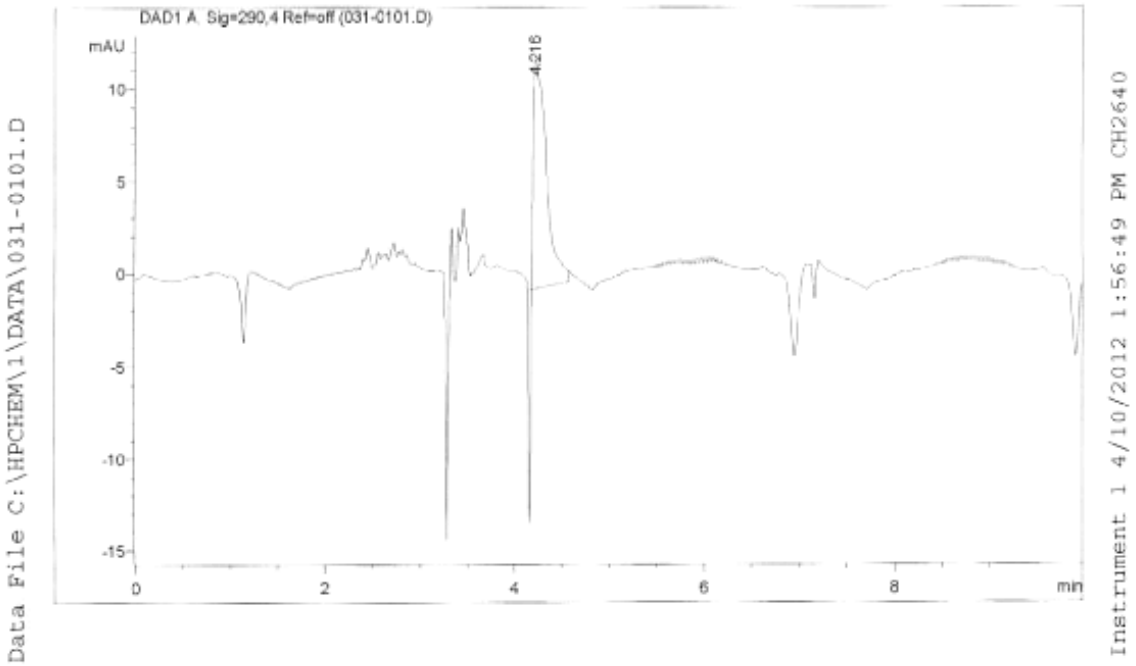

Figure 35: Mordenite (Pellet) Peak C

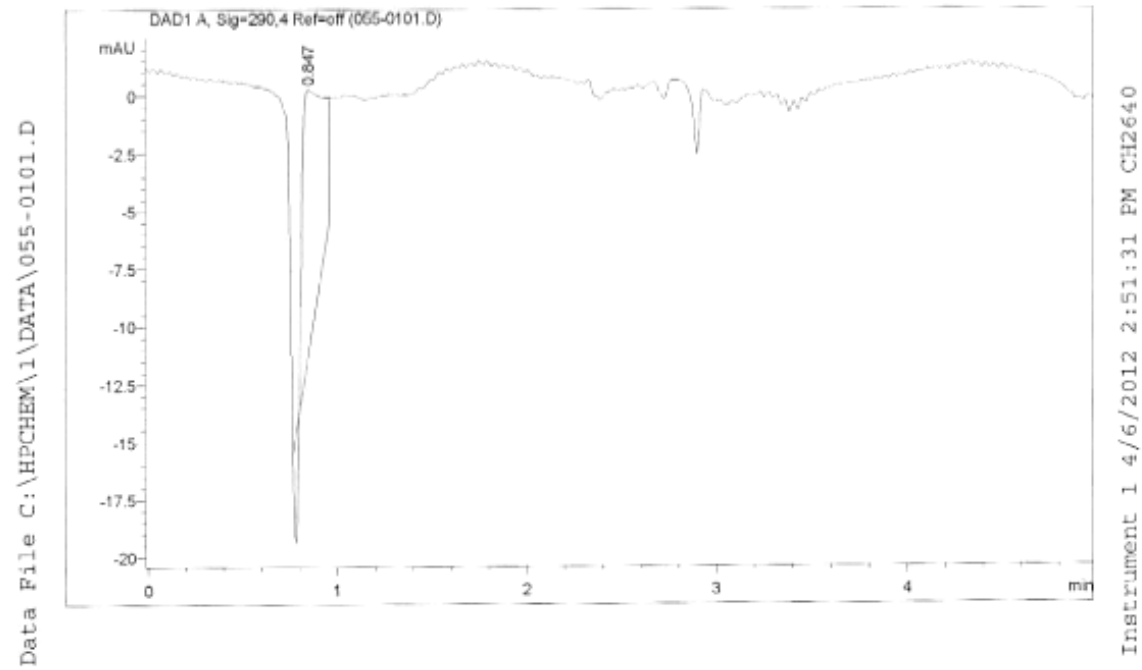

Figure 36: Mordenite (Powder) Peak A

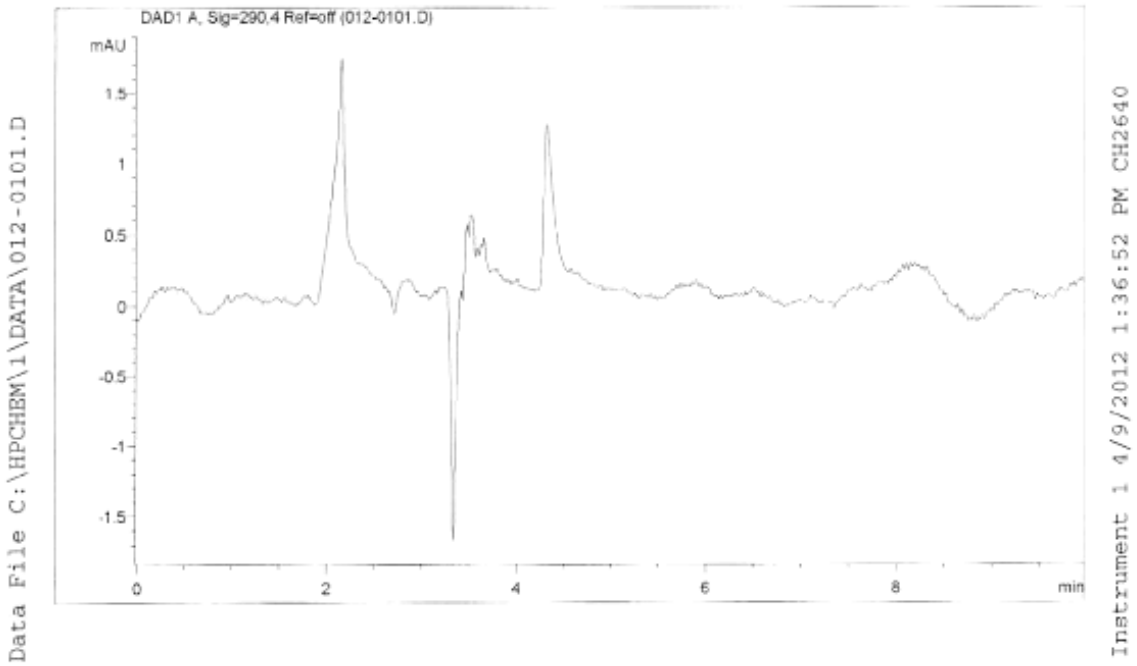

Figure 37: Mordenite (Powder) Peak B

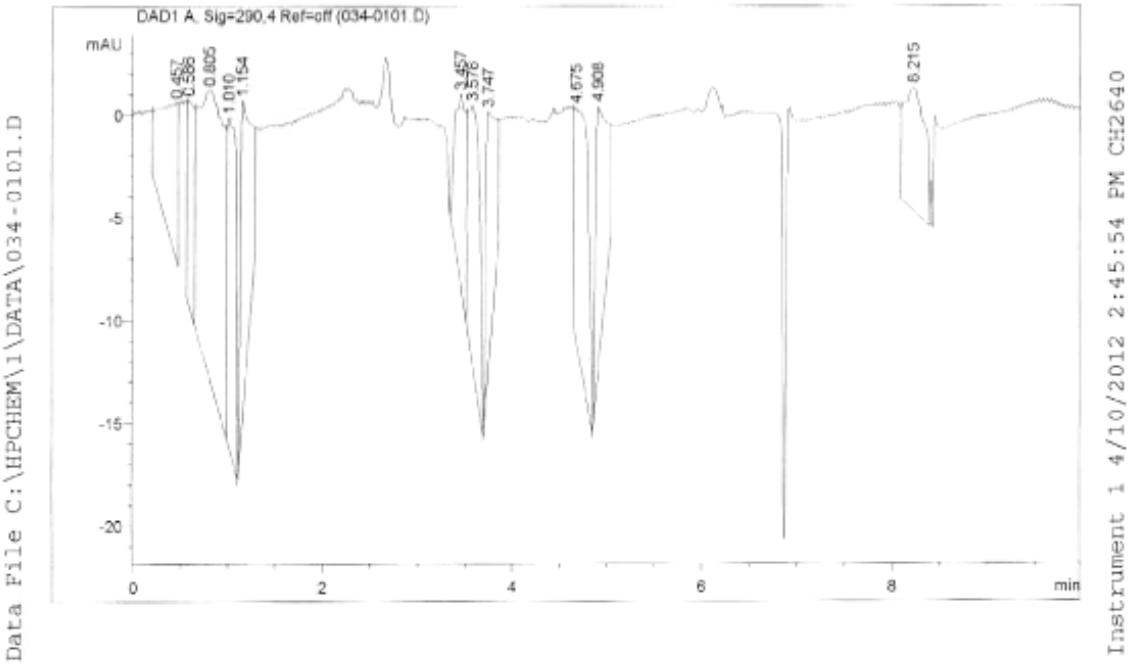

Figure 38: Mordenite (Powder) Peak C

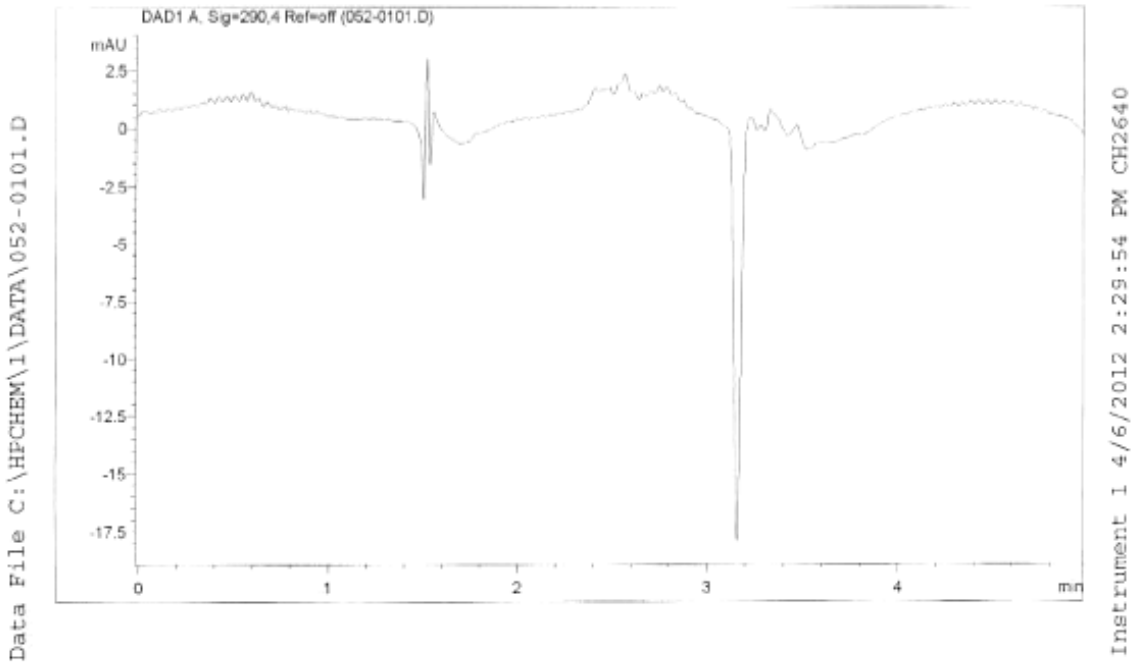

Figure 39: Zeolite Beta (Pellets) Peak A

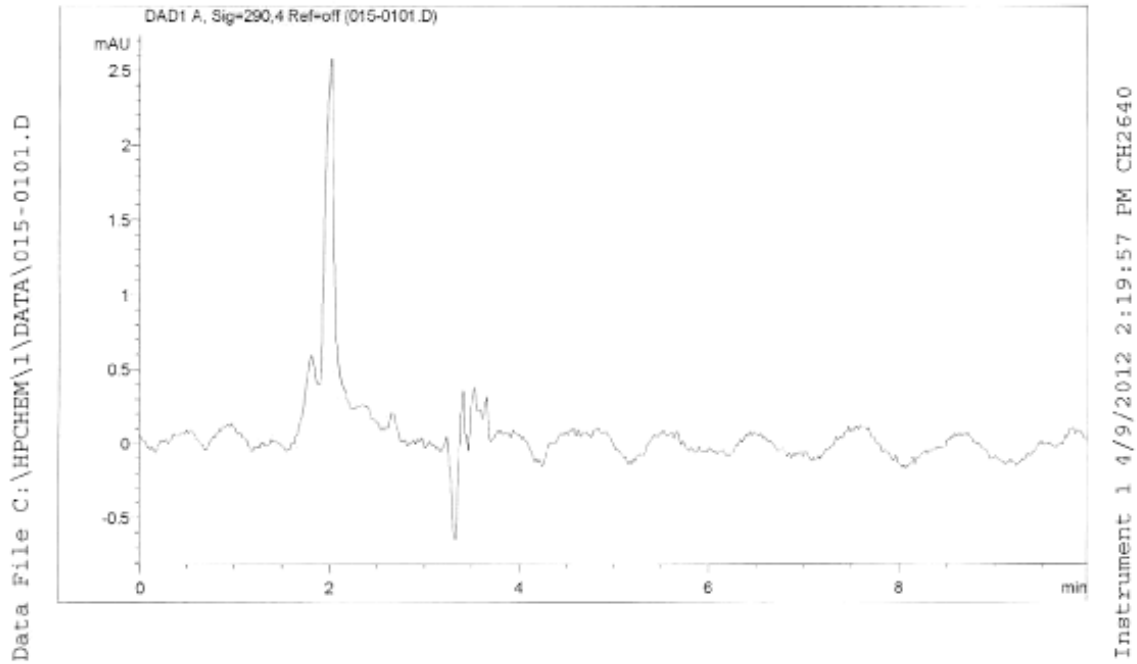

Figure 40: Zeolite Beta (Pellets) Peak B

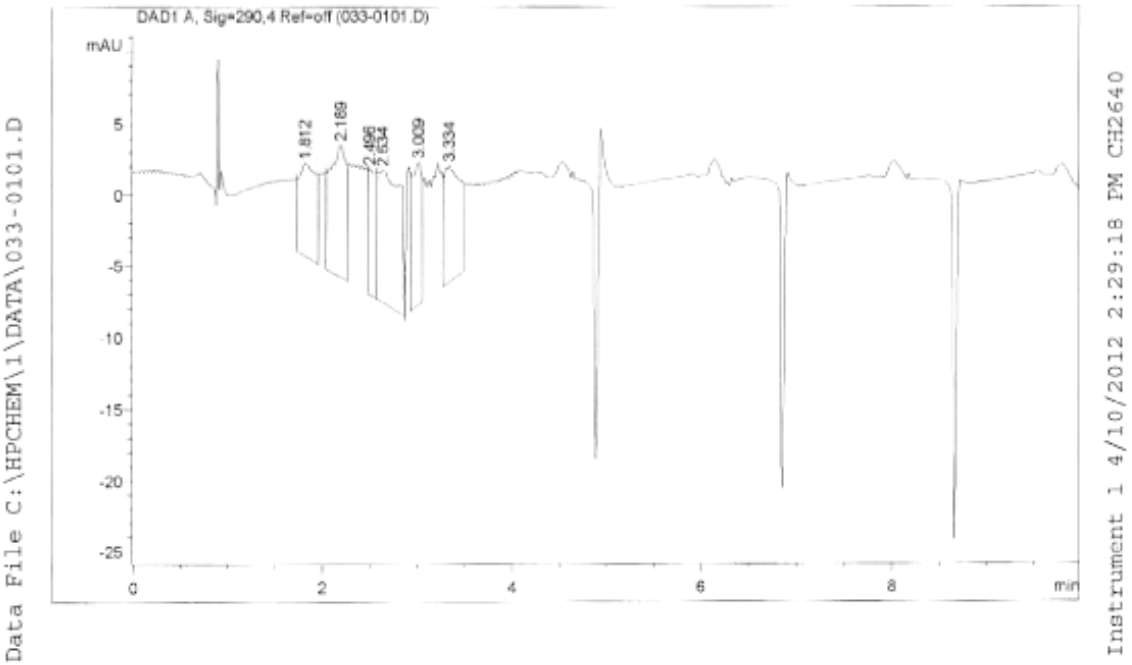

Figure 41: Zeolite Beta (Pellets) Peak C

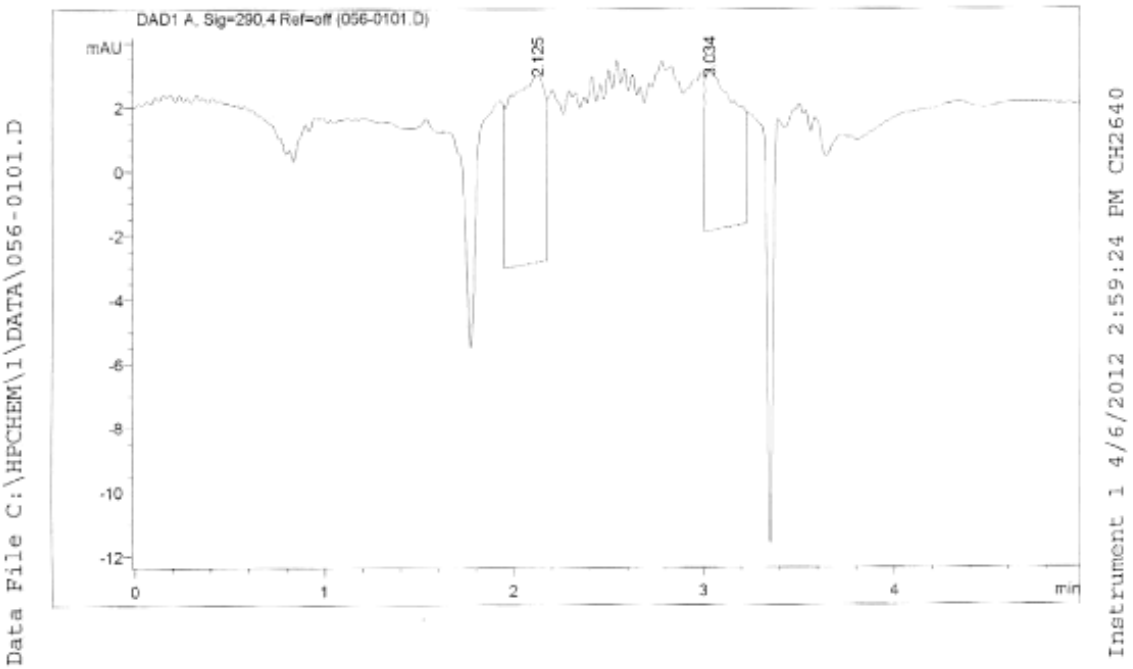

Figure 42: Zeolite Beta (Powder) Peak A

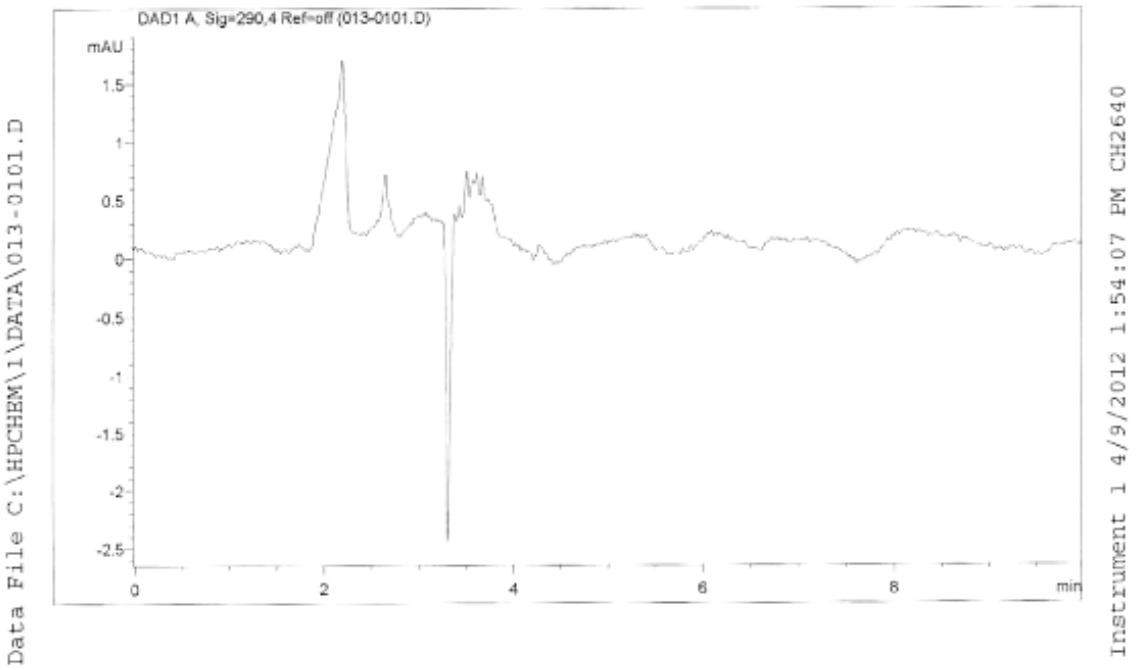

Figure 43: Zeolite Beta (Powder) Peak B

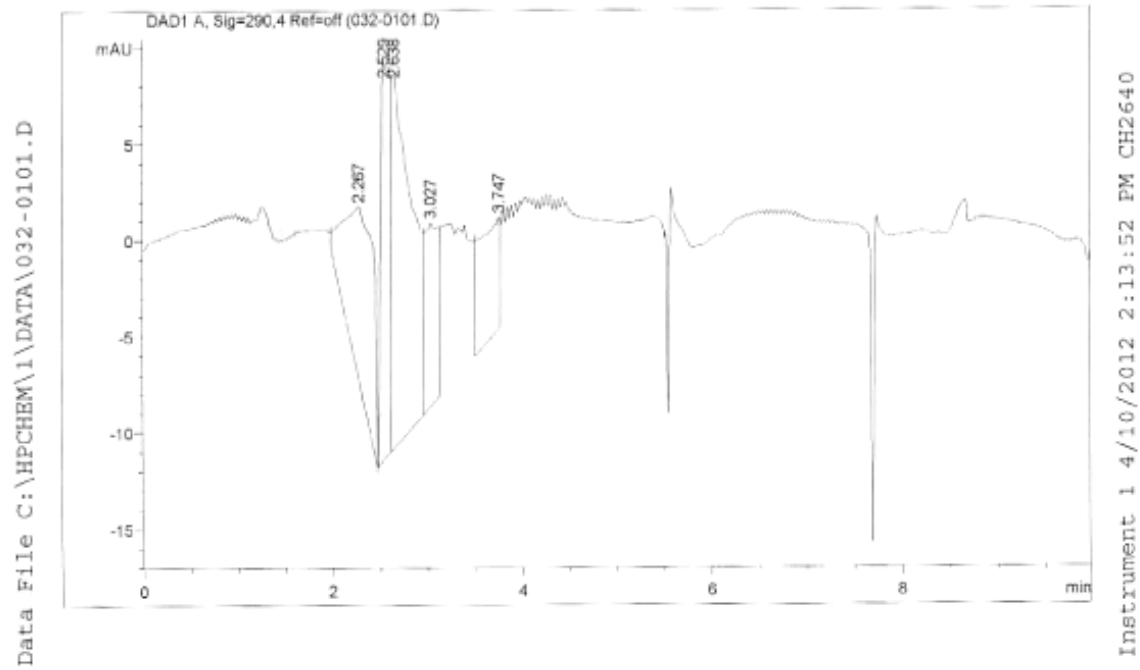

Figure 44: Zeolite Beta (Powder) Peak C

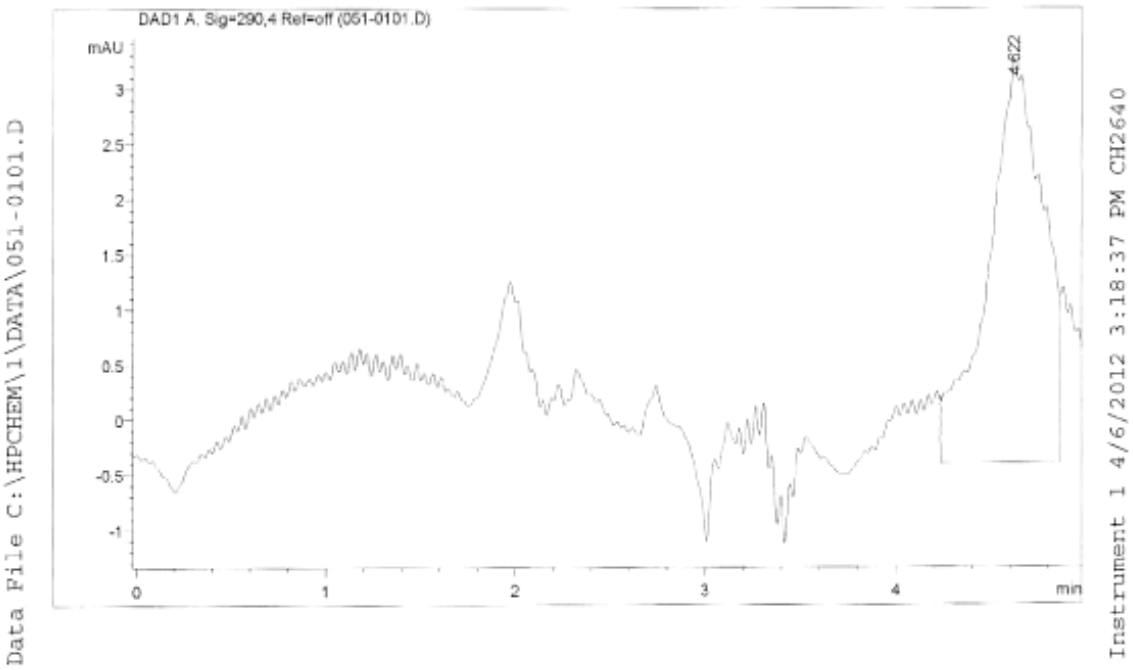

Figure 45: ZSM 5 (Pellets) Peak A

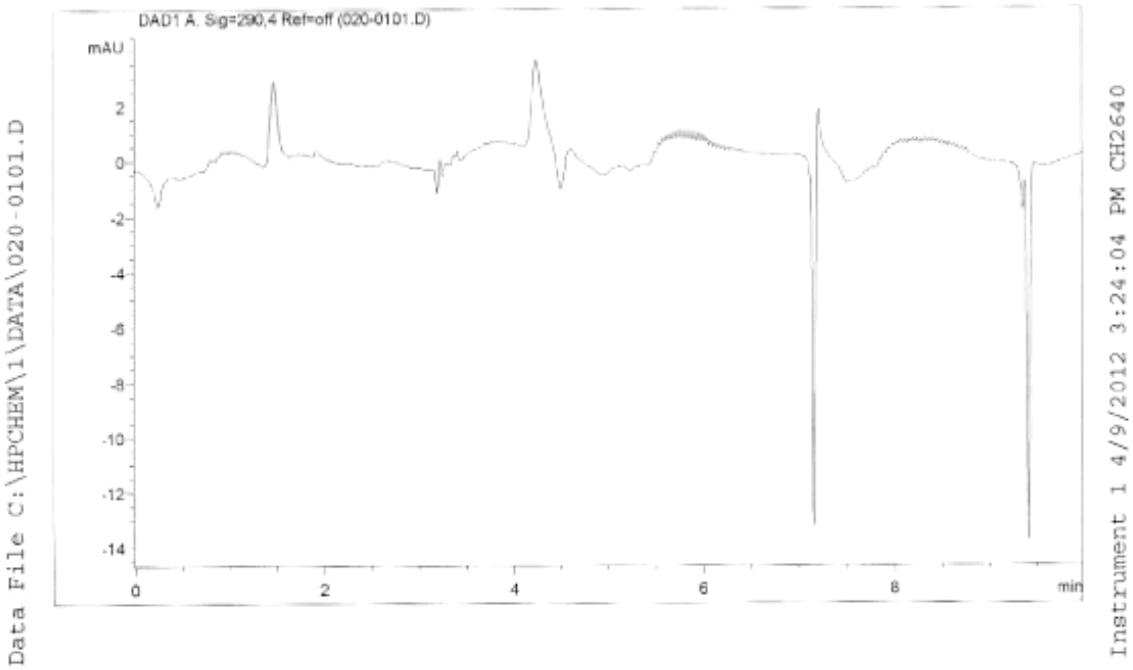

Figure 46: ZSM 5 (Pellets) Peak B

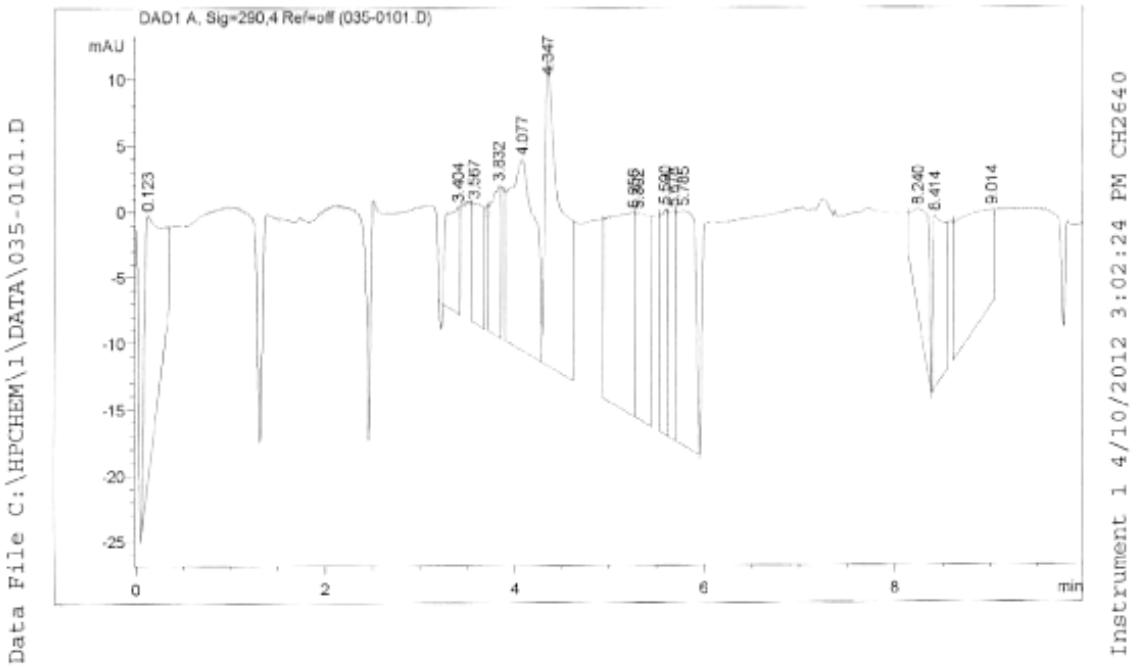

Figure 47: ZSM 5 (Pellets) Peak C

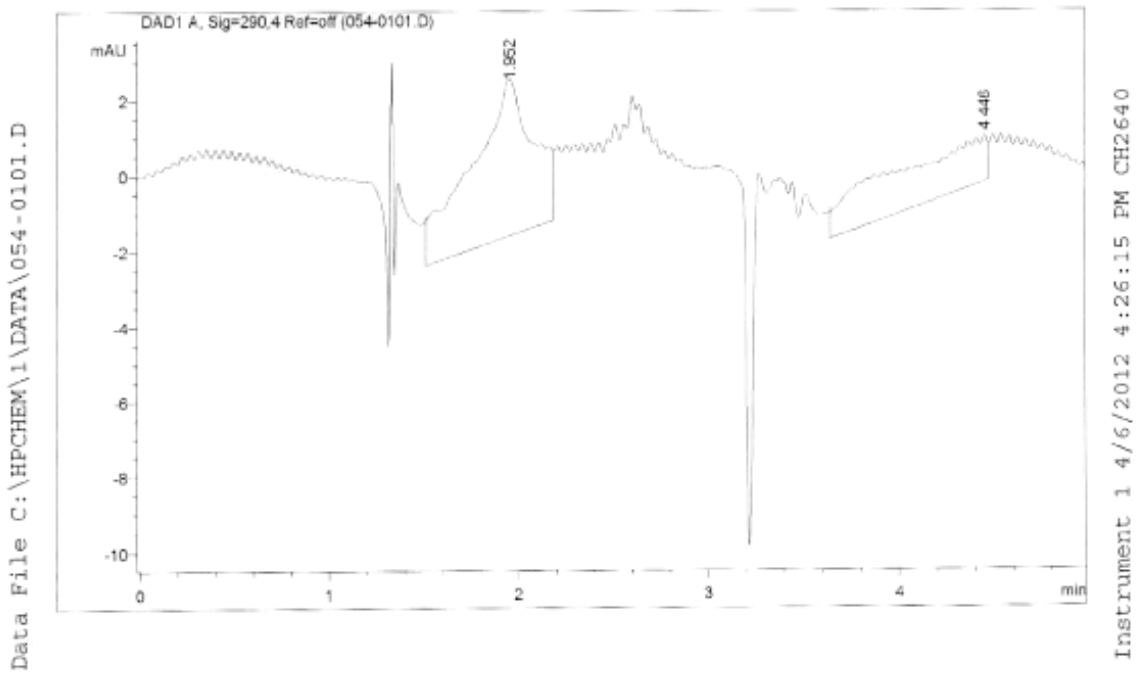

Figure 48: ZSM-5 (Powder) Peak A

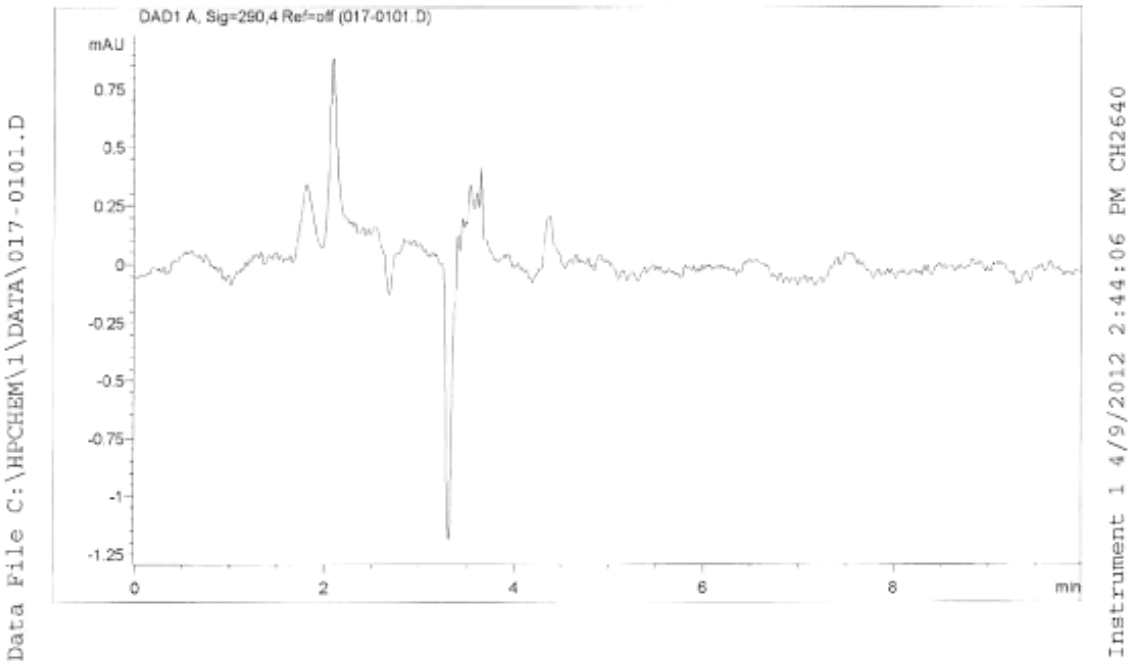

Figure 49: ZSM-5 (Powder) Peak B

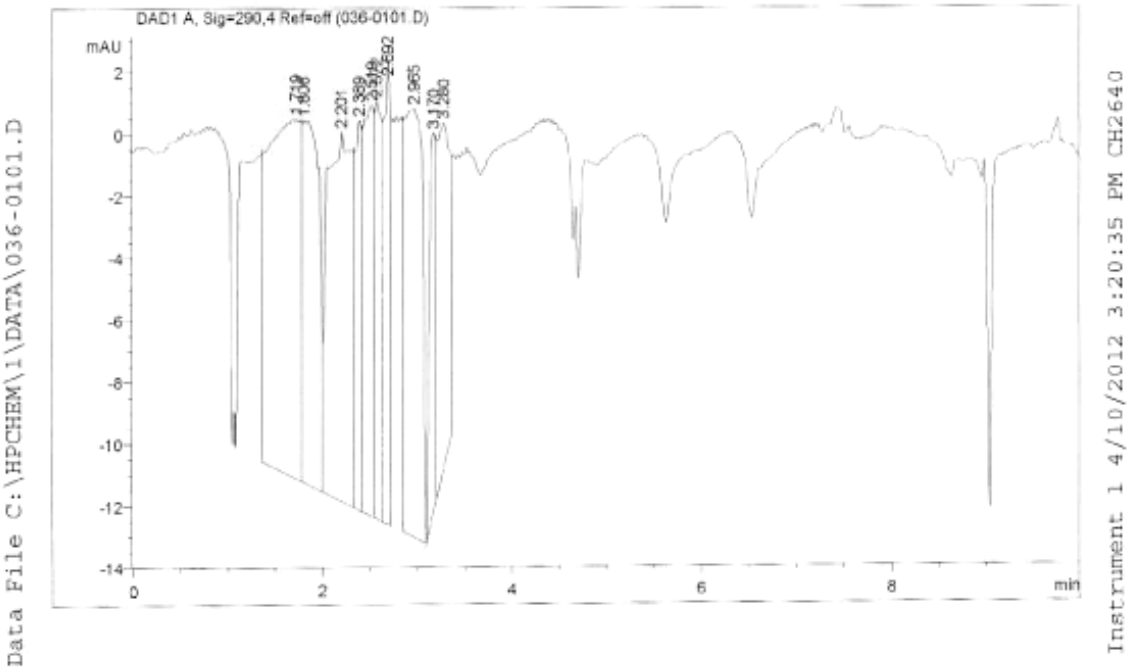

Figure 50: ZSM-5 (Powder) Peak C